

APPENDIX I

Laboratory-provided Standard Operating Procedures

STANDARD OPERATING PROCEDURES

**TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)
FOR ORGANIC AND INORGANIC ANALYTES**

SOP No.: EMAX-1311 Revision No. 0 Date: 06-May-02

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Laboratory Director

Control Number: 1311-00-

1.0 SCOPE AND APPLICATION

- 1.1. This procedure is used to characterize the mobility of organic and inorganic analytes in aqueous, solid, and multiphase wastes. This is an adaptation of EPA Method SW1311. TCLP analytes are listed in Appendix 1.

2.0 SUMMARY OF METHOD

- 2.1. For solid wastes, an amount of extraction fluid 20 times the weight of the solids is used to extract the sample. The filtrate after extraction is defined as the TCLP extract.
- 2.2. For liquid or multiphase wastes containing more than 0.5% dry solid, the filtrate is separated from the solid phase and the solid phase is extracted like solid wastes. The filtrates, if compatible, are subsequently combined as the TCLP extract, and analyzed together. If the liquids are incompatible, they are analyzed separately and the results are mathematically combined to yield an average concentration.
- 2.3. For liquid wastes containing less than 0.5% dry solid, the waste filtered through a 0.6 to 0.8 μm glass fiber filter, the filtrate is defined as TCLP extract.
- 2.4. **Interference**
- 2.4.1. Potential interference that may be encountered during analysis is discussed in the individual analytical methods.

3.0 QUANTITATION LIMITS

- 3.1. Please refer to individual analytical methods.

4.0 DYNAMIC RANGE

- 4.1. Please refer to individual analytical methods.

5.0 PRESERVATION AND HOLDING TIME

- 5.1. All samples shall be collected using an appropriate sampling plan. Preservatives shall not be added to the samples.
- 5.2. Samples must undergo TCLP extraction within the following holding time:

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| Sample Maximum Holding Time (Days) | | | | |
|------------------------------------|---|--|--|--------------------|
| Method | From: Field collection To: TCLP extraction | From: TCLP extraction To: Preparative extraction | From: Preparative Extraction To: Determinative analysis | Total elapsed time |
| Volatiles | 14 | NA | 14 | 28 |
| Semivolatiles | 14 | 7 | 40 | 61 |
| Mercury | 28 | NA | 28 | 56 |
| Metals, except mercury | 180 | NA | 180 | 360 |

6.0 ASSOCIATED SOPs

- 6.1. EMAX-QC08 Glassware Cleaning
 6.2. EMAX-QC02 Analytical Standard Preparation
 6.3. EMAX-SM04 Analytical and QC Sample Labeling

7.0 SAFETY

- 7.1. Read all MSDS for chemicals listed in this SOP.
 7.2. All reagents, standards, and samples shall be treated as potential hazards. Observe the standard laboratory safety procedures. Protective gear, i.e., lab coat, safety glasses, gloves, shall be worn at all times when performing this procedure. All sample and standard handling shall be performed in the fume hood.
 7.3. All wastes generated during analytical process shall be placed in the waste containers. These wastes shall be endorsed to the waste disposal section for proper disposal.
 7.4. Water samples shall be neutralized to pH 7 (± 2) prior to disposal.
 7.5. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES

- 8.1. **Instruments and Supplies**
 8.1.1. Extraction Bottles – 2.2 L teflon bottles
 8.1.2. Agitator – Rotating the extraction vessel in an end over end fashion at 30 \pm 2 rpm

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- 8.1.3. ZHE (Zero-Head Space Extractor Vessel) – This device is for use to determine volatile analyte in waste. Millipore Corp. Model YT80090.
- 8.1.4. Balance – 600 g / \pm 0.01 g
- 8.1.5. pH Meter – Accurate to \pm 0.05 units @ 25°C
- 8.1.6. Filter holders – Funnel type filter holders
- 8.1.7. Filters – 0.7 μ m glass fiber
- 8.1.8. Vacuum – 50 psi
- 8.1.9. Thermometer – With high/low recorder

8.2. Chemicals and Reagents

- 8.2.1. Reagent Water – DI or NanoPure water
- 8.2.2. Hydrochloric Acid – 1N ACS reagent grade
- 8.2.3. Nitric Acid – 1N ACS reagent grade
- 8.2.4. Sodium Hydroxide – 1N ACS reagent grade
- 8.2.5. Glacial Acetic Acid – ACS reagent grade
- 8.2.6. **Extraction Fluid:**
 - 8.2.6.1. Extraction Fluid #1: Pour about 500 ml of reagent water into a 1-L volumetric flask. Add 5.7 glacial acetic acid (CH₃COOH) and 64.3 ml of 1N sodium hydroxide (NaOH) and fill the flask to mark with reagent water. The pH should be 4.93 ± 0.05 .
 - 8.2.6.2. Extraction Fluid #2: Dilute 5.7 ml glacial CH₃COOH with reagent water to a volume of 1 L. pH should be 2.88 ± 0.05 .

9.0 STANDARDS

- 9.1. Refer to Section 9 of the specific method SOP.

10.0 PROCEDURES**10.1. Sample Preparation****10.1.1. For Solid Matrices****10.1.1.1. Particle Size Determination**

- 10.1.1.1.1. If sample particle size is capable of passing through 9.5 mm of standard sieve, proceed to sample extraction, otherwise reduce the particle size by crushing, cutting, or grinding until above criteria is met. Record the activities on the sample preparation log.

10.1.1.2. Extraction Fluid Determination

- 10.1.1.2.1. Weigh 5 gram of sample and add 100 ml of reagent water. Stir the solution and check the pH.
- 10.1.1.2.2. If pH is < 5 , use Fluid #1.

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10.1.1.2.3. If pH is > 5, add 3.5 ml of 1N HCl, warm to 50°C for 10 minutes, cool down the sample then check for pH.

10.1.1.2.4. If pH is < 5, use Fluid #1.

10.1.1.2.5. If pH is > 5, use Fluid #2.

10.1.2. Leaching Process

10.1.2.1. Check the pH of the extraction fluid (Refer to Section 8.2.6)

10.1.2.2. Weigh and record 100 g of sample into a TCLP extraction bottle. Slowly add 2 liter of TCLP solution and seal the bottle tightly.

10.1.2.3. Agitate the sample for 15 minutes then vent it out under a fume hood. Repeat this process 3 times. While doing this process, calibrate the rpm of the agitator by counting the number of revolutions per minute. Record the rpm in the extraction log.

10.1.2.4. Fill one extraction bottle with 2-L TCLP solution and label it as TCLP blank.

10.1.2.5. Set the thermometer to record the highest and the lowest temperature of the extraction room for a period of 20 hours.

10.1.2.6. Place the samples, to include blank sample, in the agitator and continuously rotate the sample for 18 (\pm 2) hours.

10.1.2.7. Filter the extracts as described in Section 10.1.5.

10.1.3. TCLP Extract Preservation

- For organic analyses, store the TCLP extracts at 4°C (\pm 2°C) until it is ready for organic extraction.
- For metal analyses, add spike solution to matrix spike samples and preserve with HNO₃, pH \leq 2 after filtration.

10.1.4. For Liquid or Multiphase Wastes

10.1.4.1. Perform percent solid determination.

10.1.4.1.1. Pre-weigh the container that will receive the filtrate.

10.1.4.1.2. Mix the sample well and transfer 100-g sub-sample for filtration (refer to section ^{10.1.3} *R 12/11/07*).

10.1.4.1.3. If percent solids is < 0.5%, the filtrate is the TCLP extract.

10.1.4.1.4. Preserve the extract as described in Section 10.1.3.

10.1.4.1.5. If percent solids is \geq 0.5%, record the percentage of solid on the sample preparation logbook.

10.1.4.1.6. Determine how many samples are needed for preparative extraction.

10.1.4.1.7. Separate and save the liquid phase from the solid phase.

10.1.4.1.8. Extract the solid phase as solid samples (Section 10.1.1).

10.1.4.1.9. Combine the liquid phase and the solid phase extract to obtain the TCLP extract.

10.1.4.1.10. Preserve the extract as described in Section 10.1.3.

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- 10.1.5.1. Determine percent solids for the liquid phase. Weigh 100-ml of the supernatant using a volumetric flask. If the weight >110-g, filter by positive pressure (proceed to ~~10.1.1~~), otherwise apply vacuum filtration. (~~10.1.3.1~~). (Proceed to 10.1.5.2) ^{10.1.5.3}
- 10.1.5.2. Assemble the filtration apparatus with 0.7 um glass fiber filter. (For metals analyses acid-wash the filter by passing about 20 ml of 5% HNO₃ in reagent water solution.) Rinse the filter with two parts of 20-ml reagent water. Continue to apply air until all traces of water are gone. Replace the collector with a clean flask. Slowly pour the supernatant. Record the pH of the filtrate. ^{R 12/10/02}
- 10.1.5.3. For samples containing more than 10% solid content apply positive pressure filtration. Transfer the supernatant into a clean ZHE device and seal it properly. Connect the ZHE device into a nitrogen line and slowly apply positive pressure (1-10 psi). With the filtrate receptacle ready, slowly open the outlet valve. In the event that no filtrate is collected increase the pressure at 10-psi increments (not to exceed 50 psi) at two-minute intervals. Record the pH of the filtrate.

10.1.6. Preparation of Extract for VOA Analysis

- 10.1.6.1. Use the ZHE device to obtain TCLP extract for analysis of volatiles.
- 10.1.6.2. Prepare the ZHE with sample only once and do not open the device until the final extract has been collected.
- 10.1.6.3. For solid waste, weigh out a subsample of 25 gm (maximum) of the waste. Quantitatively transfer the entire sample quickly to the ZHE. Tighten all ZHE fittings according to manufacturer's instructions and place the device in a vertical position.
- 10.1.6.4. Transfer the appropriate amount of extraction fluid #1 to the vessel via a tube connecting extraction fluid reservoir to the liquid inlet/outlet valve.
- 10.1.6.5. When the appropriate amount of fluid has been introduced into the device, close the liquid inlet/outlet valve and disconnect the extraction fluid line. Pressurize the ZHE to 5-10 psi and slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced due to addition of extraction fluid.
- 10.1.6.6. Re-pressurize the ZHE to 5-10 psi and check all ZHE fittings to ensure that they are closed.
- 10.1.6.7. Place the ZHE in the rotatory agitation apparatus and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature shall be maintained at $23 \pm 2^\circ\text{C}$.
- 10.1.6.8. Following the 18 ± 2 hour agitation period, the material in the vessel is separated into its component liquid and solid phase. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same container (i.e., Tedlar bag) holding the initial phase of the waste. A separate filtration collection container must be used if combining would create multiple phases.
- 10.1.6.9. Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4°C until analyzed.

10.2. Instrument Parameters

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10.2.1. The agitator should be set at 30 ± 2 rpm. Tumbling end-over-end fashion at temperature of $23 \pm 2^\circ\text{C}$.

10.3. Calibration

10.3.1. Refer to individual analytical methods.

10.4. Analysis

10.4.1. Refer to individual analytical methods.

10.5. Calculations

10.5.1. Calculation for Percent Solids

$$\% \text{Solids} = \frac{\text{Weight of solids}}{\text{Total area of sample}} \times 100 \quad \text{Eq.-10.5.1}$$

10.5.2. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm 0.5\%$), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final analyte concentration} = \frac{V_1 \times C_1 + V_2 \times C_2}{V_1 + V_2} \quad \text{Eq.-10.5.2}$$

where:

V_1, V_2 – volumes of first and second phase

C_1, C_2 – concentration of analytes in the first and second phase

11.0 QUALITY CONTROL

11.1. The maximum number of sample in an analytical batch shall be 20 samples unless otherwise specified by the project.

11.2. A preparation blank, using TCLP solution shall be prepared in every analytical batch unless otherwise specified by the project.

11.3. All labwares to be used in the sample preparation shall be properly treated as specified in EMAX-QC07.

12.0 CORRECTIVE ACTION

12.1. Refer to individual method SOP for internal QC procedure corrective action plan for out of control situations.

12.2. If room temperature went out-of-control during the TCLP extraction, record it in the extraction log and inform the Project Manager for further action.

13.0 POLLUTION PREVENTION

13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped in the laboratory sink.

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- 13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to the WDU for proper disposal.

14.0 WASTE MANAGEMENT

- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to the WDU shall be disposed of in accordance to EMAX-SM03

15.0 SUPPLEMENTARY NOTES

- 15.1. The method recommends using 100 gm in sample preparation, under certain circumstances, such as when only limited amount of sample is available, or unusual matrix, a smaller amount of sample, may be utilized with the client's concurrence.

15.2. Definition of Terms

- 15.2.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagent. **Preparation batch** is composed of 1 to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. **Analytical batch** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.2.2. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyze to monitor the accuracy of the analytical system.
- 15.2.3. Matrix – is a component or form of a sample.
- 15.2.4. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyze to monitor matrix effect on a method's recovery efficiency.
- 15.2.5. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.2.6. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.2.7. Re-extract/digest – is a repeated sample preparation process identified with the Lab Sample ID suffixed with "R".
- 15.2.8. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.2.9. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.2.10. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

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16.0 REFERENCES16.1. "Test Methods for Evaluation of Solid Wastes", Method 1311, USEPA SW846, 3rd Edition, as updated.**17.0 APPENDIX**

- | | |
|------------------|---|
| 17.1. Appendix 1 | TCLP Analyte List with Regulatory Limits and Reporting Limits |
| 17.2. Appendix 2 | Sample Preparation Log |

TCLP Analyte List with Regulatory Limits and Reporting Limits

| Method | Parameters | Regulatory Level (mg/L) | TCLP RL (mg/L) | Solid RL (mg/kg) |
|----------------|----------------------|-------------------------|----------------|------------------|
| VOA by 8260B | Benzene | 0.50 | 0.05 | 0.50 |
| | 2-Butanone | 200 | 0.50 | 5.00 |
| | Carbon Tetrachloride | 0.50 | 0.05 | 0.50 |
| | Chlorobenzene | 100 | 0.05 | 0.50 |
| | Chloroform | 6 | 0.05 | 0.50 |
| | 1,4-Dichlorobenzene | 7.5 | 0.05 | 0.50 |
| | 1,2-Dichloroethane | 0.50 | 0.05 | 0.50 |
| | 1,1-Dichloroethene | 0.70 | 0.05 | 0.50 |
| | Tetrachloroethene | 0.70 | 0.05 | 0.50 |
| | Trichloroethene | 0.50 | 0.05 | 0.50 |
| Vinyl Chloride | 0.20 | 0.05 | 0.50 | |

| Method | Parameters | Regulatory Level (mg/L) | TCLP RL (mg/L) | Solid RL (mg/kg) |
|---------------------------|------------|-------------------------|----------------|------------------|
| Metals by 6010B/ 7470A | Arsenic | 5.0 | 1.0 | 20 |
| | Barium | 100 | 1.0 | 20 |
| | Cadmium | 10 | 1.0 | 20 |
| | Chromium | 5.0 | 1.0 | 20 |
| | Lead | 5.0 | 1.0 | 20 |
| | Mercury | 0.2 | 0.02 | 0.2 |
| | Selenium | 1.0 | 1.0 | 20 |
| | Silver | 5.0 | 1.0 | 20 |

| Method | Parameters | Regulatory Level (mg/L) | TCLP RL (mg/L) | Solid RL (mg/kg) |
|------------------------|-------------------|-------------------------|----------------|------------------|
| Herbicides by 8151A | 2,4-D | 10 | 0.005 | 0.084 |
| | 2,4,5-TP (Silvex) | 1.0 | 0.002 | 0.033 |

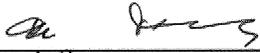
| Method | Parameters | Regulatory Level (mg/L) | TCLP RL (mg/L) | Solid RL (mg/kg) |
|--------------------------|--------------------|-------------------------|----------------|------------------|
| Pesticides by SW8081A | Endrin | 0.02 | 0.001 | 0.033 |
| | Lindane | 0.4 | 0.0005 | 0.017 |
| | Methoxychlor | 10 | 0.005 | 0.17 |
| | Heptachlor | 0.008 | 0.0005 | 0.017 |
| | Heptachlor Epoxide | 0.008 | 0.0005 | 0.017 |
| | Toxaphene | 0.5 | 0.010 | 0.33 |
| | Chlordane (β or γ) | 0.03 | 0.0005 | 0.017 |

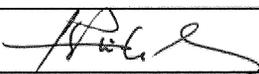
| Method | Parameters | Regulatory Level (mg/L) | TCLP RL (mg/L) | Solid RL (mg/kg) |
|-----------------------|-----------------------|-------------------------|----------------|------------------|
| SVOA by SW8270C | 2,4-Dinitrotoluene | 0.13 | 0.10 | 1.65 |
| | Hexachlorobenzene | 0.13 | 0.10 | 1.65 |
| | Hexachlorobutadiene | 0.50 | 0.10 | 1.65 |
| | Hexachloroethane | 3.0 | 0.10 | 1.65 |
| | Nitrobenzene | 2.0 | 0.10 | 1.65 |
| | Pentachlorophenol | 0.7 | 0.50 | 3.3 |
| | Pyridine | 5.0 | 0.50 | 3.3 |
| | 2-Methylphenol | 200 | 0.10 | 1.65 |
| | 4-Methylphenol | 200 | 0.10 | 1.65 |
| | 2,4,5-Trichlorophenol | 400 | 0.50 | 3.3 |
| 2,4,6-Trichlorophenol | 2.0 | 0.10 | 1.65 | |

STANDARD OPERATING PROCEDURES

VOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8260 Revision No. 4 Effective Date: 03-Sep-07

Prepared By: W. Tu Nisamaneepong  Date: 08-17-07

Approved By: Kenette Pimentel  Date: 08-17-07
QA Manager

Approved By: Caspar Pang  Date: 8/17/07
Laboratory Director

Control Number: **8260-04-**

1.0 SCOPE AND APPLICATION

- 1.1. This analytical method is used to determine the concentration of volatile organic compounds whose boiling points are below 200°C and are water insoluble or slightly water-soluble found in solid or liquid samples. The list of compounds is summarized in Tables 7 and 8. Additional analytes may be added after verification. This SOP is an adaptation of Method 8260B.

2.0 SUMMARY OF METHOD

- 2.1. This method provides gas chromatography with a mass spectrometer for the detection and quantitation of volatile organic compounds. The samples are introduced to GC by using a purge and trap concentration technique to increase the sensitivity of the method. The identification is based on the characteristic electron impact mass spectra. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a calibration curve.
- 2.2. **Interferences**
- 2.2.1. Contamination may occur by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through sample container septum during shipment and storage. Trip blanks and storage blanks can serve as means of monitoring.
- 2.2.2. Glassware and other sample processing materials in which the samples come into contact with are possible sources of contamination. All glassware and other materials used must be purchased pre-cleaned or decontaminated prior to use.
- 2.2.3. Solvents and reagents are possible sources of contamination. All solvents and reagents must be GC grade and must pass the QC checks prior to use.
- 2.2.4. Contamination by carry-over can occur whenever high concentration samples are analyzed in sequence with a low concentration sample. To reduce potential carry-over, the concentrator must be thoroughly baked-out between samples and the sample syringe and purging device must be thoroughly rinsed with an appropriate solvent between samples.
- 2.2.5. Another possible source of contamination is the analytical instrument itself. This can be monitored by analyzing an instrument blank prior to any analysis.

3.0 QUANTITATION LIMITS**3.1. Method Detection Limit (MDL)**

- 3.1.1. Prepare a minimum of seven samples for each matrix preferably at 1 µg/L (Ketones at 5 µg/L) spike level for 25-ml purge and 5µg/L (Ketones at 10µg/L) for 5-ml purge. Other concentration levels may be used to obtain a credible MDL value. Prepare a method blank and LCS as described in Section 10.

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3.1.2. Analyze the samples as described in Section 10.4 and calculate the results as described in Section 10.6.

3.1.3. Refer to EMAX-QA04 for MDL evaluation and verification.

3.2. Reporting Limit (RL)

3.2.1. Reporting limit shall be defined by the lowest calibration point unless otherwise specified by the project.

4.0 DYNAMIC RANGE

4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point (see Sec. 9.4). All samples analyzed above this concentration are considered "over-range" and shall require dilution to properly quantitate.

4.2. The concentration in the diluted sample should be at or above the project reporting limit. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required to properly quantitate.

4.3. Typical Dynamic Range

4.3.1. Water: 5 µg/L to 200 µg/L (5 ml purge)
1 µg/L to 40 µg/L (25 ml purge)

4.3.2. Soil: 5 µg/kg to 200 µg/kg

5.0 SAMPLE HOLDING TIME & PRESERVATION**5.1. Aqueous Samples**

5.1.1. Samples received in the laboratory should be contained in 40 ml vials with teflon lined septa with zero headspace.

Note: The size of any bubble caused by degassing upon cooling the sample should not exceed 6 mm.¹

5.1.2. All samples must be stored at 4°C (±2°C).

5.1.3. Samples preserved in HCL shall be analyzed within 14 days from the date of sampling. Samples with no chemical preservative must be analyzed within 7 days from the date of sampling.

5.2. Soil Samples

5.2.1. Samples receive in a glass jars or brass tubes shall be stored at 4°C (±2°C). Samples must be analyzed within 14 days from sampling date.

5.2.2. Samples received in encore tubes are frozen, preserved with sodium bisulfate or extracted with methanol prior to analysis.

- Frozen encore tubes must be analyzed within 14 days from sampling date.
- Samples preserved with sodium bisulfate within 48 hours from sampling date must be analyzed within 14 days from sampling date.

¹ Referenced from SW846 Method 5030B, Section 6.1.

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- Methanol extracts shall be analyzed within 14 days from sampling date.
- Preserved samples and extracts shall be stored at 4 °o ($\pm 2^{\circ}\text{C}$).

6.0 ASSOCIATED SOPs

- 6.1. EMAX-5030 - Purge and Trap For Aqueous Samples
- 6.2. EMAX-5035 - Closed-System Purge and Trap For Solid Samples
- 6.3. EMAX-DM01 - Data Flow and Review
- 6.4. EMAX-QA04 -Method Detection Limit Study
- 6.5. EMAX_QA08 -Corrective Action
- 6.6. EMAX-QC01 - Quality Control for Chemicals
- 6.7. EMAX-QC02 - Analytical Standard Preparation
- 6.8. EMAX-QC07 - Glassware Cleaning
- 6.9. EMAX-SM03 - Waste Disposal
- 6.10. EMAX-SM04 - Analytical and QC Sample Labeling

7.0 SAFETY

- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. All reagents, standards, and samples shall be treated as potential hazards. Observe standard laboratory safety procedures. Protective gear, i.e., lab coat, safety glasses, and gloves, shall be worn at all times when performing this procedure. All sample and standard handling shall be performed in the fume hood.
- 7.3. All waste generated during analytical process shall be placed in the waste containers. Waste shall be endorsed to the waste disposal section for proper disposal.
- 7.4. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES**8.1. Instruments and Supplies**

| | |
|---------------------------|--|
| Gas Chromatography | HP 5890 Series II or equivalent |
| Detector | HP 5970 MSD or equivalent |
| Column | RTX 502.2 (0.32 mm x 60 m), 1.8um thickness or equivalent after verification that the four gases (chloromethane, bromomethene, chloroethane, and vinyl chloride) can be resolved > 90% from each other in the total ion chromatogram |
| Data Acquisition Software | ChemStation or equivalent |
| Purge & Trap Device | 2000/OI 4460A/Dyna Tech/EST or equivalent |

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| | |
|-------------------------|---|
| Multiple purging module | DynaTech/Archon or equivalent |
| Gases | Ultra-high purity helium/Air |
| Syringes | 5-ml, 25-ml Luerlok gas-tight |
| Microsyringes | 1, 10, 20, 25, 50, 100, and 1000 μ L (Hamilton 702N or equivalent) for dilution purposes |
| Volumetric Flasks | 2,5,10, 50, and 100 ml with ground glass stopper |
| Heated Jacket | Tekmar or O.I. Automatic sample heating jacket or equivalent |

8.2. **Chemicals and Reagents**

| | |
|--------------------|---|
| Extraction Solvent | Purge & Trap Grade Methanol or equivalent |
| Reagent Water | Organic-free water |
| Reagent Soil | Organic-free Ottawa Sand or equivalent |
| Preservative | Sodium Bisulfate |

9.0 **ANALYTICAL STANDARDS**

9.1. Standard preparation for VOA is summarized in Tables 1 to 4. Refer to EMAX-QC02 for proper analytical standard preparation. Other concentration levels may be prepared provided it complies with the method and project requirements.

9.2. **Stock Standard**

- 9.2.1. Purchase Stock Standards as certified solutions.
- 9.2.2. Purchase one set of calibration standard (Refer to Tables 1) for calibration and a secondary source Stock Standard for calibration verification (Refer to Table 2).
- 9.2.3. Purchase Surrogate Mix at 2500 mg/L and Internal Standard at 2000 mg/L (Refer to Table 3).
- 9.2.4. Purchase BFB as Tuning Standard at 5000 mg/L (refer to Table 4).
- 9.2.5. After opening, transfer in inert vials with minimal headspace and store at -10°C to -20°C .

9.3. **Intermediate Standards**

9.3.1. Using the stock standard solutions, prepare intermediate standards in methanol according to Tables 1 to 4 and store with minimal headspace in an inert vial. Intermediate standard concentrations are prepared as suggested below.

| | |
|-----------------------------------|---------|
| BFB | 50 mg/L |
| Internal Standards and Surrogates | 50 mg/L |
| VOA Compounds | 50 mg/L |
| Internal Standard | 50 mg/L |
| Matrix Spike | 50 mg/L |

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VOLATILE ORGANICS BY GC/MSSOP No.: EMAX-8260 Revision No. 4 Effective Date: 03-Sep-07**9.4. Initial Calibration Standards (ICAL)****9.4.1. ICAL for 5-ml Purge**

9.4.1.1. Using intermediate standards, prepare a minimum of 6 calibration standards, as suggested below:

| VOA | IS | Surrogate | Final Volume | Final Conc. |
|--------|------|-----------|--------------|-------------|
| 0.5 µl | 5 µl | 0.5 µl | 5 ml | 5 µg/L |
| 1 µl | 5 µl | 1.0 µl | 5 ml | 10 µg/L |
| 2 µl | 5 µl | 2.0 µl | 5 ml | 20 µg/L |
| 5 µl | 5 µl | 5.0 µl | 5 ml | 50 µg/L |
| 10 µl | 5 µl | 10.0 µl | 5 ml | 100 µg/L |
| 20 µl | 5 µl | 20.0 µl | 5 ml | 200 µg/L |

9.4.2. ICAL for 25-ml Purge

9.4.2.1. Using intermediate standards, prepare a minimum of 6 calibration standards, as suggested below:

| VOA | IS | Surrogate | Final Volume | Final Conc. |
|--------|------|-----------|--------------|-------------|
| 0.5 µl | 5 µl | 0.5 µl | 25 ml | 1 µg/L |
| 1 µl | 5 µl | 1.0 µl | 25 ml | 2 µg/L |
| 2 µl | 5 µl | 2.0 µl | 25 ml | 4 µg/L |
| 5 µl | 5 µl | 5.0 µl | 25 ml | 10 µg/L |
| 10 µl | 5 µl | 10.0 µl | 25 ml | 20 µg/L |
| 20 µl | 5 µl | 20.0 µl | 25 ml | 40 µg/L |

9.5. Initial Calibration Verification Standard (ICV)

9.5.1. Using the Intermediate Standard prepared from the secondary source, spike 10µL into 5-ml or 25-ml purge. Refer to Table 5 for concentration levels for each analyte.

9.6. Daily Calibration Check Standard (DCC)

9.6.1. Using the Intermediate Standard prepared from the same source as the ICAL Standard, spike 10µL into 5-ml or 25-ml purge. Spike 5µL of IS and 5µL of Surrogate Standard. Refer to Table 5 for concentration levels for each analyte.

9.7. LCS and Matrix Spike Standard

9.7.1. For spike standards use the ICV standard unless otherwise specified by the project. Refer to Table 5.

10.0 PROCEDURES**10.1. Sample Preparation**

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10.1.1. Refer to EMAX-5030 and EMAX-5035

10.2. Instrument Parameters

10.2.1. From the main gas supply (gas Tanks) regulate gas pressure at 80 psi.

10.1.1. Fine-tune the instrument guided by the parameter conditions suggested below. Adjust the parameter conditions accordingly to obtain optimum condition. Print the instrument parameter and post it on the instrument for daily routine maintenance check.

10.2.2. Typical GC Parameters

| | |
|----------------------------------|------------------------|
| Carrier gas flow (column) helium | 1 - 5 ml/min |
| Initial Temp | 40°C; hold for 1 min. |
| Rate | 6°C/min. |
| Final Temp | 200°C; hold for 1 min. |
| Inject Port | 160°C |
| Interface | 250°C |

10.2.3. Mass Spectrometer Parameter

| | |
|----------------------|--------------|
| Scan Start | 0.5 min. |
| Splitless value time | 0 min. |
| Mass Range | 35 to 300 |
| Multiplier | 1200 to 2700 |

10.2.4. Typical Purge and Trap Condition

10.2.4.1. Purge samples at 40°C for 11 minutes, desorbed at 250°C for 2 minutes and then bake the trap at 260°C for 11 minutes.

10.3. Calibration

10.3.1. Set GC/MS operating condition as described in Section 10.2.

10.3.2. Perform Tune Check10.3.2.1. Introduce a BFB² to yield 50ng on column by either direct injection or purge and trap in 5-ml or 25-ml organic-free water.

10.3.2.2. Evaluate the tune check by a single scan or the average of 3 scans (before, at, and after the apex) with a background subtraction using a single scan no more than 20 scans prior to the elution of BFB.

10.3.2.3. Check Table 6 for acceptance criteria or follow the manufacturer's recommendation for tuning. A valid tune check expires after 12 hours.

10.3.2.4. If the system failed to meet the acceptance criteria, stop the analysis, correct the problem and repeat the procedure until all criteria are met. Consider the following suggestions to correct the problem:

- Dry purge
- Prepare a new standard

² Alternatively, BFB in DCC can be used to evaluate tuning.

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10.3.3. Initial Calibration (ICAL)

10.3.3.1. Perform ICAL when one of the conditions occurs.

- Instrument is new
- Instrument undergoes a major repair
- DCC failed to meet the acceptance criteria

10.3.3.2. Analyze minimum of 6-point initial calibration curve (Refer to 9.4) after a valid tune check.

10.3.3.3. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:

- Check the established retention time window
- Check the relative intensity of major ions
- Adjust accordingly if necessary.

10.3.3.4. Establish a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRF_m), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq-10.6.1.1, Eq-10.6.1.2 and Eq-10.6.1.3 respectively.

10.3.3.5. Evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.3.6. Evaluate the ICAL for appropriate quantitation method.

- Use RRF_m - if the RSD of individual analyte $\leq 15\%$.
- If the RSD of individual analyte $\geq 15\%$ use first order linear regression when the correlation coefficient (R) ≥ 0.995 .
- If the RSD of individual analyte $\geq 15\%$ and the correlation coefficient (R) ≤ 0.995 , use second order regression when COD ≥ 0.99 based on a minimum of six calibration points.
- Higher order regression is acceptable base on a minimum of seven calibration points.

10.3.3.7. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.4. Initial Calibration Verification (ICV)

10.3.4.1. Analyze ICV to verify the concentration of the ICAL standards (refer to 9.5).

10.3.4.2. Check for completeness of analytes as described in 10.4.2.

10.3.4.3. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of ± 30 seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.4.4. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

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10.3.4.5. Refer to Appendix 1 for ICV acceptance criteria and/or corrective action.

10.3.4.6. When non-compliant consider the following to correct the problem.

- Dry purge and re-analyze the ICV.
- Dry purge, prepare a new standard and re-analyze the ICV.
- Dry purge, clean the source and establish a new ICAL.

10.3.5. **Daily Continuing Calibration (DCC)**

10.3.5.1. Analyze DCC to check the validity of the ICAL (refer to 9.6).

10.3.5.2. Check for completeness of analytes as described in 10.4.2.

10.3.5.3. Evaluate System Performance Check Compounds (SPCC) and Calibration Check Compounds (CCC) as specified in Appendix 1.

10.3.5.4. Compare the retention times of the internal standards to the ICAL mid-point. Excursion of ± 30 seconds indicates instrument malfunction. When non-compliant check the column head pressure, gas supply or leaks. Corrective action is required prior to further analysis.

10.3.5.5. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

10.3.5.6. Establish RRF of each analyte, calculate %D (Eq-10.6.2.1) against the ICAL.

10.3.5.7. Refer to Appendix 1 for DCC acceptance criteria and/or corrective action.

10.3.5.8. When non-compliant consider the following to correct the problem.

- Dry purge and re-analyze the DCC.
- Dry purge, prepare a new standard and re-analyze the DCC.
- Dry purge, clean the source and establish a new ICAL.

10.4. **Analysis**

10.4.1. **Analytical Sequence**

10.4.1.1. Analyze BFB and evaluate tuning

10.4.1.2. Analyze DCC and check ICAL validity

10.4.1.3. Analyze Lab Control Sample

10.4.1.4. Analyze Lab Control Sample Duplicate (if required)

10.4.1.5. Analyze Method Blank

10.4.1.6. Analyze samples to a maximum number of 12-hours from the time of BFB injection.

10.4.1.7. Analyze a pair of matrix spikes (MS/MSD) for every 20 samples of the same matrix.

10.4.1.8. Record analytical sequence in the analytical run log.

10.4.2. **Sample Result Evaluation**

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10.4.2.1. Check the QC criteria as soon as the data is available.

- Check surrogate recoveries against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 QC limits.
- Check concentration of target analytes if calibration range is exceeded.
- If any of the above checkpoints indicate a problem, re-analysis is required. Note observations on the analytical run log. When results arise to questionable result, e.g. inconsistency from the first analysis, consult the Supervisor for further action.

10.4.3. **Qualitative Identification**

- ◆ The intensities of the characteristic ions maximize in the same scan or within one scan of each other.
- ◆ The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
- ◆ The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.
- ◆ Check the chromatogram for possible misidentified analytes. Investigate visible peaks in the chromatogram that were not identified in the data output. Manually integrate the peak if necessary.

10.4.3.1. For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative identification³ (TIC). Execute *FTICB exe (HP RTE-1000 program) or LSC (Chem Station program) to initiate the library search using NIST/EPA/MSDC mass spectral library. Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting following the guidelines below.

- Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
- The relative intensities of the major ions should agree within + 20%.
Example: for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30 and 70%.
- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting analytes.
- Ions present in the reference spectrum but not present in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting analytes. Data system library reduction programs can sometimes create these discrepancies.

10.4.3.2. Reporting TICs

- If the library search produces a match at or above 85%, report the analyte.
- If the library search produces more than one analyte at or above 85%, report the first analyte (highest).
- If the library search produces no matches at or above 85%, the compound should be reported as unknown.

³ Library search is performed only when indicated in the PSR.

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- ◆ Apply the appropriate quantitation method (Section 10.3.3.6). Calculate the concentration of any positively identified target analyte using Eq-10.6.3. Apply the dilution factor for diluted samples to calculate for the final concentration of the sample.

10.4.5. Manual Integration

10.4.5.1. Refer to EMAX-DM01, Section 4.4.3.

10.4.6. Dealing with Carryover

10.4.6.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.6.2. If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.

10.4.6.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.6.4. For decontaminating the sample purger consider the following suggestion:

- Rinse the purging apparatus and the contaminated port with organic-free reagent water containing 10% methanol. Dry purge the system overnight and analyze reagent blank prior to sample analysis. Repeat the process until no evidence of contamination is observed.

10.5. Data Reduction

10.5.1. Make a copy of the analytical run log and highlight the data to be reported.

10.5.2. Check that all positively identified analytes are within the calibration range.

10.5.3. Collate the reportable raw data separating the QC results from the sample results.

10.5.4. Keep all other data generated with the analytical folder marked with "For record only".

10.5.5. Proceed to report generation.

10.6. Calculations**10.6.1. Initial Calibration**

10.6.1.1. Calculate the Relative Response Factor (RRF)

$$RRF = \frac{A_x C_{is}}{A_{is} C_x} \quad \text{Eq.-10.6.1.1}$$

where:

A_x – Area of characteristic ion for the compound being measured

A_{is} – Area of characteristic ion for the specific internal standard

C_x – Concentration of the compound being measured

C_{is} – Concentration of the specific internal standard

10.6.1.2. Calculate the Average Relative Response Factor (RRF_m).

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$$RRF_m = \frac{\sum RRF}{n} \quad \text{Eq-10.6.1.2}$$

where:

RRF_m – average response factor

$\sum RRF$ – summation of response factors

n – number of measurements

10.6.1.3. Calculate the Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq.-10.6.1.3}$$

where:

SD – standard deviation

x_i – result at i^{th} measurement

\bar{x} - mean

n - number of measurements

10.6.1.4. Calculate the % relative standard deviation (%RSD).

$$\%RSD = \frac{SD}{RRF_m} * 100\% \quad \text{Eq-10.5.4}$$

where:

SD – standard deviation

RRF_m – average response factor

10.6.2. Calibration Check/Continuing Calibration

10.6.2.1. Calculate Percent Difference (%D)

$$\%D = \frac{[RRF_c - RRF_m]}{RRF_m} * 100\% \quad \text{Eq-10.6.2.1}$$

where:

RRF_c – response factor from continuing calibration standard

RRF_m – average response factor

10.6.2.2. % Drift

$$\%Drift = \frac{[found Conc. - true Conc.]}{true Conc.} * 100\% \quad \text{Eq-10.6.2.2}$$

10.6.3. Calculation of Sample Concentration (Water and Soil/Sediment Samples). When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the

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EICP of the primary characteristic ion.

10.6.3.1. Water Samples

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)}{(A_{is})(RRF_m)} \times DF \quad \text{Eq-10.6.3.1}$$

where:

A_x – area of characteristic ion for the compound to be measured

I_s – concentration of internal standard added in $\mu\text{g/L}$

A_{is} – area of characteristic ion for the internal standard

RRF_m – average response factor

DF – dilution factor = $\frac{\text{purge volume in ml (5 ml or 25 ml)}}{\text{sample amount in ml}}$

10.6.3.2. Soil/Sediment Samples (Dry weight basis)

$$\text{Concentration (ug/kg)} = \frac{(A_x)(I_s)}{(A_{is})(RRF_m)(DW)} \times DF \quad \text{Eq-10.6.3.2}$$

where:

A_x – area of characteristic ion for the compound to be measured

I_s – concentration of internal standard added in $\mu\text{g/L}$

A_{is} – area of characteristic ion for the internal standard

RRF_m – average response factor

DF – dilution factor = $\frac{5 \text{ g}}{(\text{sample amount in g})}$

DW – % solid = $\frac{100 - \% \text{moisture}}{100}$

10.6.3.3. Extracted Soil/Sediment Samples (Dry weight basis)

$$\text{Concentration (ug/kg)} = \frac{(A_x)(I_s)}{(A_{is})(RRF_m)(DW)} \times DF \quad \text{Eq-10.6.3.3}$$

where:

A_x – area of characteristic ion for the compound to be measured

I_s – concentration of internal standard added in $\mu\text{g/L}$

A_{is} – area of characteristic ion for the internal standard

RRF_m – average response factor

DF – dilution factor = $\frac{(\text{purged volume in } \mu\text{L})(5 \text{ g})}{(\text{extract aliquot in } \mu\text{L})(\text{sample amount in g})}$

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$$DW - \% \text{ solid} = \frac{100 - \% \text{moisture}}{100}$$

- 10.6.4. Alternatively, the regression line (area ratio of A_x/A_i s versus concentration using first degree or higher regression) fitted to the initial calibration may be used for determination of the sample concentration when RSD of the analyte is greater than 15 (Section 10.3.3.6)
- 10.6.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:
- 10.6.5.1. The area A_x and A_i s are derived from total ion chromatogram. A_i s refers to the closest internal standard (IS) free of interference.
- 10.6.5.2. RRF of the TIC is 1.
- 10.6.6. Method Proficiency
- 10.6.6.1. Percent Recovery

$$\% \text{ Recovery} = \frac{C_f - C}{C_s} * 100\% \quad \text{Eq-10.6.6.1}$$

where:

 C_f – concentration found C – concentration of sample C_s – concentration of spike

- 10.6.6.2. Relative Percent Difference (%RPD)

$$\% \text{ RPD} = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq-10.6.6.2}$$

where:

 RPD – Relative Percent Difference C_1 – Measured concentration of the first sample aliquot C_2 – Measured concentration of the second sample aliquot

- 10.6.7 Calculate the MDL

$$\text{MDL} = T_7 \text{SD} \quad \text{Eq-10.6.7}$$

where:

MDL – the Method Detection Limit

 T_7 – degrees of freedom for 7 measurements, which is 3.14

SD – Standard deviation for 7 measurements

10.7. Report Generation

- 10.7.1. Generate the method.txt file using WBDX⁴.exe.

⁴ X - version number

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- 10.7.2. Generate Lab Chronicle using Labchron.exe
- 10.7.3. Generate the sample results using F1VX³.exe
- 10.7.4. Generate the QC summary using QCVX³.exe

10.8. Data Review

- 10.8.1. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.
 - ◆ Sample Results
 - ◆ LCS Summary
 - ◆ MS/MSD Summary
 - ◆ ICAL Summary
 - ◆ ICV Summary
 - ◆ DCC Summary
- 10.8.2. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
 - ◆ Check internal standard area. They should be within -50 to +100% of DCC to be acceptable.
 - ◆ Check surrogate recoveries against project specific criteria (PSR). In the absence of PSR, default to in-house QC limits.
 - ◆ Check concentration of target analytes if calibration range is exceeded.
 - ◆ If any of the above checkpoints indicate a problem, re-analysis is required.
- 10.8.3. Generate the case narrative to include discussion of the following as found in the review process:
 - ◆ Number of samples analyzed
 - ◆ Analytical method(s) applied
 - ◆ Holding Time – That samples extracted and analyzed within holding time (7 days for unpreserved water, 14days for preserved water and soil samples). For non-compliance, state the number of days that the sample(s) were off from holding time.
 - ◆ Instrument Tuning Check – That instrument tuning was checked and tuning acceptance criteria was met as specified by the method/project.
 - ◆ Internal Standard Area and Retention Time – That retention time is within ± 30 seconds from retention time of the mid-point std. in the ICAL. That EICP areas are within -50% to +100% of ICAL mid-point std.
 - ◆ Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
 - ◆ Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
 - ◆ Method Blank – That MB was analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s)

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affected and the associated sample results were flagged with “B”.

- ◆ Surrogate – That surrogate was added to MB, LCS, MS/MSD (if applicable) and every sample prior to analysis, and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. sample result form or QC Summary form, and that non-compliant results were indicated by an asterisk “*”.
- ◆ Lab Control Samples – That LCS (LCD if applicable) was analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Furthermore, if corrective action is not possible (e.g., no more samples to re-analyze) state that results were qualified.
- ◆ Matrix Spike Samples – That MS/MSD (if applicable) were extracted with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- ◆ Sample Analysis – That samples were analyzed in conformance to the method and project requirements. That all positive results were qualitatively identified in accordance to the method and applied quantitation is based on the initial calibration. That pH for all preserved water samples were checked prior to analysis. For samples that are preservation non-compliant and are out of the 7-day holding time, specify the sample(s) in the discussion and that NCR was generated and forwarded to the PM for client information.
- ◆ Other Anomalies (if any) – Shall be discussed on a case by case basis concurred by the Supervisor or the Lab Director. Include NCR in the data package when required by the project, otherwise archive the NCR with the analytical folder.

10.8.4. Submit the analysis package for secondary review.

10.9. Preventive Maintenance

10.9.1. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality.

11.0 QUALITY CONTROL**11.1. Sample Preparation QC**

- 11.1.1. A preparation batch shall consist of a MB, LCS, MS/MSD and ≤ 20 field samples.
- 11.1.2. All lab wares used in the sample preparation shall be properly treated as specified in EMAX-QC07.
- 11.1.3. All solvents and reagents shall undergo quality control check in the stationary laboratory prior to its use.

11.2. Sample Analysis QC

- 11.2.1. Initial Calibration must be established and verified to be considered valid.
- 11.2.2. Analytical batch shall consist of a valid ICAL, QC samples and field samples with Tune Check and DCC every 12-hour analytical sequence.

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11.2.3. A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing organic free water and identifying its result as instrument blank, or using the tune check as instrument blank.

11.2.4. Organic free water shall be used for method blank and LCS for both water and soil matrix.

11.3. Method QC

11.3.1. Method Detection Limit Study must be established before the analytical procedure can be used.

11.3.2. Retention Time Window must be established and updated accordingly.

11.3.3. Method proficiency must be established before the analytical procedure can be used.

11.3.4. All analysts conducting this analysis must have established demonstration of proficiency.

12.0 CORRECTIVE ACTION

12.1. Implement corrective action as described in Appendix 1.

12.2. Initial Calibration

12.2.1. If the %RSD is out of acceptance criteria, review the results and identify presence of an outlier.

- ◆ If one of the standards returns a bias low or bias high on all of the analytes then that point is considered an out-liner. Prepare a standard at that ICAL point and re-analyze.
- ◆ If the highest ICAL point appears to be saturated, drop the highest point.
- ◆ If the lowest point returns a bias low response or the peaks are not distinct and sharp, drop the lowest point.

Note : The lowest calibration point identifies the reporting limit (RL). Therefore, check that the RL is in conformance to the current projects where the ICAL will be used.

12.3. A Non-Conformance Report (NCR) shall be required when anomalies other than specified in Appendix 1 is observed. Refer to EMAX-QA08 for NCR details.

12.4. Discuss water samples that are labeled preserved having a pH value > 2 in the case narrative.

13.0 POLLUTION PREVENTION

13.1. Endorse all unused samples to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.

13.2. Separate and properly identify all unused expired analytical standards prior to endorsing them to WDU for proper disposal.

14.0 WASTE MANAGEMENT

14.1. Dispose all unused samples, expired analytical standards and other waste generated during the analytical process in accordance to EMAX-SM-03.

15.0 SUPPLEMENTARY NOTES

15.1. **Definition of Terms**

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- 15.1.1. Batch – A group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
- 15.1.1.1 **Preparation batch** is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
- 15.1.1.2 **Analytical batch** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.2. Calibration – A determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.3. Carry-over – Are contaminants retained in the instrument/apparatus from a highly contaminated sample that is passed into the succeeding sample(s).
- 15.1.4. CCC – Calibration check compounds that evaluate the integrity of the system. Variability of these compounds may indicate system leak or reactive sites in the column.
- 15.1.5. Instrument Method – A file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.6. Instrument Blank – A target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.7. Lab Control Sample (LCS) – A target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.8. Matrix – is a component or form of a sample.
- 15.1.9. Matrix Spike (MS) –A sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.10. Matrix Spike Duplicate (MSD) – A replicate of MS analyzed to monitor precision or recovery.
- 15.1.11. Method Blank – A target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.12. MSDS – Material safety data sheet is where the physical data, toxicology and safety precaution of a certain substance is listed.
- 15.1.13. Response Factor – The ratio of the peak area of the target compound in the sample or sample extracts to the peak area of the internal standard in the sample or sample extract.
- 15.1.14. Sample – A specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.15. Sample Duplicate – A replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.16. SPCC – System performance check compounds are compounds that are used to check compound stability and to check for degradation cause by contaminated lines or active sites in the system.

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15.2. Application of EMAX QC Procedures

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing volatile analysis by GC/MS. The standard analyte list and RL are presented in Tables 7 & 8. In instances where there is a project or program QAPP, the requirements given in the project shall take precedence over this SOP.

15.3. Air Force Center for Environmental Excellence (AFCEE) Projects

15.3.1. When samples from AFCEE sponsored projects are analyzed for volatiles by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the Quality Assurance Project Plan, the latest version.

15.4. U.S. Army Corps of Engineers (USACE) Projects

15.4.1. When samples from USACE sponsored projects are analyzed for volatiles by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, Shell Document latest version shall be applied.

15.5. Naval Facilities Engineering Service Center (NFESC) Projects

15.5.1. When samples from NFESC sponsored projects are analyzed for volatiles by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, NFESC Laboratory Quality Assurance Guide latest version shall be applied.

15.6. Department of Energy Basic Ordering Agreement (DOE-BOA) Projects

15.6.1. For samples from DOE-BOA sponsored projects follow BOA Guidance Document, latest version in the absence of project QAPP.

16.0 REFERENCES

- 16.1. U.S. EPA Method 8260B; SW846, as updated.
- 16.2. EMAX Quality Systems Manual, as updated.

17.0 FIGURES, TABLES & APPENDICES FORMS**17.1. Figures**

- 17.1.1. Figure 1 Peak Evaluation Technique
- 17.1.2. Figure 2 Typical Chromatogram
- 16.1.1. Figure 3 Typical ICAL Summary
- 16.1.2. Figure 4 Typical Instrument Performance Check (Tuning)
- 16.1.3. Figure 5 Typical Internal Standard Area and Retention Time Summary
- 16.1.4. Figure 6 Typical Sample Result Summary
- 16.1.5. Figure 7 Typical LCS Report Summary
- 16.1.6. Figure 8 Typical MS/MSD Report Summary
- 16.1.7. Figure 9 Typical Case Narrative

17.2. Tables

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- 17.2.1. Table 1 Initial Calibration Standard Preparation
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 - 17.2.5. Table 5 Analyte Concentration Levels of Calibration and QC Standards
 - 17.2.6. Table 6 BFB Key Ion Abundance Criteria
 - 17.2.7. Table 7 Standard Analyte List I
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- 17.3. **Appendices**
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- 17.4.1. Analytical Run Log
 - 17.4.2. Instrument Maintenance Log

Figure 1 - Peak Evaluation Technique

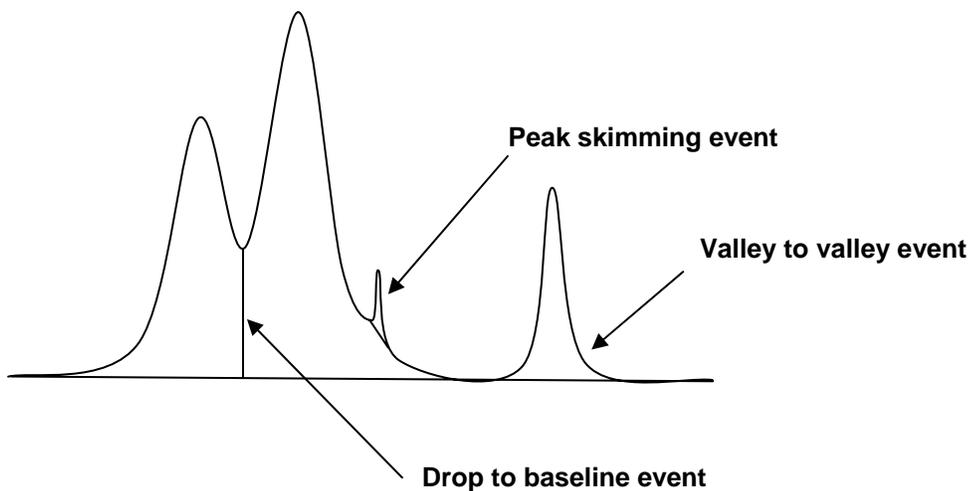


Figure 2 - TYPICAL CHROMATOGRAM

Data File : D:\INSTRUM\1\DATA\0611\0611170.D
Date : 6 Sep 2006 11:16 am
Sample : 470000779 10/20/50ppb
File : I:\ppm\470000779\0611\0611170.D
MS Integration Param: 512KBIT, 0
Print Time: Sep 6 11:44 2006
Vial: 2
Operator: JH
Inst: 3
Multiplier: 1.00
Output Results File: 470000779.D

Method : D:\INSTRUM\1\METHODS\0611\0611170.D (MS Integration)
Title : METHOD 8260
Last Update : Mon Aug 07 10:07:06 2006
Response via : Initial Calibration

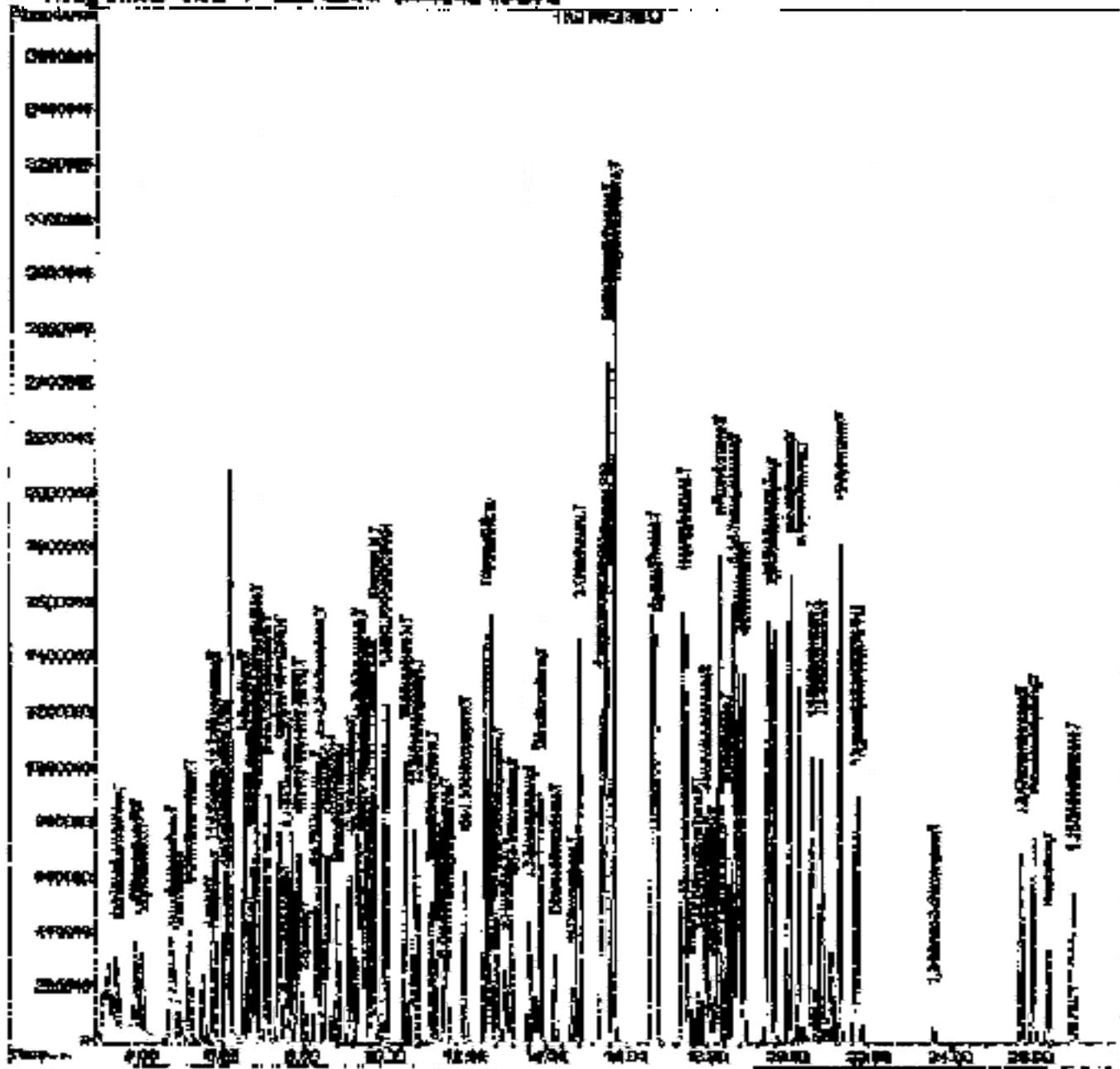


Figure 3 – TYPICAL ICAL SUMMARY
INITIAL_CALIBRATION - RELATIVE_RESPONSE_FACTOR

Instrument ID :D3
Beginning Date/Time :08/07/06 11:07
Spike Units :PPB
IC File :RHE008

Column Spec :RTX502.2 ID :0.32MM
Ending Date/Time :08/07/06 16:31
HPCHEM Method :VOD3H07

| M | IDX | Parameters | .3 | .5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | Av_RRF | %_RSD | Av_Rt_M |
|----|---------------------------------------|------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|---------|---------|---------|
| | | | 11:07 RHE003 | 11:42 RHE004 | 12:19 RHE005 | 12:55 RHE006 | 13:31 RHE007 | 14:07 RHE008 | 14:43 RHE009 | 15:19 RHE010 | 15:55 RHE011 | 16:31 RHE012 | | | |
| 1 | 1,4-DIFLUOROBENZENE | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 9.9722 |
| 2 | Dichlorodifluoromethane | 0.274 | 0.315 | 0.372 | 0.419 | 0.410 | 0.363 | 0.344 | 0.303 | 0.313 | 0.346 | 14.27 | 3.3830 | | |
| 3 | Chloromethane | 0.482 | 0.557 | 0.611 | 0.649 | 0.630 | 0.560 | 0.455 | 0.453 | 0.492 | 0.543 | 13.95 | 3.8441 | | |
| 4 | Vinyl chloride | 0.362 | 0.417 | 0.492 | 0.525 | 0.484 | 0.360 | 0.350 | 0.329 | 0.301 | 0.320 | 0.321 | 13.23 | 4.7101 | |
| 5 | Bromomethane | 0.244 | 0.277 | 0.320 | 0.368 | 0.376 | 0.350 | 0.329 | 0.301 | 0.320 | 0.321 | 13.23 | 4.7101 | | |
| 6 | Chloroethane | 0.211 | 0.225 | 0.280 | 0.320 | 0.322 | 0.288 | 0.286 | 0.254 | 0.261 | 0.272 | 14.08 | 4.8043 | | |
| 7 | Trichlorofluoromethane | 0.316 | 0.321 | 0.390 | 0.452 | 0.458 | 0.407 | 0.386 | 0.348 | 0.355 | 0.381 | 13.56 | 5.1910 | | |
| 8 | sec-Propyl alcohol | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.0000 | |
| 9 | Acrolein | 0.015 | 0.020 | 0.021 | 0.021 | 0.021 | 0.021 | 0.021 | 0.020 | 0.020 | 0.020 | 10.20 | 5.7219 | | |
| 10 | 1,1,2-Trichloro-1,2,2-trifluoroethane | 0.204 | 0.210 | 0.186 | 0.198 | 0.227 | 0.217 | 0.222 | 0.226 | 0.210 | 0.195 | 0.210 | 6.64 | 5.7630 | |
| 11 | Acetone | 0.065 | 0.051 | 0.043 | 0.043 | 0.039 | 0.037 | 0.034 | 0.034 | 0.043 | 0.043 | 26.09 | 5.7942 | | |
| 12 | 1,1-Dichloroethene | 0.476 | 0.462 | 0.390 | 0.455 | 0.503 | 0.496 | 0.523 | 0.486 | 0.434 | 0.428 | 0.465 | 8.62 | 6.0337 | |
| 13 | tert-Butyl alcohol | 0.007 | 0.008 | 0.009 | 0.010 | 0.012 | 0.012 | 0.012 | 0.012 | 0.013 | 0.010 | 20.88 | 6.0454 | | |
| 14 | Methyl acetate | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.0000 | |
| 15 | Iodomethane | 0.268 | 0.407 | 0.508 | 0.514 | 0.541 | 0.504 | 0.463 | 0.472 | 0.460 | 19.05 | 6.4879 | | | |
| 16 | Methylene chloride | 0.586 | 0.473 | 0.478 | 0.480 | 0.446 | 0.454 | 0.433 | 0.394 | 0.397 | 0.460 | 12.38 | 6.6503 | | |
| 17 | Carbon disulfide | 1.181 | 1.159 | 1.067 | 1.301 | 1.425 | 1.347 | 1.389 | 1.304 | 1.165 | 1.150 | 1.249 | 9.58 | 6.7773 | |
| 18 | Acrylonitrile | 0.040 | 0.045 | 0.051 | 0.050 | 0.050 | 0.048 | 0.047 | 0.048 | 0.047 | 0.048 | 0.047 | 7.30 | 6.7612 | |
| 19 | tert-Butyl methyl ether (MTBE) | 0.419 | 0.473 | 0.420 | 0.414 | 0.440 | 0.413 | 0.427 | 0.460 | 0.466 | 0.486 | 0.442 | 6.12 | 6.7997 | |
| 20 | trans-1,2-Dichloroethene | 0.483 | 0.506 | 0.444 | 0.485 | 0.542 | 0.519 | 0.531 | 0.532 | 0.486 | 0.468 | 0.500 | 6.36 | 7.0421 | |
| 21 | Acetonitrile | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.0000 | |
| 22 | Isopropyl ether (DIPE) | 1.146 | 1.184 | 1.131 | 1.142 | 1.149 | 1.101 | 1.113 | 1.185 | 1.148 | 1.190 | 1.149 | 2.63 | 7.3485 | |
| 23 | Vinyl acetate | 0.188 | 0.173 | 0.174 | 0.193 | 0.200 | 0.226 | 0.246 | 0.249 | 0.206 | 0.206 | 14.85 | 7.4911 | | |
| 24 | 1,1-Dichloroethane | 0.540 | 0.560 | 0.494 | 0.574 | 0.651 | 0.635 | 0.660 | 0.632 | 0.571 | 0.574 | 0.589 | 9.10 | 7.5835 | |
| 25 | tert-Butyl ethyl ether (ETBE) | 0.740 | 0.786 | 0.754 | 0.767 | 0.751 | 0.729 | 0.740 | 0.788 | 0.780 | 0.817 | 0.765 | 3.58 | 7.8750 | |
| 26 | 2-Butanone | 0.061 | 0.062 | 0.060 | 0.057 | 0.056 | 0.059 | 0.058 | 0.061 | 0.059 | 0.059 | 3.47 | 8.0497 | | |
| 27 | 2,2-Dichloropropane | 0.205 | 0.211 | 0.220 | 0.269 | 0.280 | 0.322 | 0.343 | 0.334 | 0.322 | 0.278 | 19.88 | 8.3129 | | |
| 28 | cis-1,2-Dichloroethene | 0.483 | 0.517 | 0.439 | 0.479 | 0.535 | 0.517 | 0.542 | 0.542 | 0.496 | 0.493 | 0.504 | 6.49 | 8.3703 | |
| 29 | tert-Butyl formate (TBF) | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.0000 | |
| 30 | Chloroform | 0.510 | 0.520 | 0.454 | 0.503 | 0.555 | 0.534 | 0.556 | 0.555 | 0.516 | 0.500 | 0.520 | 6.15 | 8.5726 | |
| 31 | Bromochloromethane | 0.158 | 0.187 | 0.194 | 0.231 | 0.256 | 0.243 | 0.256 | 0.242 | 0.223 | 0.228 | 0.222 | 14.60 | 8.8106 | |
| 32 | Tetrahydrofuran | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.0000 | |
| 33 | 1,1,1-Trichloroethane | 0.367 | 0.390 | 0.350 | 0.368 | 0.416 | 0.409 | 0.418 | 0.422 | 0.401 | 0.375 | 0.392 | 6.45 | 9.1214 | |
| 34 | Cyclohexane | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.0000 | |
| 35 | tert-Amyl methyl ether (TAME) | 0.653 | 0.691 | 0.665 | 0.655 | 0.653 | 0.630 | 0.641 | 0.687 | 0.681 | 0.712 | 0.667 | 3.81 | 9.3936 | |
| 36 | 1,2-Dichloroethane-d4 | 0.158 | 0.173 | 0.172 | 0.198 | 0.199 | 0.183 | 0.195 | 0.184 | 0.177 | 0.182 | 7.44 | 9.5027 | | |
| 37 | CHLOROBENZENE-D5 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 15.3832 | |
| 38 | 1,1-Dichloropropene | 0.196 | 0.185 | 0.169 | 0.174 | 0.186 | 0.183 | 0.185 | 0.193 | 0.187 | 0.180 | 0.184 | 4.40 | 9.3177 | |
| 39 | Carbon tetrachloride | 0.366 | 0.375 | 0.331 | 0.357 | 0.395 | 0.399 | 0.406 | 0.416 | 0.398 | 0.373 | 0.382 | 6.74 | 9.4903 | |
| 40 | 1,2-Dichloroethane | 0.237 | 0.252 | 0.241 | 0.238 | 0.261 | 0.258 | 0.260 | 0.273 | 0.266 | 0.260 | 0.255 | 4.82 | 9.6271 | |
| 41 | Benzene | 1.598 | 1.612 | 1.458 | 1.533 | 1.600 | 1.573 | 1.570 | 1.640 | 1.593 | 1.557 | 1.573 | 3.20 | 9.6955 | |
| 42 | Methylcyclohexane | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.0000 | |
| 43 | Trichloroethene | 0.420 | 0.421 | 0.392 | 0.406 | 0.419 | 0.416 | 0.420 | 0.438 | 0.427 | 0.415 | 0.418 | 2.89 | 10.5210 | |
| 44 | 1,2-Dichloropropane | 0.367 | 0.390 | 0.369 | 0.380 | 0.399 | 0.393 | 0.398 | 0.416 | 0.407 | 0.404 | 0.392 | 4.10 | 10.7590 | |
| 45 | Bromodichloromethane | 0.355 | 0.347 | 0.327 | 0.344 | 0.378 | 0.376 | 0.389 | 0.408 | 0.403 | 0.397 | 0.372 | 7.46 | 11.1353 | |
| 46 | Dibromomethane | 0.114 | 0.132 | 0.127 | 0.134 | 0.152 | 0.147 | 0.152 | 0.156 | 0.151 | 0.146 | 0.142 | 9.51 | 11.2498 | |
| 47 | 2-Chloroethyl vinyl ether | 0.034 | 0.023 | 0.025 | 0.025 | 0.025 | 0.028 | 0.028 | 0.028 | 0.030 | 0.028 | 14.05 | 11.4632 | | |
| 48 | 4-Methyl-2-pentanone | 0.210 | 0.191 | 0.192 | 0.184 | 0.175 | 0.188 | 0.193 | 0.206 | 0.192 | 0.192 | 5.91 | 11.5115 | | |
| 49 | cis-1,3-Dichloropropene | 0.444 | 0.488 | 0.450 | 0.461 | 0.481 | 0.486 | 0.493 | 0.524 | 0.516 | 0.518 | 0.486 | 5.74 | 11.9638 | |
| 50 | Toluene-d8 | 1.086 | 1.168 | 1.229 | 1.335 | 1.366 | 1.210 | 1.297 | 1.232 | 1.209 | 1.237 | 6.94 | 12.4477 | | |
| 51 | Toluene | 0.924 | 0.930 | 0.840 | 0.906 | 0.931 | 0.921 | 0.918 | 0.946 | 0.903 | 0.894 | 0.911 | 3.21 | 12.5929 | |
| 52 | Ethyl methacrylate | 0.269 | 0.256 | 0.262 | 0.261 | 0.254 | 0.261 | 0.288 | 0.290 | 0.306 | 0.272 | 6.66 | 12.6807 | | |
| 53 | trans-1,3-Dichloropropene | 0.278 | 0.305 | 0.288 | 0.305 | 0.320 | 0.326 | 0.333 | 0.361 | 0.358 | 0.366 | 0.324 | 9.50 | 12.8131 | |
| 54 | 2-Hexanone | 0.133 | 0.125 | 0.124 | 0.118 | 0.113 | 0.120 | 0.122 | 0.131 | 0.123 | 0.123 | 5.43 | 13.0360 | | |
| 55 | 1,1,2-Trichloroethane | 0.162 | 0.193 | 0.173 | 0.184 | 0.187 | 0.183 | 0.186 | 0.201 | 0.200 | 0.205 | 0.188 | 6.98 | 13.1432 | |
| 56 | 1,3-Dichloropropane | 0.365 | 0.381 | 0.355 | 0.360 | 0.368 | 0.364 | 0.370 | 0.394 | 0.391 | 0.398 | 0.374 | 4.08 | 13.6043 | |
| 57 | Tetrachloroethene | 0.329 | 0.308 | 0.284 | 0.299 | 0.306 | 0.306 | 0.304 | 0.313 | 0.305 | 0.300 | 0.305 | 3.69 | 13.8378 | |

| M | IDX | Parameters | .3 | .5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 50 | Av_RRF | %_RSD | Av_Rt_M |
|---|-----|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|-------|---------|
| | | | RHE003 | RHE004 | RHE005 | RHE006 | RHE007 | RHE008 | RHE009 | RHE010 | RHE011 | RHE012 | | | |
| | 58 | Dibromochloromethane | 0.192 | 0.187 | 0.188 | 0.203 | 0.220 | 0.222 | 0.230 | 0.248 | 0.249 | 0.251 | 0.219 | 11.63 | 14.2186 |
| | 59 | 1,2-Dibromoethane | 0.166 | 0.186 | 0.177 | 0.182 | 0.188 | 0.184 | 0.187 | 0.198 | 0.200 | 0.205 | 0.187 | 6.24 | 14.6306 |
| | 60 | 2-Ethyl-1-butanol | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | 0.000 | 0.00 | 0.0000 |
| | 61 | 1-Chlorohexane | 0.727 | 0.690 | 0.628 | 0.685 | 0.711 | 0.701 | 0.705 | 0.722 | 0.689 | 0.686 | 0.694 | 3.98 | 14.7912 |
| | 62 | Chlorobenzene | 0.933 | 0.941 | 0.861 | 0.908 | 0.936 | 0.914 | 0.918 | 0.938 | 0.906 | 0.901 | 0.915 | 2.64 | 15.4635 |
| | 63 | 1,1,1,2-Tetrachloroethane | 0.264 | 0.286 | 0.269 | 0.284 | 0.287 | 0.289 | 0.290 | 0.307 | 0.303 | 0.309 | 0.289 | 5.13 | 15.5141 |
| | 64 | Ethylbenzene | 1.818 | 1.782 | 1.588 | 1.738 | 1.805 | 1.754 | 1.747 | 1.760 | 1.680 | 1.656 | 1.733 | 4.13 | 15.5201 |
| 2 | 65 | m-Xylene & p-Xylene | 1.415 | 1.373 | 1.225 | 1.385 | 1.397 | 1.354 | 1.342 | 1.341 | 1.268 | 1.258 | 1.336 | 4.81 | 15.6718 |
| | 66 | o-Xylene | 1.311 | 1.293 | 1.163 | 1.337 | 1.364 | 1.321 | 1.317 | 1.325 | 1.266 | 1.271 | 1.297 | 4.28 | 16.6266 |
| | 67 | Styrene | 0.895 | 0.907 | 0.843 | 0.954 | 0.996 | 0.966 | 0.980 | 0.989 | 0.943 | 0.956 | 0.943 | 5.08 | 16.6891 |
| | 68 | 1,2-DICHLOROBENZENE-D4 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 21.6599 |
| | 69 | Bromoform | 0.243 | 0.236 | 0.240 | 0.234 | 0.261 | 0.273 | 0.287 | 0.327 | 0.334 | 0.334 | 0.277 | 14.93 | 17.5146 |
| | 70 | Isopropylbenzene | 4.912 | 4.822 | 4.185 | 4.483 | 4.698 | 4.648 | 4.624 | 4.848 | 4.659 | 4.417 | 4.630 | 4.75 | 17.3822 |
| | 71 | 1,1,2,2-Tetrachloroethane | 0.575 | 0.668 | 0.607 | 0.577 | 0.600 | 0.600 | 0.609 | 0.684 | 0.691 | 0.697 | 0.631 | 7.69 | 17.7392 |
| | 72 | 4-Bromofluorobenzene | 1.141 | 1.130 | 1.129 | 1.138 | 1.224 | 1.262 | 1.123 | 1.222 | 1.177 | 1.126 | 1.167 | 4.37 | 17.9772 |
| | 73 | 1,2,3-Trichloropropane | ----- | 0.118 | 0.123 | 0.127 | 0.134 | 0.135 | 0.135 | 0.149 | 0.153 | 0.152 | 0.136 | 9.49 | 18.0832 |
| | 74 | trans-1,4-Dichloro-2-butene | ----- | ----- | 0.087 | 0.093 | 0.106 | 0.106 | 0.110 | 0.122 | 0.124 | 0.127 | 0.109 | 13.14 | 18.1842 |
| | 75 | n-Propylbenzene | 6.590 | 6.394 | 5.699 | 6.067 | 6.366 | 6.332 | 6.268 | 6.422 | 6.113 | 5.857 | 6.211 | 4.43 | 18.2910 |
| | 76 | Bromobenzene | 0.951 | 0.919 | 0.816 | 0.856 | 0.882 | 0.883 | 0.895 | 0.942 | 0.914 | 0.889 | 0.895 | 4.46 | 18.4814 |
| | 77 | 1,3,5-Trimethylbenzene | 3.837 | 3.691 | 3.355 | 3.637 | 3.826 | 3.755 | 3.753 | 3.781 | 3.590 | 3.476 | 3.670 | 4.29 | 18.6435 |
| | 78 | 2-Chlorotoluene | 3.278 | 3.461 | 2.747 | 2.921 | 3.316 | 3.206 | 3.211 | 3.243 | 3.169 | 2.995 | 3.155 | 6.64 | 18.7566 |
| | 79 | 4-Chlorotoluene | 3.484 | 3.172 | 3.046 | 3.214 | 3.365 | 3.095 | 3.074 | 3.248 | 3.022 | 3.005 | 3.173 | 4.98 | 18.8517 |
| | 80 | tert-Butylbenzene | 3.577 | 3.482 | 3.022 | 3.217 | 3.447 | 3.401 | 3.394 | 3.454 | 3.320 | 3.181 | 3.349 | 4.96 | 19.4794 |
| | 81 | 1,2,4-Trimethylbenzene | 3.729 | 3.735 | 3.356 | 3.592 | 3.820 | 3.699 | 3.731 | 3.701 | 3.523 | 3.427 | 3.631 | 4.17 | 19.5731 |
| | 82 | sec-Butylbenzene | 5.655 | 5.310 | 4.770 | 5.059 | 5.414 | 5.309 | 5.299 | 5.430 | 5.174 | 4.927 | 5.235 | 4.96 | 19.9792 |
| | 83 | p-Isopropyltoluene | 4.437 | 4.360 | 3.966 | 4.202 | 4.547 | 4.472 | 4.423 | 4.453 | 4.241 | 4.086 | 4.319 | 4.36 | 20.2826 |
| | 84 | 1,3-Dichlorobenzene | 2.026 | 1.970 | 1.796 | 1.828 | 1.937 | 1.896 | 1.903 | 1.943 | 1.874 | 1.843 | 1.902 | 3.67 | 20.5845 |
| | 85 | 1,4-Dichlorobenzene | 1.926 | 1.933 | 1.694 | 1.810 | 1.898 | 1.833 | 1.836 | 1.861 | 1.802 | 1.772 | 1.836 | 3.98 | 20.8240 |
| | 86 | n-Butylbenzene | 4.586 | 4.549 | 4.210 | 4.475 | 4.769 | 4.688 | 4.640 | 4.552 | 4.381 | 4.222 | 4.507 | 4.15 | 21.2553 |
| | 87 | 1,2-Dichlorobenzene | 1.577 | 1.667 | 1.478 | 1.542 | 1.595 | 1.556 | 1.573 | 1.607 | 1.558 | 1.539 | 1.569 | 3.14 | 21.7268 |
| | 88 | 1,2-Dibromo-3-chloropropane | ----- | ----- | 0.062 | 0.066 | 0.077 | 0.078 | 0.084 | 0.092 | ----- | ----- | 0.077 | 14.25 | 23.5818 |
| | 89 | 1,2,4-Trichlorobenzene | 1.068 | 1.114 | 1.000 | 1.067 | 1.112 | 1.102 | 1.124 | 1.071 | 1.060 | 1.083 | 1.080 | 3.37 | 25.7293 |
| | 90 | Hexachlorobutadiene | 0.715 | 0.673 | 0.616 | 0.664 | 0.721 | 0.725 | 0.725 | 0.735 | 0.711 | 0.693 | 0.698 | 5.32 | 26.0536 |
| | 91 | Naphthalene | 1.649 | 1.692 | 1.515 | 1.553 | 1.589 | 1.572 | 1.632 | 1.580 | 1.625 | 1.683 | 1.609 | 3.54 | 26.3852 |
| | 92 | 1,2,3-Trichlorobenzene | 0.795 | 0.886 | 0.812 | 0.857 | 0.906 | 0.882 | 0.904 | 0.864 | 0.865 | 0.883 | 0.866 | 4.21 | 26.9832 |

Spike Amount = Nominal Amount * M

Ave_%RSD : 7.7 Max_%RSD : 26.1

Use Least Square Linear Regression with weighting factor of inverse concentration for comps with %_RSD > 15

Resp_Ratio = x0 + x1 * Amt_Ratio

| IDX | Parameter | x0 | x1 | CCF |
|-----|---------------------|----------|---------|---------|
| 4 | Vinyl chloride | 0.00366 | 0.41636 | 0.9830* |
| 11 | Acetone | 0.01471 | 0.03344 | 0.9987 |
| 13 | tert-Butyl alcohol | -0.00409 | 0.01232 | 0.9988 |
| 15 | Iodomethane | -0.01397 | 0.49320 | 0.9977 |
| 27 | 2,2-Dichloropropane | -0.01157 | 0.32898 | 0.9985 |

Use Quadratic Regression of inv conc w.f. for comps of linear reg of inv conc w.f. with CCF < .995

Resp_Ratio = x0 + x1 * Amt_Ratio + x2 * Amt_Ratio * Amt_Ratio

| IDX | Parameter | x0 | x1 | x2 | CCF2 |
|-----|----------------|----------|---------|----------|--------|
| 4 | Vinyl chloride | -0.01475 | 0.61251 | -0.12159 | 0.9996 |

Figure 6 – TYPICAL SAMPLE RESULT SUMMARY

SW 5030B/8260B
VOLATILE ORGANICS BY GC/MS

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=====
Client       : XYZ, INC.                Date Collected: 08/30/06
Project      : CLEAN LAND PROJECT      Date Received: 08/31/06
Batch No.    : 06H308                  Date Extracted: 09/06/06 15:29
Sample ID    : V13TB1298              Date Analyzed: 09/06/06 15:29
Lab Samp ID  : H308-01                Dilution Factor: 1
Lab File ID  : RIE146                 Matrix          : WATER
Ext Btch ID  : VOD3I12                % Moisture     : NA
Calib. Ref.  : RHE008                 Instrument ID   : T-OD3
=====

```

| PARAMETERS | RESULTS (ug/L) | RL (ug/L) | MDL (ug/L) |
|-----------------------------|-------------------|--------------|---------------|
| 1,1,1-TRICHLOROETHANE | ND | 1.0 | 0.20 |
| 1,1,2,2-TETRACHLOROETHANE | ND | 0.50 | 0.20 |
| 1,1,2-TRICHLOROETHANE | ND | 1.0 | 0.20 |
| 1,1-DICHLOROETHANE | ND | 1.0 | 0.20 |
| 1,1-DICHLOROETHENE | ND | 1.0 | 0.20 |
| 1,2-DICHLOROBENZENE | ND | 0.50 | 0.20 |
| 1,2-DICHLOROETHANE | ND | 1.0 | 0.20 |
| 1,2-DICHLOROPROPANE | ND | 0.50 | 0.20 |
| 1,2-ETHYLENEDIBROMIDE | ND | 1.0 | 0.20 |
| 1,1,2,2,2-TRIFLUOROETHANE | ND | 1.0 | 0.20 |
| 1,3-DICHLOROBENZENE | ND | 1.0 | 0.20 |
| 1,4-DICHLOROBENZENE | ND | 1.0 | 0.20 |
| BENZENE | ND | 0.40 | 0.20 |
| BROMODICHLOROMETHANE | ND | 0.50 | 0.20 |
| BROMOFORM | ND | 1.0 | 0.30 |
| BROMOMETHANE | ND | 3.0 | 0.20 |
| CARBON TETRACHLORIDE | ND | 1.0 | 0.20 |
| CHLOROBENZENE | ND | 0.50 | 0.20 |
| CHLOROETHANE | ND | 1.0 | 0.20 |
| CHLOROFORM | ND | 0.30 | 0.20 |
| CHLOROMETHANE | ND | 1.0 | 0.20 |
| CIS-1,2-DICHLOROETHENE | ND | 1.0 | 0.20 |
| CIS-1,3-DICHLOROPROPENE | ND | 0.50 | 0.20 |
| DIBROMOCHLOROMETHANE | ND | 0.50 | 0.20 |
| ETHYLBENZENE | ND | 1.0 | 0.20 |
| M,P-XYLENE | ND | 2.0 | 0.50 |
| METHYLENE CHLORIDE | ND | 1.0 | 0.50 |
| O-XYLENE | ND | 1.0 | 0.20 |
| STYRENE | ND | 1.0 | 0.20 |
| TETRACHLOROETHYLENE | ND | 1.0 | 0.20 |
| TOLUENE | ND | 1.0 | 0.20 |
| TRANS-1,2-DICHLOROETHENE | ND | 1.0 | 0.20 |
| TRANS-1,3-DICHLOROPROPENE | ND | 1.0 | 0.20 |
| TRICHLOROETHENE | ND | 1.0 | 0.20 |
| TRICHLOROFUOROMETHANE | ND | 1.0 | 0.50 |
| VINYL CHLORIDE | ND | 1.0 | 0.20 |
| ACETONE | ND | 10 | 5.0 |
| 2-BUTANONE | ND | 10 | 5.0 |
| MTBE | ND | 1.0 | 0.20 |
| METHYL ISOBUTYL KETONE | ND | 10 | 5.0 |
| 2-HEXANONE | ND | 5.0 | 2.5 |
| CARBON DISULFIDE | ND | 1.0 | 0.20 |
| DIISOPROPYLETHER (DIPE) | ND | 5.0 | 0.20 |
| ETHYL-T-BUTYLETHER (ETBE) | ND | 5.0 | 0.20 |
| T-BUTANOL (TBA) | ND | 10 | 5.0 |
| TERT-AMYLMETHYLETHER (TAME) | ND | 5.0 | 0.20 |
| VINYL ACETATE | ND | 1.0 | 0.20 |

| SURROGATE PARAMETERS | % RECOVERY | QC LIMIT |
|-----------------------|------------|----------|
| 1,2-DICHLOROETHANE-D4 | 101 | 72-119 |
| TOLUENE-D8 | 105 | 81-120 |
| 4-BROMOFLUOROBENZENE | 107 | 76-119 |

RL: Reporting Limit

Figure 7 – TYPICAL LCS RESULT SUMMARY

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
BATCH NO.: 06H308
METHOD: SW 5030B/8260B

MATRIX: WATER % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: MBLK1W
LAB SAMP ID: VOD3I12Q VOD3I12L VOD3I12C
LAB FILE ID: RIE144 RIE141 RIE142
DATE EXTRACTED: 09/06/0614:17 09/06/0612:28 09/06/0613:05 DATE COLLECTED: NA
DATE ANALYZED: 09/06/0614:17 09/06/0612:28 09/06/0613:05 DATE RECEIVED: 09/06/06
PREP. BATCH: VOD3I12 VOD3I12 VOD3I12
CALIB. REF: RHE008 RHE008 RHE008

ACCESSION:

| PARAMETER | BLNK RSLT (ug/L) | SPIKE AMT (ug/L) | BS RSLT (ug/L) | BS % REC | SPIKE AMT (ug/L) | BSD RSLT (ug/L) | BSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|---------------------------------------|---------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|------------|-----------------|----------------|
| 1,1,1-Trichloroethane | ND | 10.0 | 9.27 | 93 | 10.0 | 9.43 | 94 | 2 | 67-132 | 20 |
| 1,1,2,2-Tetrachloroethane | ND | 10.0 | 11.7 | 117 | 10.0 | 11.3 | 113 | 3 | 63-128 | 20 |
| 1,1,2-Trichloroethane | ND | 10.0 | 10.9 | 109 | 10.0 | 11.2 | 112 | 3 | 75-125 | 20 |
| 1,1-Dichloroethane | ND | 10.0 | 9.35 | 94 | 10.0 | 9.28 | 93 | 1 | 69-133 | 20 |
| 1,1-Dichloroethene | ND | 10.0 | 7.61 | 76 | 10.0 | 7.67 | 77 | 1 | 68-130 | 20 |
| 1,2-Dichlorobenzene | ND | 10.0 | 9.49 | 95 | 10.0 | 9.95 | 99 | 5 | 71-122 | 20 |
| 1,2-Dichloroethane | ND | 10.0 | 9.65 | 97 | 10.0 | 9.84 | 98 | 2 | 69-132 | 20 |
| 1,2-Dichloropropane | ND | 10.0 | 10.1 | 101 | 10.0 | 10.5 | 105 | 4 | 75-125 | 20 |
| 1,2-Ethylenedibromide | ND | 10.0 | 10.5 | 105 | 10.0 | 10.7 | 107 | 2 | 80-121 | 20 |
| 1,1,2-Trichloro-2,2,2-trifluoroethane | ND | 10.0 | 8.98 | 90 | 10.0 | 9.09 | 91 | 1 | 54-154 | 20 |
| 1,3-Dichlorobenzene | ND | 10.0 | 9.64 | 96 | 10.0 | 10.0 | 100 | 4 | 75-124 | 20 |
| 1,4-Dichlorobenzene | ND | 10.0 | 9.47 | 95 | 10.0 | 9.99 | 100 | 5 | 74-123 | 20 |
| Benzene | ND | 10.0 | 8.67 | 87 | 10.0 | 9.25 | 93 | 6 | 81-122 | 20 |
| Bromodichloromethane | ND | 10.0 | 9.98 | 100 | 10.0 | 10.1 | 101 | 1 | 76-121 | 20 |
| Bromoform | ND | 10.0 | 12.0 | 120 | 10.0 | 11.7 | 117 | 2 | 69-128 | 20 |
| Bromomethane | ND | 10.0 | 10.3 | 103 | 10.0 | 11.1 | 111 | 7 | 53-141 | 20 |
| Carbon Tetrachloride | ND | 10.0 | 9.40 | 94 | 10.0 | 9.82 | 98 | 4 | 66-138 | 20 |
| Chlorobenzene | ND | 10.0 | 9.62 | 96 | 10.0 | 10.2 | 102 | 6 | 81-122 | 20 |
| Chloroethane | ND | 10.0 | 9.63 | 96 | 10.0 | 10.2 | 102 | 6 | 58-133 | 20 |
| Chloroform | ND | 10.0 | 8.78 | 88 | 10.0 | 8.91 | 89 | 1 | 69-128 | 20 |
| Chloromethane | ND | 10.0 | 8.87 | 89 | 10.0 | 10.5 | 105 | 17 | 56-131 | 20 |
| cis-1,2-Dichloroethene | ND | 10.0 | 9.18 | 92 | 10.0 | 9.26 | 93 | 1 | 72-126 | 20 |
| cis-1,3-Dichloropropene | ND | 10.0 | 9.76 | 98 | 10.0 | 10.2 | 102 | 4 | 69-131 | 20 |
| Dibromochloromethane | ND | 10.0 | 10.5 | 105 | 10.0 | 10.7 | 107 | 2 | 66-133 | 20 |
| Ethylbenzene | ND | 10.0 | 9.31 | 93 | 10.0 | 9.89 | 99 | 6 | 73-127 | 20 |
| m,p-Xylene | ND | 20.0 | 17.9 | 89 | 20.0 | 19.1 | 96 | 7 | 76-128 | 20 |
| Methylene Chloride | ND | 10.0 | 7.38 | 74 | 10.0 | 7.71 | 77 | 4 | 63-137 | 20 |
| o-Xylene | ND | 10.0 | 9.22 | 92 | 10.0 | 9.82 | 98 | 6 | 80-121 | 20 |
| Styrene | ND | 10.0 | 9.94 | 99 | 10.0 | 10.5 | 105 | 6 | 65-134 | 20 |
| Tetrachloroethylene | ND | 10.0 | 8.89 | 89 | 10.0 | 9.47 | 95 | 6 | 66-128 | 20 |
| Toluene | ND | 10.0 | 9.41 | 94 | 10.0 | 9.89 | 99 | 5 | 77-122 | 20 |
| Trans-1,2-Dichloroethene | ND | 10.0 | 8.51 | 85 | 10.0 | 8.76 | 88 | 3 | 63-137 | 20 |
| Trans-1,3-Dichloropropene | ND | 10.0 | 10.5 | 105 | 10.0 | 10.7 | 107 | 2 | 59-135 | 20 |
| Trichloroethene | ND | 10.0 | 9.28 | 93 | 10.0 | 9.96 | 100 | 7 | 70-127 | 20 |
| Trichlorofluoromethane | ND | 10.0 | 9.84 | 98 | 10.0 | 10.2 | 102 | 3 | 57-129 | 20 |
| Vinyl Chloride | ND | 10.0 | 7.55 | 75 | 10.0 | 8.26 | 83 | 9 | 50-134 | 20 |
| Acetone | ND | 20.0 | 17.0 | 85 | 20.0 | 16.6 | 83 | 2 | 40-135 | 20 |
| 2-Butanone | ND | 20.0 | 20.7 | 104 | 20.0 | 20.4 | 102 | 2 | 49-136 | 20 |
| MTBE | ND | 10.0 | 10.4 | 104 | 10.0 | 10.2 | 102 | 2 | 65-123 | 20 |
| Methyl Isobutyl Ketone | ND | 20.0 | 23.8 | 119 | 20.0 | 23.7 | 118 | 1 | 58-134 | 20 |
| 2-Hexanone | ND | 20.0 | 21.9 | 110 | 20.0 | 21.9 | 110 | 0 | 54-165 | 20 |
| Carbon Disulfide | ND | 10.0 | 7.76 | 78 | 10.0 | 8.44 | 84 | 8 | 45-154 | 20 |
| Diisopropylether (DIPE) | ND | 10.0 | 9.84 | 98 | 10.0 | 10.1 | 101 | 3 | 63-143 | 20 |
| Ethyl-t-butylether (ETBE) | ND | 10.0 | 10.3 | 103 | 10.0 | 10.4 | 104 | 1 | 63-154 | 20 |
| T-butanol (TBA) | ND | 50.0 | 46.9 | 94 | 50.0 | 44.7 | 89 | 5 | 54-154 | 20 |
| Tert-Butylmethylether (TAME) | ND | 10.0 | 10.1 | 101 | 10.0 | 10.1 | 101 | 1 | 54-154 | 20 |
| Vinyl Acetate | ND | 10.0 | 13.6 | 136 | 10.0 | 13.6 | 136 | 0 | 36-176 | 20 |

| SURROGATE PARAMETER | SPIKE AMT (ug/L) | BS RSLT (ug/L) | BS % REC | SPIKE AMT (ug/L) | BSD RSLT (ug/L) | BSD % REC | QC LIMIT (%) |
|-----------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|-----------------|
| 1,2-Dichloroethane-d4 | 10.0 | 10.4 | 104 | 10.0 | 10.0 | 100 | 72-119 |
| Toluene-d8 | 10.0 | 10.7 | 107 | 10.0 | 10.7 | 107 | 81-120 |
| 4-Bromofluorobenzene | 10.0 | 11.3 | 113 | 10.0 | 11.0 | 110 | 76-119 |

Figure 8 – TYPICAL MS/MSD RESULT SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
BATCH NO.: 07H308
METHOD: SW 5030B/8260B

MATRIX: WATER % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: V14MW4M
LAB SAMP ID: H308-11 H308-11M H308-11S
LAB FILE ID: RIE150 RIE156 RIE157
DATE EXTRACTED: 09/06/0717:54 09/06/0721:31 09/06/0722:07 DATE COLLECTED: 08/30/07
DATE ANALYZED: 09/06/0717:54 09/06/0721:31 09/06/0722:07 DATE RECEIVED: 08/31/07
PREP. BATCH: VOD3I12 VOD3I12 VOD3I12
CALIB. REF: RHE008 RHE008 RHE008

ACCESSION:

| PARAMETER | SMPL RSLT (ug/L) | SPIKE AMT (ug/L) | MS RSLT (ug/L) | MS % REC | SPIKE AMT (ug/L) | MSD RSLT (ug/L) | MSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|--------------------------------|---------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|------------|-----------------|----------------|
| 1,1,1-Trichloroethane | ND | 10.0 | 10.3 | 103 | 10.0 | 10.2 | 102 | 1 | 67-132 | 20 |
| 1,1,2,2-Tetrachloroethane | ND | 10.0 | 11.5 | 115 | 10.0 | 11.4 | 114 | 1 | 63-128 | 20 |
| 1,1,2-Trichloroethane | ND | 10.0 | 11.1 | 111 | 10.0 | 11.2 | 112 | 1 | 75-125 | 20 |
| 1,1-Dichloroethane | ND | 10.0 | 10.2 | 102 | 10.0 | 10.1 | 101 | 1 | 69-133 | 20 |
| 1,1-Dichloroethene | ND | 10.0 | 8.18 | 82 | 10.0 | 8.83 | 88 | 8 | 68-130 | 20 |
| 1,2-Dichlorobenzene | ND | 10.0 | 10.2 | 102 | 10.0 | 10.6 | 106 | 4 | 71-122 | 20 |
| 1,2-Dichloroethane | ND | 10.0 | 10.3 | 103 | 10.0 | 10.0 | 100 | 3 | 69-132 | 20 |
| 1,2-Dichloropropane | ND | 10.0 | 10.8 | 108 | 10.0 | 10.9 | 109 | 0 | 75-125 | 20 |
| 1,2-Ethylendibromide | ND | 10.0 | 10.7 | 107 | 10.0 | 10.6 | 106 | 1 | 80-121 | 20 |
| 112Trichloro122Trifluoroethane | ND | 10.0 | 9.90 | 99 | 10.0 | 9.64 | 96 | 3 | 54-154 | 20 |
| 1,3-Dichlorobenzene | ND | 10.0 | 10.4 | 104 | 10.0 | 10.9 | 109 | 5 | 75-124 | 20 |
| 1,4-Dichlorobenzene | ND | 10.0 | 10.2 | 102 | 10.0 | 10.7 | 107 | 5 | 74-123 | 20 |
| Benzene | 0.266F | 10.0 | 9.72 | 95 | 10.0 | 9.95 | 97 | 2 | 81-122 | 20 |
| Bromodichloromethane | ND | 10.0 | 10.5 | 105 | 10.0 | 10.4 | 104 | 1 | 76-121 | 20 |
| Bromoform | ND | 10.0 | 11.7 | 117 | 10.0 | 11.4 | 114 | 3 | 69-128 | 20 |
| Bromomethane | ND | 10.0 | 9.64 | 96 | 10.0 | 11.6 | 116 | 19 | 53-141 | 20 |
| Carbon Tetrachloride | ND | 10.0 | 10.4 | 104 | 10.0 | 10.2 | 102 | 2 | 66-138 | 20 |
| Chlorobenzene | ND | 10.0 | 10.5 | 105 | 10.0 | 10.8 | 108 | 2 | 81-122 | 20 |
| Chloroethane | ND | 10.0 | 9.39 | 94 | 10.0 | 10.7 | 107 | 13 | 58-133 | 20 |
| Chloroform | ND | 10.0 | 9.64 | 96 | 10.0 | 9.40 | 94 | 3 | 69-128 | 20 |
| Chloromethane | ND | 10.0 | 9.17 | 92 | 10.0 | 11.1 | 111 | 19 | 56-131 | 20 |
| cis-1,2-Dichloroethene | 21.3 | 10.0 | 33.6 | 123 | 10.0 | 33.5 | 122 | 0 | 72-126 | 20 |
| cis-1,3-Dichloropropene | ND | 10.0 | 10.1 | 101 | 10.0 | 10.2 | 102 | 2 | 69-131 | 20 |
| Dibromochloromethane | ND | 10.0 | 10.5 | 105 | 10.0 | 10.7 | 107 | 2 | 66-133 | 20 |
| Ethylbenzene | ND | 10.0 | 10.2 | 102 | 10.0 | 10.7 | 107 | 5 | 73-127 | 20 |
| m,p-Xylene | ND | 20.0 | 19.4 | 97 | 20.0 | 20.7 | 103 | 6 | 76-128 | 20 |
| Methylene Chloride | ND | 10.0 | 7.83 | 78 | 10.0 | 8.34 | 83 | 6 | 63-137 | 20 |
| o-Xylene | ND | 10.0 | 10.0 | 100 | 10.0 | 10.6 | 106 | 6 | 80-121 | 20 |
| Styrene | ND | 10.0 | 9.78 | 98 | 10.0 | 10.4 | 104 | 6 | 65-134 | 20 |
| Tetrachloroethylene | ND | 10.0 | 9.83 | 98 | 10.0 | 9.97 | 100 | 1 | 66-128 | 20 |
| Toluene | ND | 10.0 | 10.3 | 103 | 10.0 | 10.6 | 106 | 3 | 77-122 | 20 |
| Trans-1,2-Dichloroethene | 0.746F | 10.0 | 10.2 | 95 | 10.0 | 10.1 | 94 | 1 | 63-137 | 20 |
| Trans-1,3-Dichloropropene | ND | 10.0 | 10.4 | 104 | 10.0 | 10.5 | 105 | 2 | 59-135 | 20 |
| Trichloroethene | ND | 10.0 | 10.5 | 105 | 10.0 | 10.5 | 105 | 0 | 70-127 | 20 |
| Trichlorofluoromethane | ND | 10.0 | 9.45 | 95 | 10.0 | 11.0 | 110 | 15 | 57-129 | 20 |
| Vinyl Chloride | 8.33 | 10.0 | 18.7 | 104 | 10.0 | 19.3 | 110 | 6 | 50-134 | 20 |
| Acetone | ND | 20.0 | 28.6 | 143* | 20.0 | 18.2 | 91 | 44* | 40-135 | 20 |
| 2-Butanone | ND | 20.0 | 19.6 | 98 | 20.0 | 18.5 | 93 | 6 | 49-136 | 20 |
| MTBE | ND | 10.0 | 10.2 | 102 | 10.0 | 10.1 | 101 | 1 | 65-123 | 20 |
| Methyl Isobutyl Ketone | ND | 20.0 | 19.6 | 98 | 20.0 | 19.6 | 98 | 0 | 58-134 | 20 |
| 2-Hexanone | ND | 20.0 | 20.1 | 101 | 20.0 | 19.8 | 99 | 2 | 54-165 | 20 |
| Carbon Disulfide | ND | 10.0 | 8.05 | 81 | 10.0 | 9.14 | 91 | 13 | 45-154 | 20 |
| Diisopropylether (DIPE) | ND | 10.0 | 10.3 | 103 | 10.0 | 10.5 | 105 | 2 | 63-143 | 20 |
| Ethyl-t-butylether (ETBE) | ND | 10.0 | 10.5 | 105 | 10.0 | 10.6 | 106 | 1 | 63-154 | 20 |
| T-butanol (TBA) | ND | 50.0 | 44.9 | 90 | 50.0 | 43.0 | 86 | 4 | 54-154 | 20 |
| Tert-Amylmethylether (TAME) | ND | 10.0 | 10.1 | 101 | 10.0 | 10.2 | 102 | 2 | 54-154 | 20 |
| Vinyl Acetate | ND | 10.0 | 15.6 | 156 | 10.0 | 15.0 | 150 | 4 | 36-176 | 20 |

| SURROGATE PARAMETER | SPIKE AMT (ug/L) | MS RSLT (ug/L) | MS % REC | SPIKE AMT (ug/L) | MSD RSLT (ug/L) | MSD % REC | QC LIMIT (%) |
|-----------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|-----------------|
| 1,2-Dichloroethane-d4 | 10.0 | 10.2 | 102 | 10.0 | 9.41 | 94 | 72-119 |
| Toluene-d8 | 10.0 | 10.7 | 107 | 10.0 | 10.7 | 107 | 81-120 |
| 4-Bromofluorobenzene | 10.0 | 11.4 | 114 | 10.0 | 11.1 | 111 | 76-119 |

Figure 9 – TYPICALCASE NARRATIVE

CASE NARRATIVE

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
SDG: 07H308

**METHOD SW8260B
VOLATILE ORGANICS BY GC/MS**

A total of 1 water and 19 soil samples were received on 7/9/2007 for Volatile Organics by GC/MS Method SW8260B in accordance with USEPA SW846, 3rd Edition.

HOLDING TIME

Samples were analyzed within holding time as prescribed by the project.

INITIAL CALIBRATION

Multi-calibration points were analyzed to establish ICAL. Least square linear regression was applied to all compounds having %RSD > 20. All project calibrations requirements were satisfied. Refer to Initial Calibration Relative Response Factor summary form.

Initial Calibration was verified using a secondary source. All analytes met the project requirement. Refer to the ICV following the ICAL report.

INSTRUMENT TUNING CHECK

Instrument tuning was checked prior to sample analysis and tuning acceptance criteria was met as specified by the method. Refer to Volatile Organic Instrument Performance Check - Bromofluorobenzene (BFB).

INTERNAL STANDARD & RETENTION TIME

Retention Time for Internal Standards for all samples is within ± 30 seconds compared to the retention time of the ICAL mid-point standard. Area counts of Internal standard for all samples are within -50 to +100% compared to the ICAL mid-point standard. Refer to Instrument Performance Check and Internal Standard Area and RT Summary respectively.

METHOD BLANK

Method Blank was analyzed at a frequency specified by the project. Results are compliant to project requirements.

SURROGATE

Surrogates were added to MB, LCS/LCD, MS/MSD and every sample prior to analysis. Surrogate results are included in the sample report forms. All percent recoveries met the project QC limits. Refer to sample report forms for detail.

LAB CONTROL SAMPLES

LCS/LCD were analyzed at a frequency specified by the project. All percent recoveries met the project QC limits.

MATRIX SPIKE

MS/MSD were analyzed at a frequency specified by the project. All percent recoveries met the project QC limits.

SAMPLE ANALYSIS

Samples were analyzed according to the method and project requirements except as aforementioned. Since both runs of sample 07G065-03 one internal standard is out, it may be due to matrix interference.

All positive results were qualitatively identified in accordance to the method and applied quantitation is based on the initial calibration.

All water samples were labeled preserved with HCl. The pH results are <2. Refer to analytical runlog.

TABLE 1
INITIAL CALIBRATION STANDARD PREPARATION
VOLATILE ORGANICS BY GC/MS

| ICAL/DCC Intermediate Standard | Stock Standard | | | Preparation (Solvent: Methanol) | | Intermediate Standard |
|--------------------------------|--------------------------|--------------|--------------|---------------------------------|-----------------|-----------------------|
| | Standard Name | Source | Conc. (mg/L) | Aliquot (µL) | Final Vol. (ml) | Final Conc. (mg/L) |
| I | M502A-R-10X | AccuStandard | 2000 | 50 | 2 | 50 |
| | 1-Chlorohexane | NSI | 1000 | 100 | 2 | 50 |
| | 2-Chloroethylvinyl Ether | Supelco | 5000 | 20 | 2 | 50 |
| | Carbon Disulfide | Supelco | 5000 | 20 | 2 | 50 |
| | Ethyl Methacrylate | AccuStandard | 2000 | 50 | 2 | 50 |
| | Freon113 | Supelco | 1000 | 100 | 2 | 50 |
| | Iodomethane | Supelco | 2000 | 50 | 2 | 50 |
| | T-1,4-Dichloro-2-Butene | Supelco | 2000 | 50 | 2 | 50 |
| | Vinyl Acetate | AccuStandard | 2000 | 50 | 2 | 50 |
| | Oxygenate Gas Additive | AccuStandard | 2000-10000 | 50 | 2 | 50-250 |
| II | M502B-10X | AccuStandard | 2000 | 50 | 2 | 50 |
| III | TCL VOL MIX 1 | Supelco | 2000 | 100 | 2 | 100 |
| | Acrolein/Acrylonitrile | AccuStandard | 5000 | 40 | 2 | 100 |

TABLE 2
INITIAL CALIBRATION VERIFICATION STANDARD PREPARATION
VOLATILE ORGANICS BY GC/MS

| ICV/LCS/MS Intermediate Standard | Stock Standard | | | Preparation (Solvent: Methanol) | | Intermediate Standard |
|----------------------------------|--------------------------|-----------------|--------------|---------------------------------|-----------------|-----------------------|
| | Standard Name | Source | Conc. (mg/L) | Aliquot (µL) | Final Vol. (ml) | Final Conc. (mg/L) |
| I | 502/524 Vol-Org-Mix | Supelco | 2000 | 50 | 2 | 50 |
| | Vinyl Acetate | Restek | 2000 | 50 | 2 | 50 |
| | Iodomethane | Restek | 2000 | 50 | 2 | 50 |
| | Carbon Disulfide | Restek | 2000 | 50 | 2 | 50 |
| | 1-Chlorohexane | UltraScientific | 1000 | 100 | 2 | 50 |
| | T-1,4-Dichloro-2-Butene | Restek | 2000 | 50 | 2 | 50 |
| | Freon113 | Restek | 2000 | 50 | 2 | 50 |
| | Ethyl Methacrylate | Restek | 1000 | 100 | 2 | 50 |
| | 2-Chloroethylvinyl Ether | UltraScientific | 2000 | 50 | 2 | 50 |
| | Cal. Oxygenate Mix 1 | Restek | 2000-10000 | 50 | 2 | 50-250 |
| II | VOA Org. Compound Mix#6 | Supelco | 2000 | 50 | 2 | 50 |
| III | Voa Cal Mix #1 | Restek | 5000 | 40 | 2 | 100 |
| | Acrolein/Acrylonitrile | UltraScientific | 2000 | 100 | 2 | 100 |

TABLE 3
SURROGATE/INTERNAL STANDARD PREPARATION
VOA BY GC/MS

| Surrogate/IS Intermediate Standard | Stock Standard | | | Preparation (Solvent: Methanol) | | Intermediate Standard |
|--|-----------------------|--------------|-----------------|------------------------------------|--------------------|--------------------------|
| | Standard Name | Source | Conc. (mg/L) | Aliquot (µL) | Final Vol. (ml) | Final Conc. (mg/L) |
| Surrogate | Surrogate Spike Mix | Restek | 2500 | 40 | 2 | 50 |
| Internal Standard | Internal Standard Mix | AccuStandard | 2000 | 50 | 2 | 50 |

TABLE 4
TUNING SOLUTION STANDARD PREPARATION
VOA BY GC/MS

| Tuning Intermediate Standard | Stock Standard | | | Preparation (Solvent: Methanol) | | Intermediate Standard |
|------------------------------------|----------------|--------|-----------------|------------------------------------|--------------------|--------------------------|
| | Standard Name | Source | Conc. (mg/L) | Aliquot (µL) | Final Vol. (ml) | Final Conc. (mg/L) |
| Tuning Compound | BFB | Restek | 5000 | 20 | 2 | 50 |

TABLE 5

| Analytes | ICAL ANALYTE CONCENTRATIONS (ug/L) 25-ml Purge | | | | | | | | | ICV/DCC | LCS/MS |
|-------------------------------|--|-----|---|----|----|----|-----|-----|-----|---------|--------|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | | |
| 1,1,1,2-Tetrachloroethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,1,1-Trichloroethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,1,2,2-Tetrachloroethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,1,2-Trichloroethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,1-Dichloroethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,1-Dichloroethene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,1-Dichloropropene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2,3-Trichlorobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2,3-Trichloropropane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2,4-Trichlorobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2,4-Trimethylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2-Dibromo-3-chloropropane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2-Dibromoethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2-Dichlorobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2-Dichloroethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2-Dichloropropane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,3,5-Trimethylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,3-Dichlorobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,3-Dichloropropane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,4-Dichlorobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1-Chlorohexane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 2,2-Dichloropropane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 2-Butanone (MEK) | 0.6 | 1 | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 40 | 20 |
| 2-Chloroethyl vinyl ether | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 2-Chlorotoluene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 2-Hexanone | 0.6 | 1 | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 40 | 20 |
| 4-Chlorotoluene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 4-Methyl-2-pentanone (MIBK) | 0.6 | 1 | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 40 | 20 |
| Acetone | 0.6 | 1 | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 40 | 20 |
| Acrolein | 0.6 | 1 | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 40 | 20 |
| Acrylonitrile | 0.6 | 1 | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 40 | 20 |
| Benzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Bromobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Bromochloromethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Bromodichloromethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Bromoform | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Bromomethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Carbon disulfide | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Carbon tetrachloride | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Chlorobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Chloroethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Chloroform | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Chloromethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| cis-1,2-Dichloroethene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| cis-1,3-Dichloropropene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Dibromochloromethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Dibromomethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Dichlorodifluoromethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Diisopropyl ether (DIPE) | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Ethyl Methacrylate | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Ethylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Ethyl-tert-butyl ether (ETBE) | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Freon 113 | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Hexachlorobutadiene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Iodomethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Isopropylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| m/p-Xylene | 0.6 | 1 | 2 | 4 | 10 | 20 | 40 | 60 | 80 | 40 | 20 |
| Methylene chloride | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Methyl-t-butyl ether (MTBE) | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Naphthalene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| n-Butylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| n-Propylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| o-Xylene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| p-Isopropyltoluene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| sec-Butylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Styrene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| tert-Amylmethyl ether (TAME) | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| tert-Butanol | 1.5 | 2.5 | 5 | 10 | 25 | 50 | 100 | 150 | 200 | 100 | 50 |
| tert-Butylbenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Tetrachloroethene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Toluene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| trans-1,2-Dichloroethene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| trans-1,3-Dichloropropene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| trans-1,4-Dichloro-2-butene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Trichloroethene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Trichlorofluoromethane | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Vinyl acetate | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Vinyl chloride | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| 1,2-Dichloroethane-d4 | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |
| Bromofluorobenzene | 0.3 | 0.5 | 1 | 2 | 5 | 10 | 20 | 30 | 40 | 20 | 10 |

| Analytes | ICAL ANALYTE CONCENTRATIONS (ug/L) 5-ml Purge | | | | | | | | ICV/DCC | LCS/MS |
|---------------------------------------|---|----|----|-----|-----|-----|-----|------|---------|--------|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | | |
| 1,1,1,2-Tetrachloroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,1,1-Trichloroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,1,2,2-Tetrachloroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,1,2-Trichloroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,1-Dichloroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,1-Dichloroethene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,1-Dichloropropene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2,3-Trichlorobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2,3-Trichloropropane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2,4-Trichlorobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2,4-Trimethylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2-Dibromo-3-chloropropane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2-Dibromoethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2-Dichlorobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2-Dichloroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2-Dichloropropane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,3,5-Trimethylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,3-Dichlorobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,3-Dichloropropane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,4-Dichlorobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1-Chlorohexane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 2,2-Dichloropropane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 2-Butanone (MEK) | 8 | 20 | 40 | 80 | 200 | 320 | 400 | 800 | 200 | 40 |
| 2-Chloroethyl vinyl ether | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 2-Chlorotoluene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 2-Hexanone | 8 | 20 | 40 | 80 | 200 | 320 | 400 | 800 | 200 | 40 |
| 4-Chlorotoluene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 4-Methyl-2-pentanone (MIBK) | 8 | 20 | 40 | 80 | 200 | 320 | 400 | 800 | 200 | 40 |
| Acetone | 8 | 20 | 40 | 80 | 200 | 320 | 400 | 800 | 200 | 40 |
| Acrolein | 8 | 20 | 40 | 80 | 200 | 320 | 400 | 800 | 200 | 40 |
| Acrylonitrile | 8 | 20 | 40 | 80 | 200 | 320 | 400 | 800 | 200 | 40 |
| Benzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Bromobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Bromochloromethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Bromodichloromethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Bromoform | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Bromomethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Carbon disulfide | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Carbon tetrachloride | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Chlorobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Chloroethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Chloroform | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Chloromethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| cis-1,2-Dichloroethene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| cis-1,3-Dichloropropene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Dibromochloromethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Dibromomethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Dichlorodifluoromethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Diisopropyl ether (DIPE) | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Ethyl Methacrylate | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Ethylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Ethyl-tert-butyl ether (ETBE) | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Hexachlorobutadiene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Iodomethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Isopropylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| m/p-Xylene | 4 | 10 | 20 | 40 | 100 | 160 | 200 | 400 | 200 | 40 |
| Methylene chloride | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Methyl-t-butyl ether (MTBE) | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Naphthalene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| n-Butylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| n-Propylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| o-Xylene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| p-Isopropyltoluene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| sec-Butylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Styrene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| tert-Amyl methyl ether (TAME) | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| tert-Butanol | 10 | 25 | 50 | 100 | 250 | 400 | 500 | 1000 | 500 | 100 |
| tert-Butylbenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Tetrachloroethene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Toluene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| trans-1,2-Dichloroethene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| trans-1,3-Dichloropropene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| trans-1,4-Dichloro-2-butene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Trichloroethene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Trichlorofluoromethane | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Vinyl acetate | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Vinyl chloride | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| 1,2-Dichloroethane-d4 | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Bromofluorobenzene | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |
| Toluene-d8 | 2 | 5 | 10 | 20 | 50 | 80 | 100 | 200 | 100 | 20 |

TABLE 6

BFB KEY ION ABUNDANCE CRITERIA

| M/z | Required Intensity (relative abundance) |
|-----|--|
| 50 | 15 to 40% of m/z 95 |
| 75 | 30 to 60% of m/z 95 |
| 95 | Base peak, 100% relative abundance |
| 96 | 5 to 9% of m/z 95 |
| 173 | Less than 2% of m/z 174 |
| 174 | Greater than 50% of m/z 95 |
| 175 | 5 to 9% of m/z 174 |
| 176 | Greater than 95% but less than 101% of m/z 174 |
| 177 | 5 to 9% of m/z 176 |

Table 7

| TARGET ANALYTE STANDARD LIST & REPORTING LIMITS | | | |
|--|--------------------------|---------------------------|-------------------|
| Parameters | Water (5 ml) µg/L | Water (25 ml) µg/L | Soil µg/kg |
| 1,1,1-Trichloroethane | 5 | 1 | 5 |
| 1,1,2,2-Tetrachloroethane | 5 | 1 | 5 |
| 1,1,2-Trichloroethane | 5 | 1 | 5 |
| 1,1-Dichloroethane | 5 | 1 | 5 |
| 1,1-Dichloroethene | 5 | 1 | 5 |
| 1,2-Dichloroethane | 5 | 1 | 5 |
| 1,2-Dichloropropane | 5 | 1 | 5 |
| 2-Butanone(MEK) | 20 | 20 | 20 |
| 2-Hexanone | 20 | 20 | 20 |
| 4-Methyl-2-Pentanone(MIBK) | 10 | 10 | 10 |
| Acetone | 20 | 10 | 20 |
| Benzene | 5 | 1 | 5 |
| Bromochloromethane | 5 | 1 | 5 |
| Bromodichloromethane | 5 | 1 | 5 |
| Bromoform | 5 | 1 | 5 |
| Bromomethane | 5 | 1 | 10 |
| Carbon Disulfide | 5 | 1 | 5 |
| Carbon Tetrachloride | 5 | 1 | 5 |
| Chlorobenzene | 5 | 1 | 5 |
| Chloroethane | 5 | 1 | 5 |
| Chloroform | 5 | 1 | 5 |
| Chloromethane | 5 | 1 | 5 |
| cis-1,2-Dichloroethene | 5 | 1 | 5 |
| cis-1,3-Dichloropropene | 5 | 1 | 5 |
| Dibromochloromethane | 5 | 1 | 5 |
| Ethylbenzene | 5 | 1 | 5 |
| m,p-Xylene | 10 | 1 | 10 |
| Methyl Tert-Butyl Ether | 5 | 1 | 5 |
| Methylene Chloride | 10 | 1 | 10 |
| o-Xylene | 5 | 1 | 5 |
| Styrene | 5 | 1 | 5 |
| Tetrachloroethene | 5 | 1 | 5 |
| Toluene | 5 | 1 | 5 |
| trans-1,2-Dichloroethene | 5 | 1 | 5 |
| trans-1,3-Dichloropropene | 5 | 1 | 5 |
| Trichloroethene | 5 | 1 | 5 |
| Vinyl Chloride | 5 | 1 | 5 |

Table 8

| TARGET ANALYTE EXTENDED LIST & REPORTING LIMITS | | | |
|--|--------------------------|---------------------------|-------------------|
| Parameters | Water (5 ml) µg/L | Water (25 ml) µg/L | Soil µg/kg |
| 1,1,1,2-Tetrachloroethane | 5 | 1 | 5 |
| 1,1,1-Trichloroethane | 5 | 1 | 5 |
| 1,1,2,2-Tetrachloroethane | 5 | 1 | 5 |
| 1,1,2-Trichloroethane | 5 | 1 | 5 |
| 1,1-Dichloroethane | 5 | 1 | 5 |
| 1,1-Dichloroethene | 5 | 1 | 5 |
| 1,1-Dichloropropene | 5 | 1 | 5 |
| 1,2,3-Trichlorobenzene | 5 | 1 | 5 |
| 1,2,3-Trichloropropane | 5 | 1 | 5 |
| 1,2,4-Trichlorobenzene | 5 | 1 | 5 |
| 1,2,4-Trimethylbenzene | 5 | 1 | 5 |
| 1,2-Dibromo-3-chloropropane | 10 | 2 | 10 |
| 1,2-Dibromoethane | 5 | 1 | 5 |
| 1,2-Dichlorobenzene | 5 | 1 | 5 |
| 1,2-Dichloroethane | 5 | 1 | 5 |
| 1,2-Dichloropropane | 5 | 1 | 5 |
| 1,3,5-Trimethylbenzene | 5 | 1 | 5 |
| 1,3-Dichlorobenzene | 5 | 1 | 5 |
| 1,3-Dichloropropane | 5 | 1 | 5 |
| 1-Chlorohexane | 5 | 1 | 5 |
| 2,2-Dichloropropane | 5 | 1 | 5 |
| 2-Chlorotoluene | 5 | 1 | 5 |
| 4-Chlorotoluene | 5 | 1 | 5 |
| Benzene | 5 | 1 | 5 |
| Bromobenzene | 5 | 1 | 5 |
| Bromochloromethane | 5 | 1 | 5 |
| Bromodichloromethane | 5 | 1 | 5 |
| Bromoform | 5 | 1 | 5 |
| Bromomethane | 5 | 1 | 5 |
| Carbon Tetrachloride | 5 | 1 | 5 |
| Chlorobenzene | 5 | 1 | 5 |
| Chloroethane | 5 | 1 | 5 |
| Chloroform | 5 | 1 | 5 |
| Chloromethane | 5 | 1 | 5 |
| cis-1,2-Dichloroethene | 5 | 1 | 5 |
| cis-1,3-Dichloropropene | 5 | 1 | 5 |
| Dibromochloromethane | 5 | 1 | 5 |
| Dibromomethane | 5 | 1 | 5 |
| Dichlorodifluoromethane | 5 | 1 | 5 |
| Ethylbenzene | 5 | 1 | 5 |
| Hexachlorobutadiene | 5 | 1 | 5 |
| Isopropyl Benzene | 5 | 1 | 5 |
| m,p-Xylene | 10 | 1 | 10 |
| Methyl Tert-Butyl Ether | 5 | 1 | 5 |
| Methylene Chloride | 10 | 1 | 10 |
| n-Butylbenzene | 5 | 1 | 5 |
| n-Propylbenzene | 5 | 1 | 5 |
| Naphthalene | 5 | 1 | 5 |
| o-Xylene | 5 | 1 | 5 |
| p-Isopropyltoluene | 5 | 1 | 5 |
| sec-Butylbenzene | 5 | 1 | 5 |
| Styrene | 5 | 1 | 5 |
| tert-Butylbenzene | 5 | 1 | 5 |
| Tetrachloroethene | 5 | 1 | 5 |
| Toluene | 5 | 1 | 5 |
| trans-1,2-Dichloroethene | 5 | 1 | 5 |
| trans-1,3-Dichloropropene | 5 | 1 | 5 |
| Trichloroethene | 5 | 1 | 5 |
| Trichlorofluoromethane | 5 | 1 | 5 |
| Vinyl Chloride | 5 | 1 | 5 |

**TABLE 9
CHARACTERISTIC IONS**

| Analyte | Primary Characteristic Ion | Secondary Characteristic Ion(s) |
|-----------------------------------|----------------------------|---------------------------------|
| Acetone | 43 | 58 |
| Acetonitrile | 41 | 40, 39 |
| Acrolein | 56 | 55 |
| Acrylonitrile | 53 | 52, 51 |
| Benzene | 78 | 77,52 |
| Benzyl chloride | 91 | 126, 65, 125 |
| Bromobenzene | 156 | 51,158 |
| Bromochloromethane | 49 | 128,130 |
| Bromoform | 173 | 171, 175 |
| Bromomethane | 94 | 96 |
| n-Butylbenzene | 91 | 92, 134 |
| Sec-Butylbenzene | 105 | 134 |
| Tert-Butylbenzene | 119 | 91, 134 |
| Carbon disulfide | 76 | 78 |
| Carbon tetrachloride ⁵ | 119 | 117 |
| Chlorobenzene | 112 | 51,77, 114 |
| Chloroethane | 64 | 49,66 |
| 2-Chloroethyl vinyl ether | 63 | 65, 106 |
| Chloroform | 83 | 85,47 |
| Chloromethane | 50 | 52 |
| 2-Chlorotoluene | 91 | 126 |
| 4-Chlorotoluene | 91 | 126 |
| 1,2-Dibromo-3-chloropropane | 157 | 155,175 |
| Dibromochloromethane | 129 | 127,131 |
| 1,2-Dibromoethane | 107 | 109 |
| Dibromomethane | 93 | 95, 174 |
| 1,2-Dichlorobenzene | 146 | 111, 148 |
| 1,3-Dichlorobenzene | 146 | 111, 148 |
| 1,4-Dichlorobenzene | 146 | 111, 148 |
| Cis-1,4-Dichloro-2-butene | 75 | 53, 77, 124, 89 |
| Trans-1,4-Dichloro-2-butene | 53 | 88 |
| Dichlorodifluoromethane | 85 | 87,50 |
| 1,1-Dichloroethane | 63 | 65, 83 |
| 1,2-Dichloroethane | 62 | 98,64 |
| 1,1-Dichloroethene | 61 | 63,96 |
| Cis-1,2-Dichloroethene | 96 | 61, 98 |
| Trans-1,2-Dichloroethene | 61 | 96,98 |
| 1,2-Dichloropropane | 63 | 41,76 |
| 1,3-Dichloropropane | 76 | 63,78 |
| 2,2-Dichloropropane | 77 | 97,79,41 |
| 1,1-Dichloropropene ⁶ | 77 | 110, 75 |
| Cis-1,3-Dichloropropene | 75 | 77, 39,110 |
| Trans-1,3-Dichloropropene | 75 | 77, 39,110 |
| Ethylbenzene | 91 | 106 |

⁵ Quantitation ion was changed due to co-elution (Carbon Tetrachloride - 117 to 119)

⁶ Quantitation ion was changed due to co-elution (1,1-Dichloropropene - 75 to 77)

**TABLE 9
 CHARACTERISTIC IONS**

| Analyte | Primary Characteristic Ion | Secondary Characteristic Ion(s) |
|--------------------------------------|-----------------------------------|--|
| Hexachlorobutadiene | 225 | 223, 227 |
| Hexachloroethane | 201 | 166, 199, 203 |
| 2-Hexanone | 43 | 58,100 |
| Iodomethane | 142 | 127, 141 |
| Isopropylbenzene | 105 | 120,79,103 |
| Methyl-t-butyl ether | 73 | 57 |
| Methylene chloride | 49 | 84, 86 |
| Methyl ethyl ketone | 43 | 72 |
| 4-Methyl-2-pentanone | 43 | 58, 85,100 |
| Naphthalene | 128 | 127 |
| n-Propylbenzene | 91 | 65,120 |
| Styrene | 104 | 78,103 |
| 1,2,3-Trichlorobenzene | 180 | 182, 145 |
| 1,2,4-Trichlorobenzene | 180 | 182, 145 |
| 1,1,1,2-Tetrachloroethane | 131 | 133, 119,117 |
| 1,1,2,2-Tetrachloroethane | 83 | 131, 85 |
| Tetrachloroethene | 164 | 129, 131, 166 |
| Toluene | 92 | 91 |
| 1,1,1-Trichloroethane | 97 | 99, 61 |
| 1,1,2-Trichloroethane | 97 | 83, 85,99 |
| Trichloroethene | 130 | 97, 132,95 |
| Trichlorofluoromethane | 101 | 103 |
| 1,2,3-Trichloropropane | 75 | 61,77 |
| 1,2,4-Trimethylbenzene | 105 | 120 |
| 1,3,5-Trimethylbenzene | 105 | 120,119 |
| Vinyl acetate | 43 | 86 |
| Vinyl chloride | 62 | 64 |
| o-Xylene | 91 | 106 |
| m-Xylene | 91 | 106 |
| p-Xylene | 91 | 106 |
| INTERNAL STANDARDS/SURROGATES | | |
| 1,4-Difluorobenzene | 114 | 88 |
| Chlorobenzene-d ₅ | 117 | 82,119 |
| 1,2-Dichlorobenzene-d ₄ | 152 | 150 |
| 4-Bromofluorobenzene | 95 | 174, 176 |
| 1,2-Dichloroethane-d ₄ | 65 | 102 |
| Toluene-d ₈ | 98 | 100 |

TABLE 10
INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS AND SURROGATES
ASSIGNED FOR QUANTITATION

| 1,4-DIFLUOROBENZENE | CHLOROBENZENE-D₅ | 1,2-DICHLOROBENZENE-D₄ |
|---------------------------------------|------------------------------------|--|
| Dichlorodifluoromethane | 1,1-Dichloropropene | Bromoform |
| Chloromethane | Carbon Tetrachloride | Isopropyl Benzene |
| Vinyl Chloride | Benzene | Bromofluorobenzene |
| Bromomethane | 1,2-Dichloroethane | Bromobenzene |
| Chloroethane | Trichloroethene | 1,1,2,2-Tetrachloroethane |
| Trichlorofluorometh | 1,2-Dichloropropane | 1,2,3-Trichloropropane |
| 1,1-Dichloroethene | Bromodichloromethane | 1,4-Dichloro-2-butene |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | Dibromomethane | n-Propylbenzene |
| Acetone | 2-Chloroethyl Vinyl Ether | 2-Chlorotoluene |
| Iodomethane | cis-1,3-Dichloropropene | 4-Chlorotoluene |
| Carbon Disulfide | 4-Methyl-2-Pentanone | 1,3,5-Trimethylbenzene |
| Methylene Chloride | Toluene-d ₈ | tert-Butylbenzene |
| Acrylonitrile | Toluene | 1,2,4-Trimethylbenzen |
| trans-1,2-Dichloroe | trans-1,3-Dichloropropene | Sec-Butylbenzene |
| Acrolein | Ethyl Methacrylate | 1,3-Dichlorobenzene |
| MTBE | 1,1,2-trichloroethane | 1,4-Dichlorobenzene |
| 1,1-Dichloroethane | Tetrachloroethene | p-Isopropyltoluene |
| Vinyl Acetate | 1,3-Dichloropropane | 1,2-Dichlorobenzene |
| 2,2-Dichloropropane | 2-Hexanone | n-Butylbenzene |
| cis-1,2-Dichloroeth | Dibromochloromethane | 1,2-Dibromo-3-Chloropropane |
| 2-Butanone | 1,2-Dibromoethane | 1,2,4-Trichlorobenzene |
| Bromochloromethane | Chlorobenzene | Hexachlorobutadiene |
| Chloroform | 1-Chlorohexane | Naphthalene |
| 1,1,1-Trichloroetha | 1,1,1,2-Tetrachloroethane | 1,2,3-Trichlorobenzene |
| 1,2-Dichloroethane-d ₄ | Ethylbenzene | |
| | m/p-Xylenes | |
| | o-Xylene | |
| | Styrene | |

EMAX IN-HOUSE QUALITY CONTROL

| QC PROCEDURE | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1st Rvw | 2nd Rvw |
|---|---|---|---|--|---------|---------|
| Check of mass spectral ion intensities using BFB | Prior to initial calibration and calibration verification | Refer to criteria listed in Table 6 | Retune instrument and verify | | | |
| Five-point initial calibration for all analytes | Initially; as needed | SPCCs : RF ≥ 0.1 for Bromoform, Chloromethane and 1,1-Dichloroethane RF ≥ 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane CCCs: Chloroform, 1,1-DCE, 1,2-DCP, Ethylbenzene, Toluene and Vinyl Chloride. %RSD $\leq 30\%$ and one option below. 1). linear- mean RSD for all analytes $\leq 15\%$ 2). linear – least squares regression $r \geq 0.995$, when RSD $> 15\%$ 3). non-linear – COD > 0.990 (6 points shall be used for second order, 7 points shall be used for third) | Correct the problem then repeat initial calibration | | | |
| Second-source calibration verification | After initial calibration | All analytes within $\pm 25\%$ of expected value except for the following compounds due to erratic chromatographic behavior: Bromomethane, Chloroethane, Chloromethane, Dichlorodifluoromethane within $\pm 35\%$ of expected value. | Correct the problem then repeat initial calibration | | | |
| Retention time window calculated for each analyte | Each sample | Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT of the standard component | Correct the problem then reanalyze all samples analyzed since the last retention time check | | | |
| Calibration verification | Daily, before sample analysis and every 12 hours of analysis time | SPCCs : Min. RF same as ICAL CCC : %Diff $\leq 20\%$ (when using RFs) or drift (when using least squares regression or non-linear calibration) | Correct the problem then repeat initial calibration | | | |
| ISs | Immediately after or during data acquisition for each sample | Retention time ± 30 seconds from retention time of the mid-point std. in the ICAL.EICP area within -50% to +100% of ICAL mid-point std. | Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning | | | |
| Method blank | One per preparation batch | No analytes detected \geq RL | No analytes detected \geq RL Re-prep and re-analyze method blank and all samples processed with the contaminated blank | Apply B to specific analyte(s) on all associated samples | | |
| LCS | One LCS per preparation | Within EMAX QC Limits | Re-prep and re-analyze the LCS and all associated samples | | | |
| MS/MSD | One MS/MSD per every 20 project samples per matrix | Within EMAX QC Limits | None | | | |
| Surrogate | Every Sample, MB, LCS, MS/MSD, DCC | Within EMAX In-house QC Limits | Correct the problem then re-analyze | | | |
| Results reported between MDL and RL | None | None | none | Apply J to all values between MDL and RL | | |
| Comments: | | | | Reviewed by: | | |
| | | | | Date: | | |

DEMONSTRATION OF CAPABILITY

Final Report - Water Pollution Proficiency Testing

Study: WP0107

Opening Date: January 8, 2007 - Closing Date: February 22, 2007

Laboratory: EMAX Laboratories
1825 205th Street
Torrance, CA 90501

Contact: Ms. Kenette Pimentel
310-618-8889 ext. 205

EPA Lab ID: CA00291

| Volatiles (PT-VOA-WP) cont'd | | | | | | | | | Lot #: 8058-26 |
|------------------------------|-----------------------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|----------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 4700 | trans-1,2-Dichloroethene | 10107207 | EPA824 | µg/L | 86.9 | 83.5 | 65.2 - 108 | 54.5 - 119 | Acceptable |
| 4700 | trans-1,2-Dichloroethene | 10184802 | SW5030B/SW8280B | µg/L | 86.9 | 83.5 | 65.2 - 108 | 54.5 - 119 | Acceptable |
| 4655 | 1,2-Dichloropropane | 10107207 | EPA824 | µg/L | 0.00 | 0 | | | Acceptable |
| 4655 | 1,2-Dichloropropane | 10184802 | SW5030B/SW8280B | µg/L | 0.00 | 0 | | | Acceptable |
| 4685 | trans-1,3-Dichloropropene | 10107207 | EPA824 | µg/L | 0.00 | 0 | | | Acceptable |
| 4685 | trans-1,3-Dichloropropene | 10184802 | SW5030B/SW8280B | µg/L | 0.00 | 0 | | | Acceptable |
| 4765 | Ethylbenzene | 10107207 | EPA824 | µg/L | 89.3 | 89.3 | 71.1 - 106 | 62.4 - 114 | Acceptable |
| 4765 | Ethylbenzene | 10184802 | SW5030B/SW8280B | µg/L | 89.3 | 89.3 | 71.1 - 106 | 62.4 - 114 | Acceptable |
| 4975 | Methylene Chloride | 10107207 | EPA824 | µg/L | 29.1 | 32 | 21.6 - 37.5 | 17.6 - 41.5 | Acceptable |
| 4975 | Methylene Chloride | 10184802 | SW5030B/SW8280B | µg/L | 29.1 | 32 | 21.6 - 37.5 | 17.6 - 41.5 | Acceptable |
| 4995 | 4-Methyl-2-pentanone (MIBK) | 10107207 | EPA824 | µg/L | 143 | 117 | 94.9 - 188 | 71.7 - 211 | Acceptable |
| 4995 | 4-Methyl-2-pentanone (MIBK) | 10184802 | SW5030B/SW8280B | µg/L | 143 | 117 | 94.9 - 188 | 71.7 - 211 | Acceptable |
| 5100 | Styrene | 10107207 | EPA824 | µg/L | 76.5 | 82 | 58.1 - 95.4 | 48.8 - 105 | Acceptable |
| 5100 | Styrene | 10184802 | SW5030B/SW8280B | µg/L | 76.5 | 82 | 58.1 - 95.4 | 48.8 - 105 | Acceptable |
| 5110 | 1,1,2,2-Tetrachloroethane | 10107207 | EPA824 | µg/L | 136 | 138 | 100 - 178 | 81.2 - 197 | Acceptable |
| 5110 | 1,1,2,2-Tetrachloroethane | 10184802 | SW5030B/SW8280B | µg/L | 136 | 138 | 100 - 178 | 81.2 - 197 | Acceptable |
| 5115 | Tetrachloroethene | 10107207 | EPA824 | µg/L | 120 | 119 | 83.5 - 142 | 68.8 - 157 | Acceptable |
| 5115 | Tetrachloroethene | 10184802 | SW5030B/SW8280B | µg/L | 120 | 119 | 83.5 - 142 | 68.8 - 157 | Acceptable |
| 5140 | Toluene | 10107207 | EPA824 | µg/L | 84.8 | 84.2 | 66.7 - 98.0 | 58.8 - 106 | Acceptable |
| 5140 | Toluene | 10184802 | SW5030B/SW8280B | µg/L | 84.8 | 84.2 | 66.7 - 98.0 | 58.8 - 106 | Acceptable |
| 5160 | 1,1,1-Trichloroethane | 10107207 | EPA824 | µg/L | 27.0 | 27.1 | 20.2 - 33.0 | 17.0 - 36.2 | Acceptable |
| 5160 | 1,1,1-Trichloroethane | 10184802 | SW5030B/SW8280B | µg/L | 27.0 | 27.1 | 20.2 - 33.0 | 17.0 - 36.2 | Acceptable |
| 5165 | 1,1,2-Trichloroethane | 10107207 | EPA824 | µg/L | 38.0 | 37.8 | 30.4 - 46.2 | 26.5 - 50.1 | Acceptable |
| 5165 | 1,1,2-Trichloroethane | 10184802 | SW5030B/SW8280B | µg/L | 38.0 | 37.8 | 30.4 - 46.2 | 26.5 - 50.1 | Acceptable |
| 5170 | Trichloroethene | 10107207 | EPA824 | µg/L | 87.3 | 86.1 | 65.2 - 104 | 55.5 - 113 | Acceptable |
| 5170 | Trichloroethene | 10184802 | SW5030B/SW8280B | µg/L | 87.3 | 86.1 | 65.2 - 104 | 55.5 - 113 | Acceptable |
| 5175 | Trichlorofluoromethane | 10107207 | EPA824 | µg/L | 0.00 | 0 | | | Acceptable |
| 5175 | Trichlorofluoromethane | 10184802 | SW5030B/SW8280B | µg/L | 0.00 | 0 | | | Acceptable |
| 5235 | Vinyl chloride | 10107207 | EPA824 | µg/L | 51.2 | 47.5 | | 20.5 - 81.9 | Acceptable |
| 5235 | Vinyl chloride | 10184802 | SW5030B/SW8280B | µg/L | 51.2 | 47.5 | | 20.5 - 81.9 | Acceptable |

Report Issue Date - 3/16/2007

DEMONSTRATION OF CAPABILITY

Final Report - Water Pollution Proficiency Testing

Study: WP0107

Opening Date: January 8, 2007 - Closing Date: February 22, 2007

Laboratory: EMAX Laboratories
1825 205th Street
Torrance, CA 90501

Contact: Ms. Kenette Pimentel
310-618-8889 ext. 205

EPA Lab ID: CA00291

| Volatiles (PT-VOA-WP) cont'd | | | | | | | | | | Lot #: 8058-26 |
|---|------------------------------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|------------|----------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation | |
| 5260 | Xylenes, total | 10107207 | EPA824 | µg/L | 223 | 213 | 157 - 270 | 128 - 298 | Acceptable | |
| 5260 | Xylenes, total | 10184802 | SW5030B/SW8260B | µg/L | 223 | 213 | 157 - 270 | 128 - 298 | Acceptable | |
| NELAC Experimental Analytes | | | | | | | | | | |
| 4630 | 1,1-Dichloroethane | 10107207 | EPA824 | µg/L | 132 | 135 | 107 - 167 | 91.8 - 182 | Acceptable | |
| 4630 | 1,1-Dichloroethane | 10184802 | SW5030B/SW8260B | µg/L | 132 | 135 | 107 - 167 | 91.8 - 182 | Acceptable | |
| 4645 | cis-1,2-Dichloroethane | 10107207 | EPA824 | µg/L | 108 | 102 | 90.3 - 133 | 79.7 - 143 | Acceptable | |
| 4645 | cis-1,2-Dichloroethane | 10184802 | SW5030B/SW8260B | µg/L | 108 | 102 | 90.3 - 133 | 79.7 - 143 | Acceptable | |
| 4680 | cis-1,3-Dichloropropylene | 10107207 | EPA824 | µg/L | 0.00 | 0 | | | Acceptable | |
| 4680 | cis-1,3-Dichloropropylene | 10184802 | SW5030B/SW8260B | µg/L | 0.00 | 0 | | | Acceptable | |
| 4860 | 2-Hexanone | | | µg/L | 120 | | 80.3 - 156 | 61.3 - 175 | NR | |
| 5000 | Methyl tert-butyl ether (MTBE) | 10107207 | EPA824 | µg/L | 88.0 | 77.9 | 67.3 - 112 | 56.3 - 123 | Acceptable | |
| 5000 | Methyl tert-butyl ether (MTBE) | 10184802 | SW5030B/SW8260B | µg/L | 88.0 | 77.9 | 67.3 - 112 | 56.3 - 123 | Acceptable | |
| Additional State Specific Analytes | | | | | | | | | | |
| 4320 | Acetonitrile | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4320 | Acetonitrile | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 4325 | Acrolein | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4325 | Acrolein | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 4340 | Acrylonitrile | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4340 | Acrylonitrile | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 4460 | Carbon disulfide | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4460 | Carbon disulfide | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 4500 | 2-Chloroethylvinylether | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4500 | 2-Chloroethylvinylether | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 4570 | 1,2-Dibromo-3-chloropropane (DBCP) | 10107207 | EPA824 | µg/L | 134 | 124 | | 108 - 161 | Acceptable | |
| 4570 | 1,2-Dibromo-3-chloropropane (DBCP) | 10184802 | SW5030B/SW8260B | µg/L | 134 | 124 | | 108 - 161 | Acceptable | |
| 4585 | 1,2-Dibromoethane (EDB) | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4585 | 1,2-Dibromoethane (EDB) | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 4595 | Dibromomethane | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4595 | Dibromomethane | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |

Report Issue Date - 3/16/2007

Final Report - Water Pollution Proficiency Testing

Study: WP0107

Opening Date: January 8, 2007 - Closing Date: February 22, 2007

Laboratory: EMAX Laboratories
1825 205th Street
Torrance, CA 90501

Contact: Ms. Kenette Pimentel
310-618-8889 ext. 205

EPA Lab ID: CA00291

| Volatiles (PT-VOA-WP) cont'd | | | | | | | | | | Lot #: 8058-26 |
|--|---------------------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|------------|----------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation | |
| Additional State Specific Analytes cont'd | | | | | | | | | | |
| 4625 | Dichlorodifluoromethane | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 4625 | Dichlorodifluoromethane | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 5105 | 1,1,1,2-Tetrachloroethane | 10107207 | EPA824 | µg/L | 40.7 | 38 | | 32.6 - 48.8 | Acceptable | |
| 5105 | 1,1,1,2-Tetrachloroethane | 10184802 | SW5030B/SW8260B | µg/L | 40.7 | 38 | | 32.6 - 48.8 | Acceptable | |
| 5180 | 1,2,3-Trichloropropane | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 5180 | 1,2,3-Trichloropropane | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |
| 5225 | Vinyl acetate | 10107207 | EPA824 | µg/L | <5 | 0 | | | Acceptable | |
| 5225 | Vinyl acetate | 10184802 | SW5030B/SW8260B | µg/L | <5 | 0 | | | Acceptable | |

DEMONSTRATION OF CAPABILITY

Final Report - Soil / Hazardous Waste PT

Study: HW0107

Opening Date: January 22, 2007 - Closing Date: March 8, 2007

Laboratory: EMAX Laboratories
1825 205th Street
Torrance, CA 90501

Contact: Ms. Kenette Pimentel
310-618-8889 ext. 205

EPA Lab ID: CA00291

| Volatiles (PT-VOA-SOIL) | | | | | | | Lot #: 7027-12 | |
|---|-----------------------------|-------------|--------------------|-------|----------------|--------|-------------------|------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 4375 | Benzene | 10184802 | SW5035/SW8260B | µg/kg | 125 | 123 | 74.2 - 176 | Acceptable |
| 4375 | Benzene | 10174808 | SW5035/SW8021B | µg/kg | 125 | 117 | 74.2 - 176 | Acceptable |
| 4395 | Bromodichloromethane | 10184802 | SW5035/SW8260B | µg/kg | 92.5 | 86.6 | 57.8 - 127 | Acceptable |
| 4400 | Bromofom | 10184802 | SW5035/SW8260B | µg/kg | 139 | 125 | 89.6 - 209 | Acceptable |
| 4465 | Carbon tetrachloride | 10184802 | SW5035/SW8260B | µg/kg | 79.6 | 80.6 | 39.1 - 120 | Acceptable |
| 4475 | Chlorobenzene | 10184802 | SW5035/SW8260B | µg/kg | 104 | 98 | 75.5 - 132 | Acceptable |
| 4575 | Chlorodibromomethane | 10184802 | SW5035/SW8260B | µg/kg | 95.7 | 88.4 | 62.0 - 129 | Acceptable |
| 4505 | Chloroform | 10184802 | SW5035/SW8260B | µg/kg | 86.6 | 85.5 | 43.3 - 94.0 | Acceptable |
| 4610 | 1,2-Dichlorobenzene | 10184802 | SW5035/SW8260B | µg/kg | 85.4 | 81.7 | 56.1 - 115 | Acceptable |
| 4615 | 1,3-Dichlorobenzene | 10184802 | SW5035/SW8260B | µg/kg | 93.9 | 89.5 | 63.0 - 125 | Acceptable |
| 4620 | 1,4-Dichlorobenzene | 10184802 | SW5035/SW8260B | µg/kg | 104 | 102 | 73.2 - 135 | Acceptable |
| 4630 | 1,1-Dichloroethane | 10184802 | SW5035/SW8260B | µg/kg | 77.7 | 77.9 | 46.4 - 109 | Acceptable |
| 4635 | 1,2-Dichloroethane | 10184802 | SW5035/SW8260B | µg/kg | 73.1 | 69.4 | 43.1 - 103 | Acceptable |
| 4765 | Ethylbenzene | 10184802 | SW5035/SW8260B | µg/kg | 120 | 115 | 79.1 - 160 | Acceptable |
| 4765 | Ethylbenzene | 10174808 | SW5035/SW8021B | µg/kg | 120 | 115 | 79.1 - 160 | Acceptable |
| 4975 | Methylene chloride | 10184802 | SW5035/SW8260B | µg/kg | 96.7 | 89.9 | 26.5 - 167 | Acceptable |
| 4995 | 4-Methyl-2-pentanone (MIBK) | 10184802 | SW5035/SW8260B | µg/kg | 0.00 | 0 | | Acceptable |
| 5105 | 1,1,1,2-Tetrachloroethane | 10184802 | SW5035/SW8260B | µg/kg | 122 | 114 | 84.2 - 160 | Acceptable |
| 5110 | 1,1,1,2,2-Tetrachloroethane | 10184802 | SW5035/SW8260B | µg/kg | 94.8 | 82.1 | 61.5 - 128 | Acceptable |
| 5115 | Tetrachloroethene | 10184802 | SW5035/SW8260B | µg/kg | 88.1 | 87.5 | 48.8 - 127 | Acceptable |
| 5140 | Toluene | 10184802 | SW5035/SW8260B | µg/kg | 145 | 136 | 85.2 - 204 | Acceptable |
| 5140 | Toluene | 10174808 | SW5035/SW8021B | µg/kg | 145 | 136 | 85.2 - 204 | Acceptable |
| 5160 | 1,1,1-Trichloroethane | 10184802 | SW5035/SW8260B | µg/kg | 71.3 | 69.2 | 36.0 - 107 | Acceptable |
| 5170 | Trichloroethene | 10184802 | SW5035/SW8260B | µg/kg | 81.5 | 77.6 | 48.5 - 114 | Acceptable |
| 5260 | Xylenes, total | 10184802 | SW5035/SW8260B | µg/kg | 67.8 | 64.6 | 33.0 - 103 | Acceptable |
| 5260 | Xylenes, total | 10174808 | SW5035/SW8021B | µg/kg | 67.8 | 60.6 | 33.0 - 103 | Acceptable |
| Additional State Specific Analytes | | | | | | | | |
| 4320 | Acetonitrile | 10184802 | SW5035/SW8260B | µg/kg | 125 | 0 | 0.00 - 200 | Acceptable |
| 4325 | Acrolein | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable |
| 4340 | Acrylonitrile | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable |

Report Issue Date - 3/29/2007

DEMONSTRATION OF CAPABILITY

Final Report - Soil / Hazardous Waste PT

Study: HW0107

Opening Date: January 22, 2007 - Closing Date: March 8, 2007

Laboratory: EMAX Laboratories
1825 205th Street
Torrance, CA 90501

Contact: Ms. Kenette Pimentel
310-618-8889 ext. 205

EPA Lab ID: CA00291

| Volatiles (PT-VOA-SOIL) cont'd | | | | | | | | Lot #: 7027-12 | |
|--|------------------------------------|-------------|--------------------|-------|----------------|--------|-------------------|----------------|--|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation | |
| Additional State Specific Analytes cont'd | | | | | | | | | |
| 4385 | Bromobenzene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4960 | Bromomethane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4410 | 2-Butanone (MEK) | 10184802 | SW5035/SW8260B | µg/kg | 98.1 | 102 | 0.00 - 166 | Acceptable | |
| 4460 | Carbon disulfide | 10184802 | SW5035/SW8260B | µg/kg | 86.4 | 87.4 | 0.00 - 123 | Acceptable | |
| 4465 | Chloroacetaldehyde | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4485 | Chloroethane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 5785 | bis(2-Chloroethyl)ether | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 5780 | bis(2-Chloroethoxy)methane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 5780 | bis(2-Chloroisopropyl)ether | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4500 | 2-Chloroethylvinylether | 10184802 | SW5035/SW8260B | µg/kg | 75.6 | 74.8 | 0.00 - 191 | Acceptable | |
| 4980 | Chloromethane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4570 | 1,2-Dibromo-3-chloropropane (DBCP) | 10184802 | SW5035/SW8260B | µg/kg | 99.6 | 81.6 | 62.6 - 136 | Acceptable | |
| 4585 | 1,2-Dibromoethane(EDB) | 10184802 | SW5035/SW8260B | µg/kg | 76.0 | 66.1 | 42.1 - 110 | Acceptable | |
| 4595 | Dibromomethane | 10184802 | SW5035/SW8260B | µg/kg | 109 | 102 | 64.7 - 153 | Acceptable | |
| 4625 | Dichlorodifluoromethane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4640 | 1,1-Dichloroethylene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4645 | cis-1,2-Dichloroethylene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4700 | trans-1,2-Dichloroethylene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4655 | 1,2-Dichloropropane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4680 | cis-1,3-Dichloropropylene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4685 | trans-1,3-Dichloropropylene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4800 | 2-Hexanone | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 4900 | Isopropylbenzene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 5000 | Methyl-t-butyl ether | 10184802 | SW5035/SW8260B | µg/kg | <60 | 0 | | Acceptable | |
| 5000 | Methyl-t-butyl ether | 10174808 | SW5035/SW8021B | µg/kg | <60 | 0 | | Acceptable | |
| 5005 | Naphthalene | | | µg/kg | <10 | | | NR | |
| 5100 | Styrene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 5155 | 1,2,4-Trichlorobenzene | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 5165 | 1,1,2-Trichloroethane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |

Report Issue Date - 3/29/2007

Final Report - Soil / Hazardous Waste PT

Study: HW0107

Opening Date: January 22, 2007 - Closing Date: March 8, 2007

Laboratory: EMAX Laboratories
1825 205th Street
Torrance, CA 90501

Contact: Ms. Kenette Pimentel
310-618-8889 ext. 205

EPA Lab ID: CA00291

| Volatiles (PT-VOA-SOIL) cont'd | | | | | | | | Lot #: 7027-12 | |
|--|------------------------|-------------|--------------------|-------|----------------|--------|-------------------|----------------|--|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation | |
| Additional State Specific Analytes cont'd | | | | | | | | | |
| 5175 | Trichlorofluoromethane | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |
| 5180 | 1,2,3-Trichloropropane | 10184802 | SW5035/SW8260B | µg/kg | 120 | 101 | 82.8 - 157 | Acceptable | |
| 5225 | Vinyl acetate | 10184802 | SW5035/SW8260B | µg/kg | 31.8 | 23.8 | 0.00 - 64.5 | Acceptable | |
| 5235 | Vinyl chloride | 10184802 | SW5035/SW8260B | µg/kg | <20 | 0 | | Acceptable | |

ANALYSIS LOG FOR VOLATILES

SOP EMAX-8260 Rev. No.: 2 EMAX-524.2 Rev. No.: 3 EMAX-CLP-VOA EMAX-624 Rev. No.: 1

Start Date _____ 25-ml Purge 5-ml Purge

Book # **A05-019**

| ANALYTICAL BATCH: _____ | Sample Prep. ID | Data File Name | Lab Sample ID | Sample Amount | DF | MATRIX | | NOTES | Instrument No. 05 | |
|-------------------------|-----------------|----------------|---------------|---------------|----|--------|---|-------|--|--------------|
| | | | | | | pH-W | S | | INITIAL CALIBRATION REFERENCE | |
| | 01 | | | | | | | | DATE | |
| | 02 | | | | | | | | ICAL ID | |
| | 03 | | | | | | | | STANDARDS | |
| | 04 | | | | | | | | NAME | ID |
| | 05 | | | | | | | | | CONC. (mg/L) |
| | 94 | | | | | | | | DCC | |
| | 07 | | | | | | | | DCC | |
| | 08 | | | | | | | | DCC | |
| | 09 | | | | | | | | DCC | |
| | 10 | | | | | | | | BFB | |
| | 11 | | | | | | | | IS/SURR. | |
| | 12 | | | | | | | | LCS | |
| | 13 | | | | | | | | LCS | |
| | 14 | | | | | | | | LCS | |
| | 15 | | | | | | | | LCS | |
| | 16 | | | | | | | | SOLVENT | ID |
| | 17 | | | | | | | | METHANOL | |
| | 18 | | | | | | | | ARCHIVAL | |
| | 19 | | | | | | | | Data File | |
| | 20 | | | | | | | | Comments | _____ |
| | 21 | | | | | | | | | |
| | 22 | | | | | | | | | |
| | 23 | | | | | | | | | |
| | 24 | | | | | | | | Analyzed By: | _____ |
| | 25 | | | | | | | | This page is checked during data review. | |

STANDARD OPERATING PROCEDURES

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8270 Revision No. 3 Date: 15-July-06

Prepared By: Souzan Greas *Souzan S. Greas* Date: 6/28/06

Approved By: Kenette Pimentel *Kenette Pimentel* Date: 6/28/06
QA Manager

Approved By: Kam Pang *Kam Pang* Date: 6/28/06
Laboratory Director

Control Number: 8270-03-

1.0 SCOPE AND APPLICATION

- 1.1. This method describes the process to quantify most neutral, acidic and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone. Compounds that can be determined by this method are listed in Appendix 2. It is an adaptation of method SW846 8270C.

2.0 SUMMARY OF METHOD

- 2.1. Samples are extracted with methylene chloride. Extracts are concentrated and appropriate cleanup procedure is applied if necessary.
- 2.2. Internal standards are added to an aliquot of the final extract and are qualitatively and quantitatively analyzed by gas chromatography equipped with mass spectrometry (GC/MS).
- 2.3. **Interference**
- 2.3.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interference. Determine source of interference in the preparation and/or cleanup of the samples and Perform corrective action to eliminate the problem.
- 2.3.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, rinse the sample syringe with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

3.0 QUANTITATION LIMITS**3.1. Method Detection Limits**

- 3.1.1. Prepare a minimum of seven samples for each matrix. Add MDL spike solution (preferably at the concentration of the lowest calibration point) to each of the samples and analyze the extracts as described in Section 10.4.
- 3.1.2. Refer to EMAX-QA04 for MDL evaluation and verification.

3.2. Reporting Limit

- 3.2.1. The quantitation limits of this method as written in this SOP are as follows:
- 3.2.1.1. Water = 10 µg/L to 50 µg/L
- 3.2.1.2. Soil = 330 µg/kg (on a wet weight basis) to 1670 µg/kg.

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4.0 DYNAMIC RANGE

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point. All samples analyzed above this concentration are considered "over-range" and shall require dilution for proper quantitation.
- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required for proper quantitation.
- 4.3. The linear dynamic range for this method are as follows:

| <u>Water (µg/L)</u> | <u>Soil (µg/kg)</u> |
|---------------------|---------------------|
| 10 - 160 µg/L | 330 - 5300 µg/kg |

5.0 SAMPLE PRESERVATION AND HOLDING TIME**5.1. Sample Preservation**

- 5.1.1. Store water and soil samples at 4°C (±2°C) away from light.
- 5.1.2. Store all extracts under 4°C (±2°C).

5.2. Holding Time

- 5.2.1. Extract water samples within 7 days from sampling date.
- 5.2.2. Extract soil samples within 14 days from sampling date.
- 5.2.3. Analyze all extracts within 40 days from extraction completion date.

6.0 ASSOCIATED SOPs

- | | | |
|-------|-----------|--|
| 6.1. | EMAX-QA04 | Method Detection Limit Study |
| 6.2. | EMAX-QC02 | Analytical Standard Preparation |
| 6.3. | EMAX-QA08 | Corrective Action |
| 6.4. | EMAX-SM03 | Waste Management |
| 6.5. | EMAX-SM04 | Analytical and QC Sample Labeling |
| 6.6. | EMAX-3640 | Clean Up, GPC |
| 6.7. | EMAX-3520 | Extraction of Organic Compounds by Continuous Liquid/Liquid Extraction |
| 6.8. | EMAX-3510 | Extraction of Organic Compounds by Separatory Funnel |
| 6.9. | EMAX-3550 | Extraction of Organic Compounds from Solid Samples by Pulse Sonication |
| 6.10. | EMAX-3540 | Soxhlet Extraction |
| 6.11. | EMAX-3580 | Waste Dilution |

7.0 SAFETY

- 7.1. Read all MSDS for all chemicals listed in this SOP.

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8270 Revision No. 3 Date: 15-July-06

- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. Place all wastes generated during analytical process in the waste containers. Endorse these wastes to the waste disposal section for proper disposal.
- 7.4. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS AND SUPPLIES**8.1. Instruments and Supplies**

| | |
|------------------------|--|
| Gas Chromatography | HP 5890 Series II with split/splitless injection, Shimadzu GC-17A, or equivalent |
| Mass Spectrometer | HP5970 MSD or Shimadzu GCMS – GP 5000 capable of scanning from 40 to 500 amu every 1 second using 70 volts electrode energy in the electron impact ionization mode or equivalent |
| GC/MS Interface | The interface is capillary-direct into the mass spectrometer source or equivalent |
| Chromatographic Column | ZB-5MS (20m x 18mm x 18 µm) or equivalent |
| Data System | MS-ChemStation with Enviroquant software or equivalent |
| GC Autosampler | HP7673A or Shimadzu AOC-20i capable of direct injection of 1 µl and 10 µl of extract. |
| Gases | Ultra high purity helium |
| Syringes | 10 µl, 25 µl, and 100 µl syringe Hamilton 202N or equivalent |
| Vials | Autosampler vials with teflon lined septa |

8.2. Chemicals and Reagents

- 8.2.1. Methylene chloride pesticides grade, high purity methanol and acid-washed Na₂SO₄.

9.0 STANDARDS**9.1. Standard Preparation**

- 9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX SM04, respectively.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.2. Stock Standard

- 9.2.1. Purchase stock standards as certified solutions from Accustandard or other reputable vendor (refer to Table 1 for the listing of all certified solutions).
- 9.2.2. Transfer the stock standard solutions into 2-ml amber vial with Teflon lined screw caps and store at -10°C to -20°C.

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8270 Revision No. 3 Date: 15-July-06

9.3. Intermediate Standard

9.3.1. Prepare a 10-ml 200 µg/mL intermediate standard (refer to Table 1 for details). Transfer the solution in a properly labeled 10-ml amber vial and store at -10°C to -20°C.

9.4. Internal Standard

9.4.1. The internal standard shall include 1,4-Dichlorobenzene-d₄, Naphthalene-d₈, Acenaphthalene-d₁₀, Phenanthrene-d₁₀, Chrysene-d₁₂ and Perylene-d₁₂ in methylene chloride solution.

9.4.2. Purchase internal standard solutions as certified solution from AccuStandard or other reputable vendor at 4,000 µg/ml.

9.4.3. Prepare a 10-ml of 2,000 µg/ml of working internal standard from 9.4.2. Transfer the solution in a properly labeled 10-ml amber vial and store in -10°C to -20°C.

9.5. GC/MS Tuning

9.5.1. The tuning standard shall include decafluorotriphenylphosphine (DFTPP), 4,4-DDT, Pentachlorophenol, and Benzidine.

9.5.2. Purchase tuning standard solution as certified standard at 1000 µg/ml.

9.5.3. Prepare a 10-ml of 50,000 µg/L of working standard tuning solution. Transfer the solution in a properly labeled 10-ml amber vial and store in -10°C to -20°C.

9.6. Surrogate Standard

9.6.1. Purchase surrogate stock standards in two mixtures as certified standards.

9.6.1.1. The acid surrogate mixture includes Phenol-d₅, 2-Fluorophenol, and 2,4,6-Tribromophenol at 10,000 µg/ml.

9.6.1.2. The basic neutral surrogate mixture includes Nitrobenzene-d₅, 2-Fluorobiphenyl, Terphenyl-d₁₄, and 1,2-Dichlorobenzene-d₄ at 5,000 µg/ml.

9.6.2. Prepare a 1000-ml surrogate spiking solution by adding 15 ml of the surrogate acid mixture and 10 ml the base/neutral surrogate mixture in a 1000-ml volumetric flask. Dilute to mark with Methanol. Transfer to a properly labeled amber bottle and store the surrogate solution at 4°C (± 2°C). This standard is expected to yield 50 µg/ml of each base neutral surrogate and 150 µg/ml of acid surrogate.

9.6.3. Spike each sample undergoing extraction with 1 ml of surrogate spiking standard in the final 1 ml extract. Spike volume may be adjusted to normalize with the final extract and yield the same concentration.

9.7. Calibration Standard

9.7.1. Prepare working standard solutions for initial calibration and daily calibration (refer to Table 2 for details). Transfer the solutions in 1-ml amber vial and store them at 4°C (± 2°C).

9.8. ICAL Verification Standard (Second Source Verification) (ICV)

9.8.1. Purchase a certified ICV standard from a different vendor. The ICV standard contains the same list of compounds as the stock standard (refer to Table 1-B for the standard mix and the corresponding vendors).

9.8.2. Prepare a 10-ml of 200-mg/L check standard solution. Transfer the solution in a properly labeled 10-ml amber vial and store at 4°C (± 2°C).

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8270 Revision No. 3 Date: 15-July-06

9.9. MS/MSD/LCS/LCD Spiking Standards

- 9.9.1. Purchase spiking standards as certified solutions.
- 9.9.2. Prepare 25 ml of 200 mg/L full spiking solution by adding 2.5 ml of each of Base Neutral Mix 1, B/N Mix 2, Toxic Substance Mix 1, Toxic Substance Mix 2, Phenol Mix, PAH Mix, Benzidine, Carbazole & Pyridin at 2,000 mg/L in 25 ml volumetric flask. Dilute to mark with methylene chloride and methanol (1:1 ratio).
- 9.9.3. Prepare short spike solution at 200 mg/L. Dilute 6 ml of 5000 mg/L Base Neutral matrix spike mix and 4 ml of 7500 mg/L Acid Spike Mix with methanol to a final volume of 150 ml.
- 9.9.4. Transfer the spiking solutions into a properly labeled amber bottle with Teflon lined septa cap. Store the spiking solutions at 4°C ($\pm 2^\circ\text{C}$).
- 9.9.5. Spike MS/MSD/LCS/LCD samples with 0.4 ml of full spiking solution prior to sample extraction.
- 9.9.6. Spike volume may be adjusted to normalized with the final extract volume and yield the same concentration.

10.0 PROCEDURES**10.1. Sample Preparation**

- 10.1.1. For aqueous samples, refer to EMAX-3510 or EMAX-3520.
- 10.1.2. For solid samples, refer to EMAX-3550 or EMAX-3540.
- 10.1.3. After extraction, examine the color and consistency of the extract. If the extract appears to be opaque and/or viscous, it is advisable to perform extract cleanup preferably GPC. Refer to EMAX-3640.

10.2. Instrument Parameters

- 10.2.1. Set the instrument parameters as suggested in Table 3. Fine tune the instrument to obtain optimum instrument condition.
- 10.2.2. Print and display current condition on the instrument for easy access when performing daily instrument routine check.
- 10.2.3. In the event that instruments parameters necessitate a changed, replace the instrument parameter printout with the new parameter setup and archive the previous instrument parameters in the instrument maintenance log.
- 10.2.4. Set injection volume to 1 μl to 2 μl .

10.3. Calibration

- 10.3.1. Set GC/MS operating condition as described in Section 10.2.
- 10.3.2. Perform Tune Check
 - 10.3.2.1. Analyze a solution containing 50- $\mu\text{g/ml}$ of tuning standard working solution.
 - 10.3.2.2. Evaluate the tune check by either a single scan or the average of 3 scans (before, at, and after the apex). Apply a background subtraction using a single scan no more than 20 scans prior to the elution of DFTPP. See Table 4 for acceptance criteria or follow the manufacturer's recommendation for tuning.

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8270 Revision No. 3 Date: 15-July-06

10.3.2.3. Evaluate the DDT degradation to DDE and DDD for column performance and injection port inertness using the data acquisition software.

- Degradation of DDT to DDE and DDD must be less than 20% based on area obtained from the total ion chromatogram.
- Benzidine and Pentachlorophenol must be present. Evaluate the tailing factor of benzidine and pentachlorophenol. Benzidine tailing ≤ 3 and pentachlorophenol tailing ≤ 5 . Refer to Figure-2 for peak evaluation.

10.3.3. Initial Calibration (ICAL)

10.3.3.1. Perform ICAL when one of the conditions occurs.

- Instrument is new
- Instrument undergoes a major repair
- DCC failed to meet the acceptance criteria

10.3.3.2. Analyze a 5 to 9-point initial calibration curve as suggested in Figure 5 after a valid tune check.

10.3.3.3. Evaluate System Performance Check Compounds (SPCC) and calibration Check Compounds (CCC) in accordance to Appendix 1.

10.3.3.4. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:

- Check the established retention time window
- Check the relative intensity of major ions
- Adjust accordingly if necessary.

10.3.3.5. Establish the relative retention time of each analyte with respect to the nearest internal standard.

10.3.3.6. Establish a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRFm), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq-10.6.1.1, Eq-10.6.1.2, Eq-10.6.1.3 and Eq-10.6.1.4 respectively.

10.3.3.7. Evaluate the ICAL for appropriate quantitation method.

- Use RRFm - if the RSD of individual analyte $\leq 15\%$.
- Use RRFm - if the mean RSD of all target analyte is $\leq 15\%$ provided no individual RSD is above 30%.
- Use first order linear regression if $R \geq 0.995$
- Use second order regression if $COD \geq 0.99$ (based on six calibration points)
- Higher order regression is acceptable based on a minimum of seven calibration points.

10.3.3.8. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

STANDARD OPERATING PROCEDURE

SEMIVOLATILE ORGANICS BY GC/MS

SOP No.: EMAX-8270 Revision No. 3 Date: 15-July-06

- 10.3.4. Initial Calibration Verification (ICV)
- 10.3.4.1. Verify the concentration of the ICAL by analyzing the ICV from a second source (See Table 2 for standard preparation).
 - 10.3.4.2. Check for completeness of analytes as described in 10.3.3.4.
 - 10.3.4.3. Compare the retention times of the internal standards to the ICAL mid-point. Changes greater than 30 seconds indicates instrument malfunction. Corrective action is required prior to further analysis.
 - 10.3.4.4. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).
 - 10.3.4.5. Check the project specific requirement (PSR) for ICV. Otherwise, refer to Appendix 1 for acceptance criteria or corrective action. Refer to Section 12 for possible measures for corrective action.
- 10.3.5. Daily Continuing Calibration (DCC)
- 10.3.5.1. Analyze DCC (See Table 2 for standard preparation) to check the validity of the ICAL.
 - 10.3.5.2. Check SPCCs for minimum response factor (min. 0.05)
 - 10.3.5.3. Check the CCCs for percentage difference (%Drift \leq 20%). If the CCCs are not included in the target analyte list, all analytes must meet the % drift criterion.
 - 10.3.5.4. Check for completeness of analytes as described in 10.3.3.4.
 - 10.3.5.5. Compare the retention times of the internal standards to the ICAL mid-point. Changes greater than 30 seconds indicates instrument malfunction. Corrective action is required prior to further analysis.
 - 10.3.5.6. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).
 - 10.3.5.7. Check the PSR for DCC. Otherwise, refer to Appendix 1 for acceptance criteria or corrective action. Refer to Section 12 for possible measures for corrective action.

10.4. Analysis**10.4.1. Extract Preparation**

- 10.4.1.1. Allow extracts to equilibrate with room temperature.
- 10.4.1.2. Measure 300 μ L of extract, transfer into an autosampler vial.
- 10.4.1.3. Add 6 μ L of 2000-ng/ μ L of internal standard (refer to 9.4.3).
- 10.4.1.4. Seal the vial with Teflon-lined septa cap.

10.4.2. Analytical Sequence

- 10.4.2.1. Analyze instrument blank.
- 10.4.2.2. Analyze DFTPP and evaluate tuning.
- 10.4.2.3. Analyze DCC and check ICAL validity.

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10.4.2.4. Analyze Method Blank.

10.4.2.5. Analyze Lab Control Sample and Lab Control Sample Duplicate (optional).

10.4.2.6. Analyze matrix spikes (MS/MSD) as per project requirement.

10.4.2.7. Analyze samples to a maximum number of 12-hour from the time of DFTPP injection.

10.4.3. **Sample Result Evaluation**

10.4.3.1. Check QC criteria

- Check surrogate recoveries against PSR. In the absence of PSR, default to EMAX QC limits.
- Check concentration of target analytes if calibration range is exceeded.
- If any of the above checkpoints indicate a problem, re-analysis is required.

10.4.3.2. Qualitative Identification

- The intensities of the characteristic ions must maximize in the same scan or within one scan of each other.
- The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
- The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.
- Check the chromatogram for possible misidentified analytes. Investigate visible peaks in the chromatogram that were not identified in the data output. Manually integrate the peak if necessary. The analyst prior to submission shall initial all manual integration undertaken. All original copies will be submitted as file copies.
- For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative identification¹ (TIC).
- Execute LSC (Chem Station program) to initiate the library search using NIST/EPA/MSDC mass spectral library.
- Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting.

10.4.3.3. Quantitation

- Apply the appropriate quantitation method (Section 10.6.3) to calculate the concentration of any positively identified target analyte. Apply the dilution factor for diluted samples to calculate for the final concentration of the sample.

10.4.3.4. Manual Integration

10.4.3.4.1. Refer to EMAX-DM01 for details of manual integration.

10.4.3.5. Dealing with Carryover

¹ Library search is performed only when indicated in the PSR.

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- Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
- If there was a target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carryover. If carryover is confirmed, proceed with data reduction and report the data from re-analysis.

10.5. Data Reduction

- 10.5.1. Make a copy of the analytical run log and highlight the data to be reported.
- 10.5.2. Collate the reportable raw data separating the QC results from the sample results.
- 10.5.3. Keep all other data generated with the analytical folder marked with "For Record Only".
- 10.5.4. Proceed to report generation.

10.6. Calculations

10.6.1. Initial Calibration

10.6.1.1. Calculate for Relative Response Factor (RRF)

$$RRF = \frac{A_x C_{is}}{A_{is} C_x} \quad \text{Eq.-10.6.1.1}$$

where:

- A – Area of characteristic ion for the compound being measured
- A_{is} – Area of characteristic ion for the specific internal standard
- C_x – Concentration of the compound being measured
- C_{is} – Concentration of the specific internal standard

10.6.1.2. Calculate for Average Relative Response Factor (RRF_m).

$$RRF_m = \frac{\sum RRF}{n} \quad \text{Eq-10.6.1.2}$$

where:

- RRF_m – average response factor
- $\sum RRF$ – summation of response factors

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10.6.1.3. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq.-10.6.1.3}$$

where:

 SD – standard deviation x_i – result at i^{th} measurement \bar{x} – mean n – number of measurements

10.6.1.4. Calculate for % relative standard deviation (%RSD).

$$\%RSD = \frac{SD}{RRF_m} * 100\% \quad \text{Eq.-10.6.1.4}$$

where:

 SD – standard deviation RRF_m – average response factor

10.6.1.5. Calculate for Least Square Linear Regression

$$y = ax + b \quad \text{Eq.-10.6.1.5}$$

where:

 y = Response Ratio (AX/AIS) x = Amount Ratio (CX/CIS) a = x_1 = slope of the line b = x_0 = intercept of the line

10.6.1.6. Calculate for Quadratic Regression

$$y = ax^2 + bx + c \quad \text{Eq.-10.6.1.6}$$

where:

 y = Response Ratio (AX/AIS) x = Amount Ratio (CX/CIS) a = x_1 = slope of the line

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b = x_0 = intercept of the line

c = x_2 = constant of the line

10.6.2. Calibration Check/Continuing Calibration

10.6.2.1. Calculate Percent Difference (%D) when RRF_m is used for quantitation

$$\%D = \frac{[RRF_c - RRF_m]}{RRF_m} * 100\% \quad \text{Eq-10.6.2.1}$$

where:

RRF_c – response factor from continuing calibration standard

RRF_m – average response factor

10.6.2.2. Calculate Percent Deviation (%Dt) when the first order linear regression or the second order regression is used for quantitation

$$\%D_t = \frac{\text{abs}(T_t - T_f)}{T_t} * 100\% \quad \text{Eq-10.6.2.2}$$

where:

abs – absolute value

T_t – true value of standard in µg/L

T_f – found value of standard in µg/L

10.6.3. Calculation of Sample Concentration (Water and Soil/Sediment Samples). When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the EICP of the primary characteristic ion.

10.6.3.1. Water Samples

$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(V_t)} \quad \text{Eq-10.6.3.1}$$

where:

A_x – area of characteristic ion for the compound to be measured

I_s – amount of internal standard added

V_e – extract final volume from sample extraction, usually 1-ml

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$$DF \quad - \text{dilution factor} = \frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$$

A_{is} – area of characteristic ion for the internal standard

RRF_m – average response factor

V_i – volume of extract injected in μL , usually 1- μL

V_t – volume of water extracted in ml, usually 1000-ml

10.6.3.2. Soil/Sediment Samples (Dry weight basis)

$$\text{Concentration (ug/Kg)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} \quad \text{Eq-10.6.3.2}$$

where:

A_x – area of characteristic ion for the compound to be measured

I_s – amount of internal standard injected in ng

V_e – volume of extract in ml, usually 1-ml²

$$DF \quad - \text{dilution factor} = \frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$$

A_{is} – area of characteristic ion for the internal standard

RRF_m – average response factor

V_i – volume of extract injected in μL , usually 1- μL

W_s – wet soil weight in kg

$$DW \quad - \% \text{ solid} = \frac{100 - \% \text{moisture}}{100}$$

10.6.3.3. Extract is cleaned up by GPC

$$\text{Concentration (ug/Kg)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} * \frac{V_{bg}}{V_{ig}} \quad \text{Eq-10.6.3.3}$$

where:

A_x – area of characteristic ion for the compound to be measured

I_s – amount of internal standard injected in ng

² For extracts subjected to GPC $V_i=0.5\text{-ml}$

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SEMIVOLATILE ORGANICS BY GC/MSSOP No.: EMAX-8270 Revision No. 3 Date: 15-July-06*Ve* – volume of extract in ml*DF* – dilution factor = $\frac{\text{aliquot}(\mu\text{L}) + \text{solvent}(\mu\text{L})}{\text{aliquot}(\mu\text{L})}$ *Ais* – area of characteristic ion for the internal standard*RFm* – average response factor*Vi* – volume of extract injected in μL , usually 1- μL *Ws* – wet soil weight in kg*DW* – % solid = $\frac{100 - \% \text{moisture}}{100}$ *Vbg* – total volume of extract before GPC clean-up in mL*Vig* – injected volume of extract to GPC in mL

10.6.4. Alternatively, the regression line (area ratio of A_x/A_{is} versus concentration using first degree or higher regression) fitted to the initial calibration may be used for determination of sample concentration when RSD of the analyte is greater than 15.

10.6.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:

10.6.5.1. The area “ A_x ” and “ A_{is} ” are derived from total ion chromatogram. “ A_{is} ” refers to the closest internal standard (IS) free of interference.

10.6.5.2. RRF of the TIC is 1.

10.6.6. Accuracy and Precision

10.6.6.1. Percent Recovery

$$\% \text{ Recovery} = \frac{C_f - C}{C_s} * 100 \quad \text{Eq-10.6.6.1}$$

where:

C_f – concentration found

C – concentration of sample

C_s – concentration of spike

10.6.6.2. Relative Percent Difference (%RPD)

$$\% \text{ RPD} = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq-10.6.6.2}$$

where:

RPD – Relative Percent Difference

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- C1* – Measured concentration of the first sample aliquot
C2 – Measured concentration of the second sample aliquot

10.6.7. DDT Degradation

$$\%B = \frac{A_{DDD} + A_{DDE}}{A_{DDT} + A_{DDD} + A_{DDE}} (100) \quad \text{Eq-10.6.7}$$

where:

- %B* – percent breakdown
ADDD – area of DDD
ADDE – area of DDE
ADDT – area of DDT

10.7. **Report Generation And Data Reduction & Review**

- 10.7.1. Generate the method.txt file using WDB1C.exe
- 10.7.2. Generate Lab Chronicle using Labchron1.exe
- 10.7.3. Generate the sample results using F1V3C.exe
- 10.7.4. Generate the QC summary using QCV3C.exe
- 10.7.5. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.
- Sample Results
 - LCS Summary
 - MS/MSD Summary
 - DCC Summary
 - ICV Summary
 - ICAL Summary
- 10.7.6. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
- 10.7.7. Generate the case narrative to include discussion of the following as found in the review process:
- Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples extracted and analyzed within the holding time. For non-compliance, state the number of days that the sample(s) was out of holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.

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- Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
- Method Blank – That MB was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s) affected and the associated sample results were flagged with “B”.
- Lab Control Samples (if applicable) – That LCS was prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Further more, if corrective action is not possible (e.g. no more samples to re-extract) state that results were qualified.
- Matrix Spike Samples – That MS/MSD (if applicable) were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Sample Analysis – that samples were analyzed in conformance to the method and project requirements.
- Other Anomalies (if any) – discussed on a case by case basis concurred by the Supervisor or the Lab Director.

10.7.8. Submit the analysis package for secondary review.

10.8. Preventive Maintenance

- 10.8.1. Refer to form 8270FM for daily routine maintenance check points.
- 10.8.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back to control.
- 10.8.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality.

11.0 QUALITY CONTROL**11.1. Preparative Batch**

- 11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD (when required by the project) and a maximum of 20 field samples of similar matrix.
- 11.1.2. In the absence of MS/MSD, prepare LCS/LCD to check for precision.

11.2. Analytical Batch QC

- 11.2.1. The GC/MS tuning standard must be analyzed at the beginning of every 12-hour shift. GC/MS tuning criteria is listed in Table 4 and Section 10.3.2.
- 11.2.2. A continuing calibration shall be performed before any other analysis is done, and after analysis of tuning standard. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.6.

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11.3. Method QC

- 11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.
- 11.3.2. A valid MDL must exist prior to sample analysis.
- 11.3.3. A valid ICAL must exist prior to sample analysis.
- 11.3.4. Instrument performance must be checked prior to sample analysis.
- 11.3.5. Check Appendix 1 for acceptance criteria.
- 11.3.6. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.
- 11.3.7. Surrogate standard shall be added to all samples, including method blank LCS/LCD and MS/MSD. Check PSR for QC Control Limits.

12.0 CORRECTIVE ACTIONS

- 12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures in Appendix 1. Document out-of-control event and corrective action in the analytical logbook. If the problem persists, consult the supervisor.
- 12.2. If the tune is non-compliant, consider the following suggestions to correct the problem:
 - 12.2.1. Check the instrument settings and make sure that the instrument parameters are properly set up
 - 12.2.2. Check gas flow
 - 12.2.3. Perform autotune or visual optimization
 - 12.2.4. If the problem persists, inform the supervisor
- 12.3. If initial calibration is non-compliant, consider the following suggestions to correct the problem:
 - 12.3.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.
 - 12.3.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and reanalyze.
 - 12.3.3. If the highest ICAL point appears to be saturated, drop the highest point.
 - 12.3.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

Note: The lowest calibration point identifies the reporting limit (RL). Therefore, check that the RL is in conformance to the current projects where the ICAL will be used.
 - 12.3.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:
 - 12.3.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.
 - 12.3.5.2. Check the gas flow
 - 12.3.5.3. Retune the MS
 - 12.3.5.4. Prepare a fresh standard and repeat calibration
 - 12.3.5.5. Clean the MS source and repeat the calibration

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- 12.3.6. If the problem persists, inform the supervisor.
- 12.4. If the ICV is non-compliant, consider the following suggestions to correct the problem:
 - 12.4.1. Reanalyze ICV (to rule out poor injection)
 - 12.4.2. If ICV is still out of acceptance criteria, prepare a fresh standard and reanalyze to rule out any preparation error
 - 12.4.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration
 - 12.4.4. If the problem persists, inform the supervisor
- 12.5. If the instrument blank is non-compliant, consider the following suggestions to correct problem:
 - 12.5.1. Rule out instrument contamination by performing the instrument daily maintenance , such as changing septum, cleaning liner, cleaning or using new autosampler syringe.
 - 12.5.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.
 - 12.5.3. Rule out preparation contamination by preparing a new instrument blank
 - 12.5.4. If the problem persists, inform the supervisor.
- 12.6. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:
 - 12.6.1. Change the liner
 - 12.6.2. Clean injection port
 - 12.6.3. Prepare new standard
 - 12.6.4. Cut or replace column
 - 12.6.5. Rule out leaks by checking all connections
 - 12.6.6. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL
- 12.7. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
 - 12.7.1. Rule out instrument contamination by checking instrument blank
 - 12.7.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01
 - 12.7.3. Rule out glassware contamination used for extraction as described in EMAX-QC07
 - 12.7.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents
 - 12.7.5. If the problem persists, inform the supervisor
- 12.8. If LCS is non-compliant, perform the following suggestions to correct the problem:
 - 12.8.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level
 - 12.8.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and reanalyze the LCS and the associated samples.
 - 12.8.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.
- 12.9. Execute a Non-Conformance Report (NCR) when the following circumstances occur:

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12.9.1. If corrective action needs the function of other department; eg, if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.

12.9.2. If corrective action needs the assistance of the project manager; eg. If the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.

12.10. For other problems encountered, inform the supervisor immediately for further instructions.

13.0 POLLUTION PREVENTION

13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.

13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 WASTE MANAGEMENT

14.1. All unused samples, expired analytical standards and other wastes generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES**15.1. Definition of Terms**

15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents. **Preparation batch** is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. **Analytical batch** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

15.1.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.

15.1.3. Duplicate Sample – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.1.4. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

15.1.5. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.

15.1.6. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.

15.1.7. Lab Control Sample Duplicate (LSD) – is a replicate of LCS analyzed to monitor precision when MS/MSD samples are not analyzed.

15.1.8. Matrix – is a component or form of a sample.

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- 15.1.9. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.10. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.12. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.13. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.2. Application of QC Procedures

- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all USACE Louisville projects when performing semivolatile analysis by GC/MS. In instances where there is project of program specific quality control, the requirements given in the project shall take precedence over this SOP.

15.3. Air Force Center for Environmental Excellence (AFCEE) projects

- 15.3.1. When samples from AFCEE sponsored projects are analyzed for semivolatiles by GC/MS, the calibration, QC, corrective action, and data flagging requirements shall requirements in Quality Assurance Project Plan, as updated.

15.4. U.S. Army Corps of Engineers (USACE) Projects

- 15.4.1. In the absence of project QAPP, the default QAPP is the Shell Document, latest version.

15.5. Naval Facilities Engineering Service Center (NFESC) Projects

- 15.5.1. In the absence of project QAPP, the default QAPP is the NFESC Interim Guidance Document, latest version.

15.6. Department of Energy Basic Ordering Agreement (DOE-BOA) Projects

- 15.6.1. For samples from DOE-BOA sponsored projects follow BOA Guidance Document, latest version in the absence of project QAPP.

16.0 REFERENCES

- 16.1. "Test Methods for Evaluation of Solid Wastes", EPA SW846, as updated.
- 16.2. Laboratory QA/QC Manual, as updated.

17.0 FIGURES, TABLES AND APPENDICES**17.1. Figures**

- 17.1.1. Figure 1 Peak Evaluation Technique

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- 17.1.2. Figure 2 Evaluation of Tailing Factor
- 17.1.3. Figure 3 Typical Chromatogram
- 17.1.4. Figure 4 DFTPP Chromatogram
- 17.1.5. Figure 5 Typical ICAL Summary
- 17.1.6. Figure 6 Typical ICV Summary
- 17.1.7. Figure 7 Typical Sample Result Summary
- 17.1.8. Figure 8 Typical LCS Report Summary
- 17.1.9. Figure 9 Typical MS/MSD Report Summary
- 17.1.10. Figure 10 Typical Case Narrative

17.2. Tables

- 17.2.1. Table 1 Intermediate Standard Preparation
- 17.2.2. Table 2 Working Standard Preparation
- 17.2.3. Table 3 Instrument Parameters
- 17.2.4. Table 4 DFTPP Key Ions and Ion Abundance Criteria
- 17.2.5. Table 5 Calibration Check Compounds
- 17.2.6. Table 6 System Performance Calibration Check Compounds (SPCC)

17.3. Appendices

- 17.3.1. Appendix 1 Summary of In-House Quality Control Procedures
- 17.3.2. Appendix 2 Demonstration of Capability
- 17.3.3. Appendix 3 Analyte List and Quantitation Ion
- 17.3.4. Appendix 4 Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation
- 17.3.5. Appendix 5 Target Compound List & Reporting Limits

17.4. Forms

- 17.4.1. 8270FA Analytical Run Log
- 17.4.2. 8270FS Sample Preparation Log
- 17.4.3. 8270FM Instrument Maintenance Log

Figure 1 - Peak Evaluation Technique

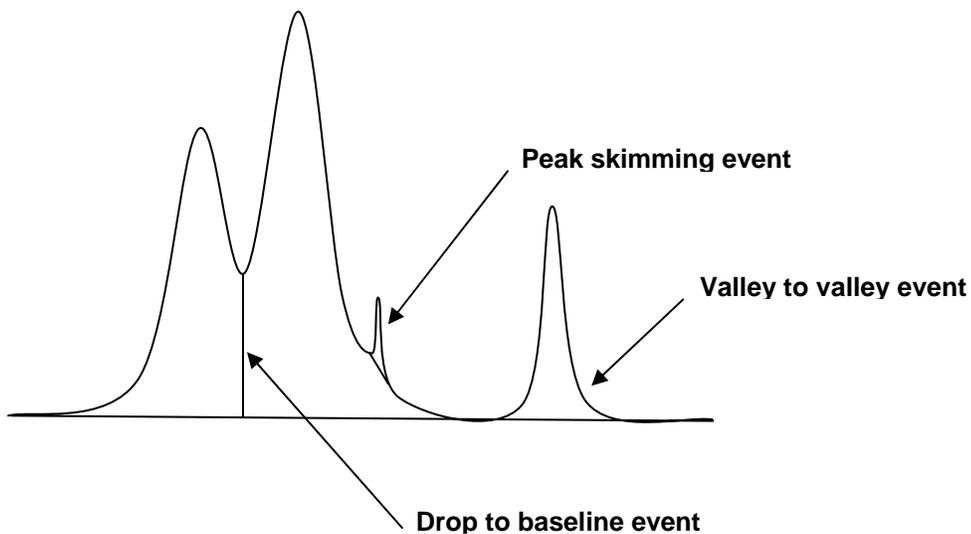


Figure 2 – Evaluation of Tailing Factor

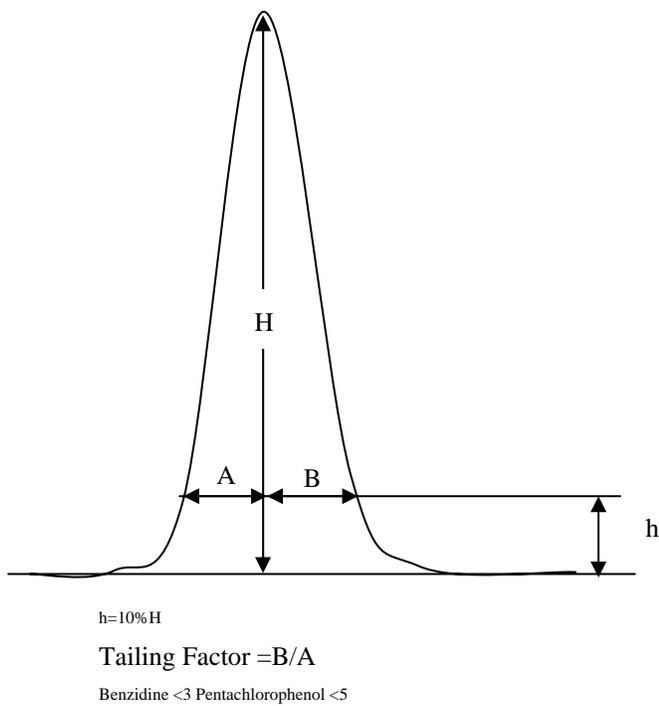


Figure 3 – Typical Chromatogram

FIGURE 4 – TYPICAL CHROMATOGRAM

Quantitation Report

Data File : C:\HPCHEM\1\DATA\06C30\RCH368.D
 Acq On : 30 Mar 2006 11:04
 Sample : CSV41C1606
 Misc :
 MS Integration Params: RTEINT.P
 Quant Time: Mar 30 11:21 2006

Vial: 3
 Operator: SG
 Inst : TO41
 Multiplr: 1.00

Quant Results File: SV41C16.RES

Method : C:\HPCHEM\1\METHODS\SV41C16.M (RTE Integrator)
 Title : METHOD 8270C
 Last Update : Fri Mar 17 17:22:53 2006
 Response via : Initial Calibration

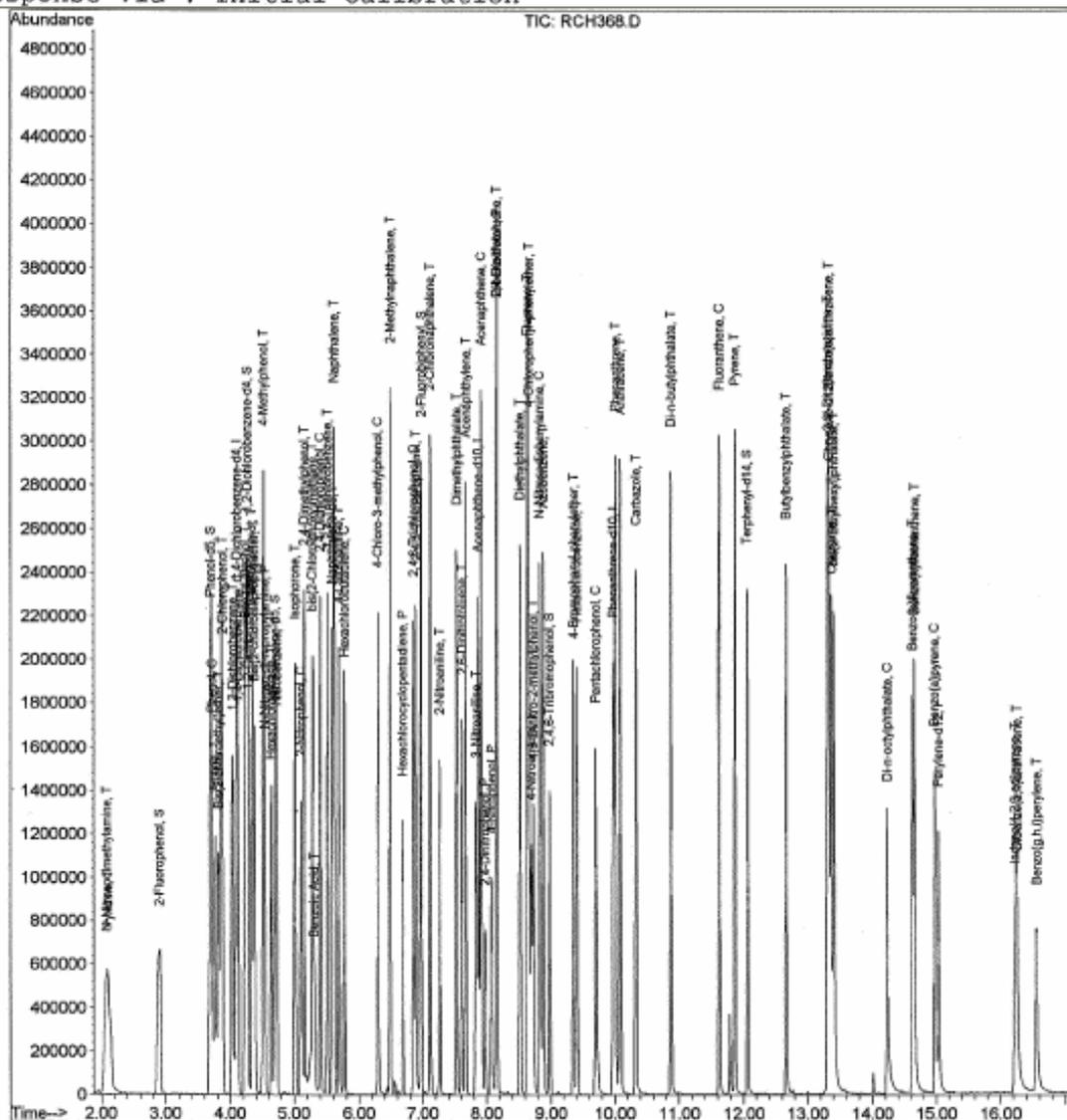
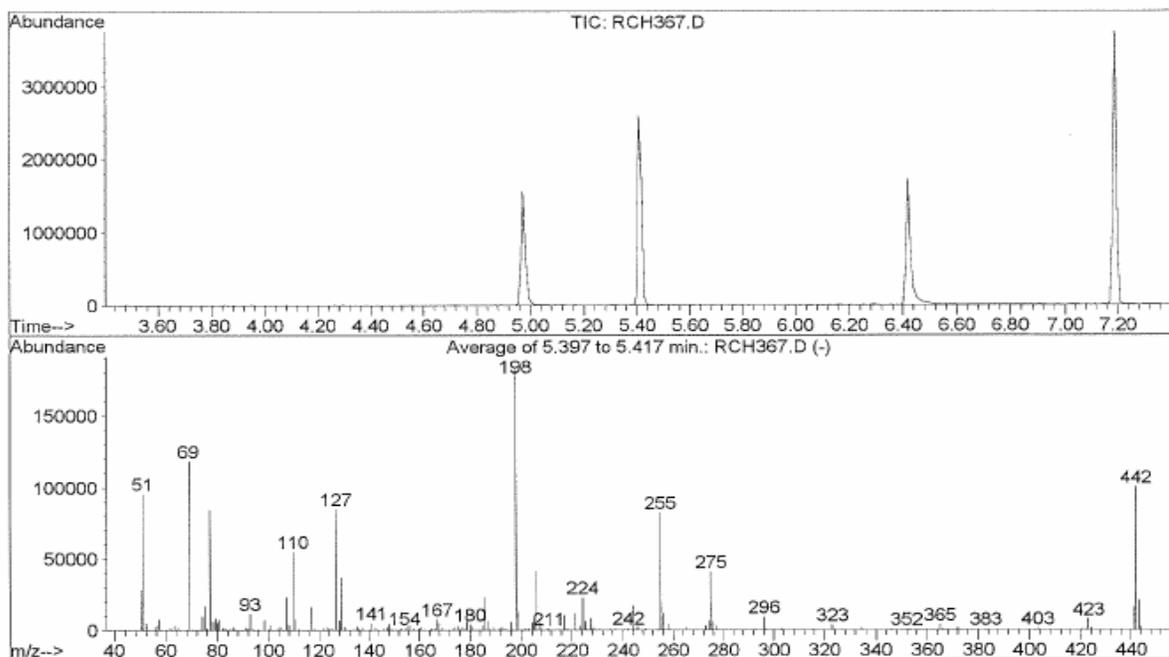


FIGURE 4 – DFTPP CHROMATOGRAM

DFTPP

Data File : C:\HPCHEM\1\DATA\06C30\RCH367.D Vial: 2
 Acq On : 30 Mar 2006 10:35 Operator: SG
 Sample : DFT41C1606 Inst : TO41
 Misc : Multiplr: 1.00
 MS Integration Params: rteint.p
 Method : C:\HPCHEM\1\METHODS\DFTPP.M (RTE Integrator)
 Title : 8270C TUNE 5970MSD-5890GC



AutoFind: Scans 304, 305, 306; Background Corrected with Scan 301

| Target Mass | Rel. to Mass | Lower Limit% | Upper Limit% | Rel. Abn% | Raw Abn | Result Pass/Fail |
|-------------|--------------|--------------|--------------|-----------|---------|------------------|
| 51 | 198 | 30 | 60 | 52.4 | 95347 | PASS |
| 68 | 69 | 0.00 | 2 | 0.0 | 0 | PASS |
| 69 | 198 | 0.00 | 100 | 64.9 | 118192 | PASS |
| 70 | 69 | 0.00 | 2 | 0.4 | 494 | PASS |
| 127 | 198 | 40 | 60 | 46.5 | 84707 | PASS |
| 197 | 198 | 0.00 | 1 | 0.0 | 0 | PASS |
| 198 | 198 | 100 | 100 | 100.0 | 181995 | PASS |
| 199 | 198 | 5 | 9 | 7.0 | 12691 | PASS |
| 275 | 198 | 10 | 30 | 22.3 | 40628 | PASS |
| 365 | 198 | 1 | 100 | 1.9 | 3539 | PASS |
| 441 | 443 | 0.01 | 100 | 76.8 | 15821 | PASS |
| 442 | 198 | 40 | 100 | 55.0 | 100111 | PASS |
| 443 | 442 | 17 | 23 | 20.6 | 20587 | PASS |

FIGURE 5 – TYPICAL ICAL SUMMARY

| INITIAL_CALIBRATION - RELATIVE_RESPONSE_FACTOR | | | | | | | | | | | | | |
|--|-----------------------------|--------|--------|--------|--------|--------|--------|--------|--------|-------|---------|-------|---------|
| Instrument ID :FO42 | | | | | | | | | | | | | |
| Beginning Date/Time :03/07/06 14:54 | | | | | | | | | | | | | |
| Spike Units :PPM | | | | | | | | | | | | | |
| IC File :RCX040 | | | | | | | | | | | | | |
| Column Spec :ZB-5MS ID :0.18MM | | | | | | | | | | | | | |
| Ending Date/Time :03/07/06 18:17 | | | | | | | | | | | | | |
| HPChem Method :SV42C07 | | | | | | | | | | | | | |
| 14:54 | 15:20 | 15:45 | 16:10 | 16:36 | 17:01 | 17:26 | 17:52 | 18:17 | Av_RRF | %_RSD | Av_Rt_M | | |
| RCX036 | RCX037 | RCX038 | RCX039 | RCX040 | RCX041 | RCX042 | RCX043 | RCX044 | | | | | |
| 5 | 10 | 20 | 40 | 50 | 80 | 100 | 120 | 160 | | | | | |
| 14:54 | 15:20 | 15:45 | 16:10 | 16:36 | 17:01 | 17:26 | 17:52 | 18:17 | Av_RRF | %_RSD | Av_Rt_M | | |
| ==== | ==== | ==== | ==== | ==== | ==== | ==== | ==== | ==== | ==== | ==== | ==== | | |
| 1 | 1,4-Dichlorobenzene-d4 | 0.863 | 0.885 | 0.855 | 0.857 | 0.891 | 0.886 | 0.874 | 0.879 | 0.872 | 0.874 | 1.48 | 3.4075 |
| 2 | N-Nitrosodimethylamine | 1.347 | 1.484 | 1.457 | 1.425 | 1.498 | 1.458 | 1.477 | 1.480 | 1.498 | 1.458 | 3.26 | 1.6110 |
| 3 | Pyridine | 1.158 | 1.204 | 1.199 | 1.202 | 1.265 | 1.249 | 1.243 | 1.241 | 1.254 | 1.224 | 2.84 | 2.6234 |
| 4 | 2-Fluorophenol | 1.908 | 1.946 | 1.898 | 1.822 | 1.870 | 1.761 | 1.645 | 1.648 | 1.631 | 1.792 | 6.96 | 3.0880 |
| 5 | Phenol | 2.000 | 2.013 | 1.994 | 1.899 | 1.986 | 1.916 | 1.905 | 1.931 | 1.985 | 1.959 | 2.32 | 3.1207 |
| 6 | Bis(2-chloroethyl)ether | 1.665 | 1.629 | 1.570 | 1.479 | 1.528 | 1.462 | 1.411 | 1.277 | 1.164 | 1.465 | 11.08 | 3.1747 |
| 7 | Phenol-d5 | 1.587 | 1.657 | 1.638 | 1.620 | 1.673 | 1.614 | 1.579 | 1.572 | 1.534 | 1.608 | 2.76 | 3.0779 |
| 8 | 2-Chlorophenol | 1.474 | 1.458 | 1.418 | 1.366 | 1.384 | 1.317 | 1.290 | 1.248 | 1.214 | 1.352 | 6.76 | 3.2140 |
| 9 | 1,3-Dichlorobenzene | 1.534 | 1.574 | 1.512 | 1.459 | 1.461 | 1.437 | 1.379 | 1.363 | 1.324 | 1.449 | 5.72 | 3.3524 |
| 10 | 1,4-Dichlorobenzene | 1.585 | 1.571 | 1.508 | 1.441 | 1.480 | 1.392 | 1.373 | 1.344 | 1.299 | 1.444 | 6.95 | 3.4255 |
| 11 | Benzyl alcohol | 0.840 | 0.907 | 0.915 | 0.873 | 0.892 | 0.806 | 0.779 | 0.752 | 0.752 | 0.835 | 7.82 | 3.5639 |
| 12 | 1,2-Dichlorobenzene-d4 | 1.034 | 1.022 | 0.956 | 0.889 | 0.898 | 0.819 | 0.804 | 0.768 | 0.739 | 0.881 | 12.19 | 3.5571 |
| 13 | 1,2-Dichlorobenzene | 1.565 | 1.494 | 1.448 | 1.327 | 1.330 | 1.218 | 1.144 | 1.076 | ----- | 1.325 | 12.99 | 3.5753 |
| 14 | 2-Methylphenol | 1.159 | 1.192 | 1.200 | 1.138 | 1.167 | 1.111 | 1.104 | 1.045 | 1.015 | 1.125 | 5.64 | 3.6719 |
| 15 | Bis(2-chloroisopropyl)ether | 3.247 | 3.178 | 3.088 | 2.969 | 2.969 | 2.878 | 2.810 | 2.792 | 2.641 | 2.953 | 6.62 | 3.6989 |
| 16 | 4-Methylphenol | 1.621 | 1.708 | 1.674 | 1.532 | 1.678 | 1.647 | 1.638 | 1.643 | 1.670 | 1.646 | 3.03 | 3.8496 |
| 17 | N-Nitroso-di-n-propylamine | 1.209 | 1.228 | 1.206 | 1.148 | 1.240 | 1.153 | 1.156 | 1.209 | 1.072 | 1.180 | 4.46 | 3.8586 |
| 18 | Hexachloroethane | 0.651 | 0.707 | 0.680 | 0.652 | 0.672 | 0.649 | 0.634 | 0.637 | 0.585 | 0.652 | 5.22 | 3.9182 |
| 19 | Naphthalene-d8 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 4.8170 |
| 20 | Nitrobenzene-d5 | 0.391 | 0.422 | 0.428 | 0.416 | 0.430 | 0.417 | 0.411 | 0.403 | 0.408 | 0.414 | 2.97 | 3.9969 |
| 21 | Nitrobenzene | 0.418 | 0.433 | 0.429 | 0.407 | 0.411 | 0.421 | 0.412 | 0.391 | 0.364 | 0.410 | 5.16 | 4.0172 |
| 22 | Isophorone | 0.803 | 0.821 | 0.808 | 0.781 | 0.796 | 0.803 | 0.797 | 0.772 | 0.763 | 0.794 | 2.31 | 4.3029 |
| 23 | 2-Nitrophenol | 0.204 | 0.224 | 0.238 | 0.234 | 0.240 | 0.239 | 0.227 | 0.219 | 0.224 | 0.228 | 5.24 | 4.3704 |
| 24 | 2,4-Dimethylphenol | 0.324 | 0.346 | 0.347 | 0.319 | 0.335 | 0.323 | 0.316 | 0.315 | 0.325 | 0.328 | 3.73 | 4.4469 |
| 25 | bis(2-Chloroethoxy)methane | 0.531 | 0.541 | 0.542 | 0.494 | 0.496 | 0.482 | 0.476 | 0.471 | 0.464 | 0.500 | 6.12 | 4.5650 |
| 26 | Benzoic Acid | 0.074 | 0.142 | 0.203 | 0.241 | 0.242 | 0.258 | 0.252 | 0.246 | 0.256 | 0.213 | 30.00 | 4.6741 |
| 27 | 2,4-Dichlorophenol | 0.322 | 0.339 | 0.352 | 0.337 | 0.341 | 0.329 | 0.320 | 0.306 | 0.302 | 0.328 | 5.03 | 4.6629 |
| 28 | 1,2,4-Trichlorobenzene | 0.389 | 0.376 | 0.368 | 0.347 | 0.346 | 0.330 | 0.313 | 0.303 | 0.294 | 0.341 | 9.81 | 4.7529 |
| 29 | Naphthalene | 1.154 | 1.122 | 1.094 | 0.981 | 0.963 | 0.928 | 0.844 | 0.836 | 0.803 | 0.969 | 13.44 | 4.8451 |
| 30 | 4-Chloroaniline | 0.485 | 0.503 | 0.518 | 0.484 | 0.472 | 0.476 | 0.448 | 0.423 | 0.422 | 0.470 | 7.06 | 4.9363 |
| 31 | Hexachlorobutadiene | 0.229 | 0.219 | 0.219 | 0.205 | 0.206 | 0.195 | 0.185 | 0.176 | 0.166 | 0.200 | 10.66 | 5.0026 |
| 32 | 4-Chloro-3-methylphenol | 0.379 | 0.400 | 0.415 | 0.396 | 0.394 | 0.384 | 0.363 | 0.357 | 0.367 | 0.384 | 4.98 | 5.5842 |
| 33 | 2-Methylnaphthalene | 0.794 | 0.803 | 0.771 | 0.694 | 0.689 | 0.658 | 0.613 | 0.593 | 0.559 | 0.686 | 12.97 | 5.7360 |
| 34 | Acenaphthene-d10 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 7.0814 |
| 35 | Hexachlorocyclopentadiene | 0.106 | 0.144 | 0.193 | 0.236 | 0.254 | 0.267 | 0.250 | 0.250 | 0.237 | 0.215 | 26.09 | 5.9340 |
| 36 | 2,4,6-Trichlorophenol | 0.404 | 0.436 | 0.442 | 0.438 | 0.451 | 0.444 | 0.426 | 0.414 | 0.396 | 0.428 | 4.47 | 6.1106 |
| 37 | 2,4,5-Trichlorophenol | 0.462 | 0.485 | 0.496 | 0.493 | 0.512 | 0.494 | 0.472 | 0.425 | 0.393 | 0.470 | 8.14 | 6.1568 |
| 38 | 2-Fluorobiphenyl | 1.451 | 1.426 | 1.412 | 1.320 | 1.305 | 1.174 | 1.126 | 1.120 | 0.952 | 1.254 | 13.58 | 6.2276 |
| 39 | 2-Chloronaphthalene | 1.237 | 1.244 | 1.226 | 1.149 | 1.163 | 1.089 | 1.065 | 0.997 | 0.899 | 1.119 | 10.52 | 6.3570 |
| 40 | 2-Nitroaniline | 0.419 | 0.470 | 0.508 | 0.532 | 0.547 | 0.570 | 0.562 | 0.542 | 0.525 | 0.520 | 9.25 | 6.5257 |
| 41 | Dimethylphthalate | 1.566 | 1.559 | 1.562 | 1.501 | 1.494 | 1.477 | 1.439 | 1.399 | 1.328 | 1.480 | 5.46 | 6.7991 |
| 42 | 2,6-Dinitrotoluene | 0.282 | 0.331 | 0.369 | 0.380 | 0.405 | 0.378 | 0.362 | 0.343 | 0.299 | 0.350 | 11.48 | 6.8733 |
| 43 | Acenaphthylene | 1.900 | 1.917 | 1.923 | 1.776 | 1.798 | 1.645 | 1.541 | 1.380 | ----- | 1.735 | 11.39 | 6.8909 |
| 44 | 3-Nitroaniline | 0.342 | 0.403 | 0.423 | 0.404 | 0.410 | 0.433 | 0.436 | 0.433 | 0.378 | 0.407 | 7.53 | 7.0882 |
| 45 | Acenaphthene | 1.279 | 1.250 | 1.202 | 1.110 | 1.098 | 1.089 | 1.023 | 0.933 | 0.846 | 1.092 | 13.06 | 7.1321 |
| 46 | 2,4-Dinitrophenol | 0.036 | 0.093 | 0.175 | 0.238 | 0.268 | 0.300 | 0.302 | 0.304 | 0.281 | 0.222 | 44.65 | 7.2333 |
| 47 | 4-Nitrophenol | 0.097 | 0.134 | 0.176 | 0.183 | 0.193 | 0.189 | 0.184 | 0.185 | 0.176 | 0.169 | 19.05 | 7.3683 |
| 48 | Dibenzofuran | 1.923 | 1.974 | 1.880 | 1.764 | 1.745 | 1.571 | 1.464 | 1.438 | 1.175 | 1.682 | 13.40 | 7.3717 |
| 49 | 2,4-Dinitrotoluene | 0.415 | 0.485 | 0.543 | 0.553 | 0.552 | 0.571 | 0.571 | 0.550 | 0.488 | 0.525 | 9.94 | 7.4122 |
| 50 | Diethylphthalate | 1.605 | 1.644 | 1.632 | 1.548 | 1.566 | 1.526 | 1.459 | 1.352 | 1.283 | 1.513 | 8.29 | 7.7766 |
| 51 | Fluorene | 1.476 | 1.551 | 1.474 | 1.418 | 1.436 | 1.347 | 1.252 | 1.164 | 1.054 | 1.352 | 12.13 | 7.8396 |
| 52 | 4-Chlorophenyl-phenylether | 0.808 | 0.815 | 0.774 | 0.732 | 0.717 | 0.708 | 0.660 | 0.597 | 0.576 | 0.710 | 12.06 | 7.8722 |
| 53 | 4-Nitroaniline | 0.375 | 0.428 | 0.474 | 0.483 | 0.496 | 0.481 | 0.470 | 0.445 | 0.436 | 0.454 | 8.28 | 7.9521 |
| 54 | 2,4,6-Tribromophenol | 0.247 | 0.273 | 0.278 | 0.279 | 0.289 | 0.279 | 0.276 | 0.267 | 0.252 | 0.271 | 4.96 | 8.1782 |
| 55 | Phenanthrene-d10 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 9.1377 |
| 56 | 4,6-Dinitro-2-methylphenol | 0.083 | 0.140 | 0.183 | 0.198 | 0.219 | 0.219 | 0.215 | 0.210 | 0.198 | 0.185 | 24.57 | 7.9757 |
| 57 | N-Nitrosodiphenylamine | 0.607 | 0.640 | 0.625 | 0.581 | 0.578 | 0.542 | 0.519 | 0.505 | 0.436 | 0.559 | 11.64 | 8.0579 |
| 58 | Azobenzene | 1.092 | 1.100 | 1.202 | 1.099 | 1.115 | 1.051 | 0.994 | 0.922 | 0.815 | 1.043 | 11.65 | 8.0916 |
| 59 | 4-Bromophenyl-phenylether | 0.286 | 0.283 | 0.281 | 0.265 | 0.262 | 0.260 | 0.249 | 0.236 | 0.217 | 0.260 | 8.91 | 8.5505 |
| 60 | Hexachlorobenzene | 0.342 | 0.346 | 0.339 | 0.316 | 0.318 | 0.303 | 0.296 | 0.284 | 0.271 | 0.313 | 8.48 | 8.5910 |
| 61 | Pentachlorophenol | 0.116 | 0.154 | 0.187 | 0.192 | 0.203 | 0.205 | 0.206 | 0.206 | 0.198 | 0.185 | 16.58 | 8.8880 |
| 62 | Phenanthrene | 1.348 | 1.346 | 1.280 | 1.147 | 1.146 | 1.080 | 1.042 | 0.994 | 0.898 | 1.142 | 13.78 | 9.1782 |
| 63 | Anthracene | 1.321 | 1.353 | 1.322 | 1.173 | 1.185 | 1.073 | 1.032 | 0.997 | 0.927 | 1.154 | 13.52 | 9.2514 |
| 64 | Carbazole | 1.238 | 1.272 | 1.241 | 1.162 | 1.163 | 1.107 | 1.055 | 1.007 | 0.937 | 1.131 | 10.10 | 9.5090 |
| 65 | Di-n-butylphthalate | 1.693 | 1.727 | 1.711 | 1.577 | 1.598 | 1.538 | 1.437 | 1.353 | 1.306 | 1.549 | 10.00 | 10.0782 |
| 66 | Fluoranthene | 1.319 | 1.372 | 1.378 | 1.263 | 1.302 | 1.205 | 1.144 | 1.119 | 1.070 | 1.241 | 9.05 | 10.8622 |
| 67 | Chrysene-d12 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 12.6429 |
| 68 | Ben-zidine | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | 0.000 | 0.000 | 0.000 | 0.0000 |
| 69 | Pyrene | 1.557 | 1.573 | 1.515 | 1.466 | 1.490 | 1.518 | 1.585 | 1.541 | 1.524 | 1.530 | 2.51 | 11.1727 |
| 70 | Terphenyl-d14 | 0.878 | 0.878 | 0.845 | 0.841 | 0.849 | 0.831 | 0.862 | 0.885 | 0.874 | 0.860 | 2.25 | 11.4134 |
| 71 | Butylbenzylphthalate | 0.677 | 0.706 | 0.706 | 0.690 | 0.707 | 0.749 | 0.730 | 0.779 | 0.747 | 0.721 | 4.50 | 12.0231 |
| 72 | 3,3'-Dichlorobenzidine | 0.368 | 0.402 | 0.421 | 0.430 | 0.443 | 0.449 | 0.458 | 0.439 | 0.488 | 0.433 | 7.88 | 12.6396 |
| 73 | Benzo(a)anthracene | 1.113 | 1.072 | 1.083 | 1.017 | 1.048 | 1.047 | 1.092 | 1.102 | 1.128 | 1.078 | 3.29 | 12.6238 |
| 74 | Chrysene | 1.195 | 1.151 | 1.158 | 1.118 | 1.137 | 1.169 | 1.196 | 1.120 | 1.144 | 1.154 | 2.48 | 12.6812 |
| 75 | bis(2-Ethylhexyl)phthalate | 0.926 | 0.942 | 0.971 | 0.936 | 0.964 | 0.981 | 0.966 | 0.976 | 0.943 | 0.956 | 2.04 | 12.7622 |
| 76 | Perylene-d12 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 14.3876 |
| 77 | Di-n-octylphthalate | 1.725 | 1.923 | 2.103 | 2.064 | 2.059 | 2.043 | 2.133 | 2.027 | 2.061 | 2.015 | 6.11 | 13.5856 |
| 78 | Benzo(b)fluoranthene | 1.384 | 1.476 | 1.467 | 1.276 | 1.448 | 1.338 | 1.378 | 1.480 | 1.792 | 1.449 | 10.09 | 13.9489 |
| 79 | Benzo(k)fluoranthene | 1.416 | 1.453 | 1.341 | 1.414 | 1.242 | 1.313 | 1.292 | 1.199 | ----- | 1.334 | 6.72 | 13.9816 |
| 80 | Benzo(a)pyrene | 1.327 | 1.363 | 1.373 | 1.264 | 1.291 | 1.243 | 1.212 | 1.224 | 1.221 | 1.281 | 4.75 | 14.3235 |
| 81 | Indeno(1,2,3-cd)pyrene | 1.168 | 1.252 | 1.310 | 1.272 | | | | | | | | |

FIGURE 6 – TYPICAL ICV SUMMARY

CONTINUE_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :TO42
IC Beginning DateTime :03/07/06 14:54
Spike Amount :50 PPM
CC/CV File :RCX045
IC File :RCX040

Column Spec :ZB-5MS ID :0.18MM
IC Ending DateTime :03/07/06 18:17
HPChem Method :SV42C07
Date_Time :03/07/06 18:59

| M_IDX | Parameters | CC_Con | CC%_D | CC_Resp | CCRRF | AvRRF | CC_Rtm | AvRtm | %_RSD | Co_X0 | Co_X1 | Co_X2 | Co_Cor |
|-------|-----------------------------|--------|-------|---------|-------|-------|--------|--------|-------|---------|--------|-------|--------|
| 1 | 1,4-Dichlorobenzene-d4 | 40.000 | 0 | 235240 | 1 | 1 | 3.403 | 3.408 | 0 | | | | |
| 2 | N-Nitrosodimethylamine | 48.185 | -3.6 | 247557 | 0.842 | 0.874 | 1.611 | 1.611 | 1.48 | | | | |
| 3 | Pyridine | 49.058 | -1.9 | 420697 | 1.431 | 1.458 | 1.611 | 1.623 | 3.26 | | | | |
| 4 | 2-Fluorophenol | | | | | | | | | | | | |
| 5 | Phenol | 49.198 | -1.6 | 518555 | 1.763 | 1.792 | 3.079 | 3.088 | 6.96 | | | | |
| 6 | Aniline | 50.776 | 1.6 | 584924 | 1.989 | 1.959 | 3.109 | 3.121 | 2.32 | | | | |
| 7 | Bis(2-chloroethyl)ether | 50.693 | 1.4 | 436767 | 1.485 | 1.465 | 3.170 | 3.175 | 11.08 | | | | |
| 8 | Phenol-d5 | | | | | | | | | | | | |
| 9 | 2-Chlorophenol | 49.585 | -0.8 | 394265 | 1.341 | 1.352 | 3.210 | 3.214 | 6.76 | | | | |
| 10 | 1,3-Dichlorobenzene | 47.185 | -5.6 | 402181 | 1.368 | 1.449 | 3.342 | 3.352 | 5.72 | | | | |
| 11 | 1,4-Dichlorobenzene | 48.152 | -3.7 | 408835 | 1.390 | 1.444 | 3.423 | 3.425 | 6.95 | | | | |
| 12 | Benzyl alcohol | 53.323 | 6.6 | 261927 | 0.891 | 0.835 | 3.555 | 3.564 | 7.82 | | | | |
| 13 | 1,2-Dichlorobenzene-d4 | | | | | | | | | | | | |
| 14 | 1,2-Dichlorobenzene | 47.880 | -4.2 | 373160 | 1.269 | 1.325 | 3.565 | 3.575 | 12.99 | | | | |
| 15 | 2-Methylphenol | 49.147 | -1.7 | 325282 | 1.106 | 1.125 | 3.666 | 3.672 | 5.64 | | | | |
| 16 | Bis(2-chloroisopropyl)ether | 46.639 | -6.7 | 809840 | 2.754 | 2.953 | 3.686 | 3.699 | 6.62 | | | | |
| 17 | 4-Methylphenol | 44.538 | -10.9 | 431039 | 1.466 | 1.646 | 3.838 | 3.850 | 3.03 | | | | |
| 18 | N-Nitroso-di-n-propylamine | 47.307 | -5.4 | 328305 | 1.116 | 1.180 | 3.848 | 3.859 | 4.46 | | | | |
| 19 | Hexachloroethane | 49.652 | -0.7 | 190305 | 0.647 | 0.652 | 3.919 | 3.918 | 5.22 | | | | |
| 20 | Naphthalene-d8 | 40.000 | 0 | 872956 | 1 | 1 | 4.810 | 4.817 | 0 | | | | |
| 21 | Nitrobenzene-d5 | | | | | | | | | | | | |
| 22 | Nitrobenzene | 48.596 | -2.8 | 434545 | 0.398 | 0.410 | 4.010 | 4.017 | 5.16 | | | | |
| 23 | Isophorone | 46.611 | -6.8 | 807359 | 0.740 | 0.794 | 4.294 | 4.303 | 2.31 | | | | |
| 24 | 2-Nitrophenol | 49.981 | -0.0 | 248400 | 0.228 | 0.228 | 4.365 | 4.370 | 5.24 | | | | |
| 25 | 2,4-Dimethylphenol | 47.533 | -4.9 | 340052 | 0.312 | 0.328 | 4.435 | 4.447 | 3.73 | | | | |
| 26 | bis(2-Chloroethoxy)methane | 49.152 | -1.7 | 535936 | 0.491 | 0.500 | 4.557 | 4.565 | 6.12 | | | | |
| 27 | Benzoic Acid | 47.056 | -5.9 | 246465 | 0.226 | 0.213 | 4.668 | 4.674 | 30.00 | | | | |
| 28 | 2,4-Dichlorophenol | 48.357 | -3.3 | 345747 | 0.317 | 0.328 | 4.658 | 4.663 | 5.03 | -0.0255 | 0.2617 | | 0.9997 |
| 29 | 1,2,4-Trichlorobenzene | 48.672 | -2.7 | 361738 | 0.332 | 0.341 | 4.749 | 4.753 | 9.81 | | | | |
| 30 | Naphthalene | 47.077 | -5.8 | 996018 | 0.913 | 0.969 | 4.840 | 4.845 | 13.44 | | | | |
| 31 | 4-Chloroaniline | 50.772 | 1.5 | 520887 | 0.477 | 0.470 | 4.931 | 4.936 | 7.06 | | | | |
| 32 | Hexachlorobutadiene | 48.806 | -2.4 | 213149 | 0.195 | 0.200 | 5.002 | 5.003 | 10.66 | | | | |
| 33 | 4-Chloro-3-methylphenol | 48.049 | -3.9 | 402562 | 0.369 | 0.384 | 5.580 | 5.584 | 4.98 | | | | |
| 34 | 2-Methylnaphthalene | 49.645 | -0.7 | 743443 | 0.681 | 0.686 | 5.731 | 5.736 | 12.97 | | | | |
| 35 | Acenaphthene-d10 | 40.000 | 0 | 509910 | 1 | 1 | 7.078 | 7.081 | 0 | | | | |
| 36 | Hexachlorocyclopentadiene | 48.255 | -3.5 | 146704 | 0.230 | 0.215 | 5.934 | 5.934 | 26.09 | -0.0215 | 0.2563 | | 0.9981 |
| 37 | 2,4,6-Trichlorophenol | 46.951 | -6.1 | 256100 | 0.402 | 0.428 | 6.106 | 6.111 | 4.47 | | | | |
| 38 | 2,4,5-Trichlorophenol | 49.184 | -1.6 | 294881 | 0.463 | 0.470 | 6.146 | 6.157 | 8.14 | | | | |
| 39 | 2-Fluorobiphenyl | | | | | | | | | | | | |
| 40 | 2-Chloronaphthalene | 48.149 | -3.7 | 686609 | 1.077 | 1.119 | 6.349 | 6.357 | 10.52 | | | | |
| 41 | 2-Nitroaniline | 50.091 | 0.2 | 331777 | 0.521 | 0.520 | 6.521 | 6.526 | 9.25 | | | | |
| 42 | Dimethylphthalate | 47.131 | -5.7 | 889438 | 1.395 | 1.480 | 6.794 | 6.799 | 5.46 | | | | |
| 43 | 2,6-Dinitrotoluene | 52.565 | 5.1 | 234481 | 0.368 | 0.350 | 6.865 | 6.873 | 11.48 | | | | |
| 44 | Acenaphthylene | 45.016 | -10.0 | 995646 | 1.562 | 1.735 | 6.885 | 6.891 | 11.39 | | | | |
| 45 | 3-Nitroaniline | 48.448 | -3.1 | 251295 | 0.394 | 0.407 | 7.078 | 7.088 | 7.53 | | | | |
| 46 | Acenaphthene | 46.486 | -7.0 | 647204 | 1.015 | 1.092 | 7.128 | 7.132 | 13.06 | | | | |
| 47 | 2,4-Dinitrophenol | 45.007 | -10.0 | 153233 | 0.240 | 0.222 | 7.230 | 7.233 | 44.65 | -0.0437 | 0.3060 | | 0.9974 |
| 48 | 4-Nitrophenol | 49.018 | -2.0 | 112248 | 0.176 | 0.169 | 7.361 | 7.368 | 19.05 | -0.0100 | 0.1878 | | 0.9989 |
| 49 | Dibenzofuran | 49.270 | -1.5 | 1056252 | 1.657 | 1.682 | 7.371 | 7.372 | 13.40 | | | | |
| 50 | 2,4-Dinitrotoluene | 45.921 | -8.2 | 307435 | 0.482 | 0.525 | 7.402 | 7.412 | 9.94 | | | | |
| 51 | Diethylphthalate | 46.068 | -7.9 | 888414 | 1.394 | 1.513 | 7.766 | 7.777 | 8.29 | | | | |
| 52 | Fluorene | 46.689 | -6.6 | 804939 | 1.263 | 1.352 | 7.837 | 7.840 | 12.13 | | | | |
| 53 | 4-Chlorophenyl-phenylether | 46.362 | -7.3 | 419393 | 0.658 | 0.710 | 7.867 | 7.872 | 12.06 | | | | |
| 54 | 4-Nitroaniline | 51.845 | 3.7 | 300225 | 0.471 | 0.454 | 7.938 | 7.952 | 8.28 | | | | |
| 55 | 2,4,6-Tribromophenol | | | | | | | | | | | | |
| 56 | Phenanthrene-d10 | 40.000 | 0 | 877096 | 1 | 1 | 9.143 | 9.138 | 0 | | | | |
| 57 | 4,6-Dinitro-2-methylphenol | 49.728 | -0.5 | 221107 | 0.202 | 0.185 | 7.969 | 7.976 | 24.57 | -0.0158 | 0.2154 | | 0.9985 |
| 58 | N-Nitrosodiphenylamine | 46.882 | -6.2 | 574886 | 0.524 | 0.559 | 8.050 | 8.058 | 11.64 | | | | |
| 59 | Azobenzene | 50.524 | 1.0 | 1155886 | 1.054 | 1.043 | 8.090 | 8.092 | 11.15 | | | | |
| 60 | 4-Bromophenyl-phenylether | 47.648 | -4.7 | 271317 | 0.247 | 0.260 | 8.546 | 8.550 | 8.91 | | | | |
| 61 | Hexachlorobenzene | 45.576 | -8.8 | 312629 | 0.285 | 0.313 | 8.586 | 8.591 | 8.48 | | | | |
| 62 | Pentachlorophenol | 46.010 | -8.0 | 198882 | 0.181 | 0.185 | 8.890 | 8.888 | 16.58 | -0.0114 | 0.2070 | | 0.9996 |
| 63 | Phenanthrene | 48.504 | -3.0 | 1215057 | 1.108 | 1.142 | 9.174 | 9.178 | 13.78 | | | | |
| 64 | Anthracene | 48.614 | -2.8 | 1229772 | 1.122 | 1.154 | 9.244 | 9.251 | 13.52 | | | | |
| 65 | Carbazole | 49.293 | -1.4 | 1222827 | 1.115 | 1.131 | 9.508 | 9.509 | 10.10 | | | | |
| 66 | Di-n-butylphthalate | 48.727 | -2.5 | 1654817 | 1.509 | 1.549 | 10.075 | 10.078 | 10.00 | | | | |
| 67 | Fluoranthene | 48.542 | -2.9 | 1321404 | 1.205 | 1.241 | 10.864 | 10.862 | 9.05 | | | | |
| 68 | Chrysene-d12 | 40.000 | 0 | 757495 | 1 | 1 | 12.646 | 12.643 | 0 | | | | |
| 69 | Benzidine | | | | | | | | | | | | |
| 70 | Pyrene | 45.216 | -9.6 | 1309956 | 1.383 | 1.530 | 11.168 | 11.173 | 2.51 | | | | |
| 71 | Terphenyl-d14 | | | | | | | | | | | | |
| 72 | Butylbenzylphthalate | 45.797 | -8.4 | 625494 | 0.661 | 0.721 | 12.028 | 12.023 | 4.50 | | | | |
| 73 | 3,3'-Dichlorobenzidine | 45.713 | -8.6 | 374967 | 0.396 | 0.433 | 12.636 | 12.640 | 7.88 | | | | |
| 74 | Benzo(a)anthracene | 44.848 | -10.3 | 915594 | 0.967 | 1.078 | 12.626 | 12.624 | 3.29 | | | | |
| 75 | Chrysene | 42.662 | -14.7 | 932542 | 0.985 | 1.154 | 12.677 | 12.681 | 2.48 | | | | |
| 76 | bis(2-Ethylhexyl)phthalate | 47.051 | -5.9 | 852038 | 0.900 | 0.956 | 12.768 | 12.762 | 2.04 | | | | |
| 77 | Perylene-d12 | 40.000 | 0 | 543525 | 1 | 1 | 14.387 | 14.388 | 0 | | | | |
| 78 | Di-n-octylphthalate | 51.864 | 3.7 | 1420290 | 2.090 | 2.015 | 13.588 | 13.586 | 6.11 | | | | |
| 79 | Benzo(b)fluoranthene | 46.122 | -7.8 | 907925 | 1.336 | 1.449 | 13.942 | 13.949 | 10.09 | | | | |
| 80 | Benzo(k)fluoranthene | 48.861 | -2.3 | 885617 | 1.304 | 1.334 | 13.983 | 13.982 | 6.72 | | | | |
| 81 | Benzo(a)pyrene | 48.979 | -2.0 | 852408 | 1.255 | 1.281 | 14.327 | 14.323 | 4.75 | | | | |
| 82 | Indeno(1,2,3-cd)pyrene | 47.412 | -5.2 | 794216 | 1.169 | 1.233 | 15.592 | 15.591 | 5.45 | | | | |
| 83 | Dibenzo(a,h)anthracene | 48.411 | -3.2 | 665602 | 0.980 | 1.012 | 15.623 | 15.624 | 6.13 | | | | |
| 84 | Benzo(g,h,i)perylene | 45.426 | -9.1 | 613493 | 0.903 | 0.994 | 15.916 | 15.915 | 9.16 | | | | |

FIGURE 7- TYPICAL SAMPLE RESULT SUMMARY

SW 3550B/8270C
 SEMI VOLATILE ORGANICS BY GC/MS

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Client      XYZ, Inc.                Date Collected: 02/21/06
Project    : CLEAN LAND PROJECT      Date Received: 02/22/06
Batch No.  : 06B164                Date Extracted: 02/24/06 15:15
Sample ID: BOR60177MS           Date Analyzed: 02/27/06 16:12
Lab Samp ID: B164-01M          Dilution Factor: 1
Lab File ID: RBH347            Matrix      : SOIL
Ext Btch ID: SVB043S           % Moisture  : 12.8
Calib. Ref.: RAH031            Instrument ID : T-041
=====
  
```

| PARAMETERS | RESULTS (ug/kg) | RL (ug/kg) | MDL (ug/kg) |
|-----------------------------|--------------------|---------------|----------------|
| ----- | ----- | ----- | ----- |
| 1,2,4-TRICHLOROBENZENE | 2030 | 378 | 192 |
| 1,2-DICHLOROBENZENE | 1840 | 378 | 192 |
| 1,3-DICHLOROBENZENE | 1820 | 378 | 192 |
| 1,4-DICHLOROBENZENE | 1810 | 378 | 192 |
| 2,4,5-TRICHLOROPHENOL | 2200 | 378 | 192 |
| 2,4,6-TRICHLOROPHENOL | 1930 | 722 | 192 |
| 2,4-DICHLOROPHENOL | 2040 | 378 | 192 |
| 2,4-DIMETHYLPHENOL | 1780 | 378 | 192 |
| 2,4-DINITROPHENOL | 1990 | 757 | 192 |
| 2,4-DINITROTOLUENE | 2650 | 378 | 192 |
| 2,6-DINITROTOLUENE | 2320 | 378 | 192 |
| 2-CHLORONAPHTHALENE | 1960 | 378 | 192 |
| 2-CHLOROPHENOL | 1920 | 378 | 192 |
| 2-METHYLNAPHTHALENE | 1900 | 378 | 192 |
| 2-METHYLPHENOL | 1830 | 378 | 192 |
| 2-NITROANILINE | 1960 | 757 | 192 |
| 2-NITROPHENOL | 1990 | 378 | 192 |
| 3,3'-DICHLOROBENZIDINE | 1250 | 757 | 192 |
| 3-NITROANILINE | 2260 | 757 | 192 |
| 4,6-DINITRO-2-METHYLPHENOL | 2630 | 757 | 192 |
| 4-BROMOPHENYL-PHENYL ETHER | 2510 | 378 | 192 |
| 4-CHLORO-3-METHYLPHENOL | 1980 | 378 | 192 |
| 4-CHLOROANILINE | 1820 | 378 | 192 |
| 4-CHLOROPHENYL-PHENYL ETHER | 2190 | 378 | 192 |
| 4-METHYLPHENOL (1) | 1720 | 378 | 192 |
| 4-NITROANILINE | 2320 | 378 | 192 |
| 4-NITROPHENOL | 1950 | 757 | 192 |
| ACENAPHTHENE | 1970 | 378 | 192 |
| ACENAPHTHYLENE | 2050 | 378 | 192 |
| ANTHRACENE | 2570 | 378 | 192 |
| BENZO(A)ANTHRACENE | 2580 | 378 | 192 |
| BENZO(A)PYRENE | 2160 | 378 | 192 |
| BENZO(B)FLUORANTHENE | 2190 | 378 | 192 |
| BENZO(K)FLUORANTHENE | 2260 | 378 | 192 |
| BENZO(G,H,I)PERYLENE | 2600 | 378 | 192 |
| BIS(2-CHLOROETHOXY)METHANE | 1900 | 378 | 192 |
| BIS(2-CHLOROETHYL)ETHER | 1870 | 378 | 192 |
| BIS(2-CHLOROISOPROPYL)ETHER | 1700 | 378 | 192 |

FIGURE 7- TYPICAL SAMPLE RESULT SUMMARY

| | | | |
|------------------------------|------|-----|-----|
| BIS (2-ETHYLHEXYL) PHTHALATE | 2550 | 378 | 192 |
| BUTYLBENZYLPHthalate | 2380 | 378 | 192 |
| CHRYSENE | 2220 | 378 | 192 |
| DI-N-BUTYLPHthalate | 2780 | 378 | 192 |
| DI-N-OCTYLPHthalate | 2390 | 378 | 192 |
| DIBENZO (A, H) ANTHRACENE | 2650 | 378 | 192 |
| DIBENZOFURAN | 1980 | 378 | 192 |
| DIETHYLPHthalate | 2540 | 378 | 192 |
| DIMETHYLPHthalate | 2330 | 378 | 192 |
| FLUORANTHENE | 2860 | 378 | 192 |
| FLUORENE | 2180 | 378 | 192 |
| HEXACHLOROBENZENE | 2630 | 378 | 192 |
| HEXACHLOROBUTADIENE | 2060 | 378 | 192 |
| HEXACHLOROCYCLOPENTADIENE | 1690 | 378 | 192 |
| HEXACHLOROETHANE | 1760 | 378 | 192 |
| INDENO (1, 2, 3-CD) PYRENE | 2600 | 378 | 192 |
| ISOPHORONE | 1990 | 378 | 192 |
| N-NITROSO-DI-N-PROPYLAMINE | 1840 | 378 | 192 |
| N-NITROSODIPHENYLAMINE (2) | 1730 | 378 | 192 |
| NAPHTHALENE | 1920 | 378 | 192 |
| NITROBENZENE | 1930 | 378 | 192 |
| PENTACHLOROPHENOL | 2200 | 757 | 192 |
| PHENANTHRENE | 2520 | 378 | 192 |
| PHENOL | 1780 | 378 | 192 |
| PYRENE | 2260 | 378 | 192 |

| SURROGATE PARAMETERS | % RECOVERY | QC LIMIT |
|------------------------|------------|----------|
| ----- | ----- | ----- |
| 2, 4, 6-TRIBROMOPHENOL | 79 | 50-155 |
| 2-FLUOROBIPHENYL | 67 | 32-114 |
| 2-FLUOROPHENOL | 64 | 30-105 |
| NITROBENZENE-D5 | 66 | 33-114 |
| PHENOL-D5 | 64 | 30-110 |
| TERPHENYL-D14 | 88 | 31-143 |

RL: Reporting Limit

(1): Cannot be separated from 3-Methylphenol

(2): Cannot be separated from Diphenylamine

FIGURE 8 – TYPICAL LCS REPORT SUMMARY

3

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN LAND PROJECT
BATCH NO.: 06B164
METHOD: SW 3550B/8270C

=====

MATRIX: SOIL % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: MBLK1S
LAB SAMP ID: SVB043SB SVB043SL SVB043SC
LAB FILE ID: RBH342 RBH343 RBH344
DATE EXTRACTED: 02/24/0615:15 02/24/0615:15 02/24/0615:15 DATE COLLECTED: NA
DATE ANALYZED: 02/27/0614:07 02/27/0614:32 02/27/0614:57 DATE RECEIVED: 02/24/06
PREP. BATCH: SVB043S SVB043S SVB043S
CALIB. REF: RAH031 RAH031 RAH031

ACCESSION:

| PARAMETER | BLNK RSLT (ug/kg) | SPIKE AMT (ug/kg) | BS RSLT (ug/kg) | BS % REC | SPIKE AMT (ug/kg) | BSD RSLT (ug/kg) | BSD % REC | RPD (%) | QC LIMIT (%) | MAX (%) |
|-----------------------------|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|--------------|-------------------|--------------|
| 1,2,4-Trichlorobenzene | ND | 2670 | 2040 | 76 | 2670 | 2030 | 76 | 0 | 25-120 | 50 |
| 1,2-Dichlorobenzene | ND | 2670 | 2020 | 76 | 2670 | 2030 | 76 | 0 | 25-115 | 50 |
| 1,3-Dichlorobenzene | ND | 2670 | 2010 | 76 | 2670 | 2010 | 75 | 0 | 26-106 | 50 |
| 1,4-Dichlorobenzene | ND | 2670 | 1980 | 74 | 2670 | 2060 | 77 | 4 | 24-123 | 50 |
| 2,4,5-Trichlorophenol | ND | 2670 | 2210 | 83 | 2670 | 2230 | 84 | 1 | 34-128 | 50 |
| 2,4,6-Trichlorophenol | ND | 2670 | 1950 | 73 | 2670 | 2050 | 77 | 5 | 35-120 | 50 |
| 2,4-Dichlorophenol | ND | 2670 | 2080 | 78 | 2670 | 2140 | 80 | 3 | 31-121 | 50 |
| 2,4-Dimethylphenol | ND | 2670 | 2070 | 77 | 2670 | 2010 | 75 | 3 | 27-119 | 50 |
| 2,4-Dinitrophenol | ND | 2670 | 1840 | 69 | 2670 | 1980 | 74 | 7 | 20-165 | 50 |
| 2,4-Dinitrotoluene | ND | 2670 | 2290 | 86 | 2670 | 2410 | 90 | 5 | 34-154 | 50 |
| 2,6-Dinitrotoluene | ND | 2670 | 2200 | 83 | 2670 | 2290 | 86 | 4 | 32-133 | 50 |
| 2-Chloronaphthalene | ND | 2670 | 2030 | 76 | 2670 | 2040 | 76 | 0 | 32-110 | 50 |
| 2-Chlorophenol | ND | 2670 | 2050 | 77 | 2670 | 2030 | 76 | 1 | 24-115 | 50 |
| 2-Methylnaphthalene | ND | 2670 | 1890 | 71 | 2670 | 1980 | 74 | 5 | 28-120 | 50 |
| 2-Methylphenol | ND | 2670 | 1890 | 71 | 2670 | 1920 | 72 | 2 | 26-123 | 50 |
| 2-Nitroaniline | ND | 2670 | 1910 | 71 | 2670 | 1970 | 74 | 3 | 29-148 | 50 |
| 2-Nitrophenol | ND | 2670 | 2100 | 79 | 2670 | 2090 | 78 | 1 | 27-126 | 50 |
| 3,3'-Dichlorobenzidine | ND | 2670 | 2300 | 86 | 2670 | 2360 | 88 | 3 | 34-137 | 50 |
| 3-Nitroaniline | ND | 2670 | 2170 | 81 | 2670 | 2170 | 81 | 0 | 30-135 | 50 |
| 4,6-Dinitro-2-Methylphenol | ND | 2670 | 2330 | 87 | 2670 | 2420 | 91 | 4 | 20-165 | 50 |
| 4-Bromophenyl-phenyl ether | ND | 2670 | 2260 | 85 | 2670 | 2230 | 84 | 1 | 34-120 | 50 |
| 4-Chloro-3-Methylphenol | ND | 2670 | 1940 | 73 | 2670 | 2080 | 78 | 7 | 28-125 | 50 |
| 4-Chloroaniline | ND | 2670 | 1900 | 71 | 2670 | 1950 | 73 | 2 | 26-121 | 50 |
| 4-Chlorophenyl-phenyl ether | ND | 2670 | 2060 | 77 | 2670 | 2040 | 76 | 1 | 34-118 | 50 |
| 4-Methylphenol | ND | 2670 | 1760 | 66 | 2670 | 1850 | 69 | 5 | 29-127 | 50 |
| 4-Nitroaniline | ND | 2670 | 2110 | 79 | 2670 | 2170 | 81 | 3 | 25-148 | 50 |
| 4-Nitrophenol | ND | 2670 | 1640 | 61 | 2670 | 1690 | 63 | 3 | 20-147 | 50 |
| Acenaphthene | ND | 2670 | 1960 | 74 | 2670 | 1960 | 74 | 0 | 29-135 | 50 |
| Acenaphthylene | ND | 2670 | 2110 | 79 | 2670 | 2110 | 79 | 0 | 33-120 | 50 |
| Anthracene | ND | 2670 | 2230 | 84 | 2670 | 2300 | 86 | 3 | 35-122 | 50 |
| Benzo(a)anthracene | ND | 2670 | 2180 | 82 | 2670 | 2200 | 83 | 1 | 33-139 | 50 |

FIGURE 8 – TYPICAL LCS REPORT SUMMARY

| | | | | | | | | | | |
|-----------------------------|----|------|------|----|------|------|----|----|--------|----|
| Benzo(a)pyrene | ND | 2670 | 1940 | 73 | 2670 | 2020 | 76 | 4 | 0-144 | 50 |
| Benzo(b)fluoranthene | ND | 2670 | 1860 | 70 | 2670 | 1970 | 74 | 5 | 24-140 | 50 |
| Benzo(k)fluoranthene | ND | 2670 | 1840 | 69 | 2670 | 1930 | 72 | 5 | 29-150 | 50 |
| Benzo(g,h,i)perylene | ND | 2670 | 2370 | 89 | 2670 | 2460 | 92 | 4 | 20-146 | 50 |
| bis(2-Chloroethoxy)methane | ND | 2670 | 1880 | 70 | 2670 | 1910 | 72 | 2 | 24-126 | 50 |
| bis(2-Chloroethyl)ether | ND | 2670 | 1840 | 69 | 2670 | 1880 | 71 | 2 | 22-121 | 50 |
| bis(2-Chloroisopropyl)ether | ND | 2670 | 1810 | 68 | 2670 | 1830 | 69 | 1 | 20-131 | 50 |
| bis(2-Ethylhexyl)phthalate | ND | 2670 | 2210 | 83 | 2670 | 2210 | 83 | 0 | 27-149 | 50 |
| Butylbenzylphthalate | ND | 2670 | 2120 | 79 | 2670 | 2100 | 79 | 1 | 26-153 | 50 |
| Chrysene | ND | 2670 | 2090 | 78 | 2670 | 2050 | 77 | 2 | 33-142 | 50 |
| Di-n-butylphthalate | ND | 2670 | 2260 | 85 | 2670 | 2430 | 91 | 7 | 36-135 | 50 |
| Di-n-octylphthalate | ND | 2670 | 2070 | 78 | 2670 | 2060 | 77 | 0 | 22-165 | 50 |
| Dibenzo(a,h)anthracene | ND | 2670 | 2390 | 90 | 2670 | 2490 | 94 | 4 | 30-148 | 50 |
| Dibenzofuran | ND | 2670 | 1880 | 71 | 2670 | 1930 | 72 | 2 | 34-120 | 50 |
| Diethylphthalate | ND | 2670 | 2190 | 82 | 2670 | 2210 | 83 | 1 | 32-126 | 50 |
| Dimethylphthalate | ND | 2670 | 2120 | 80 | 2670 | 2090 | 78 | 1 | 33-124 | 50 |
| Fluoranthene | ND | 2670 | 2450 | 92 | 2670 | 2500 | 94 | 2 | 35-122 | 50 |
| Fluorene | ND | 2670 | 2100 | 79 | 2670 | 2040 | 76 | 3 | 32-127 | 50 |
| Hexachlorobenzene | ND | 2670 | 2240 | 84 | 2670 | 2320 | 87 | 3 | 32-127 | 50 |
| Hexachlorobutadiene | ND | 2670 | 2100 | 79 | 2670 | 2080 | 78 | 1 | 27-118 | 50 |
| Hexachlorocyclopentadiene | ND | 2670 | 2000 | 75 | 2670 | 2040 | 76 | 2 | 20-123 | 50 |
| Hexachloroethane | ND | 2670 | 1990 | 75 | 2670 | 2020 | 76 | 1 | 23-107 | 50 |
| Indeno(1,2,3-cd)pyrene | ND | 2670 | 2330 | 87 | 2670 | 2440 | 91 | 5 | 28-147 | 50 |
| Isophorone | ND | 2670 | 1980 | 74 | 2670 | 2080 | 78 | 5 | 34-115 | 50 |
| n-Nitroso-di-n-propylamine | ND | 2670 | 1830 | 69 | 2670 | 2030 | 76 | 11 | 26-147 | 50 |
| n-Nitrosodiphenylamine | ND | 2670 | 1520 | 57 | 2670 | 1510 | 56 | 1 | 35-134 | 50 |
| Naphthalene | ND | 2670 | 1980 | 74 | 2670 | 2000 | 75 | 1 | 27-113 | 50 |
| Nitrobenzene | ND | 2670 | 1930 | 72 | 2670 | 1960 | 73 | 2 | 28-112 | 50 |
| Pentachlorophenol | ND | 2670 | 1810 | 68 | 2670 | 1950 | 73 | 7 | 20-163 | 50 |
| Phenanthrene | ND | 2670 | 2170 | 81 | 2670 | 2250 | 85 | 4 | 35-119 | 50 |
| Phenol | ND | 2670 | 1870 | 70 | 2670 | 1920 | 72 | 3 | 20-120 | 50 |
| Pyrene | ND | 2670 | 1990 | 75 | 2670 | 2040 | 76 | 2 | 32-147 | 50 |

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| SURROGATE PARAMETER | SPIKE AMT (ug/kg) | BS RSLT (ug/kg) | BS % REC | SPIKE AMT (ug/kg) | BSD RSLT (ug/kg) | BSD % REC | QC LIMIT (%) |
|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|-------------------|
| 2,4,6-Tribromophenol | 5000 | 3860 | 77 | 5000 | 3950 | 79 | 50-155 |
| 2-Fluorobiphenyl | 3330 | 2510 | 75 | 3330 | 2640 | 79 | 32-114 |
| 2-Fluorophenol | 5000 | 3570 | 71 | 5000 | 3850 | 77 | 30-105 |
| Nitrobenzene-d5 | 3330 | 2450 | 74 | 3330 | 2590 | 78 | 33-114 |
| Phenol-d5 | 5000 | 3480 | 70 | 5000 | 3690 | 74 | 30-110 |
| Terphenyl-d14 | 3330 | 2870 | 86 | 3330 | 2950 | 89 | 31-143 |

FIGURE 9 – TYPICAL MS/MSD REPORT SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN LAND PROJECT
BATCH NO.: 06B164
METHOD: SW 3550B/8270C

MATRIX: SOIL % MOISTURE: 12.8
DILUTION FACTOR: 1 1 1
SAMPLE ID: BOR60177
LAB SAMP ID: B164-01 B164-01M B164-01S
LAB FILE ID: RBH346 RBH347 RBH348
DATE EXTRACTED: 02/24/0615: 15 02/24/0615: 15 02/24/0615: 15 DATE COLLECTED: 02/21/06
DATE ANALYZED: 02/27/0615: 47 02/27/0616: 12 02/27/0616: 37 DATE RECEIVED: 02/22/06
PREP. BATCH: SVB043S SVB043S SVB043S
CALIB. REF: RAH031 RAH031 RAH031

ACCESSION:

| PARAMETER | SMPL RSLT (ug/kg) | SPIKE AMT (ug/kg) | MS RSLT (ug/kg) | MS % REC | SPIKE AMT (ug/kg) | MSD RSLT (ug/kg) | MSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|----------------------------------|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|------------|-----------------|----------------|
| 1, 2, 4-Tri chl orobenzene | ND | 3060 | 2030 | 66 | 3060 | 1940 | 63 | 5 | 25-120 | 50 |
| 1, 2-Di chl orobenzene | ND | 3060 | 1840 | 60 | 3060 | 1770 | 58 | 3 | 25-115 | 50 |
| 1, 3-Di chl orobenzene | ND | 3060 | 1820 | 59 | 3060 | 1680 | 55 | 8 | 26-106 | 50 |
| 1, 4-Di chl orobenzene | ND | 3060 | 1810 | 59 | 3060 | 1690 | 55 | 7 | 24-123 | 50 |
| 2, 4, 5-Tri chl orophenol | ND | 3060 | 2200 | 72 | 3060 | 2300 | 75 | 5 | 34-128 | 50 |
| 2, 4, 6-Tri chl orophenol | ND | 3060 | 1930 | 63 | 3060 | 2040 | 67 | 6 | 35-120 | 50 |
| 2, 4-Di chl orophenol | ND | 3060 | 2040 | 67 | 3060 | 2140 | 70 | 5 | 31-121 | 50 |
| 2, 4-Di methyl phenol | ND | 3060 | 1780 | 58 | 3060 | 1780 | 58 | 0 | 27-119 | 50 |
| 2, 4-Di ni trophenol | ND | 3060 | 1990 | 65 | 3060 | 2200 | 72 | 10 | 20-165 | 50 |
| 2, 4-Di ni trotol uene | ND | 3060 | 2650 | 87 | 3060 | 2680 | 88 | 1 | 34-154 | 50 |
| 2, 6-Di ni trotol uene | ND | 3060 | 2320 | 76 | 3060 | 2550 | 84 | 10 | 32-133 | 50 |
| 2-Chl oronaphthal ene | ND | 3060 | 1960 | 64 | 3060 | 2060 | 67 | 5 | 32-110 | 50 |
| 2-Chl orophenol | ND | 3060 | 1920 | 63 | 3060 | 1820 | 60 | 5 | 24-115 | 50 |
| 2-Methyl naphthal ene | ND | 3060 | 1900 | 62 | 3060 | 1920 | 63 | 1 | 28-120 | 50 |
| 2-Methyl phenol | ND | 3060 | 1830 | 60 | 3060 | 1860 | 61 | 1 | 26-123 | 50 |
| 2-Ni troani li ne | ND | 3060 | 1960 | 64 | 3060 | 2130 | 70 | 8 | 29-148 | 50 |
| 2-Ni trophenol | ND | 3060 | 1990 | 65 | 3060 | 1970 | 64 | 1 | 27-126 | 50 |
| 3, 3' -Di chl orobenzi di ne | ND | 3060 | 1250 | 41 | 3060 | 1240 | 41 | 1 | 34-137 | 50 |
| 3-Ni troani li ne | ND | 3060 | 2260 | 74 | 3060 | 2330 | 76 | 3 | 30-135 | 50 |
| 4, 6-Di ni tro-2-Methyl phenol | ND | 3060 | 2630 | 86 | 3060 | 2610 | 85 | 0 | 20-165 | 50 |
| 4-Bromophenyl -phenyl ether | ND | 3060 | 2510 | 82 | 3060 | 2480 | 81 | 1 | 34-120 | 50 |
| 4-Chl oro-3-Methyl phenol | ND | 3060 | 1980 | 65 | 3060 | 2110 | 69 | 7 | 28-125 | 50 |
| 4-Chl oroani li ne | ND | 3060 | 1820 | 60 | 3060 | 1880 | 61 | 3 | 26-121 | 50 |
| 4-Chl orophenyl -phenyl ether | ND | 3060 | 2190 | 72 | 3060 | 2230 | 73 | 1 | 34-118 | 50 |
| 4-Methyl phenol | ND | 3060 | 1720 | 56 | 3060 | 1750 | 57 | 2 | 29-127 | 50 |
| 4-Ni troani li ne | ND | 3060 | 2320 | 76 | 3060 | 2300 | 75 | 1 | 25-148 | 50 |
| 4-Ni trophenol | ND | 3060 | 1950 | 64 | 3060 | 1930 | 63 | 1 | 20-147 | 50 |
| Acenaphthene | ND | 3060 | 1970 | 64 | 3060 | 2090 | 68 | 6 | 29-135 | 50 |
| Acenaphthyl ene | ND | 3060 | 2050 | 67 | 3060 | 2180 | 71 | 6 | 33-120 | 50 |
| Anthracene | ND | 3060 | 2570 | 84 | 3060 | 2470 | 81 | 4 | 35-122 | 50 |
| Benzo(a)anthracene | ND | 3060 | 2580 | 84 | 3060 | 2530 | 83 | 2 | 33-139 | 50 |
| Benzo(a)pyrene | ND | 3060 | 2160 | 71 | 3060 | 2160 | 71 | 0 | 30-144 | 50 |
| Benzo(b)fl uoranthene | ND | 3060 | 2190 | 71 | 3060 | 2420 | 79 | 10 | 24-140 | 50 |
| Benzo(k)fl uoranthene | ND | 3060 | 2260 | 74 | 3060 | 2110 | 69 | 7 | 29-150 | 50 |
| Benzo(g, h, i)peryl ene | ND | 3060 | 2600 | 85 | 3060 | 2470 | 81 | 5 | 20-146 | 50 |
| bi s(2-Chl oroethoxy)methane | ND | 3060 | 1900 | 62 | 3060 | 1950 | 64 | 3 | 24-126 | 50 |
| bi s(2-Chl oroethyl) ether | ND | 3060 | 1870 | 61 | 3060 | 1770 | 58 | 6 | 22-121 | 50 |
| bi s(2-Chl oro i sopropyl) ether | ND | 3060 | 1700 | 56 | 3060 | 1710 | 56 | 1 | 20-131 | 50 |
| bi s(2-Ethyl hexyl) phthal ate | ND | 3060 | 2550 | 83 | 3060 | 2450 | 80 | 4 | 27-149 | 50 |
| Butyl benzyl phthal ate | ND | 3060 | 2380 | 78 | 3060 | 2490 | 81 | 4 | 26-153 | 50 |
| Chrysene | ND | 3060 | 2220 | 73 | 3060 | 2380 | 78 | 7 | 33-142 | 50 |
| Di -n-butyl phthal ate | ND | 3060 | 2780 | 91 | 3060 | 2640 | 86 | 5 | 36-135 | 50 |
| Di -n-octyl phthal ate | ND | 3060 | 2390 | 78 | 3060 | 2370 | 78 | 1 | 22-165 | 50 |
| Di benzo(a, h)anthracene | ND | 3060 | 2650 | 87 | 3060 | 2590 | 85 | 2 | 30-148 | 50 |
| Di benzofuran | ND | 3060 | 1980 | 65 | 3060 | 2080 | 68 | 5 | 34-120 | 50 |
| Di ethyl phthal ate | ND | 3060 | 2540 | 83 | 3060 | 2550 | 83 | 0 | 32-126 | 50 |

FIGURE 9 – TYPICAL MS/MSD REPORT SUMMARY

| | | | | | | | | | | |
|-------------------------------|----|------|------|----|------|------|----|---|--------|----|
| Di methyl phthal ate | ND | 3060 | 2330 | 76 | 3060 | 2400 | 78 | 3 | 33-124 | 50 |
| Fl uoranthene | ND | 3060 | 2860 | 93 | 3060 | 2710 | 89 | 5 | 35-122 | 50 |
| Fl uorene | ND | 3060 | 2180 | 71 | 3060 | 2330 | 76 | 7 | 32-127 | 50 |
| Hexachl orobenzene | ND | 3060 | 2630 | 86 | 3060 | 2600 | 85 | 1 | 32-127 | 50 |
| Hexachl orobutadi ene | ND | 3060 | 2060 | 67 | 3060 | 1880 | 61 | 9 | 27-118 | 50 |
| Hexachl orocycl opentadi ene | ND | 3060 | 1690 | 55 | 3060 | 1810 | 59 | 7 | 20-123 | 50 |
| Hexachl oroethane | ND | 3060 | 1760 | 58 | 3060 | 1670 | 55 | 5 | 23-107 | 50 |
| I ndeno(1, 2, 3-cd)pyrene | ND | 3060 | 2600 | 85 | 3060 | 2540 | 83 | 2 | 28-147 | 50 |
| I sophorone | ND | 3060 | 1990 | 65 | 3060 | 2080 | 68 | 4 | 34-115 | 50 |
| n-Ni trosodi -n-propyl ami ne | ND | 3060 | 1840 | 60 | 3060 | 1910 | 63 | 4 | 26-147 | 50 |
| n-Ni trosodi phenyl ami ne | ND | 3060 | 1730 | 57 | 3060 | 1730 | 57 | 0 | 35-134 | 50 |
| Naphthal ene | ND | 3060 | 1920 | 63 | 3060 | 1870 | 61 | 3 | 27-113 | 50 |
| Ni trobenzene | ND | 3060 | 1930 | 63 | 3060 | 1870 | 61 | 3 | 28-112 | 50 |
| Pentachl orophenol | ND | 3060 | 2200 | 72 | 3060 | 2200 | 72 | 0 | 20-163 | 50 |
| Phenanthrene | ND | 3060 | 2520 | 82 | 3060 | 2490 | 81 | 1 | 35-119 | 50 |
| Phenol | ND | 3060 | 1780 | 58 | 3060 | 1770 | 58 | 1 | 20-120 | 50 |
| Pyrene | ND | 3060 | 2260 | 74 | 3060 | 2340 | 77 | 3 | 32-147 | 50 |

=====

| SURROGATE PARAMETER | SPI KE AMT (ug/kg) | MS RSLT (ug/kg) | MS % REC | SPI KE AMT (ug/kg) | MSD RSLT (ug/kg) | MSD % REC | QC LI MI T (%) |
|-------------------------|-----------------------|--------------------|-------------|-----------------------|---------------------|--------------|---------------------|
| 2, 4, 6-Tri bromophenol | 5730 | 4520 | 79 | 5730 | 4550 | 79 | 50-155 |
| 2-Fl uorobi phenyl | 3820 | 2570 | 67 | 3820 | 2540 | 66 | 32-114 |
| 2-Fl uorophenol | 5730 | 3680 | 64 | 5730 | 3420 | 60 | 30-105 |
| Ni trobenzene-d5 | 3820 | 2510 | 66 | 3820 | 2400 | 63 | 33-114 |
| Phenol -d5 | 5730 | 3650 | 64 | 5730 | 3500 | 61 | 30-110 |
| Terphenyl -d14 | 3820 | 3350 | 88 | 3820 | 2210 | 58 | 31-143 |

FIGURE 10 – TYPICAL CASE NARRATIVE

CASE NARRATIVE

CLIENT: XYZ, INC

PROJECT: CLEAN LAND PROJECT

CLIENT SDG: 10132Q-02

EMAX SDG: 06B164

**SW 3550B/8270C
SEMI VOLATILE ORGANICS BY GC/MS**

Seven (7) soil samples were received on 02/24/06 for Semi Volatile Organic Analysis by Method 8270C in accordance with USEPA SW846, 3rd ed.

1. Holding Time

Samples were extracted and analyzed within holding time.

2. Calibration

All target analytes met calibration requirements

3. Calibration Verification

All target analytes met calibration requirements

4. Method Blank

Method Blank was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement.

5. Lab Control Sample

LCS/LCD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits.

6. Matrix Spike/Matrix Spike Duplicate

That MS/MSD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits.

7. Sample Analysis

Samples were analyzed in conformance to the method and project requirements.

TABLE 1
INTERMEDIATE STANDARD PREPARATION
METHOD: Semivolatiles by GC/MS (8270C)

A. Primary Source: Accu, Restek

| Compound Name | Stock/Internal Soln. Conc. $\mu\text{g/ml}$ | Source | Preparation | | | Final Conc. (mg/L) |
|-------------------------------------|---|-----------------|---------------------------|-------------------|-----------------|--------------------|
| | | | Aliquot (μL) | Dil. Solution | Final Vol. (ml) | |
| Base/Neutral Composite Mix | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Toxic Substance Mix #1 | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Phenol Mix | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Composite Mix 3 | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Benzidine and 3,3'dichlorobenzidine | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Acid Surrogate Mix | 7500 | Restek Standard | 267 | MeCl ₂ | 10 | 200 |
| Base/Neutral Surrogate Mix | 5000 | Restek Standard | 400 | MeCl ₂ | 10 | 200 |

B. Secondary Source: Supelco, Ultra

| Compound Name | Stock Soln. Conc. $\mu\text{g/ml}$ | Source | Preparation | | | Final Conc. mg/L |
|-----------------------|------------------------------------|------------------|--------------|-------------------|------------------|------------------|
| | | | Aliquot (ml) | Solvent | Final Volume, mL | |
| Base/Neutral Mix 1 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Base/Neutral Mix 2 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Toxic Substance Mix 1 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Toxic Substance Mix 2 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Phenol Mix | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| PAH Mixture | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Benzidine | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Carbazol | 2000 | Supelco | 1 | MeCl ₂ | 10 | 200 |
| Pyridine | 2000 | Supelco | 1 | MeCl ₂ | 10 | 200 |

TABLE 2
WORKING STANDARD CALIBRATION
METHOD: Semivolatiles by GC/MS (8270C)

| Standard Name | Intermediate Standard | Internal Standard | Amount of MeCl ₂ Needed | Final Volume | Final Conc. |
|-----------------------------------|-----------------------|--------------------|------------------------------------|--------------|--|
| Standard 1 5 mg/L Calib,Std | 12.5 µl of 200 mg/L | 10 µl of 2000 mg/L | 477.5 µl | 500 µl | 5 mg/L of Cal. Std. 40mg/L of Internal Std. |
| Standard 2 10 mg/L Calib,Std | 25 µl of 200 mg/L | 10 µl of 2000 mg/L | 465 µl | 500 µl | 10 mg/L of Cal. Std. 40mg/L of Internal Std. |
| Standard 3 20 mg/L Calib,Std | 50 µl of 200 mg/L | 10 µl of 2000 mg/L | 440 µl | 500 µl | 20 mg/L of Cal. Std. 40mg/L of Internal Std. |
| Standard 4 40 mg/L Calib,Std | 100 µl of 200 mg/L | 10 µl of 2000 mg/L | 390 µl | 500 µl | 40 mg/L of Cal. Std. 40mg/L of Internal Std. |
| Standard 5 50 mg/L Calib,Std 3 | 125 µl of 200 mg/L | 10 µl of 2000 mg/L | 365 µl | 500 µl | 50 mg/L of Cal. Std. 40mg/L of Internal Std. |
| Standard 6 80 mg/L Calib,Std | 200 µl of 200 mg/L | 10 µl of 2000 mg/L | 290 µl | 500 µl | 80 mg/L of Cal. Std. 40mg/L of Internal Std. |
| Standard 7 100 mg/L Calib,Std | 250 µl of 200 mg/L | 10 µl of 2000 mg/L | 240 µl | 500 µl | 100 mg/L of Cal Std 40mg/L of Internal Std. |
| Standard 8 120 mg/L Calib,Std | 300 µl of 200 mg/L | 10 µl of 2000 mg/L | 190 µl | 500 µl | 120 mg/L of Cal. Std. 40mg/L of Internal Std. |
| Standard 9 160 mg/L Calib,Std | 400 µl of 200 mg/L | 10 µl of 2000 mg/L | 90 µl | 500 µl | 160 mg/L of Cal. Std. 40mg/L of Internal Std. |

WORKING SECONDARY SOURCE STANDARD
METHOD: Semivolatiles by GC/MS (8270C)

| Standard Name | Intermediate Standard | Internal Standard | Amount of MeCl ₂ Needed | Final Volume | Final Conc. |
|--|-----------------------|--------------------|------------------------------------|--------------|---|
| Secondary Source Std. Mix(see Table I) | 125 µl of 200 mg/L | 10 µl of 2000 mg/L | 365 µl | 500 µl | 50 mg/L of Cal. Ver.Std. 40 mg/L of Internal Std |

³ Used as DCC standard

TABLE 3

**INSTRUMENT PARAMETERS
 METHOD 8270**

| Instrument No: | 041, 042 | 048, 052 |
|----------------------------|-------------------------------------|-------------------------------------|
| Carrier Gas | Helium at 60 psi at outlet | Helium at 90 psi at outlet |
| Column head pressure | 10-15 psi at 30°C | 80-90 psi at 45°C |
| Injection port temperature | 260°C | 260°C |
| Interface | Direct column interference at 300°C | Direct column interference at 300°C |
| Valve time | Split 2 minutes | Split 2 minute |

Oven Temperature Program

| Instrument No: | 041, 042 | 048, 052 |
|---------------------|---|--------------------------------------|
| Initial Temperature | 50°C/min; hold for 0.5min. | 45°C; hold for 0.5 minutes |
| Rate | 23°C/min to 100°C; hold for 0.0 min.; 18°C/min to 320°C; hold for 2.1 min. | 22°C/min to 340°C; hold for 0.09 min |
| Run Time | 17 minutes | 14 minutes |

Scan Parameters

| Instrument No: | 041, 042 | 048, 052 |
|-------------------------|--------------------|--------------------|
| Scan start time | After Solvent Peak | After Solvent Peak |
| Mass range | 40 to 500 AMU | 40 to 500 AMU |
| Multiplier voltage | 1000-3000 | 0.7-3 |
| Number of sampling rate | 2 | 2 |
| Threshold | 500-1500 | 500-1500 |
| Tuning File | DFTPP | DFTPP |

TABLE 4
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

| Mass | Ion Abundance Criteria |
|------|---|
| 51 | 30.0 to 60.0% of mass 198 |
| 68 | Less than 2.0% of mass 69 |
| 69 | Present |
| 70 | Less than 2.0% of mass 69 |
| 127 | 40.0 to 60.0% of mass 198 |
| 197 | Less than 1.0% of mass 198 |
| 198 | Base peak, 100% relative abundance (See Note) |
| 199 | 5.0 to 9.0% of mass 198 |
| 275 | 10.0 to 30.0% of mass 198 |
| 365 | Greater than 1.0% of mass 198 |
| 441 | Present but less than mass 443 |
| 442 | Greater than 40% of mass 198 |
| 443 | 17.0 to 23.0% of mass 442 |

NOTE: All ion abundance MUST be normalized to m/z 198, the nominal base peak.

TABLE 5
CALIBRATION CHECK COMPOUNDS (CCC)

| Base/Neutral Fraction | Acid Fraction |
|------------------------|-------------------------|
| Acenaphthene | 4-Chloro-3-methylphenol |
| 1,4-Dichlorobenzene | 2,4-Dichlorophenol |
| Hexachlorobutadiene | 2-Nitrophenol |
| N-Nitrosodiphenylamine | Phenol |
| Di-n-octylphthalate | Pentachlorophenol |
| Fluoranthene | 2,4,6-Trichlorophenol |
| Benzo(a)pyrene | |

TABLE 6
SYSTEM PERFORMANCE CALIBRATION CHECK COMPOUNDS (SPCC)

| Base/Neutral Compounds | Acid Compounds |
|----------------------------|-------------------|
| N-Nitroso-di-n-propylamine | 2,4-Dinitrophenol |
| Hexachlorocyclopentadiene | 4-Nitrophenol |

SUMMARY OF IN-HOUSE QUALITY CONTROL PROCEDURES

| QC PROCEDURE | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1st Rvw | 2nd Rvw |
|--|---|--|---|--|---------|---------|
| Check of mass spectral ion intensities using DFTPP | Prior to initial calibration and calibration verification | Refer to criteria listed in the method description (Table 4) | Retune instrument and verify | | | |
| At least 5-point calibration for all analytes | Initially; as needed | SPCCs average RF \pm 0.050 and %RSD for RFs for CCCs \leq 30% and one option below 1). linear- mean RSD for all analytes \leq 15% 2). linear – least squares regression $r \geq$ 0.995, when RSD $>$ 15% 3). non-linear – COD $>$ 0.990 (6 points shall be used for second order, 7 points shall be used for third) | Correct the problem then repeat initial calibration | | | |
| Second-source calibration verification | After initial calibration | All analytes within \pm 25% of expected value [* within \pm 35% of expected value] | Correct the problem then repeat initial calibration | | | |
| Retention time window calculated for each analyte | Each sample | Relative retention time (RRT) of the analyte within \pm 0.06 RRT units of the RRT | Correct the problem then reanalyze all samples analyzed since the last retention time check | | | |
| Calibration verification | Daily, before sample analysis and every 12 hours of analysis time | SPCCs average RF \geq 0.050; and CCCs \leq 20% difference (when using RFs) or drift (when using least squares regression or non-linear calibration) | Correct the problem then repeat initial calibration | | | |
| Internal Standard | Every sample, spiked sample, standard, and method blank | Retention time \pm 30 seconds from retention time of the mid-point std. In the ICAL. EICP area within -50% to +100% of ICAL mid-point std. | Inspect mass spectrometer and GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning | | | |
| Method blank | One per preparation batch (\leq 20 samples per matrix) | No analytes detected \geq RL | Reprep and reanalyze method blank and all samples processed with the contaminated blank | Apply B to specific analyte(s) on all associated | | |
| LCS | One LCS per preparation (\leq 20 samples per matrix) | Within EMAX In-House QC Limits | Reprep and reanalyze the LCS and all associated samples | | | |
| MS/MSD | One MS/MSD per every 20 project samples per matrix | Within EMAX In-House QC Limits | None | | | |
| Surrogate spike | Every sample, spiked sample, standard, and method blank | At least 2 out of 3 Acid and 2 out of 3 BN surrogates should be within EMAX In-house QC Limits. | If two acids or two base/neutral are out of QC limits, correct the problem then re-extract and analyze sample. | | | |
| Results reported between MDL and RL | None | None | None | Apply J to all values between MDL and RL | | |
| Comments: *Except for the following compounds due to erratic chromatographic behavior: Benzidine, 4,6-dinitro-2-methylphenol, 4-chroanaline, benzyl alcohol, n-Nitrosodimethylamine, 4-nitrophenol, 2-nitroaniline, Pyridine, Benzoic Acid, and 3-nitroaniline RL = Reporting Limit | | | | Reviewed by: | | |
| | | | | Date: | | |

DEMONSTRATION OF CAPABILITY
Method 3520C_8270C

Applicable SOP & Rev. #: EMAX-8270 Rev. # 2

Conc Unit: mg/L
Sample Amount(ml): 1000
Sample Extracted(ml): 1

Extracted date: 5/15/2006 **5/18/2006**

Analyzed date: 5/18/2006 **5/26/2006**

Extracted by: Juanita Muertigue

Analyzed by: Souzan Greas

| PARAMETER | REH077 | REH078 | REX271 | REX272 | TV | Ave. Conc. | Ave. %Rec | SD | QC Criteria | COMMENTS |
|----------------------------|--------|--------|--------|--------|----|------------|-----------|----|-------------|----------|
| 1,2,4-Trichlorobenzene | 49.1 | 55.6 | 42.6 | 52.9 | 80 | 50.1 | 63 | 6 | 30 - 130 | Passed |
| 1,2-Dichlorobenzene | 47.3 | 52.4 | 38.9 | 47.5 | 80 | 46.5 | 58 | 6 | 30 - 130 | Passed |
| 1,2-Diphenylhydrazine | 56.3 | 72.6 | 71.2 | 65.5 | 80 | 66.4 | 83 | 7 | 30 - 130 | Passed |
| 1,3-Dichlorobenzene | 45.8 | 49.4 | 34.8 | 45.1 | 80 | 43.7 | 55 | 6 | 30 - 130 | Passed |
| 1,4-Dichlorobenzene | 46.8 | 52.4 | 36.0 | 47.0 | 80 | 45.6 | 57 | 7 | 30 - 130 | Passed |
| 2,4,5-Trichlorophenol | 61.0 | 70.4 | 69.0 | 64.0 | 80 | 66.1 | 83 | 4 | 40 - 130 | Passed |
| 2,4,6-Trichlorophenol | 62.0 | 67.8 | 61.4 | 63.3 | 80 | 63.6 | 80 | 3 | 40 - 130 | Passed |
| 2,4-Dichlorophenol | 55.9 | 65.4 | 53.3 | 59.4 | 80 | 58.5 | 73 | 5 | 40 - 130 | Passed |
| 2,4-Dimethylphenol | 46.5 | 54.5 | 46.5 | 56.8 | 80 | 51.1 | 64 | 5 | 30 - 130 | Passed |
| 2,4-Dinitrophenol | 50.2 | 61.9 | 65.5 | 68.6 | 80 | 61.6 | 77 | 8 | 30 - 130 | Passed |
| 2,4-Dinitrotoluene | 67.8 | 79.4 | 80.0 | 73.3 | 80 | 75.1 | 94 | 6 | 40 - 130 | Passed |
| 2,6-Dinitrotoluene | 67.4 | 74.3 | 73.0 | 68.9 | 80 | 70.9 | 89 | 3 | 50 - 130 | Passed |
| 2-Chloronaphthalene | 59.0 | 64.2 | 56.1 | 59.7 | 80 | 59.7 | 75 | 3 | 40 - 130 | Passed |
| 2-Chlorophenol | 53.4 | 62.5 | 44.9 | 55.0 | 80 | 53.9 | 67 | 7 | 30 - 130 | Passed |
| 2-Methylnaphthalene | 50.7 | 60.4 | 50.4 | 56.4 | 80 | 54.5 | 68 | 5 | 40 - 130 | Passed |
| 2-Methylphenol | 51.6 | 63.5 | 50.2 | 54.9 | 80 | 55.1 | 69 | 6 | 30 - 130 | Passed |
| 2-Nitroaniline | 59.7 | 71.8 | 71.7 | 68.4 | 80 | 67.9 | 85 | 6 | 40 - 130 | Passed |
| 2-Nitrophenol | 55.6 | 66.4 | 51.6 | 61.4 | 80 | 58.8 | 73 | 7 | 40 - 130 | Passed |
| 3,3'-Dichlorobenzidine | 66.7 | 85.6 | 89.1 | 73.5 | 80 | 78.7 | 98 | 10 | 20 - 140 | Passed |
| 3-Nitroaniline | 59.9 | 73.0 | 71.4 | 69.5 | 80 | 68.5 | 86 | 6 | 50 - 130 | Passed |
| 4,6-Dinitro-2-methylphenol | 62.0 | 77.5 | 79.7 | 75.8 | 80 | 73.8 | 92 | 8 | 50 - 130 | Passed |
| 4-Bromophenyl-phenylether | 62.2 | 68.5 | 67.8 | 67.1 | 80 | 66.4 | 83 | 3 | 50 - 130 | Passed |
| 4-Chloro-3-methylphenol | 57.4 | 75.0 | 69.6 | 66.7 | 80 | 67.2 | 84 | 7 | 40 - 130 | Passed |
| 4-Chloroaniline | 52.0 | 57.4 | 48.6 | 52.7 | 80 | 52.7 | 66 | 4 | 30 - 130 | Passed |
| 4-Chlorophenyl-phenylether | 59.9 | 68.3 | 65.8 | 63.2 | 80 | 64.3 | 80 | 4 | 50 - 130 | Passed |
| 4-Methylphenol | 50.0 | 61.5 | 50.8 | 55.1 | 80 | 54.4 | 68 | 5 | 30 - 130 | Passed |
| 4-Nitroaniline | 64.8 | 76.5 | 78.0 | 71.2 | 80 | 72.6 | 91 | 6 | 50 - 130 | Passed |
| 4-Nitrophenol | 71.5 | 84.9 | 85.1 | 77.7 | 80 | 79.8 | 100 | 7 | 30 - 130 | Passed |
| Acenaphthene | 57.1 | 66.2 | 59.1 | 62.7 | 80 | 61.3 | 77 | 4 | 40 - 130 | Passed |
| Acenaphthylene | 60.3 | 68.8 | 64.2 | 58.7 | 80 | 63.0 | 79 | 5 | 40 - 130 | Passed |
| Aniline | 42.3 | 43.0 | 37.1 | 27.7 | 80 | 37.5 | 47 | 7 | 10 - 130 | Passed |
| Anthracene | 63.8 | 69.6 | 69.0 | 67.8 | 80 | 67.5 | 84 | 3 | 50 - 130 | Passed |
| Benzo(a)anthracene | 75.6 | 86.4 | 91.8 | 83.8 | 80 | 84.4 | 105 | 7 | 60 - 130 | Passed |
| Benzo(a)pyrene | 74.9 | 85.1 | 89.2 | 79.6 | 80 | 82.2 | 103 | 6 | 60 - 130 | Passed |
| Benzo(b)fluoranthene | 71.7 | 88.7 | 88.1 | 82.2 | 80 | 82.7 | 103 | 8 | 50 - 140 | Passed |
| Benzo(g,h,i)perylene | 70.2 | 79.1 | 83.5 | 86.0 | 80 | 79.7 | 100 | 7 | 50 - 130 | Passed |
| Benzo(k)fluoranthene | 84.4 | 90.7 | 97.5 | 72.2 | 80 | 86.2 | 108 | 11 | 50 - 130 | Passed |
| Benzoic Acid | 39.1 | 48.5 | 47.3 | 52.9 | 80 | 47.0 | 59 | 6 | 10 - 130 | Passed |
| Benzyl alcohol | 56.9 | 66.5 | 53.0 | 61.1 | 80 | 59.4 | 74 | 6 | 30 - 130 | Passed |
| bis(2-Chloroethoxy)methane | 52.7 | 64.7 | 51.4 | 60.3 | 80 | 57.3 | 72 | 6 | 40 - 130 | Passed |

DEMONSTRATION OF CAPABILITY
Method 3520C_8270C

Applicable SOP & Rev. #: EMAX-8270 Rev. # 2

Extracted date: 5/15/2006 **5/18/2006**

Conc Unit: mg/L

Analyzed date: 5/18/2006 **5/26/2006**

Sample Amount(ml): 1000

Extracted by: Juanita Muertigue

Sample Extracted(ml): 1

Analyzed by: Souzan Greas

| PARAMETER | REH077 | REH078 | REX271 | REX272 | TV | Ave. Conc. | Ave. %Rec | SD | QC Criteria | COMMENTS |
|-----------------------------|--------|--------|--------|--------|-----|------------|-----------|----|-------------|----------|
| Bis(2-chloroethyl)ether | 64.3 | 45.6 | 58.6 | 66.6 | 80 | 58.8 | 73 | 9 | 30 - 130 | Passed |
| Bis(2-chloroisopropyl)ether | 61.6 | 45.2 | 47.9 | 56.1 | 80 | 52.7 | 66 | 8 | 20 - 130 | Passed |
| bis(2-Ethylhexyl)phthalate | 82.2 | 87.9 | 87.7 | 103.6 | 80 | 90.4 | 113 | 9 | 50 - 140 | Passed |
| Butylbenzylphthalate | 88.2 | 91.7 | 90.7 | 106.1 | 80 | 94.2 | 118 | 8 | 50 - 130 | Passed |
| Carbazole | 76.8 | 77.4 | 71.2 | 78.9 | 80 | 76.1 | 95 | 3 | 60 - 130 | Passed |
| Chrysene | 78.7 | 82.3 | 80.4 | 93.8 | 80 | 83.8 | 105 | 7 | 50 - 130 | Passed |
| Di-n-butylphthalate | 81.6 | 83.8 | 85.7 | 95.7 | 80 | 86.7 | 108 | 6 | 50 - 140 | Passed |
| Di-n-octylphthalate | 65.6 | 62.4 | 60.2 | 68.8 | 80 | 64.2 | 80 | 4 | 50 - 150 | Passed |
| Dibenzo(a,h)anthracene | 82.8 | 81.5 | 69.6 | 83.6 | 80 | 79.4 | 99 | 7 | 50 - 130 | Passed |
| Dibenzofuran | 76.6 | 74.3 | 70.1 | 80.7 | 80 | 75.4 | 94 | 4 | 50 - 130 | Passed |
| Diethylphthalate | 79.8 | 80.8 | 71.1 | 79.8 | 80 | 77.9 | 97 | 5 | 50 - 130 | Passed |
| Dimethylphthalate | 91.4 | 93.1 | 85.1 | 94.4 | 80 | 91.0 | 114 | 4 | 50 - 130 | Passed |
| Fluoranthene | 77.4 | 80.1 | 71.6 | 80.0 | 80 | 77.3 | 97 | 4 | 50 - 130 | Passed |
| Fluorene | 70.3 | 68.1 | 64.2 | 71.9 | 80 | 68.6 | 86 | 3 | 40 - 130 | Passed |
| Hexachlorobenzene | 70.6 | 69.2 | 67.7 | 75.1 | 80 | 70.7 | 88 | 3 | 50 - 130 | Passed |
| Hexachlorobutadiene | 52.2 | 39.2 | 48.0 | 55.1 | 80 | 48.6 | 61 | 7 | 20 - 130 | Passed |
| Hexachlorocyclopentadiene | 43.5 | 34.4 | 43.6 | 51.3 | 80 | 43.2 | 54 | 7 | 10 - 130 | Passed |
| Hexachloroethane | 48.6 | 34.0 | 45.4 | 51.1 | 80 | 44.8 | 56 | 8 | 20 - 130 | Passed |
| Indeno(1,2,3-cd)pyrene | 87.0 | 88.0 | 87.1 | 96.7 | 80 | 89.7 | 112 | 5 | 60 - 130 | Passed |
| Isophorone | 73.6 | 64.8 | 66.1 | 75.2 | 80 | 69.9 | 87 | 5 | 40 - 130 | Passed |
| N-Nitrosodimethylamine | 58.0 | 45.8 | 53.9 | 62.4 | 80 | 55.0 | 69 | 7 | 30 - 150 | Passed |
| N-Nitroso-di-n-propylamine | 65.7 | 52.0 | 60.6 | 68.4 | 80 | 61.7 | 77 | 7 | 30 - 130 | Passed |
| N-Nitrosodiphenylamine | 60.0 | 42.9 | 51.9 | 61.6 | 80 | 54.1 | 68 | 9 | 40 - 130 | Passed |
| Naphthalene | 74.1 | 61.1 | 62.7 | 71.0 | 80 | 67.2 | 84 | 6 | 10 - 150 | Passed |
| Nitrobenzene | 53.1 | 52.3 | 51.9 | 56.7 | 80 | 53.5 | 67 | 2 | 30 - 130 | Passed |
| Pentachlorophenol | 65.5 | 66.2 | 73.2 | 81.5 | 80 | 71.6 | 90 | 7 | 30 - 130 | Passed |
| Phenanthrene | 69.3 | 68.7 | 67.7 | 74.8 | 80 | 70.1 | 88 | 3 | 50 - 130 | Passed |
| Phenol | 62.5 | 46.6 | 53.5 | 63.0 | 80 | 56.4 | 70 | 8 | 20 - 130 | Passed |
| Pyrene | 80.3 | 84.1 | 75.5 | 90.2 | 80 | 82.6 | 103 | 6 | 50 - 130 | Passed |
| Pyridine | 37.1 | 31.5 | 22.5 | 36.6 | 80 | 31.9 | 40 | 7 | 10 - 130 | Passed |
| SURROGATES | | | | | | | | | | |
| 1,2-Dichlorobenzene-d4 | 34.3 | 24.0 | 29.3 | 35.2 | 50 | 30.7 | 61 | 3 | 30 - 150 | Passed |
| 2,4,6-Tribromophenol | 126 | 125 | 108 | 125 | 150 | 120.9 | 81 | 8 | 40 - 140 | Passed |
| 2-Fluorobiphenyl | 38.6 | 33.0 | 35.2 | 41.0 | 50 | 37.0 | 74 | 6 | 30 - 130 | Passed |
| 2-Fluorophenol | 95.3 | 65.4 | 78.0 | 92.1 | 150 | 82.7 | 55 | 7 | 40 - 130 | Passed |
| Nitrobenzene-d5 | 36.9 | 29.4 | 31.7 | 36.8 | 50 | 33.7 | 67 | 4 | 40 - 130 | Passed |
| Phenol-d5 | 100 | 74.9 | 82.9 | 96.6 | 150 | 88.6 | 59 | 12 | 30 - 130 | Passed |
| Terphenyl-d14 | 53.1 | 55.1 | 51.1 | 63.2 | 50 | 55.6 | 111 | 5 | 50 - 130 | Passed |

DEMONSTRATION OF CAPABILITY
Method 3550B_8270C

Applicable SOP & Rev. #: EMAX-8270 Rev. # 2

Extracted date: 5/22/2006 5/31/2006

Conc Unit: mg/Kg

Analyzed date: 5/24/2006 6/1/2006

Sample Amount(g): 30

Extracted by: Jessie Villena

Sample Extracted(ml): 1

Analyzed by: Souzan Greas

| PARAMETER | REX212 | REX213 | RFX006 | RFX007 | TV | Ave. Conc. | Ave. %Rec | SD | QC Criteria | COMMENTS |
|----------------------------|--------|--------|--------|--------|----|------------|-----------|----|-------------|----------|
| 1,2,4-Trichlorobenzene | 61.4 | 64.4 | 60.1 | 61.3 | 80 | 61.8 | 77 | 2 | 30 - 130 | Passed |
| 1,2-Dichlorobenzene | 56.6 | 60.8 | 57.4 | 57.0 | 80 | 57.9 | 72 | 2 | 40 - 130 | Passed |
| 1,2-Diphenylhydrazine | 55.3 | 57.2 | 55.8 | 56.2 | 80 | 56.1 | 70 | 1 | 30 - 130 | Passed |
| 1,3-Dichlorobenzene | 59.3 | 62.9 | 57.2 | 57.4 | 80 | 59.2 | 74 | 3 | 40 - 130 | Passed |
| 1,4-Dichlorobenzene | 58.3 | 62.7 | 56.7 | 56.5 | 80 | 58.5 | 73 | 3 | 30 - 130 | Passed |
| 2,4,5-Trichlorophenol | 60.6 | 63.3 | 61.0 | 60.7 | 80 | 61.4 | 77 | 1 | 50 - 130 | Passed |
| 2,4,6-Trichlorophenol | 59.0 | 65.2 | 59.9 | 59.2 | 80 | 60.8 | 76 | 3 | 50 - 130 | Passed |
| 2,4-Dichlorophenol | 57.1 | 63.3 | 59.9 | 60.6 | 80 | 60.2 | 75 | 3 | 50 - 130 | Passed |
| 2,4-Dimethylphenol | 54.9 | 59.8 | 57.2 | 58.0 | 80 | 57.5 | 72 | 2 | 40 - 130 | Passed |
| 2,4-Dinitrophenol | 50.1 | 56.6 | 57.7 | 61.5 | 80 | 56.5 | 71 | 5 | 40 - 130 | Passed |
| 2,4-Dinitrotoluene | 61.4 | 69.8 | 66.1 | 68.0 | 80 | 66.4 | 83 | 4 | 30 - 130 | Passed |
| 2,6-Dinitrotoluene | 63.7 | 71.9 | 59.8 | 60.9 | 80 | 64.1 | 80 | 5 | 60 - 130 | Passed |
| 2-Chloronaphthalene | 59.1 | 66.3 | 56.9 | 55.3 | 80 | 59.4 | 74 | 5 | 50 - 130 | Passed |
| 2-Chlorophenol | 56.7 | 61.1 | 58.4 | 59.1 | 80 | 58.8 | 74 | 2 | 30 - 130 | Passed |
| 2-Methylnaphthalene | 56.7 | 58.6 | 56.3 | 57.0 | 80 | 57.1 | 71 | 1 | 40 - 130 | Passed |
| 2-Methylphenol | 55.0 | 58.6 | 58.6 | 57.2 | 80 | 57.4 | 72 | 2 | 40 - 130 | Passed |
| 2-Nitroaniline | 54.9 | 61.9 | 60.8 | 61.3 | 80 | 59.7 | 75 | 3 | 50 - 130 | Passed |
| 2-Nitrophenol | 58.8 | 64.5 | 60.0 | 61.7 | 80 | 61.2 | 77 | 2 | 50 - 130 | Passed |
| 3,3'-Dichlorobenzidine | 65.8 | 69.0 | 53.2 | 55.9 | 80 | 61.0 | 76 | 8 | 60 - 130 | Passed |
| 3-Nitroaniline | 57.1 | 65.5 | 60.0 | 62.5 | 80 | 61.3 | 77 | 4 | 60 - 130 | Passed |
| 4,6-Dinitro-2-methylphenol | 61.7 | 65.6 | 64.9 | 68.9 | 80 | 65.3 | 82 | 3 | 50 - 130 | Passed |
| 4-Bromophenyl-phenylether | 61.6 | 64.0 | 57.7 | 57.7 | 80 | 60.3 | 75 | 3 | 50 - 130 | Passed |
| 4-Chloro-3-methylphenol | 57.6 | 61.7 | 59.1 | 60.4 | 80 | 59.7 | 75 | 2 | 30 - 130 | Passed |
| 4-Chloroaniline | 57.3 | 62.0 | 57.9 | 58.7 | 80 | 59.0 | 74 | 2 | 40 - 130 | Passed |
| 4-Chlorophenyl-phenylether | 59.2 | 61.1 | 59.7 | 59.6 | 80 | 59.9 | 75 | 1 | 50 - 130 | Passed |
| 4-Methylphenol | 51.7 | 57.0 | 53.9 | 53.4 | 80 | 54.0 | 68 | 2 | 40 - 130 | Passed |
| 4-Nitroaniline | 58.6 | 64.7 | 64.0 | 66.4 | 80 | 63.4 | 79 | 3 | 60 - 130 | Passed |
| 4-Nitrophenol | 65.5 | 69.5 | 55.5 | 58.4 | 80 | 62.2 | 78 | 6 | 20 - 130 | Passed |
| Acenaphthene | 59.1 | 63.1 | 56.2 | 57.0 | 80 | 58.8 | 74 | 3 | 30 - 130 | Passed |
| Acenaphthylene | 57.4 | 63.8 | 57.0 | 57.2 | 80 | 58.8 | 74 | 3 | 30 - 130 | Passed |
| Aniline | 51.8 | 56.6 | 56.0 | 56.8 | 80 | 55.3 | 69 | 2 | 30 - 150 | Passed |
| Anthracene | 58.9 | 61.1 | 58.3 | 59.9 | 80 | 59.6 | 74 | 1 | 30 - 130 | Passed |
| Benzo(a)anthracene | 72.9 | 74.3 | 55.2 | 58.1 | 80 | 65.1 | 81 | 10 | 40 - 130 | Passed |
| Benzo(a)pyrene | 68.6 | 72.7 | 61.9 | 63.4 | 80 | 66.6 | 83 | 5 | 40 - 130 | Passed |
| Benzo(b)fluoranthene | 69.1 | 68.0 | 67.7 | 69.4 | 80 | 68.5 | 86 | 1 | 50 - 130 | Passed |
| Benzo(g,h,i)perylene | 68.9 | 73.5 | 65.3 | 65.5 | 80 | 68.3 | 85 | 4 | 40 - 140 | Passed |
| Benzo(k)fluoranthene | 61.5 | 76.5 | 60.3 | 64.0 | 80 | 65.6 | 82 | 7 | 40 - 130 | Passed |
| Benzoic Acid | 40.7 | 44.4 | 36.6 | 37.4 | 80 | 39.8 | 50 | 4 | 10 - 130 | Passed |
| Benzyl alcohol | 57.6 | 61.8 | 58.6 | 58.5 | 80 | 59.1 | 74 | 2 | 40 - 130 | Passed |
| bis(2-Chloroethoxy)methane | 56.0 | 61.1 | 58.0 | 58.9 | 80 | 58.5 | 73 | 2 | 40 - 130 | Passed |

DEMONSTRATION OF CAPABILITY
Method 3550B_8270C

Applicable SOP & Rev. #: EMAX-8270 Rev. # 2

Extracted date: 5/22/2006 **5/31/2006**

Conc Unit: mg/Kg
Sample Amount(g): 30
Sample Extracted(ml): 1

Analyzed date: 5/24/2006 **6/1/2006**

Extracted by: Jessie Villena

Analyzed by: Souzan Greas

| PARAMETER | REX212 | REX213 | RFX006 | RFX007 | TV | Ave. Conc. | Ave. %Rec | SD | QC Criteria | COMMENTS |
|-----------------------------|--------|--------|--------|--------|-----|------------|-----------|----|-------------|----------|
| Bis(2-chloroethyl)ether | 58.9 | 60.6 | 60.1 | 60.3 | 80 | 60.0 | 75 | 1 | 40 - 130 | Passed |
| Bis(2-chloroisopropyl)ether | 49.3 | 53.3 | 56.0 | 56.2 | 80 | 53.7 | 67 | 3 | 40 - 130 | Passed |
| bis(2-Ethylhexyl)phthalate | 70.7 | 77.5 | 53.8 | 56.6 | 80 | 64.6 | 81 | 11 | 50 - 130 | Passed |
| Butylbenzylphthalate | 68.6 | 77.4 | 55.5 | 58.7 | 80 | 65.1 | 81 | 10 | 60 - 130 | Passed |
| Carbazole | 58.1 | 61.0 | 60.2 | 62.2 | 80 | 60.4 | 75 | 2 | 50 - 130 | Passed |
| Chrysene | 67.8 | 71.6 | 56.4 | 58.3 | 80 | 63.5 | 79 | 7 | 40 - 130 | Passed |
| Di-n-butylphthalate | 69.9 | 76.3 | 67.1 | 68.4 | 80 | 70.4 | 88 | 4 | 50 - 130 | Passed |
| Di-n-octylphthalate | 55.0 | 60.3 | 56.5 | 55.6 | 80 | 56.9 | 71 | 2 | 50 - 130 | Passed |
| Dibenzo(a,h)anthracene | 60.9 | 70.6 | 59.6 | 61.0 | 80 | 63.0 | 79 | 5 | 40 - 130 | Passed |
| Dibenzofuran | 61.8 | 67.3 | 58.3 | 59.4 | 80 | 61.7 | 77 | 4 | 50 - 130 | Passed |
| Diethylphthalate | 61.0 | 63.7 | 58.5 | 60.8 | 80 | 61.0 | 76 | 2 | 60 - 130 | Passed |
| Dimethylphthalate | 75.5 | 78.8 | 63.9 | 65.8 | 80 | 71.0 | 89 | 7 | 60 - 130 | Passed |
| Fluoranthene | 62.7 | 65.4 | 60.6 | 63.3 | 80 | 63.0 | 79 | 2 | 40 - 130 | Passed |
| Fluorene | 59.6 | 62.4 | 57.7 | 58.2 | 80 | 59.5 | 74 | 2 | 30 - 130 | Passed |
| Hexachlorobenzene | 59.6 | 63.9 | 60.0 | 61.3 | 80 | 61.2 | 76 | 2 | 30 - 130 | Passed |
| Hexachlorobutadiene | 62.2 | 65.0 | 59.1 | 59.9 | 80 | 61.6 | 77 | 3 | 40 - 130 | Passed |
| Hexachlorocyclopentadiene | 85.0 | 89.1 | 71.7 | 73.9 | 80 | 79.9 | 100 | 8 | 10 - 130 | Passed |
| Hexachloroethane | 59.7 | 64.3 | 57.0 | 57.2 | 80 | 59.6 | 74 | 3 | 40 - 130 | Passed |
| Indeno(1,2,3-cd)pyrene | 71.4 | 76.8 | 65.8 | 66.9 | 80 | 70.2 | 88 | 5 | 40 - 130 | Passed |
| Isophorone | 62.1 | 67.1 | 63.1 | 64.2 | 80 | 64.1 | 80 | 2 | 50 - 130 | Passed |
| N-Nitrosodimethylamine | 58.5 | 58.1 | 57.5 | 58.4 | 80 | 58.1 | 73 | 0 | 30 - 150 | Passed |
| N-Nitroso-di-n-propylamine | 56.9 | 64.0 | 56.5 | 57.4 | 80 | 58.7 | 73 | 4 | 30 - 130 | Passed |
| N-Nitrosodiphenylamine | 56.0 | 59.4 | 58.9 | 59.6 | 80 | 58.5 | 73 | 2 | 30 - 140 | Passed |
| Naphthalene | 57.1 | 63.9 | 64.0 | 63.3 | 80 | 62.1 | 78 | 3 | 30 - 130 | Passed |
| Nitrobenzene | 64.2 | 67.1 | 53.0 | 52.8 | 80 | 59.3 | 74 | 7 | 40 - 130 | Passed |
| Pentachlorophenol | 60.0 | 62.6 | 59.9 | 62.2 | 80 | 61.2 | 76 | 1 | 20 - 130 | Passed |
| Phenanthrene | 56.8 | 61.5 | 57.0 | 59.0 | 80 | 58.6 | 73 | 2 | 30 - 130 | Passed |
| Phenol | 53.1 | 56.4 | 55.2 | 55.2 | 80 | 55.0 | 69 | 1 | 30 - 130 | Passed |
| Pyrene | 63.4 | 67.7 | 53.4 | 54.5 | 80 | 59.7 | 75 | 7 | 30 - 130 | Passed |
| Pyridine | 46.3 | 47.5 | 46.7 | 48.4 | 80 | 47.2 | 59 | 1 | 10 - 130 | Passed |
| SURROGATES | | | | | | | | | | |
| 1,2-Dichlorobenzene-d4 | 40.0 | 41.5 | 40.1 | 39.7 | 50 | 40.3 | 81 | 2 | 30 - 150 | Passed |
| 2,4,6-Tribromophenol | 122 | 135 | 127 | 128 | 150 | 128.3 | 86 | 1 | 40 - 130 | Passed |
| 2-Fluorobiphenyl | 41.3 | 45.2 | 41.2 | 40.7 | 50 | 42.1 | 84 | 7 | 30 - 130 | Passed |
| 2-Fluorophenol | 105 | 109 | 110 | 108 | 150 | 108.1 | 72 | 1 | 30 - 130 | Passed |
| Nitrobenzene-d5 | 38.6 | 41.1 | 41.1 | 41.3 | 50 | 40.5 | 81 | 1 | 30 - 130 | Passed |
| Phenol-d5 | 103 | 108 | 111 | 109 | 150 | 107.7 | 72 | 3 | 30 - 130 | Passed |
| Terphenyl-d14 | 52.4 | 54.9 | 43.3 | 44.1 | 50 | 48.7 | 97 | 6 | 40 - 130 | Passed |

QUANTITATION IONS

| Compound | T/S/IS | Primary Quant-Ion | Secondary Quat-Ion | Compound | T/S/IS | Primary Quant-Ion | Secondary Quant-Ion |
|------------------------------|--------|-------------------|--------------------|-----------------------------|--------|-------------------|---------------------|
| 1,2,4-Trichlorobenzene | T | 180 | 182,145 | Bis(2-ethylhexyl) phthalate | T | 149 | 167,279 |
| 1,2-Dichlorobenzene | T | 146 | 148,111 | Butylbenzylphthalate | T | 149 | 91,206 |
| 1,3-Dichlorobenzene | T | 146 | 148,111 | Carbazole | T | 167 | 166,168 |
| 1,4-Dichlorobenzene | T | 146 | 148,111 | Chrysene | T | 228 | 226,229 |
| 2,4,5-Trichlorophenol | T | 196 | 198,97,132,99 | Dibenzo(a,h)anthracene | T | 278 | 139,279 |
| 2,4,6-Trichlorophenol | T | 196 | 198,200 | Dibenzofuran | T | 168 | 139 |
| 2,4-Dichlorophenol | T | 162 | 164,98 | Diethylphthalate | T | 149 | 177,150 |
| 2,4-Dimethylphenol | T | 122 | 107,121 | Dimethylphthalate | T | 163 | 194,164 |
| 2,4-Dinitrophenol | T | 184 | 63,154 | Di-n-butylphthalate | T | 149 | 150,104 |
| 2,4-Dinitrotoluene | T | 165 | 63,89 | Di-n-octylphthalate | T | 149 | 167,43 |
| 2,6-Dinitrotoluene | T | 165 | 63,89 | Fluoranthene | T | 202 | 101,203 |
| 2-Chloronaphthalene | T | 162 | 127,164 | Fluorene | T | 166 | 165,167 |
| 2-Chlorophenol | T | 128 | 64,130 | Hexachlorobenzene | T | 284 | 142249 |
| 2-Methylnaphthalene | T | 142 | 141 | Hexachlorobutadiene | T | 225 | 223,227 |
| 2-Methylphenol | T | 107 | 108,77,79,90 | Hexachlorocyclopentadiene | T | 237 | 235,272 |
| 2-Nitroaniline | T | 65 | 92,138 | Hexachloroethane | T | 117 | 201,199 |
| 2-Nitrophenol | T | 139 | 109,65 | Hydroquinone | T | 110 | 81,53,55 |
| 3,3'-Dichlorobenzidine | T | 252 | 254,126 | Indeno(1,2,3-cd)pyrene | T | 276 | 138,227 |
| 3-Nitroaniline | T | 138 | 108,92 | Isophorone | T | 82 | 95,138 |
| 4,6-Dinitro-2-methylphenol | T | 198 | 51,105 | Naphthalene | T | 128 | 129,127 |
| 4-Bromophenyl phenyl ether | T | 248 | 250,141 | Nitrobenzene | T | 77 | 123,65 |
| 4-Chloro-3-methylphenol | T | 107 | 144,142 | N-Nitroso-dimethylamine | T | 42 | 74,44 |
| 4-Chloroaniline | T | 127 | 129,65,92 | N-Nitroso-di-n-propylamine | T | 70 | 42,101,130 |
| 4-Chlorophenyl phenyl ether | T | 204 | 206,141 | N-Nitroso-diphenylamine | T | 169 | 168,167 |
| 4-Methylphenol | T | 107 | 108,77,79,90 | Pentachlorophenol | T | 266 | 264,268 |
| 4-Nitroaniline | T | 138 | 65,108,92,80,39 | Phenanthrene | T | 178 | 179,176 |
| 4-Nitrophenol | T | 139 | 109,65 | Phenol | T | 94 | 65,66 |
| Acenaphthene | T | 154 | 153,152 | Pyrene | T | 202 | 200,203 |
| Acenaphthylene | T | 152 | 151,153 | Pyridine | T | 79 | 52 |
| Aniline | T | 93 | 66,65 | 2,4,6-Tribromophenol | S | 330 | 332141 |
| Anthracene | T | 178 | 176,179 | 2-Fluorobiphenyl | S | 172 | 171 |
| Azobenzene | T | 77 | 105,182 | 2-Fluorophenol | S | 112 | 64 |
| Benzidine | T | 184 | 92,185 | Nitrobenzene-d5 | S | 82 | 128,545 |
| Benzo(a)anthracene | T | 228 | 229,226 | Phenol-d5 | S | 99 | 42,71 |
| Benzo(a)pyrene | T | 252 | 25,3125 | Terphenyl-d14 | S | 244 | 122,212 |
| Benzo(b)fluoranthene | T | 252 | 253,125 | 1,2-Dichlorobenzene-d4 | S | 152 | 150,115 |
| Benzo(g,h,l)perylene | T | 276 | 138,277 | 1,4-Dichlorobenzene-d4 | IS | 152 | 150,154 |
| Benzo(k)fluoranthene | T | 252 | 253,125 | Acenaphthene-d10 | IS | 164 | 162,160 |
| Benzoic acid | T | 122 | 105,77 | Chrysene-d12 | IS | 240 | 120,236, |
| Benzyl Alcohol | T | 108 | 79,77 | Naphthalene-d8 | IS | 136 | 68 |
| Bis(2-chloroethoxy) methane | T | 93 | 95,123 | Perylene-d12 | IS | 264 | 260,265 |
| Bis(2-Chloroethyl) ether | T | 93 | 63,95 | Phenanthrene-d10 | IS | 188 | 94,80 |
| bis(2-chloroisopropyl) ether | T | 45 | 77,121 | | | | |

T - TARGET COMPOUND S - SURROGATE IS - INTERNAL STANDARD

SEMIVOLATILES INTERNAL STANDARDS WITH CORRESPONDING TARGET COMPOUNDS
 AND SURROGATES ASSIGNED FOR QUANTITATION

| 1,4-Dichlorobenzene-d4 | Naphthalene-d8 | Acenaphthene-d10 | Phenanthrene-d10 | Chrysene-d12 | Perylene-d12 |
|-------------------------------|----------------------------|----------------------------|----------------------------|----------------------------|------------------------|
| Phenol | Nitrobenzene | Hexachlorocyclopentadiene | 4-Bromophenyl phenol ether | Pyrene | Di-n-octyl-phthalate |
| Bis(2-Chloroethyl)ether | Isophorone | 2,4,6-Trichlorophenol | Hexachlorobenzene | Butylbenzylphthalate | Benzo(b)fluoranthene |
| 2-Chlorophenol | 2-Nitrophenol | 2,4,5-Trichlorophenol | Pentachlorophenol | 3,3'-Dichlorobenzidine | Benzo(k)fluoranthene |
| 1,3-Dichlorobenzene | 2,4-Dimethylphenol | 2-Chloronaphthalene | Carbazole | Benzo(a)anthracene | Benzo(a)pyrene |
| 1,4-Dichlorobenzene | Bis(2-Chloroethoxy)methane | 2-Nitroaniline | Phenanthrene | Bis(2-ethylhexyl)phthalate | Indeno(1,2,3-cd)pyrene |
| 1,2-Dichlorobenzene | 2,4-Dichlorophenol | Dimethylphthalate | Anthracene | Chrysene | Benzo(g,h,l)perylene |
| 2-Methylphenol | 1,2,4-Trichlorobenzene | Acenaphthylene | Di-n-butylphthalate | Terphenyl-d14 (surr) | Dibenzo(a,h)anthracene |
| 4-Methylphenol | Naphthalene | 3-Nitroaniline | Fluoranthene | Benzidine | |
| N-Nitroso-di-n-propylamine | 4-Chloroaniline | Acenaphthene | | | |
| Hexachloroethane | Hexachlorobutadiene | 2,4-Dinitrophenol | | | |
| 2-Fluorophenol (surr) | 2-Chloro-3-methylphenol | 4-Nitrophenol | | | |
| Phenol-d5 (surr) | 2-Methylnaphthalene | Dibenzofuran | | | |
| 1,2-Dichlorobenzene-d4 (surr) | Nitrobenzene-d5 (surr) | 2,4-Dinitrotoluene | | | |
| Aniline | Benzoic Acid | 2,6-Dinitrotoluene | | | |
| Benzyl Alcohol | | Diethylphthalate | | | |
| Bis(2-chloroisopropyl)ether | | 4-Chlorophenylphenylether | | | |
| N-Nitrosodimethylamine | | Fluorene | | | |
| Pyridine | | 4-Nitroaniline | | | |
| | | 2-Fluorobiphenyl (surr) | | | |
| | | 4,6-Dinitro-2-methylphenol | | | |
| | | Azobenzene | | | |
| | | N-Nitrosodiphenylamine | | | |
| | | 2,4,6-Tribromophenol(surr) | | | |

SEMIVOLATILES TARGET COMPOUND LIST & REPORTING LIMITS

| PARAMETER | REPORTING LIMITS | | PARAMETER | REPORTING LIMITS | |
|----------------------------|------------------|-------|-----------------------------|------------------|-------|
| | µg/L | µg/Kg | | µg/L | µg/Kg |
| 1,2,4-Trichlorobenzene | 10 | 330 | Benzo(g,h,i)perylene | 10 | 330 |
| 1,2-Dichlorobenzene | 10 | 330 | Benzo(k)fluoranthene | 10 | 330 |
| 1,3-Dichlorobenzene | 10 | 330 | Benzoic Acid | 20 | 420 |
| 1,4-Dichlorobenzene | 10 | 330 | Benzyl Alcohol | 10 | 330 |
| 2,4,5-Trichlorophenol | 10 | 330 | bis(2-chloroethoxy)methane | 10 | 330 |
| 2,4,6-Trichlorophenol | 10 | 330 | bis(2-chloroethyl)ether | 10 | 330 |
| 2,4-Dichlorophenol | 10 | 330 | bis(2-chloroisopropyl)ether | 10 | 330 |
| 2,4-Dimethylphenol | 10 | 330 | bis(2-Ethylhexyl)phthalate | 10 | 330 |
| 2,4-Dinitrophenol | 20 | 660 | Butylbenzylphthalate | 10 | 330 |
| 2,4-Dinitrotoluene | 10 | 330 | Carbazole | 10 | 330 |
| 2,6-Dinitrotoluene | 10 | 330 | Chrysene | 10 | 330 |
| 2-Chloronaphthalene | 10 | 330 | Di-n-butylphthalate | 10 | 330 |
| 2-Chlorophenol | 10 | 330 | Di-n-octylphthalate | 10 | 330 |
| 2-Methylnaphthalene | 10 | 330 | Dibenzo(a,h)anthracene | 10 | 330 |
| 2-Methylphenol | 10 | 330 | Dibenzofuran | 10 | 330 |
| 2-Nitroaniline | 10 | 330 | Diethylphthalate | 10 | 330 |
| 2-Nitrophenol | 10 | 330 | Dimethylphthalate | 10 | 330 |
| 3,3'-Dichlorobenzidine | 10 | 330 | Fluoranthene | 10 | 330 |
| 3-Nitroaniline | 10 | 330 | Fluorene | 10 | 330 |
| 4,6-Dinitro-2-methylphenol | 20 | 660 | Hexachlorobenzene | 10 | 330 |
| 4-Bromophenyl-phenylether | 10 | 330 | Hexachlorobutadiene | 10 | 330 |
| 4-Chloro-3-methylphenol | 10 | 330 | Hexachlorocyclopentadiene | 10 | 330 |
| 4-Chloroaniline | 10 | 330 | Hexachloroethane | 10 | 330 |
| 4-Chlorophenyl-phenylether | 10 | 330 | Indeno(1,2,3-cd)pyrene | 10 | 330 |
| 4-Methylphenol | 10 | 330 | Isophorone | 10 | 330 |
| 4-Nitroaniline | 10 | 330 | n-Nitroso-di-n-propylamine | 10 | 330 |
| 4-Nitrophenol | 20 | 660 | n-Nitrosodimethylamine | 10 | 330 |
| Acenaphthene | 10 | 330 | n-Nitrosodiphenylamine | 10 | 330 |
| Acenaphthylene | 10 | 330 | Naphthalene | 10 | 330 |
| Aniline | 20 | 660 | Nitrobenzene | 10 | 330 |
| Anthracene | 10 | 330 | Pentachlorophenol | 20 | 660 |
| Azobenzene | 10 | 330 | Phenanthrene | 10 | 330 |
| Benzidine | 50 | 830 | Phenol | 10 | 330 |
| Benzo(a)anthracene | 10 | 330 | Pyrene | 10 | 330 |
| benzo(a)pyrene | 10 | 330 | Pyridine | 20 | 400 |
| Benzo(b)fluoranthene | 10 | 330 | | | |

ANALYSIS RUN LOG FOR SEMIVOLATILES

SOP o> EMAX-8270 Rev. No. 3 o EMAX-8270SIM Rev. No. 0 o EMAX-CLPSVOA o EMAX-M8270SIM Rev. No. 1 o

Book #41- 011

Method File: _____ Tune File: _____ Start Date/Time: _____ End Date/Time: _____

| Preparative Batch | Data File Name | Run ID | DF | Matrix | | Notes | Instrument No: 41 | |
|-------------------|----------------|--------|----|--------|---|-------|---------------------------------|------|
| | | | | S | W | | INITIAL CALIBRATION REFERENCE | |
| | | | | | | | Date | |
| | | | | | | | ICAL ID | |
| | | | | | | | Standards | |
| | | | | | | | Name | ID |
| | | | | | | | Conc. (mg/L) | |
| | | | | | | | DFTPP | |
| | | | | | | | DCC | |
| | | | | | | | INT. STD. | |
| | | | | | | | Solvent | ID |
| | | | | | | | CH ₂ Cl ₂ | |
| | | | | | | | DATA FILE | |
| | | | | | | | Electronic Data Archival | |
| | | | | | | | Location | Date |
| | | | | | | | HPCHEM_SVOA/T041 | |
| | | | | | | | Comments: | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | Analyzed By: | |
| | | | | | | | Date Disposed: | |
| | | | | | | | Disposed by: | |

This page is checked during data review.

EMAX LABORATORIES, INC. 1835 W. 20th St. Torrance, CA 90501

ANALYTICAL BATCH _____

EXTRACTION LOG FOR SEMIVOLATILES

SOP o EMAX-3540 Rev. No.: 0 o EMAX-3510 Rev. No.: 1 o EMAX-3550 Rev. No.: 1 o EMAX-3520 Rev. No.: 2 o EMAX-CLP-SVOA o Book # ESV-026

Matrix: Initial Start Date/Time: End Date/Time: Final Start Date/Time: End Date/Time:

| Sample Prep ID | Lab Sample ID | Sonicator Number | Sample Amount (g/ml) | pH | Extract Volume (ml) | Clean-up [G] [F] [A] [C] | Notes | Standards | ID | Amount Added (ml) |
|----------------|---------------|------------------|----------------------|----|---------------------|--------------------------|-------|---|-------------------------------------|--------------------------|
| *01 | | | | | | | | Surrogate | | |
| *02 | | | | | | | | LCS/MS | | |
| *03 | | | | | | | | | | |
| *04 | | | | | | | | Reagent | Lot# / ID | |
| *05 | | | | | | | | CH ₂ Cl ₂ | | |
| *06 | | | | | | | | Na ₂ SO ₄ | | |
| *07 | | | | | | | | H ₂ SO ₄ | | |
| *08 | | | | | | | | NaOH | | |
| *09 | | | | | | | | Silica Sand | | |
| *10 | | | | | | | | TUNING | | |
| *11 | | | | | | | | Sonicator # | Reading | |
| *12 | | | | | | | | | | |
| *13 | | | | | | | | | | |
| *14 | | | | | | | | | | |
| *15 | | | | | | | | | | |
| *16 | | | | | | | | Concentrator | Water Bath Temperature Setting (°C) | Thermometer Reading (°C) |
| *17 | | | | | | | | 1 | | |
| *18 | | | | | | | | 2 | | |
| *19 | | | | | | | | 3 | | |
| *20 | | | | | | | | 4 | | |
| *21 | | | | | | | | 5 | | |
| *22 | | | | | | | | 6 | | |
| *23 | | | | | | | | Comments: Thermometer ID = T ₁ | | |
| *24 | | | | | | | | Prepared By: _____ | Witnessed By: _____ | |
| *25 | | | | | | | | Standard Added By: _____ | | |
| *26 | | | | | | | | Checked By: _____ | | |
| *27 | | | | | | | | Extract Received by: _____ | Location: _____ | |
| *28 | | | | | | | | Disposed by: _____ | Disposed on: _____ | |

PREPARATION BATCH: *

Clean-up Legend :[G]=GPC [A]=Acid [F]=Florisisl [S]=Silica

STANDARD OPERATING PROCEDURES

AK GASOLINE RANGE ORGANICS

SOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08
 Prepared By: Lucita Arzadon *L.P. Arzadon* Date: 03/31/08
 Approved By: Kenette Pimentel *Kenette Pimentel* Date: 03.31.08
 QA Manager
 Approved By: Caspar Pang *Caspar Pang* Date: 3/31/08
 Laboratory Director

Control Number: AK 101-01-

1.0 SCOPE AND APPLICATION

- 1.1. This method is used to determine the concentration of Gasoline Range Organics (GRO) in soil and water. This corresponds to volatile organic compounds with a comparable hydrocarbon range from beginning of C₆ peak to the beginning of the C₁₀ peak and to a boiling point range between approximately 60°C and 170°C. This method is an adaptation of Method AK 101.

2.0 SUMMARY OF METHOD

- 2.1. A known amount of sample is purged by inert gas and a specific trap retains volatile organic compounds. The trap is back flushed into the GC system equipped with flame ionization detector (FID). The instrument is calibrated with a gasoline standard for start of C₆ peak to start of the C₁₀ peak. Integration is performed using forced baseline-baseline integration.
- 2.2. Other non-petroleum compounds with similar characteristics and boiling points may also be detected with this method.
- 2.3. **Interferences**
- 2.3.1. Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to use.
- 2.3.2. Carry-over from a highly concentrated sample can be a potential source of contamination. Instrument performance must be observed keenly for possible carry-over. If this is apparent, inject solvent blank until no trace of carry-over is observed.
- 2.3.3. Deposits may adhere in the injection port/glass liner over a period of time and can cause interference. The injection port and glass liner must be routinely cleaned.
- 2.3.4. High moisture content in soil samples may cause moisture dilution resulting in results biased low. Moisture dilution is dilution of methanol preservative by moisture contained in the sample.

3.0 METHOD DETECTION LIMITS AND REPORTING LIMITS**3.1. Method Detection Limit**

- 3.1.1. Prepare a minimum of seven samples for each matrix at 0.1-mg/L (or 0.05-mg/L) spike level for 5-ml purge. Prepare a method blank and LFB as described in Section 10.
- 3.1.2. Analyze the samples as described in Section 10.4 and calculate the results as described in

STANDARD OPERATING PROCEDURES

AK GASOLINE RANGE ORGANICSSOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08

Section 10.6

3.1.3. Refer to EMAX-QA04 for MDL evaluation and verification.

3.2. **Practical Quantitation Limit (PQL)**

3.2.1. Detection level is equal to five times the MDL, unless otherwise specified by the project.

4.0 DYNAMIC RANGE

4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (refer to Section 9.7.1). Dilute and reanalyze all samples having results above this range for proper quantitation.

4.2. The lowest quantifiable range of diluted samples is equal to the lowest calibration point (refer to Section 9.7.1.) Lower the dilution factor and reanalyzed all diluted samples analyzed below this range for proper quantitation.

5.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIME

| Matrix | Container | Preservative | Holding Time |
|-----------------------------------|--|---|------------------------|
| Water | 40-ml glass vials protected from light. | pH < 2 with HCl Zero Headspace 4°C ±2 °C | 14 days |
| Soil | Pre-weighed jar with methanol- surrogate solution. Soil/Solvent ratio = 1:1 (w:w) | Soil must be totally submerged in Methanol-Surrogate solution. 25 °C | 28 days |
| Sample for Moisture Determination | Moisture proof | 4°C ±2 °C | 24-hours from delivery |

Note: Samples must be accompanied with Trip Blanks. Allow soil samples to equilibrate with methanol for at least 48 hours from preservation time. Soil samples must be accompanied with a sample for moisture determination.

6.0 ASSOCIATED SOPs

- 6.1. EMAX-5030 Purge and Trap
- 6.2. EMAX-5035 Closed System Purge and Trap
- 6.3. EMAX-QC02 Standard Preparation
- 6.4. EMAX-QC07 Glassware Cleaning

STANDARD OPERATING PROCEDURES

AK GASOLINE RANGE ORGANICSSOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08

- 6.5. EMAX-SM04 Analytical and QC Sample Labeling
- 6.6. EMAX-DM01 Data Flow and Review
- 6.7. EMAX-MCD Moisture Content Determination
- 6.8. EMAX-QA08 Corrective Action
- 6.9. EMAX-QA04 Method Detection Limit Study
- 6.10. EMAX-SM03 Waste Disposal

7.0 SAFETY

- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure.
- 7.3. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES**8.1. Instruments and Supplies**

| | |
|-----------------------|--|
| Purge and Trap System | Archon or equivalent |
| Gas Chromatography | HP 5890 Series II, GC with FID |
| Column | DB5 – 30m x .53 mm, 1.5 µm thickness, or equivalent |
| Gas | ultra-high purity helium ultra-high purity hydrogen, compressed air |
| Syringes | 5 ml Luerlok hypodermic gas-tight with shut-off valve |
| Microsyringes | 10,25,and 100 ul with a 0.006 mm ID needle (Hamilton 702N or equivalent) for dilution purposes |
| Data System | EZ-Chrom |
| Purge Trap | Supelco, Trap “G” or equivalent |

9.0 STANDARDS**9.1. Standard Preparation**

- 9.1.1. Follow the general guidelines for standard preparation as described in EMAX-QC02.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

STANDARD OPERATING PROCEDURES

AK GASOLINE RANGE ORGANICSSOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08**9.2. Stock Standard**

- 9.2.1. Purchase stock standards as certified solutions from two different vendors. Use one as primary standard and the other as secondary standard.
- 9.2.2. Transfer standards on a properly labeled inert vial with minimal headspace and store it at -10 °C to -20 °C.
- 9.2.3. Prepare calibration standards from the primary standard.
- 9.2.4. Prepare initial calibration verification standards and spiking standards from the secondary standard.

9.3. Calibration Standards

| Name | Source | Catalog # | Conc. (mg/L) | Notes |
|-------------------------|--------|-----------|--------------|---------------|
| Unleaded Gas Comp. Std. | Restek | 30081 | 2500 | or equivalent |

9.4. Surrogate Standards

| Name | Source | Catalog # | Conc. ((mg/L) | Notes |
|------------------------|------------------|-----------|---------------|---------------|
| Bromofluorobenzene/TFT | Ultra Scientific | STM-410 | 2000 | or equivalent |

9.5. LFB/Matrix Spike Standard

| Name | Source | Catalog # | Conc. (mg/L) | Notes |
|----------------------------|--------------|------------------|--------------|---------------|
| Cert. BTEX in Unleaded Gas | Accustandard | AK101.0-GCS-BTEX | 5000 | or equivalent |

9.6. Intermediate Standard

Using the stock standard solution, prepare in methanol and store with minimal headspace in an inert vial. Prepare secondary dilution standards from the stock standards at concentration levels as follows:

| | |
|------------------|-----------|
| Gasoline | 2500 mg/L |
| Surrogate | 50 mg/L |
| LFB/Matrix Spike | 5500 mg/L |

9.7. Working Standard**9.7.1. Gasoline Calibration Standard (GCS)**

Prepare initial calibration standards in 5 ml organic free water as suggested below:

| Standard | 2500 mg/L | 50 mg/L (BFB/TFT) | Final Conc. (gas, ug/L) |
|----------|-----------|-------------------|-------------------------|
| 1 | 0.04 µl | 1 µl | 20/10 |
| 2 | 0.1µl | 2 µl | 50/20 |
| 3 | 0.2 µl | 3 µl | 100/30 |
| 4 | 1 µl | 4 µl | 500/40 |
| 5 | 2 µl | 6 µl | 2000/60 |

STANDARD OPERATING PROCEDURES

AK GASOLINE RANGE ORGANICS

SOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08

9.7.2. Initial and Continuing Calibration Standard (CVS/CCS)

9.7.2.1. For ICV, prepare an intermediate standard at 5000 mg/L from the secondary stock standard. Use this standard to spike CVS sample.

9.7.2.2. For CCS, prepare an intermediate standard at 1000 mg/L from the primary stock standard. Use this standard to spike CCS samples.

9.8. **Retention Time Window Standard**

9.8.1. Prepare a standard containing a mixture of C₆ and C₁₀ at 2000 mg/L for retention time analysis. Use this standard to spike RTW sample.

10.0 PROCEDURES**10.1. Sample Preparation**

10.1.1. For water samples, measure 5-ml of sample and transfer into a clean 40-ml vial. Spike the sample with 4µL of 50 mg/L of surrogate standard.

10.1.2. For LFB, spike 5-ml reagent water with 0.5 µL of 5000 mg/L spike standard and 4µL of 50 mg/L of surrogate standard.

10.1.3. For MS, spike 5-ml water sample with 0.5 µL of 5000 mg/L spike standard and 4µL of 50 mg/L of surrogate standard.

10.1.4. For soil samples, weigh the sample jar and record the weight to the nearest 0.1g.

10.1.5. Record the sample preservation time of the sample. For best results allow the sample to equilibrate with methanol for at least 48 hours¹.

10.1.6. Gently swirl the jar and then allow the sediment to settle. Measure and transfer 100-µl of extract into 5-ml reagent water.

10.1.7. Spike each sample with 40 µg/L surrogate solution. For MS sample, spike 0.5 µL of 5000 mg/L spike standard and 4µL of 50 mg/L of surrogate standard.

10.1.8. For soil LFB, extract the total volatile organic of the five (5)-gm soils with methanol and treat the extract as a regular sample with 0.5µL of 5000 mg/L and 4 µL of 50 mg/L of surrogate standard.

10.1.9. For soil samples that are not preserved with methanol-surrogate solution, gently mix each sample. Weigh 25-g of sample into a tared 40-ml vial and record the weight to the nearest 0.1g. Add 25-ml of methanol-surrogate solution, cap and swirl for 2 minutes. Treat the sample extracts similarly as the samples preserved with methanol-surrogate solution.

Note: Qualify results of samples not preserved with methanol as estimates"²

10.1.10. For Methanol Soluble Samples or High Contaminated Soil Samples

10.1.10.1. Measure and transfer 1-g of sample into 20-ml vial. Record the weight to the nearest 0.1g.

¹ AK101, Section 9.3.5

² AK101, Section 9.4.1

STANDARD OPERATING PROCEDURES

AK GASOLINE RANGE ORGANICSSOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08

10.1.10.2. Add 5-ml of methanol-surrogate solution. Cap the vial and swirl for 2-min. Treat the extract as the samples preserved with methanol-surrogate solution.

10.1.11. Moisture Determination

10.1.11.1. Weigh a numbered moisture pan and record the number and weight of the pan.

10.1.11.2. Weigh about 5-10 g of the sample into the pre-weighed moisture pan.

10.1.11.3. Dry the samples overnight at 105°C.

10.1.11.4. Remove the samples from the oven and cool in a desiccator until sample reaches room temperature. Weigh the dried samples.

10.1.11.5. Record all weights to the nearest 0.01g.

10.2. Instrument Parameters

10.2.1. Gas Chromatographic Condition:

| | | | | |
|----------------------------------|---|---------------|------------------|------------------|
| Carrier gas flow (column) helium | 9-10 ml/min | | | |
| Make up gas (He) | 20-21 ml/min | | | |
| Helium Tank | 80 psi | | | |
| Detector Temperature | Detector A: 220°C; Detector B: 235°C | | | |
| Temperature programming: | Initial temperature: 35°C Initial time: 6 minutes | | | |
| | Level | Rate (°C/min) | Initial Temp(°C) | Final Time (min) |
| | 1 | 8.00 | 70 | 0 |
| | 2(A) | 5.0 | 120 | 0 |
| | 2(B) | 25.0 | 230 | 2.00 |

10.2.2. Purge and Trap Condition

| | |
|--------|-----------------------------|
| Purge | 10 min. at room temperature |
| Desorb | 2 min. at 180°C |
| Bake | 14 min. at 185°C |

10.3. Calibration

10.3.1. Gasoline Calibration (GCS)

10.3.1.1. Prepare initial calibration solution as described in Section 9.7.1. Analyze them as described in Section 10.4.

10.3.1.2. Refer to Section 10.5 for calculation.

10.3.1.3. Acceptance criteria are specified in Appendix 1.

10.3.1.4. Verify the initial calibration by a second source standard.

10.3.2. Gasoline Calibration Verification (GCV)

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10.3.2.1. Spike 5-ml water reagent with 0.5 µL of 5000 mg/L GCV standard and 8µL of 50 mg/L of surrogate standard. The sample result is expected to yield 1000 µg/L/40µg/L (±25%).

10.3.2.2. Analyze the GCV sample to verify the concentration of the ICAL. See Appendix 1 for acceptance criteria.

10.3.3. Continuing Calibration (CCS)

10.3.3.1. Spike 5-ml water reagent with 1.0 µL of 1000 mg/L CCS standard and 8µL of 50 mg/L of surrogate standard. The sample result is expected to yield 1000 µg/L/40µg/L (±25%).

10.3.3.2. Analyze the CCS sample to verify the validity of the ICAL. See Appendix 1 for acceptance criteria.

10.3.4. Retention Time Window Check (RTW)

10.3.4.1. Spike 5-ml water reagent with 0.5 µL of 2000 mg/L RTW standard. The sample result is expected to yield 10 mg/L for each analyte.

10.3.4.2. Analyze the RTW check sample to monitor retention time drift. See Appendix 1 for acceptance criteria.

10.4. **Analysis**

10.4.1. Analytical Sequence

10.4.1.1. Assuming that there is an existing initial calibration, for every 20 field samples, set the analytical sequence as follows:

10.4.1.1.1. Reagent Blank

10.4.1.1.2. RTW Window sample

10.4.1.1.3. Opening CCS

10.4.1.1.4. Method Blank

10.4.1.1.5. Field samples to include field blanks, field duplicates, matrix spikes (only when requested)

10.4.1.1.6. Lab Fortified Blank (LFB)

10.4.1.1.7. Closing CCS

10.4.2. Sample Result Evaluation

10.4.2.1. Check that surrogates are within the control limits. Refer to Appendix 1 for acceptance criteria and corrective action.

10.4.2.2. Dilute and re-analyze samples having concentrations greater than the highest calibration range.

10.4.2.3. Dilute and re-analyze samples having saturated peak(s) within C₆ to C₁₀. See Figure 1 for typical saturated peak.

10.4.2.4. Re-analyze samples suspected of carry-over from a preceding sample that has high concentration.

10.4.3. Identification and Quantitation

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- 10.4.3.1. Identification is based on pattern recognition. Refer to Figure 2a for typical gasoline pattern.
- 10.4.3.2. Compare sample chromatograms to reference gasoline standard chromatograms for their response hydrocarbon range and peak distribution to determine if the result resembles gasoline pattern.
- 10.4.3.3. When the elution profile of a sample does not match that of gasoline standard pattern, but falls within the retention time window, quantitate results as gasoline range organics (GRO) and denote the observed deviation in case narrative.
- 10.4.3.4. Integrate the total peak area response eluting from the peak start time for C₆ (hexane) and the peak start time for C₁₀ (decane).

10.5. Establishing Retention Time Window (RTW)

- 10.4.3 Analyze the RTW standard (refer to 10.3.4) daily within 72 hours.
- 10.4.4 Calculate the standard deviation (SD) of the RT for each analyte. The absolute RT window is established by $\pm 3 * SD$.
- 10.4.5 The lower limit of the RT window C₆ for the first eluting component and the upper limit of the RT window for C₁₀ for the last eluting compound determines the retention time range of GRO.
- 10.4.6 Update the RT window using the established absolute RT window during initial calibration.
- Note: If cases where the SD=0, use +0.05 min. in place of SD.³*
- 10.4.7 RT window must be (re)established when the following conditions occur:
- Instrument is new
 - Major repair was done
 - GC condition was changed
 - GC column was replaced

10.6. Calculations

- 10.6.1. Initial Calibration
- 10.6.1.1. Calculate the Calibration Factor (CF).

$$CF = \frac{R_a}{C_k} \quad \text{Eq. 10.6.1.1}$$

where:

CF - is the calibration factor

R_a - is the analyte response measured in peak area

C_k - is the known concentration of the analyte in µg/L (H₂O); µg/Kg (Soil)

- 10.6.1.2. Calculate the Standard Deviation

³ AK101, Section 9.9.2.2

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$$SD = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}} \quad \text{Eq. 10.6.1.2}$$

where:

- SD* - is the standard deviation
x_i - is the result at the *i*th measurement
 \bar{x} - is the mean
N - is the number of measurements

10.6.1.3. Calculate the Percent Relative Standard Deviation (%RSD).

$$\%RSD = \left[\frac{SD}{ACF} \right] 100 \quad \text{Eq. 10.6.1.3}$$

where:

- %RSD* - is the percent relative standard deviation
SD - is the standard deviation
ACF - is the average calibration factor

10.6.1.4. Calculate the Average Calibration Factor (ACF)

$$ACF = \frac{\sum CF}{N} \quad \text{Eq. 10.6.1.4.}$$

where:

- ACF* - is the average calibration factor
 $\sum CF$ - is the summation of the calibration factors
N - is the number of calibration points

10.6.2. Calculate the Percent Difference for CCS from ACF

$$\%D = @ \text{abs} \left[\frac{Ck - Cf}{Ck} \right] * 100 \quad \text{Eq. 10.6.2}$$

where:

- %D* - is the percent difference CCS from the ACF

STANDARD OPERATING PROCEDURES

AK GASOLINE RANGE ORGANICSSOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08*C_k* - known concentration of analyte, in µg/L*C_f* - is the concentration found, in µg/L

10.6.3. Calculate Sample Results

$$C = \left[\frac{R_a}{ACF} \right] \left[\frac{V_e}{S_a} \right] DF \quad \text{For water samples} \quad \text{Eq. 10.6.3.1}$$

$$C = \left[\frac{R_a}{ACF} \right] \left[\frac{V_e}{(S_a)(\%S)} \right] DF \quad \text{For soil samples} \quad \text{Eq. 10.6.3.2}$$

where:

C - is the concentration of analyte in µg/L (H₂O), µg/Kg (soil)*R_a* - is the analyte response measured in peak area*ACF* - is the average calibration factor from initial standard calibration*V_e* - is the purgeable volume in mL*S_a* - is the sample amount in mL (H₂O); g (Soil)*DF* - is the dilution factor*%S* - is the percent solid of the sample=(1-MC)*MC* -is the percent moisture content of the sample

$$MC = \frac{(WetWeight - DryWeight)}{(WetWeight - MoisturePan)} (100)$$

10.6.4. Calculate the percent recovery of spike in LFB.

$$\%R = \left[\frac{C_f}{C_o} \right] 100 \quad \text{Eq. 10.6.4}$$

where:

%R - is the percent recovery*C_f* -is the concentration found, in µg/L*C_o* - is the known concentration of spiked solution, in µg/L

10.6.5. Calculate the MS recovery

$$\%R = \left[\frac{(C_f - C_s)}{C_o} \right] 100 \quad \text{Eq. 10.6.5}$$

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AK GASOLINE RANGE ORGANICSSOP No.: EMAX-AK 101 Revision No. 1 Effective Date: 01-Apr-08*where:**%R* - is the percent recovery*C_f* - is the concentration found, in µg/L*C_s* - is the concentration of the sample, in µg/L (H₂O); in µg/Kg (Soil)*C_o* - is the known concentration of spiked solution

10.6.6. Calculate the precision.

$$\% RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq. 10.6.6}$$

*where:**RPD* - is the relative percent recovery*C1* - Measured concentration of the first sample aliquot*C2* - Measured concentration of the second sample aliquot

10.6.7. Calculate the Method Detection Limit

$$MDL = t_7SD \quad \text{Eq10.6.7}$$

*where:**MDL* - is the method detection limit*t₇* - degrees of freedom for 7 measurements, which is 3.14*SD* - is the standard deviation**10.7. Report Generation**

- 10.7.1. Generate the method.txt file using WBDX⁴.exe.
- 10.7.2. Generate Lab Chronicle using Labchrn1.exe
- 10.7.3. Generate the sample results using F1VX¹.exe
- 10.7.4. Generate the QC summary using QCVX¹.exe
- 10.7.5. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.
 - Sample Results
 - LFB Summary
 - MS/MSD Summary

⁴ X - version number

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- ICAL Summary
- GCV Summary
- CCS Summary
- Analysis Log
- Extraction Log

10.8. Data Review

- 10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
- 10.8.2. Check that identifications are done properly.
- 10.8.3. Check that samples results are integrated properly, and results over calibration range are diluted and re-analyzed within the calibration range.
- 10.8.4. Check that presence of saturated peak(s) are diluted, and quantitated properly.
- 10.8.5. Check that suspected carry-overs are confirmed.
- 10.8.6. Generate the case narrative to include discussion of the following as found in the review process:
 - Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples extracted and analyzed within holding time. For non-compliance, state the number of days that the sample(s) was out of holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Method Blank – That MB was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s) affected and the associated sample results were flagged with “B”.
 - Surrogate – That surrogate was added to CCS, CCV, MB, LFB/LFBD, MS/MSD (if applicable) and every sample prior to analysis, and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. sample result form or QC Summary form, and that non-compliant results were indicated by an asterisk “*”.
 - Laboratory Fortified Blank Samples – That LFB/LFBD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Further more, if corrective action is not possible (e.g. no more samples to re-extract) state that results were qualified.
 - Matrix Spike Samples – That MS/MSD (if applicable) were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC

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limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.

- Sample Analysis – that samples were analyzed in conformance to the method and project requirements.
- Other Anomalies (if any) – discussed on a case by case basis concurred by the Supervisor or the Lab Director.

10.8.7. Submit the analysis package for secondary review.

10.9. Preventive Maintenance

- 10.9.1. Perform daily instrument check prior to sample analysis. Refer to AK101-FM – Instrument Maintenance Log.
- 10.9.2. Check the gas flow from time to time to ensure that ideal gas flow is maintained accordingly.
- 10.9.3. Maintain an inventory of instrument parts and supplies for routinely maintenance.

11.0 QUALITY CONTROL**11.1. Sample Preparation**

- 11.1.1. A preparation batch shall consist of a MB, LFB/LFBD, MS/MSD (only when requested) and ≤ 20 field samples.
- 11.1.2. All labwares used in the sample preparation shall be properly treated as specified in EMAX-QC07
- 11.1.3. All solvents and reagents shall undergo quality control check in the stationary laboratory prior to its use.

11.2. Analytical Batch

- 11.2.1. Initial Calibration must be established and verified by daily continuing calibration at the frequency specified in Appendix 1.
- 11.2.2. Analytical batch shall consist of a valid ICAL, RB, RTW check, samples identified in every preparation batch bracketed with opening and closing CCS.
- 11.2.3. A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing a reagent blank.
- 11.2.4. Organic free water shall be used for method blank and LFB for both water and soil matrix.

11.3. Method QC

- 11.3.1. Method Detection Limit Study must be established before the analytical procedure can be used and updated when there is a significant change in instrument, method or personnel.
- 11.3.2. Retention Time Window must be established and updated as described in Section 10.4.3.
- 11.3.3. Demonstration of Capability must be established before the analytical procedure can be used.
- 11.3.4. All analysts conducting this analysis must have documented demonstration of capability.

12.0 CORRECTIVE ACTION

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12.1. Calibration

- 12.1.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:
- 12.1.1.1. If RSD > 25%, check each calibration point. If an outlier exists, re-analyze that calibration point.
- 12.1.1.2. If GVS not within the expected recovery range, review the chromatogram.
- Bias low results are indicative of poor purging or standard degradation.
 - Bias high is indicative of inaccurate standard injection or instrument contamination.
 - Consider preparing a fresh GVS standard and re-analyze the GVS.
- 12.1.1.3. If problem persists, inform the Supervisor prior to re-calibration
- 12.1.2. If the continuing calibration is non-compliant, consider the suggestions described in correcting GVS.
- 12.1.3. If instrument blank/reagent blank is non-compliant, consider the following suggestions to correct the problem:
- 12.1.3.1. Check the reagent water source e.g. same source is used by a similar analysis on a different instrument to rule out reagent contamination.
- 12.1.3.2. Bake the sample concentrator and or GC column for at least 15 min.
- 12.1.3.3. Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.
- 12.1.3.4. If problem persists, inform Supervisor prior to reanalysis.

12.2. Surrogates

- 12.2.1. If surrogates are non-compliant, and are not due to matrix effects, consider the following suggestions to correct the problem:
- 12.2.1.1. Check for calculation errors and that the concentrations of the surrogate solutions are correct.
- High recoveries may be due to co-eluting matrix interference, examine the sample chromatogram
 - Low recoveries may be due to poor purge, check the purge tube with a blank before reanalyzing the sample.
- 12.2.1.2. Check instrument performance to determine if it is within acceptable guidelines.
- 12.2.1.3. Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.3. Sample Preparation QCs

- 12.3.1. If method blank is non-compliant, consider the following suggestions to correct the problem:
- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM, otherwise perform the corrective action as specified in the PSR.
- 12.3.2. If LFB is non-compliant, consider the following suggestions to correct the problem:
- Check for errors in calculation and concentration of the analyte solution

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- Check instrument performance to determine if it is within acceptable guidelines
- Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.3.3. If the Relative percent difference between the LFB results exceeds the control limits, but meets the percent recovery criteria (Refer to Appendix 1), consider the following suggestions to correct the problem:

- Check for errors in calculation and concentration of the analyte solution
- Check instrument performance to determine if it is within acceptable guidelines
- Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.4. Technical Holding Time

12.4.1. If samples are out of technical holding time, fill-out a Non-Conformance Report (NCR) and forward it to the project manager who will consult with the client for further instruction.

12.5. Sample Preservation

12.5.1. If water samples not contained in amber vials needs to be protected from light.

12.5.2. If water samples are not labeled preserved or samples were received out of the expected range of refrigeration, inform the PM for the PM to consult with the client for further instruction.

12.5.3. If water samples are marked preserved and pH is >2, discuss it in the case narrative.

12.5.4. If soil samples were not preserved with methanol-surrogate solution, inform the PM and report the results as estimate (\geq).

13.0 **POLLUTION PREVENTION**

13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.

13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 **WASTE MANAGEMENT**

14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 **SUPPLEMENTARY NOTES**

15.1 **Application of EMAX QC Procedures**

15.1.1 The procedure and QC criteria summarized in this SOP shall be applied to all projects when performing AK-101. Project specific quality control requirements shall take precedence over this SOP.

15.6 **Definition of Terms**

15.6.1 Batch – is a group of samples that are prepared and/or analyzed at the same time using the

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- same lot of reagents. Preparation batch is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. Analytical batch is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.6.2 Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.6.2 Gasoline Calibration Standard (GCS)- is an equal-weight mixture of regular, plus, and premium grades of commercial gasoline, mixed and diluted to appropriate concentrations, used to prepare a standard curve.
- 15.6.2 Calibration Verification Standard (CVS)- is a gasoline quality control standard (Certified, or equivalent) prepared as the GCS of this method but with product from a source other than that used to prepare the GCS. This standard serves as a quality control check to verify the accuracy of calibration,
- 15.6.2 Continuing Calibration Standard (CCS)- is a mid-range working standard diluted from the GCS, used to verify that the analytical system is operating in a manner comparable to that at the time of calibration.
- 15.6.4 Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.6.5 Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.6.7 Matrix – is a component or form of a sample.
- 15.6.8 Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.6.9 Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.6.10 Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.6.10 Laboratory Fortified Blank (LFB)- is a method blank sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. The spike recovery is used to evaluate method control.
- 15.6.13 Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.6.13 Sample/Matrix Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.6.14 Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.6.14 Practical Quantitation Limit (PQL) – is defined as 5 times the MDL

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16.0 REFERENCES

- 16.1. U.S. EPA Method 8015B, SW846, as updated
- 16.2. Corporate QA/QC Manual, as updated.

17.0 FIGURES, APPENDICES AND FORMS

17.1. Figures

- 17.1.1. Figure 1 Peak Evaluation Technique
- 17.1.2. Figure 2a Typical GRO Chromatogram
- 17.1.3. Figure 2b Typical n-Alkane Chromatogram
- 17.1.4. Figure 3 Typical ICAL Summary
- 17.1.5. Figure 4 Typical Continuing Calibration Summary
- 17.1.6. Figure 5 Typical Sample Report
- 17.1.7. Figure 6 Typical LFB/LFBD Summary
- 17.1.8. Figure 7 Typical MS/MSD Summary
- 17.1.9. Figure 8 Typical Case Narrative

17.2. Appendices

- 17.2.1. Appendix 1 Summary of Quality Control Procedures
- 17.2.2. Appendix 2 Demonstration of Capability

17.3 Forms

- 17.3.1 AK101FS Sample Preparation Log
- 17.3.1 AK101 FA - Analytical Run Log
- 17.3.2 AK101 FM - Instrument Maintenance Log

Figure 1 - Peak Evaluation Technique

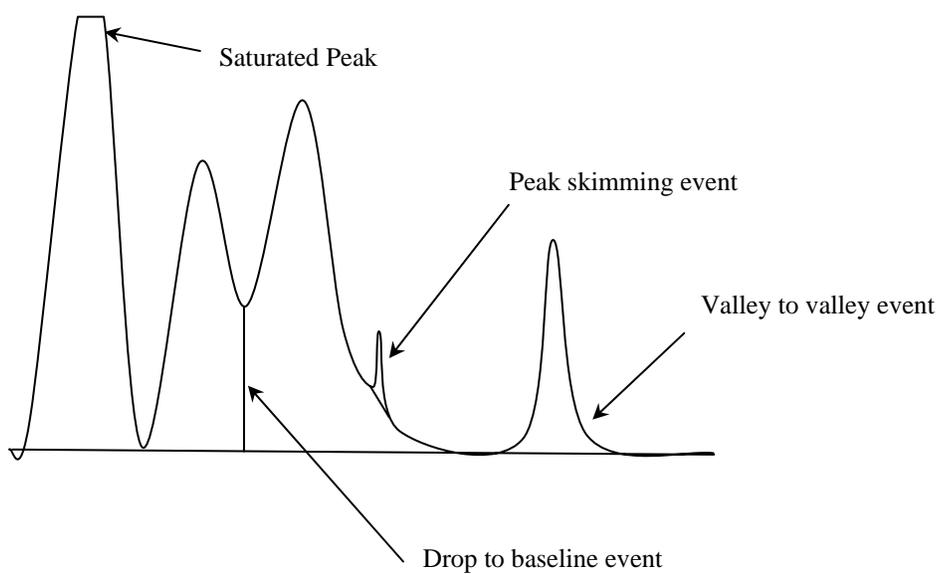


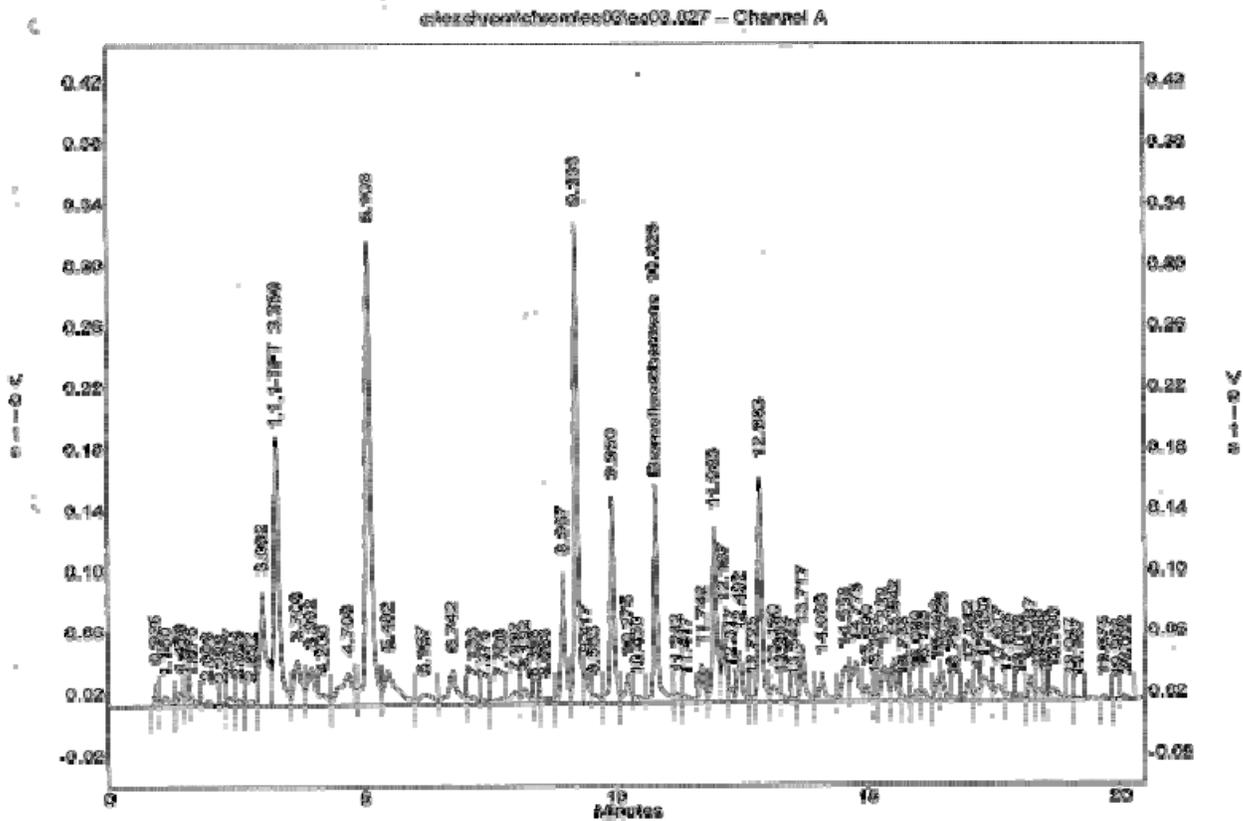
Figure 2a – Typical GRO Chromatogram

Method AE-101 by FID
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\ec03\ec03.027
 Method : c:\ezchrom\methods\vg39c03.met
 Sample ID : IVG39C0302 1000/50
 Acquired : Mar 04, 2006 04:51:45
 Printed : Mar 06, 2006 12:25:07
 User : SERGIO

Channel A Results

| # | Peak Name | Ret. Time (Min) | Area | Ave. CF | ESTD Conc. (PPB) |
|----|--------------------|-----------------|------------|---------|------------------|
| 12 | 1,1,1-TFT | 3.350 | 1078738.0 | 21531.8 | 50.10 |
| 35 | Bromofluorobenzene | 10.825 | 814390.0 | 15026.0 | 54.20 |
| G2 | GRO (C6-C10) | | 11260569.0 | 12418.6 | 906.75 |



Handwritten signature: D. 03/06/06

Figure 3 Typical ICAL Summary

INITIAL CALIBRATION
 Method AK-101

Lab Name : EMAX Inc
 Instrument ID : GCT39
 GC Column : DB-5
 Column size ID : 30MX.53MM
 LFIID & Datetime: EC03019A 03/03/06 23:46
 LFIID & Datetime: EC03020A 03/04/06 00:24
 LFIID & Datetime: EC03021A 03/04/06 01:02
 LFIID & Datetime: EC03022A 03/04/06 01:40
 LFIID & Datetime: EC03023A 03/04/06 02:18
 LFIID & Datetime: EC03024A 03/04/06 02:57
 LFIID & Datetime: EC03025A 03/04/06 03:35
 CONC UNIT: ppb

| COMPOUND | CONC X | CALIBRATION FACTORS | | | | | | | MEAN | %RSD |
|------------------------|-----------|---------------------|-------|-------|--------|--------|---------|---------|---------|------|
| | | 1.00X | 2.50X | 5.00X | 25.00X | 50.00X | 100.00X | 150.00X | | |
| GRO(C6-C10) | 20.00 | 9660 | 10361 | 13007 | 12779 | 13750 | 15695 | 13678 | 12418.6 | 13.7 |
| SURROGATE | X | 1.00X | 2.00X | 3.00X | 4.00X | 5.00X | 7.50X | 10.00X | MEAN | %RSD |
| Bromofluorobenzene | 10.00 | 12063 | 13106 | 13108 | 14879 | 17078 | 17312 | 17635 | 15026.0 | 15.5 |
| 1,1,1-Trifluorotoluene | 10.00 | 17166 | 19380 | 19227 | 21362 | 23275 | 24612 | 25700 | 21531.8 | 14.6 |

10/20/07 MET

Figure 4 Typical Continuing Calibration Summary

CONTINUING CALIBRATION VERIFICATION

Method: MS-101

Lab Name : EMAX
 Instrument ID : GC39
 GC Column : DB-5
 Column size ID : 30MX.53MM
 Mid Conc Init LFID & Datetime: EC03023A 03/04/2006 02:18
 Conc Cont LFID & Datetime: EC03027A 03/04/2006 04:51
 CONC UNIT : ppb

| COMPOUND | RT | RT WINDOW | | TRUE CONC | AVERAGE CF | RESULT | | | QL | %D LIMITS |
|------------------------|---------|-----------|--------|-----------|------------|----------|--------|----|----|-----------|
| | MINUTES | FROM | TO | | | AREA | CONC | %D | | |
| GRO(C6-C10) | 0.000 | 0.000 | 0.000 | 1000.0 | 12418.6 | 11260569 | 906.75 | -9 | | 15 |
| SURROGATE | MINUTES | FROM | TO | TRUECON | CF | AREA | CONC | %D | QL | LIMITS |
| Bromofluorobenzene | 10.825 | 10.763 | 10.887 | 50.0 | 15026.0 | 814390 | 54.20 | 0 | | 15 |
| 1,1,1-Trifluorotoluene | 3.350 | 3.249 | 3.451 | 50.0 | 21531.8 | 1078738 | 50.10 | 0 | | 15 |

VG39C03.MET

Figure 5 Typical Sample Report

METHOD AK101 GASOLINE RANGE ORGANICS

Client : XYZ, INC
 Project : CLEAN LAND PROJECT
 Batch No. : 06D008

Matrix : SOIL
 Instrument ID : GCT039

| SAMPLE ID | EMAX SAMPLE ID | RESULTS (mg/kg) | SURR (%) | DLF | MOIST | RL (mg/kg) | MDL (mg/kg) | Analysis DATETIME | Extraction DATETIME | LFID | CAL REF | PREP BATCH | Collection DATETIME | Received DATETIME |
|-------------|-------------------|--------------------|-------------|-----|-------|---------------|----------------|----------------------|------------------------|----------|----------|------------|------------------------|----------------------|
| MBLK1S | VM39C03B | ND | 80 | 1 | NA | 1 | .5 | 04/19/0602:04 | 04/06/0602:04 | ED06065A | EC01063A | VG39C03 | NA | 04/03/06 |
| LCS1S | VM39C03L | 22.2 | 93 | 1 | NA | 1 | .5 | 04/19/0602:39 | 04/06/0602:39 | ED06066A | EC01063A | VG39C03 | NA | 04/03/06 |
| LCD1S | VM39C03C | 22.8 | 94 | 1 | NA | 1 | .5 | 04/19/0603:14 | 04/06/0603:14 | ED06067A | EC01063A | VG39C03 | NA | 04/03/06 |
| MBLK2S | VM39C04B | ND | 83 | 1 | NA | 1 | .5 | 04/19/0618:03 | 04/06/0618:03 | ED06003A | EC03002A | VG39C04 | NA | 04/03/06 |
| LCS2S | VM39C04L | 21.1 | 77 | 1 | NA | 1 | .5 | 04/19/0618:38 | 04/06/0618:38 | ED06004A | EC03002A | VG39C04 | NA | 04/03/06 |
| LCD2S | VM39C04C | 26 | 117 | 1 | NA | 1 | .5 | 04/19/0619:12 | 04/06/0619:12 | ED06005A | EC03002A | VG39C04 | NA | 04/03/06 |
| B-1@5FT | D008-01 | ND | 78 | 1 | 11.6 | 1.1 | .57 | 04/19/0603:48 | 04/06/0603:48 | ED06068A | EC01063A | VG39C03 | 03/31/06 | 04/03/06 |
| B-1@10FT | D008-02 | ND | 81 | 1 | 3.8 | 1 | .52 | 04/19/0604:23 | 04/06/0604:23 | ED06069A | EC01063A | VG39C03 | 03/31/06 | 04/03/06 |
| B-1@15FT | D008-03 | ND | 80 | 1 | 1.9 | 1 | .51 | 04/19/0604:57 | 04/06/0604:57 | ED06070A | EC01063A | VG39C03 | 03/31/06 | 04/03/06 |
| B-3@5FT | D008-04 | ND | 78 | 1 | 3.0 | 1 | .52 | 04/19/0605:32 | 04/06/0605:32 | ED06071A | EC01063A | VG39C03 | 03/31/06 | 04/03/06 |
| B-3@10FT | D008-05 | ND | 75 | 1 | 3.0 | 1 | .52 | 04/19/0606:06 | 04/06/0606:06 | ED06072A | EC01063A | VG39C03 | 03/31/06 | 04/03/06 |
| B-3@15FT | D008-06 | ND | 78 | 1 | 2.1 | 1 | .51 | 04/19/0606:41 | 04/06/0606:41 | ED06073A | EC01063A | VG39C03 | 03/31/06 | 04/03/06 |
| B-6@15FTMS | D008-15M | 24.2 | 94 | 1 | 2.4 | 1.02 | .512 | 04/19/0613:36 | 04/06/0613:36 | ED06085A | EC01075A | VG39C03 | 03/31/06 | 04/03/06 |
| B-6@15FTMSD | D008-15S | 20.5 | 88 | 1 | 2.4 | 1.02 | .512 | 04/19/0614:09 | 04/06/0614:09 | ED06086A | EC01075A | VG39C03 | 03/31/06 | 04/03/06 |

Figure 6 Typical LFB/LFBD Summary

EMAX QUALITY CONTROL DATA LFB/LFBD ANALYSIS

CLIENT: XYZ, INC.
 PROJECT: CLEAN LAND PROJECT
 BATCH NO.: 06D008
 METHOD: METHOD AK 101

```

=====
MATRIX: SOIL % MOISTURE: NA
DILUTION FACTOR: 1 1
SAMPLE ID: MBLK1S
LAB SAMP ID: VG39C03B VG39C03L VG39C03C
LAB FILE ID: ED06065A ED06066A ED06067A
DATE EXTRACTED: 04/06/0602:04 04/06/0602:39 04/06/0603:14 DATE COLLECTED: NA
DATE ANALYZED: 04/19/0602:04 04/19/0602:39 04/19/0603:14 DATE RECEIVED: 04//03/06
PREP. BATCH: VM39C03 VM39C03 VM39C03
CALIB. REF: EC01063A EC01063A EC01063A
  
```

ACCESSION:

| PARAMETER | BLNK RSLT (mg/kg) | SPIKE AMT (mg/kg) | BS RSLT (mg/kg) | BS % REC | SPIKE AMT (mg/kg) | BSD RSLT (mg/kg) | BSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|-----------|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|------------|-----------------|----------------|
| GRO | ND | 25 | 22.2 | 89 | 25 | 22.8 | 91 | 2 | 60-120 | 20 |

```

=====
SURROGATE PARAMETER SPIKE AMT BS RSLT BS SPIKE AMT BSD RSLT BSD QC LIMIT
(mg/kg) (mg/kg) % REC (mg/kg) (mg/kg) % REC (%)
-----
Bromofluorobenzene 2 1.86 93 2 1.87 94 60-120
  
```

Figure 7 Typical MS/MSD Summary

EMAX QUALITY CONTROL DATA MS/MSD ANALYSIS

CLIENT: XYZ, INC
 PROJECT: CLEAN LAND PROJECT
 BATCH NO.: 06D008
 METHOD: METHOD AK 101

```

=====
MATRIX: SOIL % MOISTURE: 2.4
DILUTION FACTOR: 1 1 1
SAMPLE ID: B-6@15FT
LAB SAMP ID: D008-15 D008-15M D008-15S
LAB FILE ID: ED06084A ED06085A ED06086A
DATE EXTRACTED: 04/03/0613:01 04/03/0613:36 04/03/0614:09 DATE COLLECTED: 03/31/06
DATE ANALYZED: 04/19/0613:01 04/19/0613:36 04/19/0614:09 DATE RECEIVED: 04/03/06
PREP. BATCH: VG39C03 VG39C03 VG39C03
CALIB. REF: ED06075A ED06075A ED06075A
  
```

ACCESSION:

| PARAMETER | SMPL RSLT (mg/kg) | SPIKE AMT (mg/kg) | MS RSLT (mg/kg) | MS % REC | SPIKE AMT (mg/kg) | MSD RSLT (mg/kg) | MSD % REC |
|-----------|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|
| GRO | ND | 25.6 | 24.2 | 94 | 25.6 | 20.5 | 80 |

```

=====
SURROGATE PARAMETER SPIKE AMT MS RSLT MS SPIKE AMT MSD RSLT MSD QC LIMIT
(mg/kg) (mg/kg) % REC (mg/kg) (mg/kg) % REC ( % )
-----
Bromofluorobenzene 2.05 1.93 94 2.05 1.79 88 50-150
  
```

Figure 8 Typical Case Narrative

CLIENT: XYZ, INC
PROJECT: CLEAN LAND PROJECT
SDG: 06D008

METHOD AK 101 GASOLINE RANGE ORGANICS

Eight (8) soil samples were received on 04/27/06 for Determination of Gasoline Range Organics (GRO) by Method AK101, version 04/08/02.

1. Holding Time

Samples were extracted and analyzed within holding time.

2. Calibration

Target analyte met calibration requirements

3. Calibration Verification

Target analyte met calibration requirements

4. Method Blank

Method Blank was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement.

5. Lab Fortified Blank/Duplicate Sample

LFB/LFBD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits.

6. Surrogate

Surrogate was added to CCS, CCV, MB, LFB/LFBD and MS/MSD and every sample prior to analysis, and that recoveries met the project QC limits.

7. Matrix Spike/Matrix Spike Duplicate

That MS/MSD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits.

8. Sample Analysis

Samples were analyzed in conformance to the method and project requirements.

SUMMARY OF INHOUSE QUALITY CONTROL PROCEDURES

| QC Procedure | Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | 1 st Rvw | 2 nd Rvw |
|---|---|--|---|-------------------------------|---------------------|---------------------|
| Five-point initial calibration for all analytes | Initially; as needed | RSD \leq 25% | Correct the problem then repeat initial calibration | | | |
| Calibration Verification Standard (CVS) | After initial calibration | Within \pm 25% of expected value | Correct the problem then repeat initial calibration | | | |
| Initial calibration verification (ICV) | Daily, before sample analysis | Within \pm 25% of expected value | Correct the problem then repeat initial calibration | | | |
| Continuing Calibration Samples (CCS) | Every 12 hours of analysis time and at the end of analysis sequence | Within \pm 25% of expected value | Correct the problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification | | | |
| Laboratory Fortified Blank/Duplicate (LFB/LFBD) | Daily, before sample analysis and every 20 samples | Recovery: 60%-120% RPD >20% | Correct the problem and reanalyze all associated samples | | | |
| Method blank | One per preparation batch | No analytes detected \geq PQL | Reprep and reanalyze method blank and all samples processed with the contaminated blank | Apply B to associated samples | | |
| Surrogate spike | Every sample, spiked sample, standard, and method blank | Recovery: 60%-120% (MB,LFB/LFBD, ICV,CVS, CCS); 50%-150% (MS/MSD, field samples) | Correct the problem then re-extract/re-analyze the sample | | | |
| MS/MSD | Analyze as specified by project. | None | None | | | |
| Retention Time Window Standard | Daily or \leq 20 samples per preparation batch | RTW= ABS(Ave RT) \pm 3XSD or For SD=0; RTW: \pm 0.05 min. | Update as necessary | | | |
| Chromatogram | All sample results | Within calibration range NO SATURATED PEAK(s) | Dilute and re-analyze all samples over the calibration range Diluted and re-analyzed all samples demonstrating saturated peak(s) even if the total integrated peaks do not exceed the calibration range. | | | |
| Preservative | All field samples | Water: HCl; 4°C (\pm 2°C) Soil: Methanol-Surrogate; <25°C | Inform PM/Client and discuss in the case narrative Report unpreserved soil as ">" | | | |
| Holding Time | All field samples | Water: 14 days Soil: 28 days | Inform PM/Client and discuss in the case narrative | | | |
| Comments: PQL= 5X MDL | | | | Reviewed By | | |
| | | | | Date | | |

DEMONSTRATION OF CAPABILITY



6390 Joyce Drive Phone 303-940-0033
 # 100 Fax 866-283-0269
 Golden, CO 80403 www.wibby.com

**Final Report - Rapid Return™
 Study: RR-02994**

Opening Date: March 31, 2006 - Closing Date: April 26, 2006

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-618-8888

EPA Lab ID: CA00291

| Alaska GRO in Water (RR-AKGRO-USTW) | | | | | | | Lot #: RR-02994-28 | |
|-------------------------------------|-------------------------|-------------|--------------------|-------|----------------|--------|--------------------|------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 9408 | Gasoline Range Organics | | AK101 | µg/L | 523 | 341 | 335 - 651 | Acceptable |
| Alaska DRO in Water (RR-AKDRO-USTW) | | | | | | | Lot #: RR-02994-27 | |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 9369 | Diesel Range Organics | | AK102 | µg/L | 1310 | 1510 | 1070 - 1510 | Acceptable |
| Alaska GRO in Soil (RR-AKGRO-USTS) | | | | | | | Lot #: RR-02994-30 | |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 9408 | Gasoline Range Organics | | AK101 | mg/kg | 706 | 634 | 528 - 1020 | Acceptable |
| Alaska DRO in Soil (RR-AKDRO-USTS) | | | | | | | Lot #: RR-02994-29 | |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 9369 | Diesel Range Organics | | AK102 | mg/kg | 447 | 577 | 354 - 686 | Acceptable |
| Alaska RRO in Soil (RR-AKRRO-USTS) | | | | | | | Lot #: RR-02994-26 | |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| - | Residual Range Organics | | AK103 | mg/kg | 540 | 410 | 405 - 675 | Acceptable |

AK101FS-SAMPLE PREPARATION LOG

EXTRACTION LOG FOR NONHALOGENATED VOLATILES

SOP EMAX-5035 Rev.#: 1 EMAX-8015G Rev.#: 1 EMAX-BTEXM Rev.#: 1

Book #: E39-011

Matrix: _____ Start Date: _____ Time: _____ End Date: _____ Time: _____

| Data File Name | Lab Sample ID | W ₁ (g) | W _r (g) | W _s (g) | DF | Notes | Standards / Reagents | ID / Lot# | Amount Added (ml) | Conc. (mg/L) |
|----------------|---------------|--------------------|--------------------|--------------------|----|-------|--|-----------|-------------------|--------------|
| * 1 | | | | | | | Surrogate | | | |
| * 2 | | | | | | | LCS/MS | | | |
| * 3 | | | | | | | Methanol | | | |
| * 4 | | | | | | | Silica Sand | | | |
| * 5 | | | | | | | | | | |
| * 6 | | | | | | | | | | |
| * 7 | | | | | | | | | | |
| * 8 | | | | | | | | | | |
| * 9 | | | | | | | W ₁ = Weight of Vial+Solvent | | | |
| * 0 | | | | | | | W _r = Weight of Vial+Solvent+Sample | | | |
| * 1 | | | | | | | W _s = Weight of Sample | | | |
| * 2 | | | | | | | Comments: | | | |
| * 3 | | | | | | | | | | |
| * 4 | | | | | | | | | | |
| * 5 | | | | | | | | | | |
| * 6 | | | | | | | | | | |
| * 7 | | | | | | | | | | |
| * 8 | | | | | | | | | | |
| * 9 | | | | | | | | | | |
| * 0 | | | | | | | | | | |
| * 1 | | | | | | | | | | |
| * 2 | | | | | | | | | | |

Prepared By: _____

Standard Added By: _____

Checked By: _____


EMAX LABORATORIES, INC. 1835 W. 203rd St. Torrance, CA 90501

PREPARATION BATCH # _____

AK101FA-ANALYTICAL RUN LOG

ANALYSIS RUN LOG FOR NONHALOGENATED VOLATILES

SOP: □ □ EMAX-5030B Rev. No. 1 □ □ EMAX-BTEXM Rev. No. 1 □ □ EMAX-8015G Rev. No. 1 □ □

| Starting Date: | Time: | Ending Date: | Time: | Book # A39-024 | | | | | | | |
|--------------------|-----------------|----------------|---------------|----------------|--------------|----|--------|-------|-------------------------------|--------------|-------------|
| ANALYTICAL BATCH * | Sample Prep. ID | Data File Name | Lab Sample ID | Sample Amount | Purge Volume | pH | Matrix | Notes | Instrument No: | 39 | |
| | *01 | | | | | | | | Initial Calibration Reference | | |
| | *02 | | | | | | | | FIDChannel A | PIDChannel B | |
| | *03 | | | | | | | | Method File | | |
| | *04 | | | | | | | | Date | | |
| | *05 | | | | | | | | ICAL ID | | |
| | *06 | | | | | | | | ICV ID | | |
| | *07 | | | | | | | | | | |
| | *08 | | | | | | | | Std. | ID | Conc.(mg/L) |
| | *09 | | | | | | | | DCC GAS | | |
| | *10 | | | | | | | | DCC BTEX | | |
| | *11 | | | | | | | | BFB/TFT | | |
| | *12 | | | | | | | | LCS/LCSD | | |
| | *13 | | | | | | | | MS/MSD | | |
| | *14 | | | | | | | | | | |
| | *15 | | | | | | | | Solvent | ID | |
| | *16 | | | | | | | | Methanol | | |
| | *17 | | | | | | | | Electronic Data Archival | | |
| | *18 | | | | | | | | Location | Date | |
| | *19 | | | | | | | | EZC-3-BTEX | | |
| | *20 | | | | | | | | Comments: _____ | | |
| | *21 | | | | | | | | _____ | | |
| | *22 | | | | | | | | _____ | | |
| | *23 | | | | | | | | _____ | | |
| | *24 | | | | | | | | _____ | | |
| | *25 | | | | | | | | _____ | | |
| | *26 | | | | | | | | _____ | | |
| | *27 | | | | | | | | _____ | | |
| | *28 | | | | | | | | Analyzed By: _____ | | |
| | *29 | | | | | | | | Disposed on: | By: _____ | |
| *30 | | | | | | | | _____ | | | |

STANDARD OPERATING PROCEDURES
AK DIESEL RANGE ORGANICS

SOP No.: EMAX-AK102 Revision No. 2 Effective Date: 01-Aug-09
 Prepared By: Lucita Arzadon *R. R. Arzadon* Date: 07-29-09
 Approved By: Kenette Pimentel *K. Pimentel* Date: 07.29.09
 QA Manager
 Approved By: Caspar Pang *C. Pang* Date: 7/29/09
 Laboratory Director

Control Number: AK102-02-

1.0 SCOPE AND APPLICATION

- 1.1. This method is designed to measure the concentration of Diesel Range Organics (DRO) in water and soil. This method is limited to provide semiquantitative results on those extractable hydrocarbons with a comparable aliphatic hydrocarbon range from the beginning of C₁₀ peak to the beginning of the C₂₅ peak, and a boiling point range of approximately 170°C to 400°C. This is an adaptation of Method AK 102

2.0 SUMMARY OF METHOD

- 2.1. Petroleum hydrocarbons are extracted in methylene chloride, analyzed by flame ionization detector (FID) in gas chromatograph and quantified as diesel fuel from the beginning of C₁₀ peak to the beginning of the C₂₅ peak. The hydrocarbons that fall in this range are defined as Diesel Range Organics (DRO). Integration is performed using forced baseline-baseline integration.
- 2.2. Other non-petroleum compounds with similar characteristics and boiling points may also be detected with this method.
- 2.3. **Interference**
- 2.3.1. Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to its use.
- 2.3.2. Carry-over from a highly concentrated sample can be a potential source of contamination. Instrument performance must be observed keenly for possible carry-over. If this is apparent, inject solvent blank until no trace of carry-over is observed.
- 2.3.3. Deposits may adhere in the injection port/glass liner over a period of time and can cause interference. The injection port and glass liner must be routinely cleaned.

3.0 METHOD DETECTION LIMITS AND QUANTITATION LIMITS

3.1. **Method Detection Limit (MDL)**

- 3.1.1. Prepare a minimum of eight samples. Add spike standard at 10 to 20 mg/L spike level to seven samples and treat one as method blank.
- 3.1.2. Analyze the samples as described in Section 10 and calculate the results as described in Section 10.6.
- 3.1.3. Refer to EMAX QA04 for MDL evaluation and verification.

3.2. **Practical Quantitation Limit**

- 3.2.1. Detection level is equal to five times the MDL, unless otherwise specified by the project.

4.0 DYNAMIC RANGE

- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (See Section 9.7.1). Dilute and reanalyze all samples having results above this range to properly quantitate.

STANDARD OPERATING PROCEDURES
AK DIESEL RANGE ORGANICS

SOP No.: EMAX-AK102 Revision No. 2 Effective Date: 01-Aug-09

- 4.2. The lowest quantifiable range of diluted samples is equal to the lowest calibration point (See Section 9.7.1.) Lower the dilution factor and reanalyzed all diluted samples analyzed below this range to properly quantitate

5.0 SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIME

| Matrix | Container | Preservative | Holding Time |
|--------|--|------------------------------|-------------------------------|
| Water | 1L amber glass with Teflon lined screw caps | pH < 2 with HCl 4°C ±2 °C | Extraction - 14 days |
| Soil | Core tube or 4 or 8 oz amber glass jar with Teflon-lined lid | 4°C ±2 °C | Analyses of extracts - 40days |

6.0 ASSOCIATED SOPs

| | |
|------------|--|
| EMAX-QC02 | Analytical Standard Preparation |
| EMAX-SM04 | Analytical and QC Sample Labeling |
| EMAX-QA04 | Method Detection Limit Study |
| EMAX-QA08 | Corrective Action |
| EMAX-DM01 | Data Flow & Review |
| EMAX-SM03 | Waste Disposal |
| EMAX-LUFTE | Total Petroleum Hydrocarbons by Extraction |
| EMAX-3510 | Extraction, Separatory Funnel |
| EMAX-3520 | Extraction, Continuous Liquid/Liquid |
| EMAX-3540 | Extraction, Soxhlet |
| EMAX-3550 | Extraction , Pulse Sonication |
| EMAX-3580 | Waste Dilution |

7.0 SAFETY

- 7.1. Read all MSDS for chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. Place all wastes generated during analytical process in the waste containers. Endorse these wastes to the waste disposal section for proper disposal.
- 7.4. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

STANDARD OPERATING PROCEDURES
AK DIESEL RANGE ORGANICS

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8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES

8.1. Instruments and Supplies

- 8.1.1. Gas Chromatography: GC HP5890 II with FID and 7673HP Autosampler, or equivalent
- 8.1.2. Detector: FID
- 8.1.3. Column: DB5, (0.25 mm x 30 m) 0.25 μ m thickness, or equivalent
- 8.1.4. Data Acquisition: EZ Chrom, or equivalent
- 8.1.5. Syringes: 10, 25, 100 μ L microsyringe
- 8.1.6. Boiling chips
- 8.1.7. Analytical balance capable of accurately weighing 0.001 g ;top-loading balance capable of weighing to the nearest 0.01 g.
- 8.1.8. Stainless steel spatula
- 8.1.9. Glassware
 - 8.1.9.1. Volumetric Flask: 10, 100 and 1000 mL
 - 8.1.9.2. Vials: 2,10 and 40 ml, amber
 - 8.1.9.3. Separatory funnel-2000 mL with Teflon stopcock
 - 8.1.9.4. Continuous liquid-liquid extractor-equipped with Teflon or glass connecting joints and stopcocks
 - 8.1.9.5. Bottle: 250-ml (amber)
- 8.1.10. Ultrasonic cell disrupter/Horn-type sonicator equipped with a titanium tip.
- 8.1.11. Soxhlet extraction apparatus as described in SW-846, Method 3540.

8.2. Chemicals and Reagents

- 8.2.1. Methylene Chloride; Acetone
- 8.2.2. Sodium Sulfate
- 8.2.3. High purity He, H₂, Air

9.0 STANDARDS

9.1. Standard Preparation

- 9.1.1. The procedure for analytical standard preparation is detailed in EMAX-QC02.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.2. Stock Standard

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AK DIESEL RANGE ORGANICS

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- 9.2.1. Purchase stock standards as certified solutions from two different vendors. Use one as primary standard and the other as secondary standard.
- 9.2.2. Transfer standards on a properly labeled inert vial with minimal headspace and store it at -10 °C to -20 °C.
- 9.2.3. Prepare calibration standards from the primary standard.
- 9.2.4. Prepare initial calibration verification standards and spiking standards from the secondary standard.

9.3. **Calibration Standards**

| Name | Source | Catalog # | Conc. (mg/L) | Notes |
|-----------|--------|-----------|--------------|---------------|
| Diesel #2 | Restek | 31259 | 50,000 | or equivalent |

9.4. **Surrogate Standards**

| Name | Source | Catalog # | Conc. ((mg/L) | Notes |
|-----------------|---------|--------------|---------------|---------------|
| Ortho-terphenyl | Aldrich | AK102 SS-10X | 2000 | or equivalent |

9.5. **LFB/Matrix Spike Standard**

| Name | Source | Catalog # | Conc. (mg/L) | Notes |
|-----------|--------------|-----------|--------------|---------------|
| Diesel #2 | Accustandard | FU-009N | neat | or equivalent |

9.6. **Intermediate Standard**

Using the stock standard solution, prepare in methanol or Acetone and store with minimal headspace in an inert vial. Prepare secondary dilution standards from the stock standards at concentration levels as follows:

| | |
|------------------|-----------|
| Diesel | 5000 mg/L |
| Surrogate | 250 mg/L |
| LFB/Matrix Spike | 5000 mg/L |

9.7. **Working Standard**

9.7.1. Diesel Calibration Standard (DCS)

Prepare initial calibration standards in 1 ml MeCl₂ as suggested below:

| Standard | 50000 mg/L | 250 mg/L Ortho-terphenyl | Final Conc. (mg/L) | |
|----------|------------|--------------------------|---------------------|-----------|
| | | | Diesel | Surrogate |
| 1 | 0.2 µl | 20 µl | 10 | 5 |
| 2 | 2.0 µl | 40µl | 100 | 10 |
| 3 | 10.0 µl | 60 µl | 500 | 15 |
| 4 | 20.0 µl | 100 µl | 1000 | 25 |
| 5 | 30.0 µl | 140 µl | 1500 | 35 |
| 6 | 60.0 µl | 220 µl | 3000 | 55 |

9.7.2. Initial and Continuing Calibration Standard (CVS/CCS)

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AK DIESEL RANGE ORGANICS

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9.7.2.1. For CVS, prepare an intermediate standard at 500 mg/L from the secondary stock standard.

9.7.2.2. For CCS, prepare an intermediate standard at 1,500 mg/L from the primary stock standard.

9.8. Retention Time Window Standard

9.8.1. Prepare a standard containing a mixture of C₁₀ and C₂₅ at 20 mg/L for retention time analysis. Use this standard to spike RTW sample.

10.0 PROCEDURES

10.1. Sample Preparation

10.1.1. The preferred method for water extraction is SW-846 Method 3510 (Separatory Funnel Liquid-Liquid Extraction) and for soil samples Method 3540 (Soxhlet Extraction). Equivalent extraction techniques which meet the quality assurance requirements of this method can be used.

10.1.2. For water samples, measure 1-L of sample and transfer to a 2-L separatory funnel. Spike the sample with 1 mL of 250 mg/L of surrogate standard.

10.1.3. For LFBs and duplicate LFBs spike 1-L reagent water with 1 mL of 5000 mg/L Diesel spike standard and 1 mL of 250 mg/mL of surrogate standard.

10.1.4. For MS, spike 1-L water sample with 1-mL of 5,000 mg/L Diesel spike standard and 1 mL of 250 mg/mL of surrogate standard.

10.1.5. Prepare a method blank using 1-L of reagent water. Spike the sample with 1 mL of 250 mg/mL of surrogate standard.

10.1.6. For water samples, rinse the inner walls of the sample bottle by adding 60 mL methylene chloride after the sample has been transferred to the separatory funnel. Transfer solvent to the separatory funnel. Extract the sample by shaking it for no less than two minutes with frequent ventilation.

10.1.7. Allow the layers to separate (approximately 10 minutes rest after shaking).

10.1.8. Drain the bottom layer (methylene chloride)

10.1.9. Repeat Section 10.1.6 to Section-10.1.8 two more times and concentrate extracts to 10 mL at a temperature not to exceed 40°C.

10.1.10. Transfer extracts to GC vials for analysis. Store extracts in a freezer at <-10°C. Record information for the extraction and concentration steps.

10.1.11. For soil samples, weigh 10 g of sample into an extraction thimble and add an equal weight of anhydrous sodium sulfate and stir the mixture well with a spatula.

10.1.12. Place loaded thimbles in extractors and spike 1 mL of 250 mg/mL of surrogate standard to both field and quality control samples.

10.1.13. For LFBs and duplicate LFBs, spike 10 g. of methylene chloride rinsed Ottawa sand or alternative standard soil with 1 mL at 5000 mg/L of spike standard and 1 mL of 250 mg/mL of surrogate standard.

10.1.14. Prepare a method blank using 10 g. of methylene chloride rinsed Ottawa sand or alternative standard soil. Spike the sample with 1 mL of 250 mg/mL of surrogate standard.

10.1.15. Add 300 mL of methylene chloride to the 500 mL extraction flask. Add boiling chips to the flask. Connect the extractors to the flask and the condenser to the extractor. Extract for 18-24 hours.

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10.1.16. Transfer extract into a clean concentrator tube and concentrate extracts to 10 mL at a temperature not to exceed 40°C. Transfer to GC vials for analysis. Record information for the extraction and concentration steps.

10.1.17. Moisture Determination for Solids

10.1.17.1. Weigh a numbered moisture pan and record the number and weight of the pan.

10.1.17.2. Weigh about 5-10 g of the sample into the pre-weighed moisture pan.

10.1.17.3. Dry the samples overnight at 105°C.

10.1.17.4. Remove the samples from the oven and cool in a desiccator until sample reaches room temperature. Weigh the dried samples.

10.1.17.5. Record all weights to the nearest 0.01g.

10.2. Instrument Parameters

10.2.1. Fine tune the instrument guided by the parameter conditions as listed below:

| Instrument | Temp (°C) | Rate (°C/min) | Time (min) | Temperature (°C) | | Injection Volume (µL) | Head Pressure (psi) |
|------------|-----------|---------------|------------|------------------|----------|-----------------------|---------------------|
| | | | | Injector | Detector | | |
| GC105 | 55 | 0 | 0.5 | 280 | 320 | 2 | 21 |
| | 320 | 60 | 5.08 | | | | |

10.3. Calibration

10.3.1. Diesel Calibration (DCS)

10.3.1.1. Prepare initial calibration solution as described in Section 9.7.1. Analyze them as described in Section 10.4.

10.3.1.2. Refer to Section 10.6 for calculation.

10.3.1.3. Acceptance criteria are specified in Appendix 1.

10.3.1.4. Verify the initial calibration by a second source standard.

10.3.2. Diesel Calibration Verification (CVS)

10.3.2.1. Take 300 µL of intermediate standard at 5000mg/L and 100 µL of 250 mg/L of surrogate standard in 1.0 ml MeCl₂. The sample result is expected to yield 1500 mg/L /25 mg/L (±25%).

10.3.2.2. Analyze the CVS sample to verify the concentration of the ICAL. See Appendix 1 for acceptance criteria.

10.3.3. Continuing Calibration (CCS)

10.3.3.1. Take 30 µL of stock standard at 50,000mg/L of Diesel #2 and 100 µL of 250 mg/L of surrogate standard in 1.0 ml MeCl₂. The sample result is expected to yield 1500 mg/L/25 mg/L (±25%).

10.3.3.2. Analyze the CCS sample to verify the validity of the ICAL. See Appendix 1 for acceptance criteria.

10.3.4. Retention Time Window Check (RTW)

10.3.4.1. Prepare standard containing 1000 mg/L RTW standard.

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10.3.4.2. Analyze the RTW check sample to monitor retention time drift. See Appendix 1 for acceptance criteria.

10.4. Analysis

10.4.1. Extract Preparation

10.4.1.1. Allow the extracts to equilibrate with the room temperature.

10.4.1.2. Transfer about 1-mL of extracts into Autosampler vials.

10.4.2. Analytical Sequence

10.4.2.1. Assuming that there is an existing initial calibration, for every 20 field samples, set the analytical sequence as follows:

10.4.2.1.1. Instrument Blank

10.4.2.1.2. RTW Window sample

10.4.2.1.3. Opening CCS

10.4.2.1.4. Method Blank

10.4.2.1.5. Field samples to include field blanks, field duplicates, matrix spikes (only when requested)

10.4.2.1.6. Lab Fortified Blank (LFB)

10.4.2.1.7. Closing CCS

10.4.3. Sample Result Evaluation

10.4.3.1. Check that surrogates are within the control limits. Refer to Appendix 1 for acceptance criteria and corrective action.

10.4.3.2. Dilute and re-analyze samples having concentrations greater than the highest calibration range.

10.4.3.3. Dilute and re-analyze samples having saturated peak(s) within C₁₀ to C₂₅. See Figure 1 for typical saturated peak.

10.4.3.4. Re-analyze samples suspected of carry-over from a preceding sample that has high concentration.

10.4.4. Identification and Quantitation

10.4.4.1. Identification is based on pattern recognition. Refer to Figure 2a for typical diesel pattern.

10.4.4.2. Compare sample chromatograms to reference diesel standard chromatograms for their response hydrocarbon range and peak distribution to determine if the result resembles diesel pattern.

10.4.4.3. When the elution profile of a sample does not match that of diesel standard pattern, but falls within the retention time window, quantitate results as diesel range organics (DRO) and denote the observed deviation in case narrative.

10.4.4.4. Integrate the total peak area response eluting at the beginning of C₁₀ peak to the beginning of C₂₅ peak baseline to baseline and quantitate the diesel concentration using equation 10.6.4.

10.5. Establishing Retention Time Window (RTW)

10.5.1. Analyze the RTW standard (refer to 10.3.4) daily within 72 hours.

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10.5.2. Calculate the standard deviation (SD) of the RT C₁₀, C₂₅ and surrogate. The absolute RT window is established by $\pm 3 * SD$.

10.5.3. The lower limit of the RT window C₁₀ for the first eluting component and the upper limit of the RT window for C₂₅ for the last eluting compound determine the retention time range of DRO.

10.5.4. Update the RT window using the established absolute RT window during initial calibration.

Note: If cases where the SD=0, use +0.05 min. in place of SD.¹

10.5.5. RT window must be (re)established when the following conditions occur:

- Instrument is new
- Major repair was done
- GC condition was changed
- GC column was replaced

10.6. Calculations

10.6.1. Calculate for Percent Moisture for Soils

$$\% \text{Moisture} = \frac{(A - C)}{(A - B)} * 100 \quad \text{Eq-10.6.1}$$

where:

- A weight of boat + wet sample
 B weight of boat
 C weight of boat + dry sample

10.6.2. Initial Calibration

10.6.2.1. Calculate for Calibration Factor

$$CF = \frac{R_t}{C_v} \quad \text{Eq-10.6.2.1}$$

where:

- CF – Calibration Factor
 R_t – Total response of the integrated peaks
 C_v – Known value of the standard concentration, mg/L

10.6.2.2. Calculate for the Average Calibration Factor

$$ACF = \frac{\sum CF}{n} \quad \text{Eq-10.6.2.2}$$

where:

- ACF – Average Calibration Factor
 $\sum CF$ – Summation of Calibration Factors
 n – Number of measurements

¹ AK101, Section 9.9.2.2

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10.6.2.3. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq-10.6.2.3}$$

where:

- SD – Standard Deviation
- x_i – Result at the i^{th} measurement
- \bar{x} – mean
- n – number of measurements

10.6.2.4. Calculate for Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{ACF} * 100 \quad \text{Eq-10.6.2.4}$$

where:

- $\%RSD$ – Percent Relative Standard Deviation
- SD – Standard Deviation
- ACF – Average Calibration Factor

10.6.3. Calculate for Percent Difference for CCS from ACF

$$\%D = \frac{C_f - C_k}{C_k} * 100 \quad \text{Eq-10.6.3}$$

where:

- $\%D$ – Percent Difference CCS from known concentration
- C_k – Known concentration of the analyte, in mg/L
- C_f – Found concentration, in mg/L

10.6.4. Calculate for Sample Concentration

$$C = \frac{(R_t)(V_e)(DF)}{(ACF)(A_s)(\%S)} \quad \text{Eq-10.6.4}$$

where:

- C – Concentration of the sample, mg/L or mg/Kg
- R_t – Total response of the integrated peaks
- V_e – Volume of Extract, ml
- A_s – Sample amount, ml or g
- DF – Dilution Factor
- ACF – Average Calibration Factor
- $\%S$ – Percent solids (%S for water = 1)

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10.6.5. Calculate for Precision

$$\% RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq-10.6.5}$$

where:

- RPD* – Relative Percent Difference
- C1* – Measured concentration of the first sample aliquot
- C2* – Measured concentration of the second sample aliquot

10.7. Report Generation

- 10.7.1. Generate the method.txt file using WDBX².exe.
- 10.7.2. Generate Lab Chronicle using LABCHRNX.exe
- 10.7.3. Generate the sample results using F1VX.exe
- 10.7.4. Generate the QC summary using QCVX.exe
- 10.7.5. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.
 - Sample Results
 - LFB Summary
 - MS/MSD Summary
 - ICAL Summary
 - CCV Summary
 - CCS Summary
 - Analysis Log
 - Extraction Log

10.8. Date Review

- 10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
- 10.8.2. Generate the case narrative to include discussion of the following as found in the review process:
 - Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples extracted and analyzed within holding time. For non-compliance, state the number of days that the sample(s) was out of holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.

²X - version number

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- Method Blank – That MB was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s) affected and the associated sample results were flagged with “B”.
- Surrogate – That surrogate was added to CCS, CCV, MB, LFB/LFBD, MS/MSD (if applicable) and every sample prior to analysis, and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. sample result form or QC Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Laboratory Fortified Blank Samples – That LFB/LFBD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Further more, if corrective action is not possible (e.g. no more samples to re-extract) state that results were qualified.
- Matrix Spike Samples – That MS/MSD (if applicable) were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Sample Analysis – that samples were analyzed in conformance to the method and project requirements.
- Other Anomalies (if any) – discussed on a case by case basis concurred by the Supervisor or the Lab Director.

10.8.3. Submit the analysis package for secondary review.

10.9. Preventive Maintenance

- 10.9.1. Perform daily instrument check prior to sample analysis. Refer to AK102-FM – Instrument Maintenance Log.
- 10.9.2. Check the gas flow from time to time to ensure that ideal gas flow is maintained accordingly.
- 10.9.3. Maintain an inventory of instrument parts and supplies for routinely maintenance.

11.0 QUALITY CONTROL

11.1. Sample Preparation

- 11.1.1. A preparation batch shall consist of a MB, LFB/LFBD, MS/MSD (only when requested) and ≤ 20 field samples.
- 11.1.2. All labware used in the sample preparation shall be properly treated as specified in EMAX-QC07
- 11.1.3. All solvents and reagents shall undergo quality control check in the stationary laboratory prior to its use.

11.2. Analytical Batch

- 11.2.1. Initial Calibration must be established and verified by daily continuing calibration at the frequency specified in Appendix 1.
- 11.2.2. Analytical batch shall consist of a valid ICAL, RTW check, samples identified in every preparation batch bracketed with opening and closing CCS.
- 11.2.3. A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing an instrument blank.

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11.2.4. Organic free water shall be used for method blank and LFB for both water and soil matrix.

11.3. Method QC

11.3.1. Method Detection Limit Study must be established before the analytical procedure can be used.

11.3.2. Retention Time Window must be established and updated as described in Section 10.4.3.

11.3.3. Demonstration of Capability must be established before the analytical procedure can be used.

11.3.4. All analysts conducting this analysis must have documented demonstration of capability.

12.0 CORRECTIVE ACTION

12.1. Calibration

12.1.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

12.1.1.1. If RSD > 25%, check each calibration point. If an outlier exists, re-analyze that calibration point.

12.1.1.2. If CVS is not within the expected recovery range, review the chromatogram.

- Bias low results are indicative of poor purging or standard degradation.
- Bias high is indicative of inaccurate standard injection or instrument contamination.
- Consider preparing a fresh CVS standard and re-analyze the CVS.

12.1.1.3. If problem persists, inform the Supervisor prior to re-calibration

12.1.2. If the continuing calibration is non-compliant, consider the suggestions described in correcting CVS.

12.1.3. If instrument blank/reagent blank is non-compliant, consider the following suggestions to correct the problem:

12.1.3.1. Check the reagent water source e.g. same source is used by a similar analysis on a different instrument to rule out reagent contamination.

12.1.3.2. Bake the sample concentrator and or GC column for at least 15 min.

12.1.3.3. Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.1.3.4. If problem persists, inform Supervisor prior to reanalysis.

12.2. Surrogates

12.2.1. If surrogates are non-compliant, and are not due to matrix effects, consider the following suggestions to correct the problem:

12.2.1.1. Check for calculation errors and that the concentrations of the surrogate solutions are correct.

12.2.1.2. Check instrument performance to determine if it is within acceptable guidelines.

12.2.1.3. Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.3. Sample Preparation QCs

12.3.1. If method blank is non-compliant, consider the following suggestions to correct the problem:

- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM, otherwise perform the corrective action as specified in the PSR.

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- 12.3.2. If LFB is non-compliant, consider the following suggestions to correct the problem:
- Check for errors in calculation and concentration of the analyte solution
 - Check instrument performance to determine if it is within acceptable guidelines
 - Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.4. Technical Holding Time

- 12.4.1. If samples are out of technical holding time, fill-out a Non-Conformance Report (NCR) and forward it to the project manager who will consult with the client for further instruction.

12.5. Sample Preservation

- 12.5.1. If water samples not contained in amber vials needs to be protected from light.
- 12.5.2. If water samples are not labeled preserved or samples were received out of the expected range of refrigeration, inform the PM for the PM to consult with the client for further instruction.
- 12.5.3. If water samples are marked preserved and pH is >2, discuss it in the case narrative.

13.0 POLLUTION PREVENTION

- 13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.
- 13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 WASTE MANAGEMENT

- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. Application of EMAX QC Procedures

- 15.1.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing AK-102. Project or program specific quality control shall take precedence over this SOP.

15.2. Definition of Terms

- 15.2.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents. Preparation batch is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. Analytical batch is compose of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.2.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.2.3. Diesel Calibration Standard (DCS)- is a Commercial #2 diesel fuel or equivalent hydrocarbon mixture in which greater than 95% of the hydrocarbon mass elutes within the diesel change diluted to appropriate concentrations in methylene chloride. The DCS serves as a calibration

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standard for DRO.

- 15.2.4. Calibration Verification Standard (CVS)- is a quality control standard but with a diesel range hydrocarbon mixture from a source other than that used to prepare the Diesel Calibration Standard. It is used by the laboratory to verify the accuracy of calibration.
- 15.2.5. Continuing Calibration Standard (CCS)- is a mid-range working standard diluted from the Diesel Calibration Standard, used to verify that the analytical system is operating in a manner comparable to that at the time initial calibration.
- 15.2.6. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.2.7. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.2.8. Matrix – is a component or form of a sample.
- 15.2.9. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.2.10. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.2.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.2.12. Laboratory Fortified Blank (LFB)- is a method blank sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. The spike recovery is used to evaluate method control.
- 15.2.13. Solvent Blank- is a target-analyte-free sample that doesn't go through the procedure and may also serve as an instrument blank. It is analyzed to demonstrate that the solvent used in the method is free from contamination.
- 15.2.14. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.2.15. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.2.16. Surrogate – compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.
- 15.2.17. Practical Quantitation Limit (PQL) – is defined as 5 times the MDL.

16.0 REFERENCES

- 16.1. Method AK 102 ; For Determination of Diesel Range Organics Version 04/08/02
- 16.2. Test Method for Evaluating Solid Waste Physical/Chemical Methods. SW-846, 3rd edition.
- 16.3. Corporate QA/QC Manual, as updated.

17.0 FIGURES, APPENDICES AND FORMS

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17.1. Figures

- | | | |
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| 17.1.2. | Figure 2a | Typical DRO Chromatogram |
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17.2. Appendices

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| 17.2.1. | Appendix 1 | Summary of Quality Control Procedures |
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17.3. Forms

- | | | |
|---------|----------|------------------------------------|
| 17.3.1. | AK102 FA | Typical Analytical Log |
| 17.3.2. | AK102 FM | Typical Instrument Maintenance Log |
| 17.3.3. | AK102 FS | Typical Sample Extraction Log |

Figure 1 - Peak Evaluation Technique

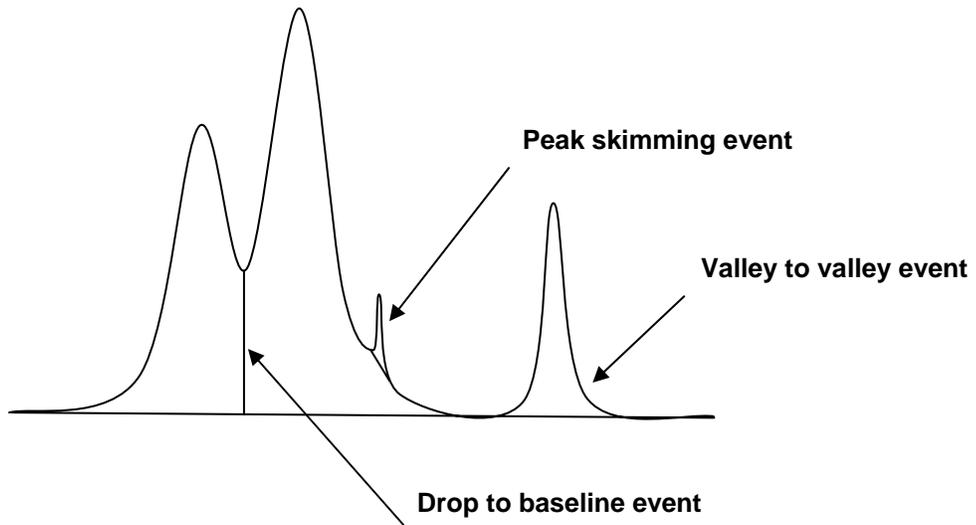


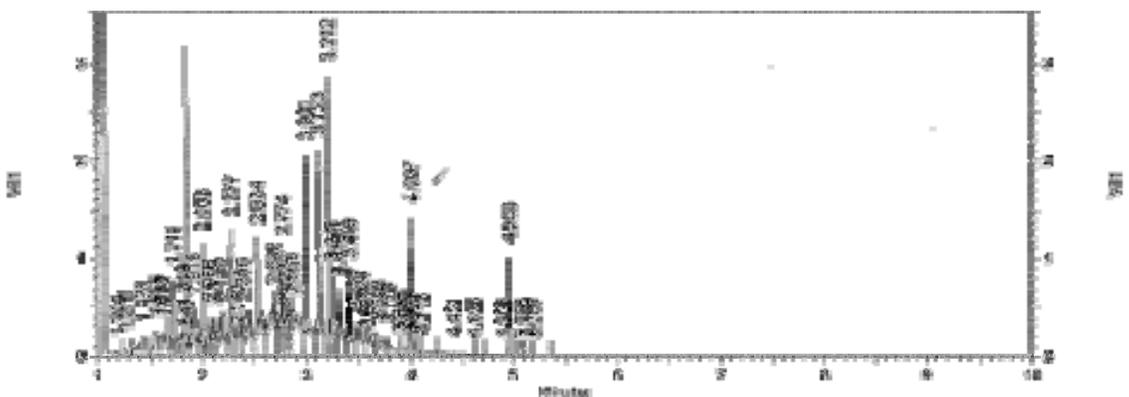
Figure 2A: Typical DRO Chromatogram

Method AK-102 by GC/FID
 EMAX Laboratories, Inc.

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Inst. Name : GC05 (Offline)
 File : D:\EZCHROM\CHROMPLI\54115.024
 Method : D:\EZCHROM\METHODS\DS105115AS1.met
 Sequence : D:\EZCHROM\SEQ\GENC\F116.seq
 Sample ID : DS105115AS01 500100/25/25
 Acquired : 00/15/06 21:01:45
 Printed : 00/20/06 09:14:17
 User : System

| A Results Name | Retention Time | Area | Average RF | ESTD concentration |
|----------------|----------------|---------|-------------|--------------------|
| o-TERPHENYL | 4.007 | 793613 | 21932.48000 | 35.000 CAL |
| DRO(C10-C15) | | 2889220 | 28802.04000 | 100.000 CAL |



Software Version: Version 3.1.7

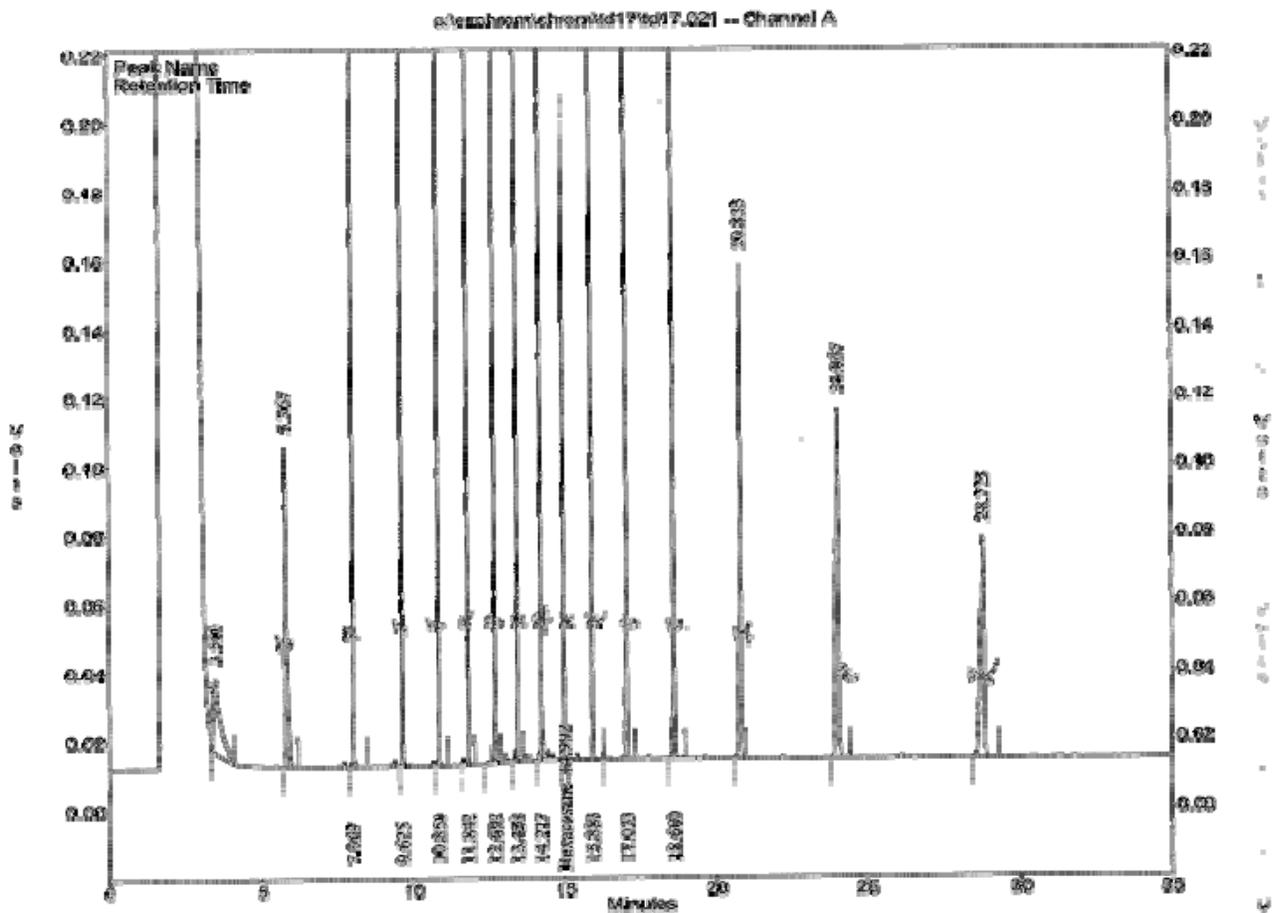
Figure 2B: TYPICAL n-ALKANE CHROMATOGRAM

Method AK-102 by GC/FID
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\td17\td17.021
 Method : c:\ezchrom\methods\ds50d17.met
 Sample ID : HC-CHAIN
 Acquired : Apr 18, 2006 08:09:47
 Printed : Apr 27, 2006 11:11:20
 User : JANE

Channel A Results

| # | Peak Name | Ret. Time (Min) | Area | Ave. CF | ESTD Conc. (ppm) |
|---|---------------|-----------------|---------|---------|------------------|
| | DRO (C10-C25) | | 3950441 | 26460.6 | 149.3 |



AK-102

Figure 3: **Typical ICAL Summary**

INITIAL CALIBRATION
METHOD AK102

Lab Name : EMAX Inc
Instrument ID : GCT105
GC Column : HP5
Column size ID : 30MX0.32MM 0.25UM
LFID & Datetime: LG21002A 07/21/08 11:10
LFID & Datetime: LG21003A 07/21/08 11:27
LFID & Datetime: LG21004A 07/21/08 11:44
LFID & Datetime: LG21005A 07/21/08 12:01
LFID & Datetime: LG21007A 07/21/08 12:34
LFID & Datetime: LG21008A 07/21/08 12:51
LFID & Datetime: LG21009A 07/21/08 13:08
CONC UNIT: ppm

| COMPOUND | CONC X | CALIBRATION FACTORS | | | | | | | MEAN | %RSD |
|-----------------|-----------|---------------------|-------|--------|--------|---------|---------|---------|---------|------|
| | | 1.00X | 2.00X | 10.00X | 20.00X | 100.00X | 300.00X | 600.00X | | |
| DIESEL(C10-C25) | 5.00 | 22348 | 22343 | 24415 | 24926 | 23902 | 22253 | 22101 | 23183.9 | 5.1 |

DS105G21AK.MET

Lab Name : EMAX Inc
Instrument ID : GCT105
GC Column : HP5
Column size ID : 30MX0.32MM 0.25UM
LFID & Datetime: LG21010A 07/21/08 13:25
LFID & Datetime: LG21011A 07/21/08 13:41
LFID & Datetime: LG21012A 07/21/08 13:58
LFID & Datetime: LG21013A 07/21/08 14:15
LFID & Datetime: LG21014A 07/21/08 14:32
LFID & Datetime: LG21015A 07/21/08 14:48
CONC UNIT: ppm

| SURROGATE | X | 1.00X | 2.00X | 4.00X | 10.00X | 30.00X | 40.00X | MEAN | %RSD |
|-------------|------|-------|-------|-------|--------|--------|--------|---------|------|
| | | | | | | | | | |
| O-TERPHENYL | 5.00 | 23277 | 24633 | 24753 | 25118 | 22951 | 24180 | 24151.9 | 3.6 |

DS105G21AK.MET

Figure 4:

Typical Continuing Calibration

CONTINUE CALIBRATION
 METHOD AK102

Lab Name : EMAX Inc
 Instrument ID : GCT105
 GC Column : HP5
 Column size ID : 30MX0.32MM 0.25UM
 Mid Conc Init LFID & Datetime: LG21007A 07/21/2008 12:34
 Conc Cont LFID & Datetime: LG24109A 07/25/2008 18:38
 CONC UNIT : ppm

| COMPOUND | RT MINUTES | RT WINDOW FROM TO | TRUE CONC | AVERAGE CF | RESULT AREA | CONC | %D | QL | LIMITS |
|-----------------|---------------|----------------------|--------------|---------------|----------------|--------|-----|----|--------|
| DIESEL(C10-C25) | NA | NA NA | 500.0 | 23183.9 | 12879002 | 555.51 | 11 | | 25 |
| SURROGATE | MINUTES | FROM TO | TRUECONC | CF | AREA | CONC | %R | QL | LIMITS |
| O-TERPHENYL | 3.947 | 3.876 4.018 | 20.0 | 24151.9 | 561963 | 23.27 | 116 | | 60-120 |

Figure 5: Typical Sample Report

METHOD AK 102/AK 103
 TOTAL PETROLEUM HYDROCARBONS BY EXTRACTION

```

=====
Client       : XYZ, INC.                      Date Collected: 07/22/08
Project      : ANY WASTE SAMPLING             Date Received: 07/24/08
Batch No.    : 08G256                         Date Extracted: 07/25/08 10:20
Sample ID    : W-08-203-0089                 Date Analyzed: 07/25/08 14:42
Lab Samp ID  : G256-01                       Dilution Factor: 1
Lab File ID  : LG24095A                      Matrix          : SOIL
Ext Btch ID  : DSG034S                       % Moisture     : 14.5
Calib. Ref. : LG24082A                       Instrument ID   : GCT105
=====
  
```

| PARAMETERS | RESULTS (mg/kg) | RL (mg/kg) | MDL (mg/kg) |
|------------|--------------------|---------------|----------------|
| DRO | ND | 12 | 5.8 |
| RRO | ND | 23 | 5.8 |

| SURROGATE PARAMETERS | % RECOVERY | QC LIMIT |
|----------------------|------------|----------|
| O-TERPHENYL | 75 | 50-150 |

RL : Reporting Limit
 Carbon Range:
 DRO: Diesel Range Organic (C10-C25)
 RRO: Residuel Range Organic (C25-C36)

Figure 6:

Typical LFB/LFBD Summary

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: ANY WASTE SAMPLING
BATCH NO.: 08G256
METHOD: METHOD AK 102/AK 103

=====

MATRIX: SOIL % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: MBLK1S
LAB SAMP ID: DSG034SB DSG034SL DSG034SC
LAB FILE ID: LG24087A LG24088A LG24089A
DATE EXTRACTED: 07/25/0810:20 07/25/0810:20 07/25/0810:20 DATE COLLECTED: NA
DATE ANALYZED: 07/25/0812:27 07/25/0812:44 07/25/0813:01 DATE RECEIVED: 07/25/08
PREP. BATCH: DSG034S DSG034S DSG034S
CALIB. REF: LG24082A LG24082A LG24082A

ACCESSION:

| PARAMETER | BLNK RSLT (mg/kg) | SPIKE AMT (mg/kg) | BS RSLT (mg/kg) | BS % REC | SPIKE AMT (mg/kg) | BSD RSLT (mg/kg) | BSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|-----------|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|--------------|-------------------|------------------|
| DRO | ND | 500 | 420 | 84 | 500 | 423 | 85 | 1 | 60-120 | 20 |
| RRO | NA | NA | NA | NA | NA | NA | NA | NA | 60-120 | 20 |

=====

| SURROGATE PARAMETER | SPIKE AMT (mg/kg) | BS RSLT (mg/kg) | BS % REC | SPIKE AMT (mg/kg) | BSD RSLT (mg/kg) | BSD % REC | QC LIMIT (%) |
|---------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|-------------------|
| O-Terphenyl | 20.0 | 21.5 | 108 | 20.0 | 21.7 | 108 | 60-120 |

Figure 7:

Typical MS/MSD Summary

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: ANY WASTE SAMPLING
BATCH NO.: 08G256
METHOD: METHOD AK 102/AK 103

=====

MATRIX: SOIL % MOISTURE: 18.6
DILUTION FACTOR: 1 1 1
SAMPLE ID: W-08-203-0090
LAB SAMP ID: G256-03 G256-03M G256-03S
LAB FILE ID: LG24099A LG24100A LG24101A
DATE EXTRACTED: 07/25/0810:20 07/25/0810:20 07/25/0810:20 DATE COLLECTED: 07/22/08
DATE ANALYZED: 07/25/0815:50 07/25/0816:06 07/25/0816:23 DATE RECEIVED: 07/24/08
PREP. BATCH: DSG034S DSG034S DSG034S
CALIB. REF: LG24097A LG24097A LG24097A

ACCESSION:

| PARAMETER | SMPL RSLT (mg/kg) | SPIKE AMT (mg/kg) | MS RSLT (mg/kg) | MS % REC | SPIKE AMT (mg/kg) | MSD RSLT (mg/kg) | MSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|-----------|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|--------------|-------------------|------------------|
| DRO | ND | 614 | 457 | 74 | 614 | 446 | 73 | 2 | 50-140 | 20 |

=====

| SURROGATE PARAMETER | SPIKE AMT (mg/kg) | MS RSLT (mg/kg) | MS % REC | SPIKE AMT (mg/kg) | MSD RSLT (mg/kg) | MSD % REC | QC LIMIT (%) |
|---------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|-------------------|
| O-Terphenyl | 24.6 | 26.2 | 107 | 24.6 | 24.5 | 100 | 50-150 |

Figure 8: Typical Case Narrative

CASE NARRATIVE

Client : XYZ, INC.
Project : ANY WASTE SAMPLING
SDG : 08G256

METHOD AK 102 TOTAL PETROLEUM HYDROCARBONS BY EXTRACTION

A total of six (6) soil samples were received on 07/24/08 for TPH Diesel & Motor Oil analysis, Method AK 102 in accordance with State of Alaska Method For Determination of Diesel Range Organics, Version 04/08/02.

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Calibration was performed as prescribed by the method and was verified using a secondary source. All calibration requirements were within acceptance criteria.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

Two (2) sets of LCS/LCD were analyzed with the samples in this SDG. Percent recoveries for DSG034SL/C were all within QC limits. Percent recoveries for DSG035SL/C were all within QC limits.

Matrix QC Sample

One (1) set of MS/MSD were analyzed with the samples in this SDG. Percent recoveries for G256-03M/S were within project QC limits.

Surrogate

Surrogate was added on QC and field samples. Surrogate recoveries were within project QC limits.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameter.

Appendix 1:

SUMMARY OF QUALITY CONTROL PROCEDURES

| QC Procedure | Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | 1 st Rvw | 2 nd Rvw |
|--|--|---|---|--|---------------------|---------------------|
| Minimum five-point initial calibration | Initially; as needed | Using RF: mean RSD $\leq 25\%$ Using Calibration Curve: Linear fit with $R_2 \geq 0.995$; Quadratic fit with $R_2 \geq 0.995$ | Correct the problem then repeat initial calibration | | | |
| Calibration Verification Standard (CVS) | After initial calibration | Within $\pm 25\%$ of expected value Recovery of Hydrocarbons eluting between the retention time markers $> 95\%$ | Correct the problem then repeat initial calibration | | | |
| Initial calibration verification (ICV) | Daily, before sample analysis using mid-point CCS | Within $\pm 25\%$ of expected value | Correct the problem then repeat initial calibration | | | |
| Continuing Calibration Samples (CCS) | Every 12 hours of analysis time and end of analysis sequence | Within $\pm 25\%$ of expected value | Correct the problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification | | | |
| Method blank | One per preparation batch | No analyte detected \geq PQL | Reprep and reanalyze method blank and all samples processed with the contaminated blank | Apply B to specific analyte(s) on all associated samples | | |
| Lab Fortified Blank/Duplicate (LFB/LFBD) | One per preparation batch | Recovery: 60% - 120% RPD $\leq 20\%$ | Correct the problem then re-extract and analyze sample | | | |
| Surrogate spike | Every sample, spiked sample, standard, and method blank | Recovery: 60%-120% (MB,LFB/LFBD, ICV,CVS, CCS) 50%-150% (MS/MSD, field samples) | Correct the problem then re-extract and analyze sample | | | |
| MS/MSD | Analyze as specified by project. | None | None | | | |
| Retention Time Window Standard | Daily or ≤ 20 samples per preparation batch | RTW= ABS(Ave RT) ± 3 XSD or For SD=0; RTW: ± 0.05 min. | Update as necessary | | | |
| Chromatogram | All sample results | Within calibration range NO SATURATED PEAK(s) | Dilute and re-analyze all samples over the calibration range Diluted and re-analyzed all samples demonstrating saturated peak(s) even if the total integrated peaks do not exceed the calibration range. | | | |
| Results reported between MDL and RL | None | None | None | | | |
| Comments: PQL= 5X MDL | | | | Reviewed By | | |
| | | | | Date | | |

Appendix 2:

DEMONSTRATION OF CAPABILITY



6390 Joyce Drive
100
Golden, CO 80403

Phone 303-940-0033
Fax 866-283-0269
www.wibby.com

Final Report - Underground Storage Tank PT

Study: UST0109

Opening Date: January 19, 2009 - Closing Date: March 5, 2009

Laboratory: EMAX Laboratories
1825 205th Street
Torrance, CA 90501

Contact: Kenette Pimentel, Quality Assurance Manager
310-818-8889 ext. 205

EPA Lab ID: CA00291

| Alaska DRO in Water (PT-AKDRO-USTW) | | | | | | | | Lot #: 6035-27 | |
|-------------------------------------|-----------------------|-------------|--------------------|-------|----------------|--------|-------------------|----------------|--|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation | |
| 9369 | Diesel Range Organics | AK 102 | AK 102 | µg/L | 1040 | 992 | 844 - 1440 | Acceptable | |
| Alaska DRO in Soil (PT-AKDRO-USTS) | | | | | | | | Lot #: 6035-29 | |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation | |
| 9369 | Diesel Range Organics | AK 102 | AK 102 | mg/kg | 638 | 611 | 505 - 979 | Acceptable | |

AK102FS:

TYPICAL SAMPLE EXTRACTION LOG

Page 1



EXTRACTION LOG FOR TPH

SOP EMAX-3550 Rev. No.2 EMAX-3520 Rev. No.2 EMAX-LUFT E Rev. No.3 EMAX-3540 Rev. No.0 EMAX-3510 Rev. No. 1

| Matrix | Start Date: | Time: | End Date: | Time: | Book # ED\$-043 | | | | | |
|---------------------------|----------------|---------------|------------------|------------------------|---------------------|---------------------|-------|-----------------------------------|-----------------------------------|-------------------|
| PREPARATION BATCH # _____ | Sample Prep ID | Lab Sample ID | Sonicator Number | Sample Amount (g ml) | Extract Volume (ml) | Silica Gel Clean-up | Notes | Standards | ID | Amount Added (ml) |
| | 01 | | | | | | | Surrogate | | |
| | 02 | | | | | | | LCSMS | | |
| | 03 | | | | | | | Reagent | Lot# / ID | |
| | 04 | | | | | | | CH ₂ Cl ₂ | | |
| | 05 | | | | | | | Na ₂ SO ₄ | | |
| | 06 | | | | | | | HCl | | |
| | 07 | | | | | | | Silica Sand | | |
| | 08 | | | | | | | Silica Gel | | |
| | 09 | | | | | | | TUNING | | |
| | 10 | | | | | | | Sonicator # | Reading | |
| | 11 | | | | | | | | | |
| | 12 | | | | | | | | | |
| | 13 | | | | | | | | | |
| | 14 | | | | | | | Concentrator Water Bath Temp. (C) | | |
| | 15 | | | | | | | 1 | | |
| | 16 | | | | | | | 2 | | |
| | 17 | | | | | | | 3 | | |
| | 18 | | | | | | | 4 | | |
| | 19 | | | | | | | 5 | | |
| | 20 | | | | | | | 6 | | |
| | 21 | | | | | | | Comments: | Test thermometer = T ₁ | |
| | 22 | | | | | | | | | |
| | 23 | | | | | | | Prepared By: | Standard Added By: | |
| | 24 | | | | | | | Witnessed By: | Checked By: | |
| | 25 | | | | | | | Extract Received by: | Extract Location: | |
| | 26 | | | | | | | Disposal Date: | Disposed By: | |
| 27 | | | | | | | | | | |

This page is checked during data review.

STANDARD OPERATING PROCEDURES
AK RESIDUAL RANGE ORGANICS

SOP No.: EMAX-AK103 Revision No. 1 Effective Date: 02-Jul-07
 Prepared By: Lucita Arzadon *L.R. Arzadon* Date: 06-27-07
 Approved By: Kenette Pimentel *Kenette Pimentel* Date: 06-27-07
 QA Manager
 Approved By: Kam Pang *Kam Pang* Date: 6/27/07
 Laboratory Director

Control Number: AK103-01-

1.0 SCOPE AND APPLICATION

- 1.1. This method is designed to measure the concentration of Residual Range Organics (RRO) in soil. This method is limited to provide semiquantitative results on those extractable hydrocarbons with a comparable aliphatic hydrocarbon range from the beginning of C₂₅ to the end of C₃₆, and compounds with boiling points from approximately 400°C to 500°C. This is an adaptation of Method AK 103

2.0 SUMMARY OF METHOD

- 2.1. Petroleum hydrocarbons are extracted in methylene chloride and analyzed by flame ionization detector (FID) in gas chromatograph and quantified as lubricating or motor oil at C₂₅ to C₃₆ range. The hydrocarbons that fall in this range are defined as Residual Range Organics (RRO). Integration is performed using forced baseline-baseline integration.
- 2.2. Components greater than C₃₆ present in products such as asphalts, and mid-range boiling point products such as diesel and bunker C, are also detectable under the conditions of the method.
- 2.3. **Interference**
- 2.3.1. Glassware can be a potential source of contamination. They must be scrupulously cleaned prior to its use.
- 2.3.2. Carry-over from a highly concentrated sample can be a potential source of contamination. Instrument performance must be observed keenly for possible carry-over. If this is apparent, inject solvent blank until no trace of carry-over is observed.
- 2.3.3. Deposits may adhere in the injection port/glass liner over a period of time and can cause interference. The injection port and glass liner must be routinely cleaned.

3.0 METHOD DETECTION LIMITS AND QUANTITATION LIMITS

3.1. **Method Detection Limit (MDL)**

- 3.1.1. Prepare a minimum of eight samples. Add spike standard at 100mg/kg spike level to seven samples and treat one as method blank.
- 3.1.2. Analyze the samples as described in Section 10 and calculate the results as described in Section 10.6.
- 3.1.3. Refer to EMAX QA04 for MDL evaluation and verification.

3.2. **Reporting Limit**

- 3.2.1. Detection level is equal to the concentration of the lowest calibration point, unless otherwise specified by the project.

4.0 DYNAMIC RANGE

STANDARD OPERATING PROCEDURES
AK RESIDUAL RANGE ORGANICS

SOP No.: EMAX-AK 103 Revision No. 1 Effective Date: 02-Jul-07

- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (See Section 9.7.1). Dilute and reanalyze all samples having results above this range to properly quantitate.
- 4.2. The lowest quantifiable range of diluted samples is equal to the lowest calibration point (See Section 9.7.1.) Lower the dilution factor and reanalyzed all diluted samples analyzed below this range to properly quantitate

5.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIME

| Matrix | Container | Preservative | Holding Time |
|--------|--|------------------------------|---|
| Water | 1L amber glass with Teflon lined screw caps | pH < 2 with HCl 4°C ±2 °C | Extraction - 14 days Analyses of extracts - 40days |
| Soil | Core tube or 4 or 8 oz amber glass jar with Teflon-lined lid | 4°C ±2 °C | |

6.0 ASSOCIATED SOPs

| | |
|------------|--|
| EMAX-QC02 | Analytical Standard Preparation |
| EMAX-SM04 | Analytical and QC Sample Labeling |
| EMAX-QA04 | Method Detection Limit Study |
| EMAX-QA08 | Corrective Action |
| EMAX-DM01 | Data Flow & Review |
| EMAX-SM03 | Waste Disposal |
| EMAX-LUFTE | Total Petroleum Hydrocarbons by Extraction |
| EMAX-3540 | Extraction, Soxhlet |
| EMAX-3550 | Extraction , Pulse Sonication |
| EMAX-3580 | Waste Dilution |

7.0 SAFETY

- 7.1. Read all MSDS for chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. Place all wastes generated during analytical process in the wastes containers. Endorse these wastes to the waste disposal section for proper disposal.

STANDARD OPERATING PROCEDURES
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- 7.4. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES

8.1. Instruments and Supplies

- 8.1.1. Gas Chromatography: GC HP5890 II with FID and 7673HP Autosampler, or equivalent
- 8.1.2. Detector: FID
- 8.1.3. Column: DB5, (0.25 mm x 30 m) 0.25 μ m thickness, or equivalent
- 8.1.4. Data Acquisition: EZ Chrom, or equivalent
- 8.1.5. Syringes: 10, 25, 100 μ L microsyringe
- 8.1.6. Glasswares
 - 8.1.6.1. Graduated cylinders
 - 8.1.6.2. Disposable pipettes
 - 8.1.6.3. Volumetric Flask: 10, 100 and 1000 mL
 - 8.1.6.4. Vials: 2, 10 and 40 ml, amber
 - 8.1.6.5. Bottle: 250-ml (amber)
- 8.1.7. Stainless steel spatula
- 8.1.8. Analytical balance capable of accurately weighing 0.0001 g./ Top-loading balance capable of weighing to the nearest 0.01 g.
- 8.1.9. Boiling chips
- 8.1.10. Ultrasonic cell disrupter/Horn-type sonicator equipped with a titanium tip.
- 8.1.11. Soxhlet extraction apparatus as described in SW-846, Method 3540.

8.2. Chemicals and Reagents

- 8.2.1. Methylene Chloride, Acetone
- 8.2.2. Sodium Sulfate
- 8.2.3. High purity He, H₂, Air

9.0 STANDARDS

9.1. Standard Preparation

- 9.1.1. The procedure for analytical standard preparation is detailed in EMAX-QC02.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.2. Stock Standard

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- 9.2.1. Purchase stock standards as certified solutions from two different vendors. Use one as primary standard and the other as secondary standard.
- 9.2.2. Transfer standards on a properly labeled inert vial with minimal headspace and store it at -10 °C to -20 °C.
- 9.2.3. Prepare calibration standards from the primary standard.
- 9.2.4. Prepare initial calibration verification standards and spiking standards from the secondary standard.

9.3. **Calibration Standards**

| Name | Source | Catalog # | Conc. (mg/L) | Notes |
|-----------------------------|---------|------------------------|--------------|---------------|
| 30W and 40W Motor Oil (1:1) | AccuStd | FU-L018-4 FU-L019-4 | 50,000 | or equivalent |

9.4. **Surrogate Standards**

| Name | Source | Catalog # | Conc. ((mg/L) | Notes |
|-------------------------|---------|------------------|---------------|---------------|
| Bromobenzene/Hexacosane | Aldrich | 23,987-9/24168-7 | neat | or equivalent |

9.5. **LFB/Matrix Spike Standard**

| Name | Source | Catalog # | Conc. (mg/L) | Notes |
|-----------------------------|--------|-----------|--------------|---------------|
| 30W and 40W Motor Oil (1:1) | Restek | 56196 | neat | or equivalent |

9.6. **Intermediate Standard**

Using the stock standard solution, prepare in methanol or Acetone and store with minimal headspace in an inert vial. Prepare secondary dilution standards from the stock standards at concentration levels as follows:

30W and 40W Motor Oil (1:1) 5000 mg/L

Surrogate 1000/250 mg/L

LCS/Matrix Spike 5000 mg/L

9.7. **. Working Standard**

9.7.1. Residuals Calibration Standard (RCS)

Prepare initial calibration standards in 1 ml organic free water as suggested below.

| Standard | Motor Oil Std. 50000 mg/L | Final Conc. (mg/L) | Surrogate 1000/250 mg/L | Final Conc. (mg/L) |
|----------|------------------------------|-----------------------|----------------------------|-----------------------|
| 1 | 1 µl | 50 | 1 µl | 10/2.5 |
| 2 | 2 µl | 100 | 2.0 µl | 20/5 |
| 3 | 10 µl | 500 | 10 µl | 100/25 |
| 4 | 20 µl | 1000 | 20 µl | 200/50 |
| 5 | 30 µl | 1500 | 30 µl | 300/75 |
| 6 | 60 µl | 3000 | 60 µl | 600/150 |

9.7.2. Initial and Continuing Calibration Standard (CVS/CCS)

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9.7.2.1. For CVS, prepare an intermediate standard at 1000 mg/L from the secondary stock standard with surrogate at 200/50 mg/L

9.7.2.2. For CCS, prepare an intermediate standard at 1000 mg/L from the primary stock standard with surrogate at 200/50 mg/L

9.8. Retention Time Window Standard

9.8.1. Prepare a standard containing a mixture of C₂₅ and C₄₄ at 20 mg/L for retention time analysis. Use this standard to spike RTW sample.

10.0 PROCEDURES

10.1. Sample Preparation

10.1.1. The preferred method for soil extraction is Method 3540 (Soxhlet Extraction). Equivalent extraction techniques which meet the quality assurance requirements of this method can be used.

10.1.2. Weigh 15 g of sample into an extraction thimble and add an equal weight of anhydrous sodium sulfate and stir the mixture well with a spatula.

10.1.3. Place loaded thimbles in extractors and spike 1 mL of 1000/250 mg/mL of surrogate standard to both field and quality control samples.

10.1.4. For LFBs and duplicate LFBs, spike 10 g. of methylene chloride rinsed Ottawa sand or alternative standard soil with 1 mL at 5000 mg/L of spike standard and 1 mL of 1000/250 mg/mL of surrogate standard.

10.1.5. Prepare a method blank using 15 g. of methylene chloride rinsed Ottawa sand or alternative standard soil. Spike the sample with 1 mL of 1000/250 mg/mL of surrogate standard.

10.1.6. Add 300 mL of methylene chloride to the 500 mL extraction flask. Add boiling chips to the flask. Connect the extractors to the flask and the condenser to the extractor. Extract for 18-24 hours.

10.1.7. Transfer extract into a clean concentrator tube and concentrate extracts to 10 mL at a temperature not to exceed 40°C. Transfer to GC vials for analysis. Record information for the extraction and concentration steps.

10.1.8. Moisture Determination for Solids

10.1.8.1. Weigh a numbered moisture pan and record the number and weight of the pan.

10.1.8.2. Weigh about 5-10 g of the sample into the pre-weighed moisture pan.

10.1.8.3. Dry the samples overnight at 105°C.

10.1.8.4. Remove the samples from the oven and cool in a desiccator until sample reaches room temperature. Weigh the dried samples.

10.1.8.5. Record all weights to the nearest 0.01g.

10.2. Instrument Parameters

10.2.1. Fine tune the instrument guided by the parameter conditions as listed below:

| Instrument | Temp (°C) | Rate (°C/min) | Time (min) | Temperature (°C) | | Injection Volume (µL) | Head Pressure (psi) |
|------------|-----------|---------------|------------|------------------|----------|-----------------------|---------------------|
| | | | | Injector | Detector | | |
| | 50 | 0 | 2 | | | | |

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| | | | | | | | |
|----|-----|----|------|-----|-----|---|-------|
| 50 | 50 | 0 | 2 | 280 | 320 | 2 | 12-20 |
| | 120 | 20 | 1 | | | | |
| | 310 | 28 | 22.1 | | | | |

10.3. Calibration

10.3.1. Continuing Calibration Verification

- 10.3.1.1. Prepare initial calibration solution as described in Section 9.7.1. Analyze them as described in Section 10.4.
- 10.3.1.2. Refer to Section 10.5 for calculation.
- 10.3.1.3. Acceptance criteria are specified in Appendix 1.
- 10.3.1.4. Verify the initial calibration by a second source standard.
- 10.3.1.5.

10.3.2. Retention Time Window Check (RTW)

- 10.3.2.1. Prepare standard containing 1000 mg/L RTW standard.
- 10.3.2.2. Analyze the RTW check sample to monitor retention time drift. See Appendix 1 for acceptance criteria.

10.4. Analysis

10.4.1. Extract Preparation

- 10.4.1.1. Allow the extracts to equilibrate with the room temperature.
- 10.4.1.2. Transfer about 1-mL of extracts into Autosampler vials.

10.4.2. Analytical Sequence

- 10.4.2.1. Assuming that there is an existing initial calibration, for every 20 field samples, set the analytical sequence as follows:
 - 10.4.2.1.1. Instrument Blank
 - 10.4.2.1.2. RTW Window sample
 - 10.4.2.1.3. Opening CCS
 - 10.4.2.1.4. Method Blank
 - 10.4.2.1.5. Field samples to include field blanks, field duplicates, matrix spikes (only when requested)
 - 10.4.2.1.6. Lab Control Sample (LCS)
 - 10.4.2.1.7. Closing CCV

10.4.3. Sample Result Evaluation

- 10.4.3.1. Check that surrogates are within the control limits. Refer to Appendix 1 for acceptance criteria and corrective action.
- 10.4.3.2. Dilute and re-analyze samples having concentrations greater than the highest calibration range.

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10.4.3.3. Dilute and re-analyze samples having saturated peak(s) within C₂₅ to C₄₄. See Figure 1 for typical saturated peak.

10.4.3.4. Re-analyze samples suspected of carry-over from a preceding sample that has high concentration.

10.4.4. Identification and Quantitation

10.4.4.1. Identification is based on pattern recognition. Refer to Figure 2a for typical residual pattern.

10.4.4.2. Compare sample chromatograms to reference residual standard chromatograms for their response hydrocarbon range and peak distribution to determine if the result resembles residual pattern.

10.4.4.3. When the elution profile of a sample does not match that of residual standard pattern, but falls within the retention time window, quantitate results as residual range organics (RRO) and denote the observed deviation in case narrative.

10.4.4.4. Integrate the total peak area response eluting between the peak start of C₂₅ and the peak end of C₄₄ baseline to baseline and quantitate the residual range organics concentration using equation 10.5.3.

10.5. **Establishing Retention Time Window (RTW)**

10.5.1. Analyze the RTW standard (refer to 10.3.4) daily within 72 hours.

10.5.2. Calculate the standard deviation (SD) of the RT for each analyte. The absolute RT window is established by $\pm 3 * SD$.

10.5.3. The lower limit of the RT window C₂₅ for the first eluting component and the upper limit of the RT window for C₄₄ for the last eluting compound determines the retention time range of RRO.

10.5.4. Update the RT window using the established absolute RT window during initial calibration.

Note: If cases where the SD=0, use +0.05 min. in place of SD.¹

10.5.5. RT window must be (re)established when the following conditions occur:

- Instrument is new
- Major repair was done
- GC condition was changed
- GC column was replaced

10.6. **Calculations**

10.6.1. Calculate for Percent Moisture for Soils

$$\% \text{Moisture} = \frac{(A - C)}{(A - B)} * 100 \quad \text{Eq-10.6.1}$$

where:

- | | |
|---|-----------------------------|
| A | weight of boat + wet sample |
| B | weight of boat |
| C | weight of boat + dry sample |

¹ AK101, Section 9.9.2.2

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10.6.2. Initial Calibration

10.6.2.1. Calculate for Calibration Factor

$$CF = \frac{R_t}{C_v} \quad \text{Eq-10.6.2.1}$$

where:

- CF – Calibration Factor
- R_t – Total response of the integrated peaks
- C_v – Known value of the standard concentration, mg/L

10.6.2.2. Calculate for the Average Calibration Factor

$$ACF = \frac{\sum CF}{n} \quad \text{Eq-10.6.2.2}$$

where:

- ACF – Average Calibration Factor
- $\sum CF$ – Summation of Calibration Factors
- n – Number of measurements

10.6.2.3. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq-10.6.2.3}$$

where:

- SD – Standard Deviation
- x_i – Result at the i^{th} measurement
- \bar{x} – mean
- n – number of measurements

10.6.2.4. Calculate for Percent Relative Standard Deviation

$$\%RSD = \frac{SD}{ACF} * 100 \quad \text{Eq-10.6.2.4}$$

where:

- $\%RSD$ – Percent Relative Standard Deviation
- SD – Standard Deviation
- ACF – Average Calibration Factor

10.6.3. Calculate for Percent Difference for DCC from ACF

$$\%D = \frac{Cf - Ck}{Ck} * 100 \quad \text{Eq-10.6.3}$$

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where:

- $%D$ – Percent Difference DCC from known concentration
- Ck – Known concentration of the analyte, in mg/L
- Cf – Found concentration, in mg/L

10.6.4. Calculate for Sample Concentration

$$C = \frac{(R_t)(V_e)(DF)}{(ACF)(A_s)(\%S)} \quad \text{Eq-10.6.4}$$

where:

- C – Concentration of the sample, mg/L or mg/Kg
- R_t – Total response of the integrated peaks
- V_e – Volume of Extract, ml
- A_s – Sample amount, ml or g
- DF – Dilution Factor
- ACF – Average Calibration Factor
- $\%S$ – Percent solids (%S for water = 1)

10.6.5. Calculate for Precision

$$\% RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq-10.6.5}$$

where:

- RPD – Relative Percent Difference
- $C1$ – Measured concentration of the first sample aliquot
- $C2$ – Measured concentration of the second sample aliquot

10.7. Report Generation

- 10.7.1. Generate the method.txt file using WDBX².exe.
- 10.7.2. Generate Lab Chronicle using LABCHRNX.exe
- 10.7.3. Generate the sample results using FIVX.exe
- 10.7.4. Generate the QC summary using QCVX.exe
- 10.7.5. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.
 - Sample Results
 - LFB Summary

² X - version number

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- MS/MSD Summary
- ICAL Summary
- DCV Summary
- CCV Summary
- Analysis Log
- Extraction Log

10.8. Date Review

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

10.8.2. Generate the case narrative to include discussion of the following as found in the review process:

- Number of samples analyzed
- Analytical method(s) applied
- Holding Time – That samples extracted and analyzed within holding time. For non-compliance, state the number of days that the sample(s) was out of holding time.
- Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
- Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
- Method Blank – That MB was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s) affected and the associated sample results were flagged with “B”.
- Lab Control Samples (if applicable) – That LCS was prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Further more, if corrective action is not possible (e.g. no more samples to re-extract) state that results were qualified.
- Matrix Spike Samples – That MS/MSD (if applicable) were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Sample Analysis – that samples were analyzed in conformance to the method and project requirements.
- Other Anomalies (if any) – discussed on a case by case basis concurred by the Supervisor or the Lab Director.

10.8.3. Submit the analysis package for secondary review.

10.9. Preventive Maintenance

10.9.1. Perform daily instrument check prior to sample analysis. Refer to AK103-FM – Instrument Maintenance Log.

10.9.2. Check the gas flow from time to time to ensure that ideal gas flow is maintained accordingly.

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10.9.3. Maintain an inventory of instrument parts and supplies for routinely maintenance.

11.0 QUALITY CONTROL

11.1. Sample Preparation

- 11.1.1. A preparation batch shall consist of a MB, LFB, MS/MSD (only when requested) and ≤ 20 field samples.
- 11.1.2. All labwares used in the sample preparation shall be properly treated as specified in EMAX-QC07
- 11.1.3. All solvents and reagents shall undergo quality control check in the stationary laboratory prior to its use.

11.2. Analytical Batch

- 11.2.1. Initial Calibration must be established and verified by daily continuing calibration at the frequency specified in Appendix 1.
- 11.2.2. Analytical batch shall consist of a valid ICAL, RTW check, samples identified in every preparation batch bracketed with opening and closing CCS.
- 11.2.3. A record must be established that the analytical instrument is free from contamination prior to any analysis. This can be achieved by analyzing an instrument blank.
- 11.2.4. Organic free water shall be used for method blank and LFB for both water and soil matrix.

11.3. Method QC

- 11.3.1. Method Detection Limit Study must be established before the analytical procedure can be used.
- 11.3.2. Retention Time Window must be established and updated as described in Section 10.4.3.
- 11.3.3. Demonstration of Capability must be established before the analytical procedure can be used.
- 11.3.4. All analysts conducting this analysis must have documented demonstration of capability.

12.0 CORRECTIVE ACTION

12.1. Calibration

- 12.1.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:
 - 12.1.1.1. If RSD > 25%, check each calibration point. If an outlier exists, re-analyze that calibration point.
 - 12.1.1.2. If CVS not within the expected recovery range, review the chromatogram.
 - Bias low results are indicative of poor purging or standard degradation.
 - Bias high is indicative of inaccurate standard injection of instrument contamination.
 - Consider preparing a fresh CVS standard and re-analyze the CVS.
 - 12.1.1.3. If problem persist, inform the Supervisor prior to re-calibration
- 12.1.2. If the continuing calibration is non-compliant, consider the suggestions described in correcting CVS.
- 12.1.3. If instrument blank/reagent blank is non-compliant, consider the following suggestions to correct the problem:

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- 12.1.3.1. Check the reagent water source e.g. same source is used by a similar analysis on a different instrument to rule out reagent contamination.
- 12.1.3.2. Bake the sample concentrator and or GC column for at least 15 min.
- 12.1.3.3. Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.
- 12.1.3.4. If problem persists, inform Supervisor prior to reanalysis.

12.2. **Surrogates**

- 12.2.1. If surrogates are non-compliant, and are not due to matrix effects, consider the following suggestions to correct the problem:
 - 12.2.1.1. Check for calculation errors and that the concentrations of the surrogate solutions are correct.
 - 12.2.1.2. Check instrument performance to determine if it is within acceptable guidelines.
 - 12.2.1.3. Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.3. **Sample Preparation QCs**

- 12.3.1. If method blank is non-compliant, consider the following suggestions to correct the problem:
 - Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM, otherwise perform the corrective action as specified in the PSR.
- 12.3.2. If LFB is non-compliant, consider the following suggestions to correct the problem:
 - Check for errors in calculation and concentration of the analyte solution
 - Check instrument performance to determine if it is within acceptable guidelines
 - Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.

12.4. **Technical Holding Time**

- 12.4.1. If samples are out of technical holding time, fill-out a Non-Conformance Report (NCR) and forward it to the project manager who will consult with the client for further instruction.

12.5. **Sample Preservation**

- 12.5.1. If water samples not contained in amber vials needs to be protected from light.
- 12.5.2. If water samples are not labeled preserved or samples were received out of the expected range of refrigeration, inform the PM for the PM to consult with the client for further instruction.
- 12.5.3. If water samples are marked preserved and pH is >2, discuss it in the case narrative.

13.0 **POLLUTION PREVENTION**

- 13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.
- 13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 **WASTE MANAGEMENT**

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- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

15.1. Application of EMAX QC Procedures

- 15.1.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing diesel range organics analysis by GC. In the instance where there is project or program specific quality control, the requirements given in the project shall take precedence over this SOP.

15.2. Air Force Center for Environmental Excellence (AFCEE) projects

- 15.2.1. When samples from AFCEE sponsored projects are analyzed for diesel range organic analysis by GC, the analyte list, the reporting limits, the calibration, QC, corrective action, and data requirements shall follow the QC criteria in the Quality Assurance Project Plan, the latest version.

15.3. U.S. Army Corps of Engineers (USACE) Projects

- 15.3.1. When samples from USACE sponsored projects are analyzed for diesel range organic analysis by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, Shell Document latest version shall be applied.

15.4. Navy Restoration Sponsored Projects

- 15.4.1. When samples from Navy Restoration sponsored projects are analyzed for diesel range organic analysis by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, Navy Quality Systems Manual latest version shall be applied.

15.5. Department of Energy Basic Ordering Agreement (DOE-BOA) Projects

- 15.5.1. For samples from DOE-BOA sponsored projects follow BOA Guidance Document, latest version in the absence of project QAPP.

15.6. Definition of Terms

- 15.6.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents. Preparation batch is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. Analytical batch is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.6.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.6.3. Residual Calibration Standard (RCS)- is a blend of equal weights of 30 weight and 40 weight motor oils (1:1) and diluted to appropriate concentrations in methylene chloride. The standard serves as a calibration standard for RRO.
- 15.6.4. Calibration Verification Standard (CVS)- is a quality control standard but with a residual range hydrocarbon mixture from a source other than that used to prepare the Residual Calibration

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Standard. It is used by the laboratory to verify the accuracy of calibration.

- 15.6.5. Continuing Calibration Standard (CCS)- is a mid-range working standard diluted from the Residual Calibration Standard, used to verify that the analytical system is operating in a manner comparable to that at the time initial calibration.
- 15.6.6. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.6.7. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.6.8. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.6.9. Matrix – is a component or form of a sample.
- 15.6.10. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.6.11. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.6.12. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.6.13. Laboratory Fortified Blank (LFB)- is a method blank sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. The spike recovery is used to evaluate method control.
- 15.6.14. Solvent Blank- is a target-analyte-free sample that doesn't go through the procedure and may also serve as an instrument blank. It is analyzed to demonstrate that the solvent used in the method is free from contamination
- 15.6.15. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.6.16. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.6.17. Surrogate – compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recovery. Compounds not expected to be detected in environmental media.
- 15.6.18. Practical Quantitation Limit (PQL) – is defined as 5 times the MDL.

16.0 REFERENCES

- 16.1. Test Method for Evaluating Solid Waste Physical/Chemical Methods. SW-846, 3rd edition.
- 16.2. Corporate QA/QC Manual, as updated.

STANDARD OPERATING PROCEDURES
AK RESIDUAL RANGE ORGANICS

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17.0 FIGURES, APPENDICES AND FORMS

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17.2. Appendices

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17.3. Forms

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Figure 1 - Peak Evaluation Technique

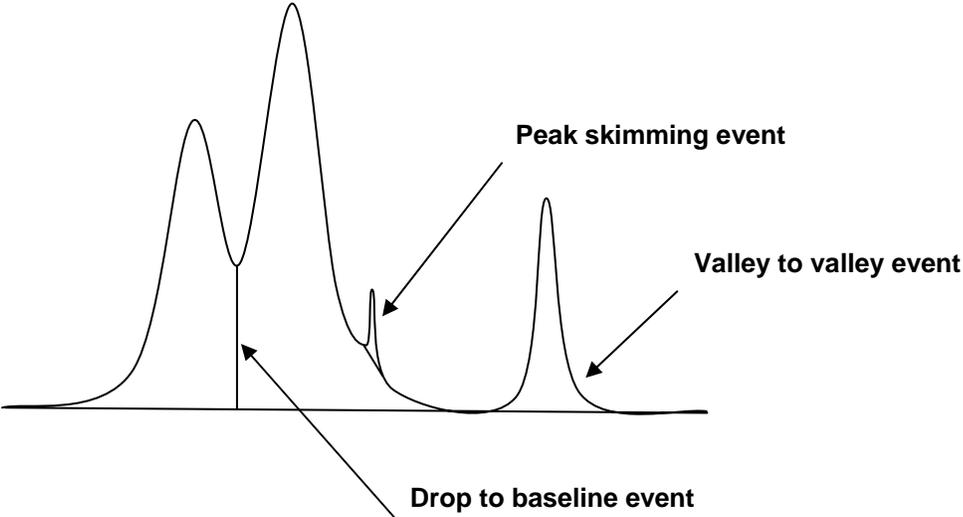


Figure 2 -Typical RRO Chromatogram

METHOD 8015 by GC/FID
EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\tl23\tl23.023
Method : c:\ezchrom\methods\m550123.met
Sample ID : IM550L2301 500PPM
Acquired : Dec 24, 2005 01:23:00
Printed : Dec 27, 2005 10:10:31
User : JANE

Channel A Results

| # | Peak Name | Ret.Time (Min) | Area | Ave. CF | ESTD Conc. (ppm) |
|----|----------------|----------------|----------|---------|------------------|
| G1 | MOTOR OIL 5W30 | | 16144312 | 31918.8 | 505.8 |

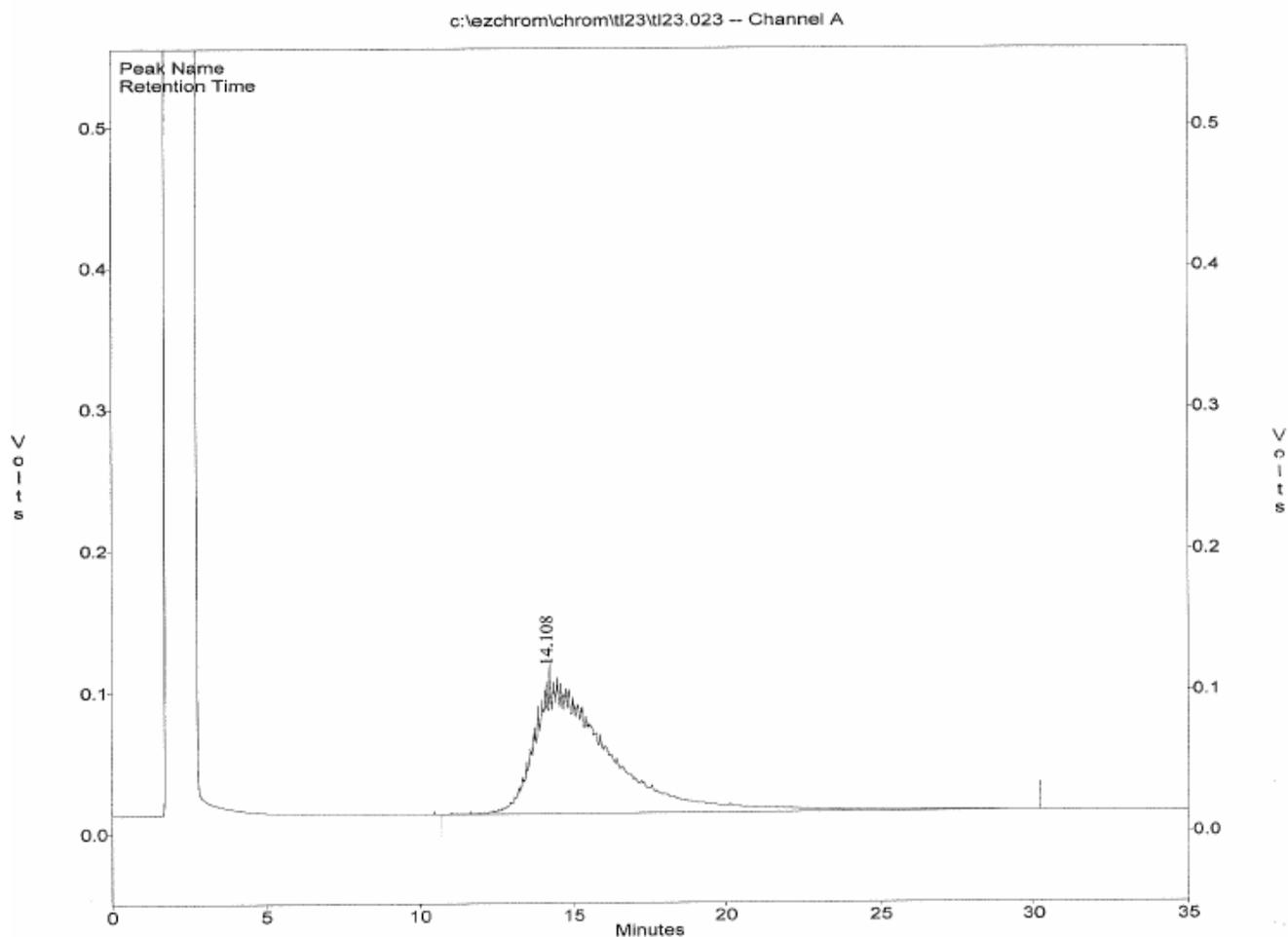


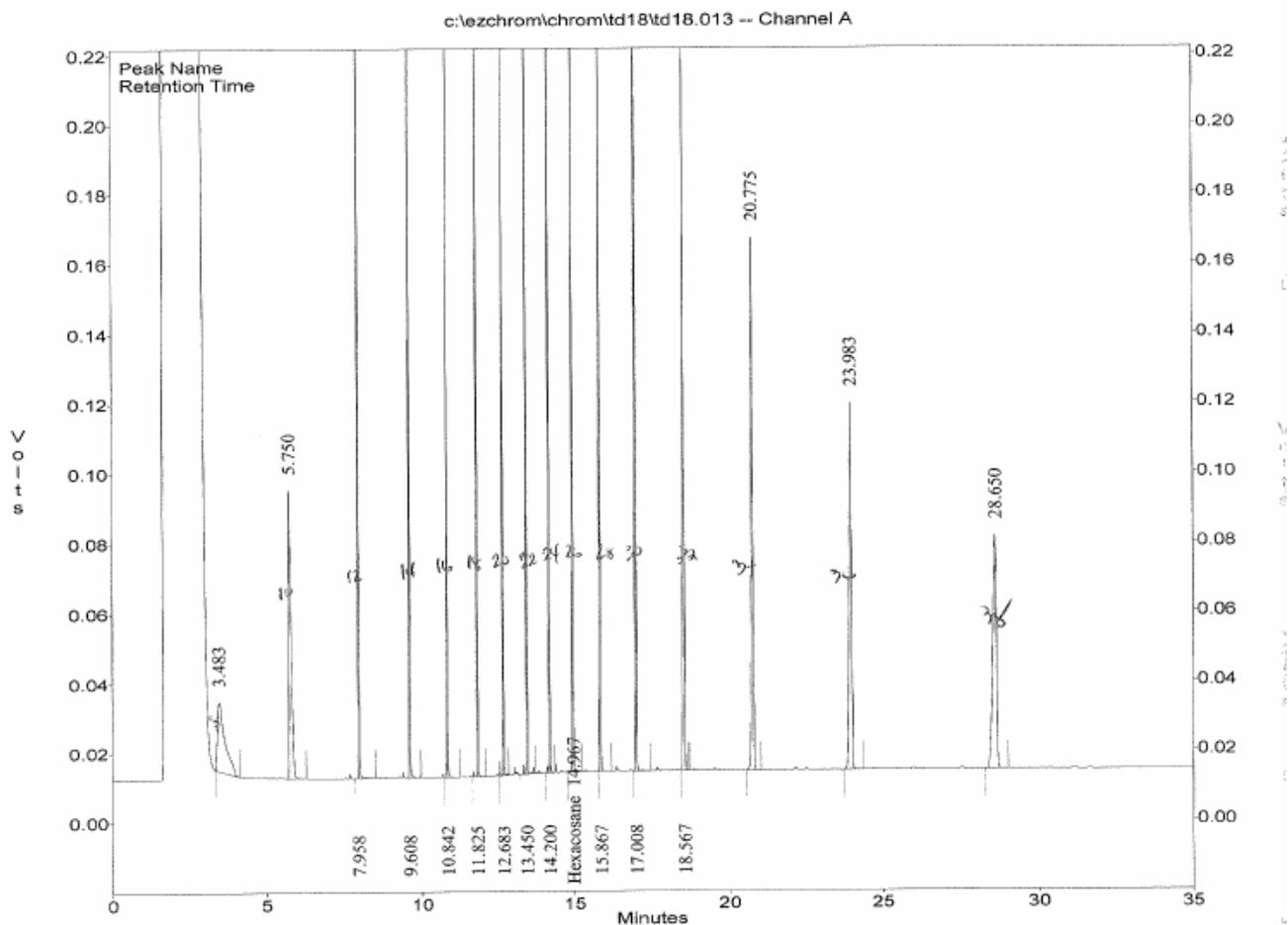
Figure 2 -Typical n-Alkane Chromatogram

METHOD 8015 by GC/FID
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\td18\td18.013
 Method : c:\ezchrom\methods\ds50d18.met
 Sample ID : HC-CHAIN
 Acquired : Apr 18, 2006 18:18:35
 Printed : Apr 19, 2006 14:46:23
 User : MICHAEL

Channel A Results

| # | Peak Name | Ret.Time (Min) | Area | Ave. CF | ESTD Conc. (ppm) |
|----|------------------|----------------|---------|---------|------------------|
| -- | Bromobenzene | 5.083 | 0 | 0.0 | 0.0 |
| 10 | Hexacosane | 14.967 | 636155 | 32776.1 | 19.4 |
| G1 | Diesel (TOTAL) | | 8185781 | 22285.1 | 367.3 |
| G2 | Diesel (C10-C24) | | 3786954 | 22203.1 | 170.6 |
| G3 | Diesel (C10-C28) | | 4825004 | 22261.9 | 216.7 |



Handwritten signature

Figure 3 – Typical ICAL Summary
 INITIAL CALIBRATION
 METHOD AK 103

Lab Name : EMAX Inc
 Instrument ID : GCT050
 GC Column : DB-5
 Column size ID : 30MX0.25MM
 LFID & Datetime: TL23016A 12/23/05 20:29
 LFID & Datetime: TL23017A 12/23/05 21:11
 LFID & Datetime: TL23018A 12/23/05 21:53
 LFID & Datetime: TL23019A 12/23/05 22:35
 LFID & Datetime: TL23020A 12/23/05 23:17
 LFID & Datetime: TL23021A 12/23/05 23:59
 LFID & Datetime: TL23022A 12/24/05 00:41
 CONC UNIT: ppm

| COMPOUND | CONC X | CALIBRATION FACTORS (AREA or HEIGHT)/UNIT | | | | | | | MEAN | %RSD |
|----------------|-----------|---|-------|-------|--------|---------|---------|---------|---------|------|
| | | 1.00X | 2.00X | 5.00X | 50.00X | 100.00X | 150.00X | 300.00X | | |
| MOTOR OIL 5W30 | 10.00 | 32792 | 33453 | 33803 | 31744 | 30539 | 29862 | 31239 | 31918.8 | 4.7 |

M550L23.MET

Figure 4 – Typical Continuing Calibration

CONTINUE CALIBRATION
 METHOD AK 103

Lab Name : EMAX
 Instrument ID : GCT050
 GC Column : DB-5
 Column size ID : 30MX0.25MM
 Mid Conc Init LFID & Datetime: TL23021A 12/23/2005 23:59
 Conc Cont LFID & Datetime: TA05003A 01/05/2006 12:40
 CONC UNIT : ppm

| COMPOUND | RT MINUTES | RT WINDOW | | TRUE CONC | AVERAGE CF | RESULT | | %D | QL | %D LIMITS |
|----------------|---------------|-----------|-------|--------------|---------------|----------|--------|----|----|--------------|
| | | FROM | TO | | | AREA | CONC | | | |
| MOTOR OIL 5W30 | 0.000 | 0.000 | 0.000 | 500.0 | 31918.8 | 14694030 | 460.36 | -8 | | 15 |

Figure 5 – Typical Sample Report

METHOD AK 103
 AK RESIDUAL RANGE ORGANICS

```

=====
Client       : XYZ, INC                      Date Collected: 03/31/06
Project      : CLEAN LAND PROJECT           Date Received: 04/01/06
Batch No.    : 06D008                       Date Extracted: 04/17/06 11: 30
Sample ID    : A77S2                         Date Analyzed: 04/19/06 11: 53
Lab Samp ID  : D008-05                       Dilution Factor: 1
Lab File ID  : TD18025A                      Matrix          : SOIL
Ext Btch ID  : DSD018S                       % Moisture     : 17.6
Calib. Ref.  : TB21024A                      Instrument ID   : GCT050
=====
  
```

| PARAMETERS | RESULTS (mg/kg) | RL (mg/kg) | MDL (mg/kg) |
|------------|--------------------|---------------|----------------|
| DI ESEL | 70 | 12 | 6.1 |
| MOTOR OIL | 410 | 24 | 6.1 |

| SURROGATE PARAMETERS | % RECOVERY | QC LIMIT |
|----------------------|------------|----------|
| BROMOBENZENE | 90 | 54-165 |
| HEXACOSANE | 132 | 54-176 |

RL : Reporting Limit
 Parameter H-C Range
 Diesel C10-C25
 Motor Oil C25-C36

Figure 6 – Typical LCS Summary

EMAX QUALITY CONTROL DATA LCS ANALYSIS

CLIENT: XYZ, INC.
 PROJECT: CLEAN LAND PROJECT
 BATCH NO.: 06D008
 METHOD: METHOD AK 103

=====

| | | | | |
|------------------|----------------|----------------|-----------------|----------|
| MATRIX: | SOIL | | % MOI STURE: | NA |
| DILUTION FACTOR: | 1 | 1 | | |
| SAMPLE ID: | MBLK1S | | | |
| LAB SAMP ID: | DSD018SB | DSD018SL | | |
| LAB FILE ID: | TD18026A | TD18025A | | |
| DATE EXTRACTED: | 04/17/0611: 30 | 04/17/0611: 30 | DATE COLLECTED: | NA |
| DATE ANALYZED: | 04/19/0606: 20 | 04/19/0605: 38 | DATE RECEIVED: | 03/31/06 |
| PREP. BATCH: | DSD018S | DSD018S | | |
| CALIB. REF: | TB21014A | TB21014A | | |

ACCESSION:

| PARAMETER | BLNK RSLT (mg/kg) | SPIKE AMT (mg/kg) | BS RSLT (mg/kg) | BS % REC | QC LIMIT (%) |
|-----------|----------------------|----------------------|--------------------|-------------|-------------------|
| Motor Oil | ND | 500 | 476 | 95 | 60-150 |

=====

| SURROGATE PARAMETER | SPIKE AMT (mg/kg) | BS RSLT (mg/kg) | BS % REC | QC LIMIT (%) |
|---------------------|----------------------|--------------------|-------------|-------------------|
| Bromobenzene | 100 | 96.5 | 96 | 60-150 |
| Hexacosane | 25 | 27.8 | 111 | 60-160 |

Figure 7 – Typical MS/MSD Summary

EMAX QUALITY CONTROL DATA
 MS/MSD ANALYSIS

CLIENT: XYZ, INC.
 PROJECT: CLEAN LAND PROJECT
 BATCH NO.: 06D008
 METHOD: METHOD AK 103

MATRIX: SOIL % MOISTURE: 22.8
 DILUTION FACTOR: 1 1 1
 SAMPLE ID: A81S10
 LAB SAMP ID: D008-10 D008-10M D008-10S
 LAB FILE ID: TD18027A TD18028A TD18029A
 DATE EXTRACTED: 04/17/0611:30 04/17/0611:30 04/17/0611:30 DATE COLLECTED: 03/31/06
 DATE ANALYZED: 04/19/0616:44 04/19/0613:17 04/19/0613:59 DATE RECEIVED: 04/01/06
 PREP. BATCH: DSD018S DSD018S DSD018S
 CALIB. REF: TB23003A TB21024A TB21024A

ACCESSION:

| PARAMETER | SMPL RSLT (mg/kg) | SPIKE AMT (mg/kg) | MS RSLT (mg/kg) | MS % REC | SPIKE AMT (mg/kg) | MSD RSLT (mg/kg) | MSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|-----------|----------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|------------|-----------------|----------------|
| Motor Oil | ND | 648 | 602 | 93 | 648 | 595 | 92 | 1 | 54-165 | 50 |

| SURROGATE PARAMETER | SPIKE AMT (mg/kg) | MS RSLT (mg/kg) | MS % REC | SPIKE AMT (mg/kg) | MSD RSLT (mg/kg) | MSD % REC | QC LIMIT (%) |
|---------------------|----------------------|--------------------|-------------|----------------------|---------------------|--------------|-----------------|
| Bromobenzene | 130 | 123 | 95 | 130 | 119 | 92 | 54-165 |
| Hexacosane | 32.4 | 38.3 | 118 | 32.4 | 35.8 | 111 | 54-176 |

Figure 8 – Typical Case Narrative

CASE NARRATIVE

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
SDG: 06D008

AK 103 AK RESIDUAL RANGE ORGANICS

Twenty (20) soil samples were received on 03/31/06 for Determination of Residual Range Organics (RRO) by Method AK 103, version 04/08/02.

1. Holding Time

Samples were extracted and analyzed within holding time.

2. Calibration

All target analytes met calibration requirements

3. Calibration Verification

All target analytes met calibration requirements

4. Method Blank

Method Blank was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement.

5. Lab Control Sample

LCS/LCD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits.

6. Matrix Spike/Matrix Spike Duplicate

That MS/MSD were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits.

7. Sample Analysis

Samples were analyzed in conformance to the method and project requirements.

SUMMARY OF QUALITY CONTROL PROCEDURES

| QC Procedure | Frequency | Acceptance Criteria | Corrective Action | Flagging Criteria | 1 st Rvw | 2 nd Rvw |
|--|--|--|---|--|------------------------|------------------------|
| Minimum five-point initial calibration | Initially; as needed | Mean RSD \leq 25% or Linear Regression @ $r^2 \geq$ 0.995 | Correct the problem then repeat initial calibration | | | |
| Second-source calibration verification | After initial calibration | Within \pm 25% of expected value | Correct the problem then repeat initial calibration | | | |
| Initial calibration verification | Daily, before sample analysis using mid-point CCS | Within \pm 25% of expected value | Correct the problem then repeat initial calibration | | | |
| Calibration verification | Every 12 hours of analysis time and end of analysis sequence | Within \pm 25% of expected value | Correct the problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification | | | |
| Method blank | One per preparation batch | No analyte detected \geq RL | Reprep and reanalyze method blank and all samples processed with the contaminated blank | Apply B to specific analyte(s) on all associated samples | | |
| LCS/LCD | One per preparation batch | Recovery: 60% - 120% RPD \leq 20% | Reprep and reanalyze the LCS and all associated samples | | | |
| Surrogate spike | Every sample, spiked sample, standard, and method blank | Recovery: 60%-120% (Lab Control Samples); 50%-150% (field samples) | Correct the problem then reextract and analyze sample | | | |
| MS/MSD | Analyze as specified by project. | Refer to EMAX QC Limits | None | | | |
| Chromatogram | All sample results | Within calibration range NO SATURATED PEAK(s) | Dilute and re-analyze all samples over the calibration range Diluted and re-analyzed all samples demonstrating saturated peak(s) even if the total integrated peaks do not exceed the calibration range. | | | |
| Results reported between MDL and RL | None | None | None | | | |
| Comments: RL= lowest calibration point | | | | Reviewed By | | |
| | | | | Date | | |

DEMONSTRATION OF CAPABILITY



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 Golden, CO 80403 www.wibby.com

Final Report - Rapid Return™

Study: RR-03452

Opening Date: March 27, 2007 - Closing Date: April 10, 2007

Laboratory: EMAX Laboratories
 1826 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-818-8889 ext. 205

EPA Lab ID: CA00291

| Alaska DRO in Soil (PT-AKDRO-USTS) | | | | | | | Lot #: RR-03452-29 | | |
|--|-------------------------|-------------|--------------------|-------|----------------|--------|--------------------|------------|--|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation | |
| 9399 | Diesel Range Organics | AK102 | AK102 | mg/kg | 618 | 563 | 420 - 816 | Acceptable | |
| Alaska Residual Range Organics in Soil (PT-AKRRO-USTS) | | | | | | | Lot #: RR-03452-26 | | |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation | |
| - | Residual Range Organics | AK103 | AK103 | mg/kg | 642 | 977 | 632 - 1050 | Acceptable | |

ANALYSIS RUN LOG FOR TPH

SOP EMAX-M8015D Revision No. 2

Book # A50-013

Starting Date:

Time:

Ending Date:

Time:

| | | | | | | | | | | | |
|-------------------------|-------------------|----------------|---------------|----|--------|---|-------|--|---------------------------------|-----------|--------------------------------|
| ANALYTICAL BATCH | Preparative Batch | Data File Name | Lab Sample ID | DF | Matrix | | Notes | Instrument No: | | 50 | |
| | | | | | S | W | | INITIAL CALIBRATION REFERENCE | | | |
| | | | | | | | | | ID | Date | |
| | | | | | | | | | Diesel | | |
| | | | | | | | | | Motor oil | | |
| | | | | | | | | | JP 5 | | |
| | | | | | | | | | | | |
| | | | | | | | | | Standards | | |
| | | | | | | | | | Name | ID | Conc. <small>(mg/L)</small> |
| | | | | | | | | | CH ₂ Cl ₂ | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | Disk Archival | | |
| | | | | | | | | | Name | Location | |
| | | | | | | | | | Comments: _____ | | |
| | | | | | | | | Analyzed By: _____ | | | |
| | | | | | | | | This page is checked during the data review process. | | | |

EXTRACTION LOG FOR TPH

SOP EMAX-3550 Rev. No.1 EMAX-3520 Rev. No.1 EMAX-LUFT E Rev. No.1 EMAX-3540 Rev. No.0 EMAX-3510 Rev. No. 0 EMAX-CLP

Matrix: _____ Start Date: _____ Time: _____ End Date: _____ Time: _____ Book # EDS-017

| PREPARATION BATCH * | Sample Prep ID | Lab Sample ID | Sonicator Number | Sample Amount (g ml) | Extract Volume (ml) | Clean-up [G] [F] [A] [S] | Notes | Standards | ID | Amount Added (ml) |
|---------------------|----------------|---------------|------------------|------------------------|---------------------|--------------------------|-------|-----------------------------------|--------------------------|-------------------|
| | 01 | | | | | | | | Surrogate | |
| 02 | | | | | | | | LCS/MS | | |
| 03 | | | | | | | | Reagent | Lot# / ID | |
| 04 | | | | | | | | CH ₂ Cl ₂ | | |
| 05 | | | | | | | | Na ₂ SO ₄ | | |
| 06 | | | | | | | | HCl | | |
| 07 | | | | | | | | Silica Sand | | |
| 08 | | | | | | | | TUNING | | |
| 09 | | | | | | | | Sonicator # | Reading | |
| 10 | | | | | | | | | | |
| 11 | | | | | | | | | | |
| 12 | | | | | | | | | | |
| 13 | | | | | | | | Concentrator Water Bath Temp. (C) | | |
| 14 | | | | | | | | 1 | | |
| 15 | | | | | | | | 2 | | |
| 16 | | | | | | | | 3 | | |
| 17 | | | | | | | | 4 | | |
| 18 | | | | | | | | 5 | | |
| 19 | | | | | | | | Comment: Test Thermometer = T1 | | |
| 20 | | | | | | | | | | |
| 21 | | | | | | | | | | |
| 22 | | | | | | | | Prepared By: _____ | Standard Added By: _____ | |
| 23 | | | | | | | | Witnessed By: _____ | Checked By: _____ | |
| 24 | | | | | | | | Extract Location: _____ | Received By: _____ | |
| | | | | | | | | Disposal Date: _____ | Disposed By: _____ | |

STANDARD OPERATING PROCEDURE

INDUCTIVE COUPLED PLASMA EMISSION SPECTROMETRIC METHOD
FOR TRACE METAL ANALYSES

SOP No.: EMAX-6010 Revision No. 5 Effective Date: 15--Jun-06
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 QA Manager
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 Laboratory Director

Control Number: 6010-05-

1.0 SCOPE AND APPLICATION

- 1.1. This procedure is applicable for trace metal quantitative analyses of drinking water, wastewater, groundwater, aqueous, extract, soil, sludge, and sediment samples using the Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) method. All matrices require proper sample preparation prior to analysis. Two different ICPs are employed in this procedure. Two are Thermo-Jarrell Ash (TJA) TRACE-ICP-61E and the other is the regular TJA ICP-61E instrument.
- 1.2. The elements and their corresponding wavelengths are listed in Table 1 and Table 1A.
- 1.3. This SOP is an adaptation of the SW846 Method 6010B.

2.0 SUMMARY OF METHOD

- 2.1. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) is employed to determine individual elements through their respective atomic or energy emission at different wavelengths. Samples are nebulized and the generated aerosol is carried through the center of the plasma torch, where the metals atoms are excited into high electronic states. Atomic and ionic line emission spectra characteristics of the particular metals are produced when the electron decays back to the lower energy levels. The emission is dispersed by a grating spectrometer and the intensities of the specific lines are monitored simultaneously by photomultiplier tubes. The photocurrent produced by the photomultiplier tube will increase in direct proportion to the concentration of the element in the sample within the linear range of a specific emission line. The photocurrent is processed by a computer system and related to concentration through a calibration procedure. The calibration procedure is made on the assumption that the instrumental response is linear with concentration within the dynamic range.
- 2.2. Calibration is performed by standardizing the instrument with a blank that is matrix matched to the digestate and one standard. The calibration curve is verified by another standard from a different source before any sample is analyzed. The intensity of emission of each element is measured and compared to a calibration curve for each element concentration versus intensity.
- 2.3. **Interference**
 - 2.3.1. **Physical Interference.** Contamination might be introduced by the memory effect of metals during previous analysis (carry over) adhering to the walls of the pumping system and the nebulization chamber. Adequate rinsing of at least 90 seconds using matrix acid blank (refer to Section 9.2) shall be performed between sample analyses to minimize system contamination.
 - 2.3.2. **Chemical Interference.** Matrix matching between digestates and all standard and samples dilution is used in sample analysis to eliminate/minimize chemical interference.
 - 2.3.3. **Spectral Interference.** Both off-peak (background) and on-peak (inter-element correction coefficients) interference corrections are applied for all affected elements to take care of spectral interference. Refer to Section 10.3.4.

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3.0 QUANTITATION LIMITS**3.1. Method Detection Limit**

3.1.1. Prepare and analyze a minimum of seven MDL samples with method blank (MB) and lab control sample (LCS) as described in Section 10. Refer to Table 2A of EMAX QA04 for suggested concentrations.

3.1.2. Refer to EMAX QA04 for acceptance criteria.

3.2. Reporting Limits (RLs)

3.2.1. Target analytes with respective reporting limits are listed in Table 4, Table 5 and Table 6.

3.3. Instrument Detection Limit (IDL)

3.3.1. Establish IDL as described in Section 10.3.5.

3.3.2. Determine IDL every three months.

4.0 LINEAR DYNAMIC RANGE (LDR)

4.1. Establish LDR for each element as described in Section 10.3.6.

4.2. Verify LDR every six months or when there is a significant change in the instrument signal.

5.0 SAMPLE HOLDING TIME AND PRESERVATION**5.1. Water Samples**

5.1.1. Water samples are collected in polyethylene or glass bottles.

5.1.2. For total recoverable metal analysis, the sample is preserved at the time of collection to pH < 2 with nitric acid.

5.1.3. For dissolved metal analysis, the sample is filtered through a 0.45 µm filter at the time of collection and preserved to pH < 2. Samples requested to be properly preserved in the lab shall observe at least 24 hours from the time preservative is added before sample digestion.

5.1.4. Samples shall be stored in the same condition as received unless specified in the project requirement.

5.2. Soil Samples

5.2.1. Soil samples are collected in glass jars or brass tubes.

5.2.2. Soil samples are stored at 4 °C (+2°C) until analysis.

5.3. Holding Time

5.3.1. All samples must be analyzed within 180 days from the collection date.

6.0 ASSOCIATED SOPs

6.1. EMAX-QC01 - Quality Control for Chemicals

6.2. EMAX-QC02 - Analytical Standard Preparation

6.3. EMAX-QA04 - IDL/MDL/RL

6.4. EMAX-QC07 - Glassware Cleaning

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- 6.5. EMAX-QA08 - Corrective Action
- 6.6. EMAX-SM04 - Analytical and QC Sample Labeling
- 6.7. EMAX-3005 - Acid Digestion, Total Recoverable or Dissolved Metals
- 6.8. EMAX-3010 - Acid Digestion, Total Metals (Aqueous)
- 6.9. EMAX-3050 - Acid Digestion, Total Metals (Solids)

7.0 SAFETY

- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves at all times when performing this procedure. Observe all chemical hygiene procedures as mentioned in the Chemical Hygiene Plan .
- 7.3. Place all wastes generated during analytical process in the waste containers. Endorse these wastes to waste disposal section for proper disposal.
- 7.4. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES**8.1. Instruments and Supplies**

- 8.1.1. ICPs: Thermo-Jarrell Ash ICAP-61E; Thermo-Jarrell Ash Trace ICAP61E
- 8.1.2. Autosampler: Thermo-Jarrell Ash 300
- 8.1.3. Computer: IBM Compatible
- 8.1.4. RF Generator: TJA RF Generators
- 8.1.5. Data Acquisition: Thermospec Software Rev. 6.2
- 8.1.6. Autosampler rack(s): 13 mm, 75 positions
- 8.1.7. Culture tubes: 13 mm, polystyrene
- 8.1.8. Volumetric Flask: 100, 250, 500, 1000 ml
- 8.1.9. Micropipets: 100, 1000, 5000, 10000 μ l
- 8.1.10. Pipet Tips: 200, 1000, 5000, 10000 μ l
- 8.1.11. Polyethylene bottles: 125, 250, 500, 1000 ml
- 8.1.12. Liquid argon
- 8.1.13. Liquid nitrogen

8.2. Chemicals and Reagents

- 8.2.1. DI water, ASTM Type II or equivalent
- 8.2.2. Nitric Acid, Trace high purity grade, concentrated
- 8.2.3. Hydrochloric acid, Trace high purity grade, concentrated

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9.1.1. The stock standards are purchased as certified single element solutions in 1000 mg/L and 10,000 mg/L or as custom-made certified mixed stock solutions in various concentrations.

9.1.2. The following table is the list of the custom-made certified mixed stock standard solutions:

| SOURCE | STANDARD | ELEMENTS | CONC. (mg/L) | MATRIX |
|--------------|-----------|---|--------------|--|
| AccuStandard | X-EMAX-4 | Ca, Mg, Na, K | 10000 | 5% HNO ₃ |
| AccuStandard | X-EMAX-6A | Sb, As, B, Mo, Sr, Tl, Ti, V, Zn, Se | 500 | 5% HNO ₃ and Trace HF/Tartaric Acid |
| | X-EMAX-6B | Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Ni, Ag | 500 | 10% HNO ₃ |
| CPI | MIX3 | Ca, Mg, K, Na | 10000 | 5% HNO ₃ |
| CPI | MIX2 | Sb, Mo | 500 | 5% HNO ₃ and Trace HF |
| CPI | MIX1 | As, Ba, Be, Bi, B, Cd, Cr, Co, Cu, Pb, ZN Mn, Ni, Se, Sr, Tl, V, Ti Ag | 500 | 5% HNO ₃ and Trace HF |
| AccuStandard | CRDL-1 | Sb | 120 | 5% HNO ₃ |
| | | Ni | 80 | |
| | | Cu | 50 | |
| | | Zn | 40 | |
| | | Mn | 30 | |
| | | Pb | 6 | |
| | | Co, V | 100 | |
| | | As, Cr, Ag, Tl | 20 | |
| | | Be, Cd, Se | 10 | |
| AccuStandard | INT-B1 | Ag, Cd, Ni, Pb, Zn | 100 | 5% HNO ₃ |
| | | Ba, Be, Co, Cr, Cu, Mn, V | 50 | |
| AccuStandard | INT-A1 | Al, Ca, Mg | 5000 | 5% HNO ₃ |
| | | Fe | 2000 | |

9.1.3. Detailed procedure of standard preparation and labeling is described in EMAX-QC02 and EMAX-SM04, respectively.

9.2. Matrix Acid Blank (S₀)

9.2.1. The matrix acid blank is used for dilutions of the standards and, digestates, if necessary.

9.2.2. Prepare this solution by mixing 6% by volume nitric acid and 5% by volume hydrochloric acid in reagent water. Transfer into a clean HDPE bottle and identify the solution as S₀.

9.3. Initial Calibration Standard

9.3.1. The initial calibration consists of a blank (S₀) and one standard.

9.3.2. Prepare Reg-S₆ by diluting 4 ml each of X-EMAX-6A and EMAX-6B and 1-ml of 10,000 mg/L Sn to a final volume of 1-liter using S₀ (blank). Transfer the standard on a clean HDPE bottle and identify the solution as ICP-S₆.

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- 9.3.3. Prepare Reg-S₃ by diluting 10 ml of X-EMAX-4, 1-ml each of 10,000 mg/L of Al and Fe to a final volume of 1-liter using S₀. Transfer the standard on a clean HDPE and identify the solution as ICP-S₃.
- 9.3.4. Prepare Trace-S₂ by diluting 2-ml of each of EMAX-6A and EMAX-6B to a final volume of 1-liter with S₀. Transfer the standard on a clean HDPE and identify the solution as Trace-S₂.
- 9.3.5. Prepare Trace-S₅ by diluting 10-ml of each of EMAX-4 and 1-ml each of 10,000 mg/L of Al and Fe to a final volume of 1-liter with S₀. Transfer the standard on a clean HDPE and identify the solution as Trace-S₅.
- 9.3.6. Prepare ICP-Low – S₂ by diluting 2-ml of X-EMAX-6A to a final volume of 1000-ml with S₀ (blank). Transfer the standard on a clean HDPE and identify as ICP-Low S₂.
- 9.3.7. Prepare ICP-Low – S₅ by diluting 2-ml of X-EMAX-6B to a final volume of 1000-ml with S₀ (blank). Transfer the standard on a clean HDPE and identify as ICP-Low S₅.
- 9.3.8. Prepare ICP-Low – S₉ by diluting 10-ml of X-EMAX-4, 1-ml each of 10,000 mg/L of Aluminum (Al) and Iron (Fe), 1-ml of 1000 mg/L of Lithium (Li) and 10-ml of 1000 mg/L of Uranium (U) to a final volume of 1000-ml with S₀ (blank). Transfer the standard on a clean HDPE and identify as ICP-Low S₉.
- 9.3.9. Refer to Tables 2, 3 and 6 for final concentrations.
- 9.4. **Initial Calibration Verification (ICV)**
- 9.4.1. Purchase ICV mixed stock standards solution from a different source other than the initial calibration standard. The concentrations of the analytes are approximately one-half of the concentration that is found in the initial calibration. Refer to Section 9.1 for the ICV analyte list.
- 9.4.2. Prepare ICV for ICP-61E by diluting 2-ml of CPI-MIX-1, 2mL CPI-MIX-2 and 5mL of CPI-MIX3 and 0.5 mL each of 10,000mg/L Al, Fe and Sn to 1-L with S₀. Transfer the solution into a clean HDPE bottle and identify the solution as ICP-ICV.
- 9.4.3. Prepare ICV for Trace by diluting 0.5-ml each of 1000-µg/ml As, Pb, Se, Tl, Cd, Mn, V, Zn, Cr, 10,000-µg/ml Al, Fe, and 5 ml each of 10,000-µg/ml Ca and Mg to a final volume of 1-liter with S₀. Transfer the solution into a clean HDPE bottle and identify the solution as ICV-Trace.
- 9.4.4. Prepare ICV for ICP-Low by diluting 1-ml of CPI Mix1 and Mix 2, 5-ml of CPI Mix3, 0.5-ml each 10,000 mg/L of Al and Fe, 0.5-ml of 1000 mg/L of Li and 5-ml of 1000 mg/L of Uranium to a final volume of 1000-ml of S₀.
- 9.4.5. Refer to Table 2, 3 and 6 for final concentrations.
- 9.5. **Continuing Calibration Verification (CCV) Standard**
- 9.5.1. Prepare ICP-CCV by diluting 7.5-ml of X-EMAX-4, 0.75-ml each of 10,000 µg/ml Al and Fe and Sn and 3-ml each of X-EMAX-6A and EMAX-6B to a final volume of 1-liter with S₀. Transfer the standard on a clean HDPE bottle and identify the solution as ICP-CCV.
- 9.5.2. Prepare CCV- Trace by diluting 0.4-ml each of 1000-µg/ml As, Pb, Se, Tl, Cd, Mn, V, Zn, Cr, 0.4-ml each of 10,000 µg/ml Al and Fe and 4-ml each of 10,000-µg/ml Ca and Mg to a final volume of 1-L with S₀. Transfer the standard on a clean HDPE and identify the solution as CCV- Trace.
- 9.5.3. Prepare CCV- ICP-Low by diluting 1.5-ml each of 1000-µg/ml X-EMAX-6A and 6B, 0.75-ml each of 10,000 µg/ml Al, Sn and Fe, 0.75-ml of 1000 µg/ml of Lithium, 7.5-ml each of X-EMAX-4 and 1000-µg/ml Uranium, to a final volume of 1-L with S₀. Transfer the standard on a clean HDPE and identify the solution as CCV- ICP-Low.

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9.5.4. Refer to Tables 2, 3 and 6 for final concentrations.

9.6. Interference Check Standard, Primary Interferents A and Analytes B (ICSAB)9.6.1. Prepare ICSA by diluting 100-ml of INT-A1 to 1,000 ml with S₀. Transfer the solution into a HPDE bottle and identify the solution as ICSA. This solution can be use for both ICP-61E and Trace.9.6.2. Prepare the ICSAB by diluting the following standards to 1,000 ml with S₀.

- 100-ml of INT-A1,
- 10-ml of INT-B1,
- 5-ml of K (10,000 mg/L), 1-ml Na (10,000 mg/L), 1-ml from each of As, Mo, Ti, (1000 mg/L individual standards), 3.6 ml Se, Tl (1,000 mg/L individual standards)
- 0.5 ml of B & Sr (1000 mg/L individual standards)
- 0.1-ml of Sb and Sn (10,000 mg/L individual standard).
- 1-ml of Li & U (1000 mg/L individual standards)

9.6.3. Transfer the solution into a HPDE bottle and identify the solution as ICP-ICSAB.

9.6.4. Dilute 100-ml of INT-A1 and 10-ml of INT-B1 and 1-ml each of As, Se, Tl to 1-L with S₀. Transfer the solution into a HDPE bottle and identify as Trace-ICSAB.

9.6.5. Refer to Table 4 and 5 for final concentrations.

9.7. Application of Analytical Standards

9.7.1. All standard concentrations described in Section 9 are typical concentrations. They may vary at any time to meet project requirement or any related requirement. Where changes are made, Method QC requirements and/or project requirement must be met.

10.0 PROCEDURES**10.1. Sample Preparation**

10.1.1. For aqueous samples to be analyzed for total recoverable metals and/or dissolved metals – refer to EMAX-3005.

10.1.2. For aqueous samples to be analyzed for total metals – refer to EMAX-3010.

10.1.3. For solid samples to be analyzed for total metals – refer to EMAX-3050.

10.2. Instrument Parameters**10.2.1. Regular ICP**

- RF Power: 1350 W
- Nebulizer Pressure: 15-45 psi
- Analysis Pump Rate: 100-200 RPM
- Flush Pump Rate: 100-200 RPM
- No. Lines: 31
- Analysis Time: 5 minutes
- No. of Replicates: 2

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- RF Power: 950 W
- Nebulizer Pressure: 15-45 psi
- Analysis Pump Rate: 100-200 RPM
- Flush Pump Rate: 100-200 RPM
- No. Lines: 33
- Analysis Time: 6 minutes
- No. of Replicates: 2
- Internal Standard: Yttrium, 5 MG/L

10.2.3. Trace ICP

- RF Power: 950 W
- Nebulizer Pressure: 15-45 psi
- Analysis Pump Rate: 100-200 RPM
- Flush Pump Rate: 100-200 RPM
- No. Lines: 15 (2 Lines each of Lead and Selenium)
- Analysis Time: 5 minutes
- No. of Replicates: 2

10.3. Calibration**10.3.1. Instrument Set-Up**

- 10.3.1.1. Set up the ICP-61E or TRACE-ICP with proper operating parameters. Refer to Section 10.2.1 and 10.2.2 respectively.
- 10.3.1.2. Ignite the plasma and allow the instrument to become thermally stable at least 1 hour.
- 10.3.1.3. Check the peristaltic pump to deliver a steady flow.

10.3.2. Profile the Instrument

| Instrument | Profile Solution | Vernier Position |
|------------|----------------------------|------------------|
| ICP-61E | 10.0 µg/ml copper solution | ± 0.05 or better |
| TRACE-ICP | 1.0 µg/ml arsenic solution | ± 0.05 or better |
| ICP-Low | 1.0 µg/ml arsenic solution | ± 0.05 or better |

10.3.3. Initial Calibration

- 10.3.3.1. Set the system to flush with the matrix acid blank between each analysis run for a minimum of 90 seconds.
- 10.3.3.2. Run the calibration standards as described in Section 10.4. Refer to Appendix 1 for acceptance criteria.

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- 10.3.4.1. Analyze a 100-mg/L single element. For elements such as Fe, Mg, Ca and Al, use a 500-mg/L concentration. The analytical results of these target analyte shall fall below $\frac{1}{2}$ of the reporting limit.
- 10.3.4.2. Calculate the IEC factor by dividing the apparent target analyte concentration to the actual concentration of the interference check solution found in the analysis.
- 10.3.4.3. Follow the same procedure for uncommon elements that may contribute spectral interference.
- 10.3.4.4. Verify and update all IEC factors or multivariate correction matrices every six months or when major instrumentation maintenance is performed (e.g., cleanup/changes of torch, nebulizer, injector, or plasma conditions)

10.3.5. Establishing Instrument Detection Limit (IDL)

- 10.3.5.1. Analyze a minimum of seven consecutive method blanks.
- 10.3.5.2. Repeat the process within three non-consecutive days.
- 10.3.5.3. Calculate the standard deviation of each run.
- 10.3.5.4. The average of the standard deviation of the three runs determines the IDL for each analyte.

10.3.6. Establishing Linear Dynamic Range (LDR)

- 10.3.6.1. Analyze a minimum of three standards at different concentrations, one of which is close to the upper limit of the linear range. Refer to Table 5 for suggested LDR concentration levels.
- 10.3.6.2. The upper limit of the linear range is an observed signal no more than 10% below the level extrapolated from lower standards.
- 10.3.6.3. Prepare a standard at the determined LDR, analyze and quantitate against the normal calibration curve to verify the LDR. Percent recovery must be within $\pm 10\%$ of the expected value. If non-compliant, re-establish the LDR.

10.4. Analysis**10.4.1. Analytical Sequence.**

- 10.4.1.1. Input the analytical sequence into the instrument using data acquisition software. Refer to Table 7.
- 10.4.1.2. Set QC limits on QC samples for easy verification while analytical samples are on the run.
- 10.4.1.3. Using the analytical sequence, shake the sample container then aliquot about 7 ml of standards/samples into the sample tubes placing them on the same order as the analytical sequence.
- 10.4.1.4. Dilution Test sample is prepared at 5 times dilution. Pipette 1.4- ml of sample , add 5.6- ml of S_0 into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.

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- 10.4.1.5. Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.
- 10.4.1.6. Set the prepared analytical samples into the auto-sampler and start the analytical run.
- 10.4.1.7. Check the initial calibration verification for compliance (see Appendix 1 for QC criteria). If non-compliant, stop the run, correct the problem and recalibrate.
- 10.4.1.8. Check the sample rack to ensure that the Autosampler did not skip any sample.

10.5. Data Reduction

- 10.5.1. Dilute and reanalyzed all samples that exceeded the DLR.
- 10.5.2. Check the MS/MSD, serial dilution and the post digestion spike results. If matrix interference is indicated, check the PSR if MSA is waived, otherwise refer to 10.5.3.

10.5.3. Method of Standard Addition (MSA)¹

- 10.5.3.1. Perform MSA for all EP extracts, samples for delisting petition and whenever a new matrix is encountered.
- 10.5.3.2. Prepare two sample solutions (Ms1 and Ms2) with identical sample aliquot (V_x). Add reagent water to Ms1 and spike standard to Ms2. Choose a concentration and volume of spike standard so that the response of Ms1 is twice of Ms2 avoiding excessive dilution of the sample.

- $Ms1 = V_x + V_w$
- $Ms2 = V_x + V_s$

where:

V_x - volume of sample

V_s - volume of spike

V_w - volume of reagent water

Example: Sample concentration is tentatively determined at 5 mg/L.

Ms1 – take 5-ml of sample then add 5-ml of reagent water. This solution will have an approximate concentration of 2.5 mg/L.

Ms2 – take 5-ml of sample then add 5-ml of 5-mg/L spike standard. This solution will have an approximate concentration of 5 mg/L.

- 10.5.3.3. Analyze Ms1 and Ms2 and calculate the results using Eq-10.6.6.
- 10.5.3.4. For TCLP extracts, perform MSA* if;
- ✓ The recovery of the matrix spike is $\leq 50\%$ and the concentration of the contaminant does not exceed the regulatory level.
 - ✓ When the measured concentration of the analyte in the extract is within 20% of the appropriate regulatory level.

Note: *Matrix spike standards must be added after filtration (pre-digestion) of the TCLP extract and prior to preservation.

¹ Adopted from Section 8.7.1 USEPA SW846 Method 7000

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10.6.1. The computer software is designed to calculate the concentrations in the digestates, based on the assumption that the initial calibration is linear through the origin. Thus, for aqueous samples, the computer-produced results represent the concentration of the sample.

10.6.2. For water samples, if the initial sample taken was V_s , different from 100 mL, then calculate the concentration using the following equation:

$$C_s = C_i \left(\frac{V_e}{V_s} \right) DF \quad \text{Eq-10.6.2}$$

where:

C_s - concentration in the sample, mg/L

C_i - concentration in the digestate, mg/L

V_s - volume of sample taken, ml

V_e - volume of digestate, ml

DF - dilution factor

10.6.3. For solids, use the following equation to calculate the concentration.

$$C_s = C_i \left(\frac{V_e}{W_s} \right) \left(\frac{100}{100 - \%H_2O} \right) DF \quad \text{Eq-10.6.3}$$

Where:

C_s - concentration in the sample, mg/Kg

C_i - concentration in the digestate (computer generated), mg/Kg

W_s - sample amount taken, g

V_e - volume of digestate, ml

$\%H_2O$ - percent moisture of the sample

DF - dilution factor

10.6.4. Calculate the percent recovery (%R)

$$\% \text{ Recovery} = \frac{C_f - C}{C_s} * 100 \quad \text{Eq-10.6.4}$$

where:

C_f - concentration found

C - concentration of sample

C_s - concentration of spike

10.6.5. Relative Percent Difference (%RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2} \right)} \times 100 \quad \text{Eq-10.6.5}$$

Where:

RPD = Relative Percent Difference

C_1 = Measured concentration of the first sample aliquot

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- 10.6.6. Calculate for sample result from determined from MSA

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x} \quad \text{Eq-10.6.6}$$

Where:

 C_x – concentration of the sample C_s – concentration of the spike S_1 - analytical signal of Ms1 S_2 - analytical signal of Ms2 V_x – volume of sample aliquot V_s – volume of spike/reagent water**10.7. Report Generation**

- 10.7.1. Print the summary of the analytical run, perform a data transfer into a disk, and convert the instrument electronic output file into an ASCII file format.
- 10.7.2. Run the ICPCHK.exe program for calibration check.
- 10.7.3. Identify samples that need to be re-analyzed, if any, and report all samples that met the analytical requirements.
- 10.7.4. Generate the report using the following in-house reporting program:

| Executable Files | Required Support Files | Output |
|------------------------|---|--|
| WDBX ² .exe | Login File (requires network) Seq_name.sq; Gcints.txt | Method.txt [this file integrates the login sample information and the analytical sample information] |
| IF1VX.exe | Method.txt; Method.met; Method.crf; Project.pln; Qcell.txt | Sample Results (Form1) |
| IQCVX.exe | Method.txt; Method.crf; Method.qc; Project.pln; Qcell.txt | QC Summary for LCS and MS |
| QCX | Method.txt; Method.crf; Method.qc; Project.pln; Qcell.txt | Summary for Dilution Test |

10.7.5. Data Review

- 10.7.5.1. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated and re-analyses.

- Sample Results
- LCS Summary
- MS/MSD Summary

² X – latest program version

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- CCV Summary
- ICV Summary
- ICAL Summary

10.7.6. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- ✓ Review the ICPCHK.exe output file to ensure that it agrees with the instrument output. Check Project Specific Requirement (PSR) or Appendix 1 for acceptance criteria.
- ✓ Check frequency of calibration verification. Verify results to be within acceptance limits.
- ✓ Check of target analytes concentration to be within linear range.
- ✓ Verify interference check results to be within acceptance limits.

10.7.6.1. If any of the above checkpoints is non-compliant, re-analysis is required.

10.7.7. Generate the case narrative to include discussion of the following as found in the review process:

- Number of samples analyzed
- Analytical method(s) applied
- Holding Time – That samples were analyzed within holding time. For non-compliance, state the number of days/hours that the sample(s) were off from holding time.
- Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
- Initial Calibration Verification (ICV) – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
- Method Blank – That MB was analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte affected and the associated sample results were flagged with “B”.
- Lab Control Sample – That LCS was analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Furthermore, if corrective action is not possible (e.g., no more samples to re-analyze) state that results were qualified.
- Matrix Spike/Matrix Spike Duplicate – That MS/MSD is extracted with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Dilution Test and Post Digestion Spike – That dilution test and post digestion spike are analyzed for every preparation batch. That positive results of dilution test are evaluated to meet the method requirement and that Post Digestion Spike is evaluated for analytes that are not detected. For non-compliant analytes, reference the associated form, (Serial Dilution Test or Analytical Spike) and that non-compliant results were indicated by “*” or refer to MSA result.

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- Sample Analysis – That samples were analyzed in conformance to the method and project requirements. That positive results were qualitatively identified in accordance to the method and applied quantitation is based on the initial calibration.
- Other Anomalies (if any) – Shall be discussed on a case to case basis concurred by the Supervisor or the Lab Director. Include the NCR in the data package if required by the project, otherwise archive the NCR with the analytical folder.

10.7.8. Submit the analysis package for secondary review.

10.8. Preventive Maintenance

10.8.1. Perform daily instrument check prior to sample analysis. Refer to 6010-F3 – Instrument Maintenance Log.

10.8.2. Maintain an inventory of instrument parts and supplies for routinely maintenance.

11.0 QUALITY CONTROL ANALYSES**11.1. Sample Preparation QC**

11.1.1. A preparative batch consists of 20 or fewer samples of the same matrix, that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.

11.1.2. Every preparative batch shall have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. These QC samples shall be digested together with the field samples.

11.1.3. All reagents shall be subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2. Sample Analysis QC

11.2.1. Every analytical run shall be preceded with an initial calibration and initial calibration verification (ICV). Obtain the ICV standard from a different source from that of the initial calibration. Analyze an instrument calibration blank (ICB) after the ICV. No further analysis shall be valid unless acceptance criteria are met.

11.2.2. Verify inter-element and background correction factors with ICSA and ICSAB standards after ICB.

11.2.3. Verify calibration with continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the analytical run.

11.2.3.1. Dilution Test shall be performed whenever a new or unusual sample matrix is encountered.

11.2.3.2. Evaluate Post Digestion Spike result if the dilution test failed to meet the acceptance criteria.

11.2.4. Refer to Appendix 1 for acceptance criteria.

11.3. Method QC

11.3.1. Method Detection Limit Study must be established before the analytical procedure can be used.

11.3.2. Method proficiency must be established before the analytical procedure can be used.

11.3.3. All analysts conducting this analysis must have established demonstration of proficiency.

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- 11.3.4. Perform dynamic range study at least every six months or whenever there is a significant change in instrument response. The analytically determined concentration of this standard must be within 10% of the expected value.

12.0 CORRECTIVE ACTION

- 12.1. Quality control procedures and corresponding corrective actions are summarized in Appendix 1.
- 12.2. When ICV is non-compliant consider the following options to correct the problem prior to recalibration:
- Check the gas flow if it is conforming to the instrument parameter setting and correct if necessary.
 - Check the pump tubings and replace if necessary.
 - Check the nebulizer for clogs and clean if necessary.
 - Prepare new standards and re-calibrate
- 12.3. When ICB/CCB is non-compliant, consider the following suggestions to correct the problem:
- Replace the instrument blank (S0) with a fresh reagent water and re-analyze the blank.
 - If problem persist, check the pump tubings and the nebulizer for clogs and perform maintenance as necessary.
- 12.4. When Method Blank (MB) is non-compliant, consider the following suggestions to correct the problem:
- Check if associated samples detected the same analytes detected in the MB.
 - If analytes detected on the MB is not detected in the associated samples, report the results and flag the analytes detected on the MB as required by the project.
 - If similar analytes are detected at concentration levels greater than 10X the detection limits, refer the issue to the PM if the result is reportable, otherwise check the source of contamination, correct the problem and re-digest all associated samples.
 - Prepare and analyze a reagent blank to contain the same acid strength as the digested method blank. If contamination exists, check reagent to identify where the contamination is coming from. Use new reagents as necessary and re-digest/re-analyze all associated samples.
- 12.5. A Non-Conformance Report (NCR) is required when the following circumstances occur:
- 12.5.1. Corrective action needs the function of other department, e.g. the sample needs to be re-digested. Refer to EMAX-QA08 for details of completing an NCR.
- 12.5.2. Corrective action needs the assistance of the Project Manager, e.g. sample passed the holding time, insufficient amount of sample, etc.
- 12.5.3. Corrective action prescribed in the IQC Summary does not correct the problem.
- 12.6. For other problems encountered, inform the supervisor immediately for further instruction.

13.0 POLLUTION PREVENTION

- 13.1. Unused samples, digestates and instrument wastes are very acidic and are very corrosive. Endorse the wastes to the Waste Management Unit for proper treatment or disposal.

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14.0 WASTE MANAGEMENT

- 14.1. All unused samples expired analytical standards and other waste generated during the analytical process, endorse to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES**15.1. Definition of Terms**

- 15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
- 15.1.1.1. **Preparation batch** is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
- 15.1.1.2. **Analytical batch** is compose of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.2. Calibration – is defined as an instrument response per unit measure. It is an experimental value by measuring the response of an instrument per unit target analyte under the method specific condition. A determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.3. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.4. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.5. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyze to monitor the accuracy of the analytical system.
- 15.1.6. Matrix – is a physical state of a sample. Most of environmental samples are classified as water, soil or air.
- 15.1.7. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyze to monitor matrix effect on a method's recovery efficiency.
- 15.1.8. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.9. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical procedure to monitor contamination.
- 15.1.10. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.11. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

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15.1.12. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.2. Application of EMAX QC Procedures

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing metals analysis by ICP. In instances where there is a project or program QAPP, the requirements given in the project shall take precedence over this SOP.

15.3. Air Force Center for Environmental Excellence (AFCEE) Projects

15.3.1. When samples from AFCEE sponsored projects are analyzed for metals by ICP, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the Quality Assurance Project Plan, the latest version.

15.4. U.S. Army Corps of Engineers (USACE) Projects

15.4.1. When samples from USACE sponsored projects are analyzed for metals by ICP, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, Shell Document latest version shall be applied.

15.5. Naval Facilities Engineering Service Center (NFESC) Projects

15.5.1. When samples from NFESC sponsored projects are analyzed for metals by ICP, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, NFESC Laboratory Quality Assurance Guide latest version shall be applied

15.6. Department of Energy Basic Ordering Agreement (DOE-BOA) Projects

15.6.1. For samples from DOE-BOA sponsored projects follow BOA Guidance Document, latest version in the absence of project QAPP.

16.0 REFERENCES

- 16.1. Test Methods for Evaluating Solid Waste, Method 6010B, Physical and Chemical Methods, US EPA SE-846, 3rd edition, 1992.
- 16.2. Quality Assurance Manual, EMAX Laboratories, as updated.

17.0 TABLES AND APPENDICES

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TABLE 1
ICP ELEMENTS & WAVELENGTHS

| ELEMENT | SYMBOL | WAVELENGTHS | | |
|------------|--------|-------------|----------------------------|----------------------------------|
| | | ICP-61E | TRACE-ICP | |
| | | | Wavelength | Channel |
| Aluminum | Al | 308.215 | 308.215 | |
| Antimony | Sb | 206.838 | | |
| Arsenic | As | 193.696 | 189.042 | |
| Barium | Ba | 493.409 | | |
| Beryllium | Be | 313.042 | | |
| Boron | B | 249.678 | | |
| Cadmium | Cd | 228.802 | 228.800 | |
| Calcium | Ca | 317.933 | 317.933 | |
| Chromium | Cr | 267.716 | | |
| Cobalt | Co | 228.616 | | |
| Copper | Cu | 324.754 | 324.754 | |
| Iron | Fe | 259.940 | 271.441 | |
| Lead | Pb | 220.353 | 220.351 (1) 220.352 (1) | Channel 2203/1 Channel 2203/2 |
| Magnesium | Mg | 279.078 | 279.078 | |
| Manganese | Mn | 257.610 | 257.610 | |
| Molybdenum | Mo | 202.030 | | |
| Nickel | Ni | 231.604 | | |
| Potassium | K | 766.491 | | |
| Selenium | Se | 196.026 | 196.021 (2) 196.022 (2) | Channel 1960/1 Channel 1960/2 |
| Silver | Ag | 328.068 | | |
| Sodium | Na | 588.995 | | |
| Strontium | Sr | 421.552 | | |
| Thallium | Tl | 190.864 | 190.862 | |
| Tin | Sn | 189.989 | | |
| Titanium | Ti | 334.941 | | |
| Vanadium | V | 292.402 | 292.402 | |
| Zinc | Zn | 213.856 | 213.856 | |

TABLE 1-A
ICP-Low ELEMENTS & WAVELENGTHS

| ELEMENT | SYMBOL | WAVELENGTH | HIGH STANDARD | LOW STANDARD |
|------------|--------|------------|----------------|----------------|
| 1960_1 | -- | 196.090 | S ₂ | S ₀ |
| 1960_2 | -- | 196.091 | S ₂ | S ₀ |
| 2203_1 | -- | 220.353 | S ₅ | S ₀ |
| 2203_2 | -- | 220.354 | S ₅ | S ₀ |
| Aluminum | Al | 308.215 | S ₉ | S ₀ |
| Antimony | Sb | 206.833 | S ₂ | S ₀ |
| Arsenic | As | 189.042 | S ₂ | S ₀ |
| Barium | Ba | 493.409 | S ₅ | S ₀ |
| Beryllium | Be | 313.042 | S ₅ | S ₀ |
| Boron | B | 249.678 | S ₂ | S ₀ |
| Cadmium | Cd | 226.502 | S ₅ | S ₀ |
| Calcium | Ca | 317.933 | S ₉ | S ₀ |
| Chromium | Cr | 267.716 | S ₅ | S ₀ |
| Cobalt | Co | 228.616 | S ₅ | S ₀ |
| Copper | Cu | 324.754 | S ₅ | S ₀ |
| Iron | Fe | 271.441 | S ₉ | S ₀ |
| Lead | Pb | 220.355 | None | None |
| Lithium | Li | 670.784 | S ₉ | S ₀ |
| Magnesium | Mg | 279.079 | S ₉ | S ₀ |
| Manganese | Mn | 257.610 | S ₅ | S ₀ |
| Molybdenum | Mo | 202.030 | S ₂ | S ₀ |
| Nickel | Ni | 231.604 | S ₅ | S ₀ |
| Potassium | K | 766.490 | S ₉ | S ₀ |
| Selenium | Se | 196.092 | None | None |
| Silver | Ag | 328.068 | S ₅ | S ₀ |
| Sodium | Na | 588.995 | S ₉ | S ₀ |
| Strontium | Sr | 421.552 | S ₂ | S ₀ |
| Thallium | Tl | 190.864 | S ₂ | S ₀ |
| Tin | Sn | 189.989 | S ₅ | S ₀ |
| Titanium | Ti | 334.941 | S ₂ | S ₀ |
| Uranium | U | 409.014 | S ₉ | S ₀ |
| Vanadium | V | 292.402 | S ₂ | S ₀ |
| Zinc | Zn | 213.856 | S ₂ | S ₀ |

Table 2
CALIBRATION STANDARD PREPARATION
METHOD 6010B (ICAP-61E)

| Standard # | Mixed Standard Name | Conc. (mg/L) | Source | Preparation | | |
|------------|---------------------|--------------|----------|--------------|-----------------|--------------------|
| | | | | Aliquot (mL) | Final Vol. (ml) | Final Conc. (mg/L) |
| S3 | EMAX-4 | 10000 | Accu std | 10.0 | 1000 | 100 |
| | Fe | 10000 | Accu std | 1.0 | | 10 |
| | Al | 10000 | Accu std | 1.0 | | 10 |
| S6 | EMAX-6A | 500 | Accu std | 4.0 | 1000 | 2 |
| | EMAX-6B | 500 | Accu std | 4.0 | | 2 |
| | Sn | 10000 | Accu std | 1.0 | | 10 |
| ICV | EMAX-MIX-3 | 10000 | CPI | 5.0 | 1000 | 50.0 |
| | EMAX-MIX-2 | 500 | CPI | 2.0 | | 1.0 |
| | EMAX-MIX-1 | 500 | CPI | 2.0 | | 1.0 |
| | Al | 10000 | CPI | 0.5 | | 5.0 |
| | Fe | 10000 | CPI | 0.5 | | 5.0 |
| | Sn | 1000 | CPI | 5.0 | | 5.0 |
| CCV | EMAX-4 | 10000 | Accu std | 7.5 | 1000 | 75 |
| | Al | 10000 | Accu std | .75 | | 7.5 |
| | Fe | 10000 | Accu std | .75 | | 7.5 |
| | Sn | 10000 | Accu std | .75 | | 7.5 |
| | EMAX-6A & 6B | 500 | Accu std | 3 | | 1.5 |

Table 3
CALIBRATION STANDARD PREPARATION
METHOD 6010B (TRACE-ICP)

| Standard # | Mixed Standard Name | Conc. (mg/L) | Source | Preparation | | |
|------------|---------------------------------------|--------------|---------|--------------|-----------------|-------------|
| | | | | Aliquot (mL) | Final Vol. (ml) | Final Conc. |
| S2 | EMAX-6A | 500 | AccuStd | 2 | 1000 | 1.0 |
| | EMAX-6B | 500 | AccuStd | 2 | 1000 | 1.0 |
| S5 | EMAX-4 | 10000 | AccuStd | 10.0 | 1000 | 100 |
| | Al | 10000 | AccuStd | 1.0 | | 10.0 |
| | Fe | 10000 | AccuStd | 1.0 | | 10.0 |
| ICV | (As, Pb, Se, Tl, Cd, Cr, Mn, V, Zn) | 1000 | CPI | 0.5 | 1000 | 0.5 |
| | Al, Fe | 10000 | CPI | 0.5 | | 5.0 |
| | | 10000 | CPI | 5.0 | | 50 |
| CCV | (As, Pb, Se, Tl, Cd, Cr, Mn, V, Zn) | 1000 | AccuStd | 0.4 | 1000 | 0.4 |
| | Al, Fe | 10000 | AccuStd | 0.4 | | 4.0 |
| | Ca, Mg | 10000 | AccuStd | 4.0 | | 40 |
| | | | | | | |

TABLE 4

CALIBRATION STANDARDS CONCENTRATION AND REPORTING LIMIT FOR ICP-61E

| ELEMENT | S3 (mg/L) | S6 (mg/L) | ICV /CCV (mg/L) | CCV (mg/L) | ICSA (mg/L) | ICSAB (mg/L) | RL (Water) (mg/L) | RL (Soil) (mg/Kg) |
|----------------|----------------------|----------------------|--------------------------------|-----------------------|------------------------|-------------------------|----------------------------------|------------------------------|
| Aluminum | 10 | | 5 | 7.5 | 500 | 500 | 0.20 | 20 |
| Antimony | | 2 | 1 | 1.5 | 0 | 1 | 0.10 | 10 |
| Arsenic | | 2 | 1 | 1.5 | 0 | 1 | 0.10 | 10 |
| Barium | | 2 | 1 | 1.5 | 0 | 0.5 | 0.01 | 1 |
| Beryllium | | 2 | 1 | 1.5 | 0 | 0.5 | 0.01 | 1 |
| Boron | | 2 | 1 | 1.5 | 0 | 0.5 | 0.10 | 10 |
| Cadmium | | 2 | 1 | 1.5 | 0 | 1 | 0.01 | 1 |
| Calcium | 100 | | 50 | 75 | 500 | 500 | 1.00 | 100 |
| Chromium | | 2 | 1 | 1.5 | 0 | 0.5 | 0.02 | 2 |
| Cobalt | | 2 | 1 | 1.5 | 0 | 0.5 | 0.02 | 2 |
| Copper | | 2 | 1 | 1.5 | 0 | 0.5 | 0.01 | 2 |
| Iron | 10 | | 5 | 7.5 | 200 | 200 | 1.00 | 20 |
| Lead | | 2 | 1 | 1.5 | 0 | 1 | 0.10 | 10 |
| Magnesium | 100 | | 50 | 75 | 500 | 500 | 1.00 | 100 |
| Manganese | | 2 | 1 | 1.5 | 0 | 0.5 | 0.1 | 1 |
| Molybdenum | | 2 | 1 | 1.5 | 0 | 1 | 0.1 | 5 |
| Nickel | | 2 | 1 | 1.5 | 0 | 1 | 0.02 | 2 |
| Potassium | 100 | | 50 | 75 | 0 | 50 | 5.00 | 500 |
| Selenium | | 2 | 1 | 1.5 | 0 | 3.6 | 1.0 | 10 |
| Silver | | 2 | 1 | 1.5 | 0 | 1.0 | 0.02 | 2 |
| Sodium | 100 | | 50 | 75 | 0 | 10 | 1.00 | 100 |
| Strontium | | 2 | 1 | 1.5 | 0 | 0.5 | 0.02 | 1 |
| Thallium | | 2 | 1 | 1.5 | 0 | 3.6 | 0.10 | 5 |
| Tin | | 10 | 5 | 7.5 | 0 | 1 | 0.10 | 5 |
| Vanadium | | 2 | 1 | 1.5 | 0 | 0.5 | 0.01 | 2 |
| Zinc | | 2 | 1 | 1.5 | 0 | 1 | 0.02 | 1 |

**TABLE 5
CALIBRATION STANDARDS CONCENTRATION AND REPORTING LIMIT FOR TRACE-ICP**

| ELEMENT | S2 (µg/L) | S5 (µg/L) | ICV (µg/L) | CCV (µg/L) | ICSA (µg/L) | ICSAB (µg/L) | RL (Water) (µg/L) | RL (Soil) (mg/Kg) |
|-----------|--------------|--------------|---------------|---------------|----------------|-----------------|-------------------------|----------------------|
| Aluminum | 0 | 10000 | 5000 | 4000 | 500000 | 500000 | 200.00 | 20.00 |
| Arsenic | 1000 | 0 | 500 | 400 | 0 | 1000 | 10.00 | 1.00 |
| Cadmium | 1000 | 0 | 500 | 400 | 0 | 1000 | 5.00 | 0.50 |
| Calcium | 0 | 100000 | 50000 | 40000 | 500000 | 500000 | 1000.00 | 100.00 |
| Copper | 1000 | 0 | 500 | 400 | 0 | 1000 | 5.00 | 0.50 |
| Iron | 0 | 10000 | 5000 | 4000 | 200000 | 200000 | 200.00 | 20 |
| Lead | 1000 | 0 | 500 | 400 | 0 | 1000 | 10.00 | 1.00 |
| Magnesium | 0 | 100000 | 50000 | 40000 | 500000 | 500000 | 1000.00 | 100.00 |
| Manganese | 1000 | 0 | 500 | 400 | 0 | 500 | 5.00 | 1.00 |
| Selenium | 1000 | 0 | 500 | 400 | 0 | 1000 | 10.00 | 1.00 |
| Thallium | 1000 | 0 | 500 | 400 | 0 | 1000 | 10.00 | 1.00 |
| Vanadium | 1000 | 0 | 500 | 400 | 0 | 500 | 10.00 | 1.00 |
| Zinc | 1000 | 0 | 500 | 400 | 0 | 1000 | 20.00 | 1.00 |

**TABLE 6
CALIBRATION STANDARDS CONCENTRATION AND REPORTING LIMIT FOR ICP-Low**

| ELEMENT | S2 (µg/L) | S5 (µg/L) | S9 (µg/L) | ICV /CCV (µg/L) | CCV (µg/L) | ICSA (µg/L) | ICSAB (µg/L) | RL (Water) (µg/L) | RL (Soil) (µg/Kg) |
|------------|--------------|--------------|--------------|-----------------------|---------------|----------------|-----------------|-------------------------|-------------------------|
| Aluminum | | | 10000 | 5000 | 7500 | 500000 | 500000 | 200 | 20000 |
| Antimony | 1000 | | | 500 | 750 | 0 | 1000 | 100 | 10000 |
| Arsenic | 1000 | | | 500 | 750 | 0 | 1000 | 10 | 1 |
| Barium | | 1000 | | 500 | 750 | 0 | 500 | 10 | 1000 |
| Beryllium | | 1000 | | 500 | 750 | 0 | 500 | 10 | 1000 |
| Boron | 1000 | | | 500 | 750 | 0 | 500 | 100 | 10000 |
| Cadmium | | 1000 | | 500 | 750 | 0 | 1000 | 5 | 0.5 |
| Calcium | | | 100000 | 50000 | 75000 | 500000 | 500000 | 1000 | 100000 |
| Chromium | | 1000 | | 500 | 750 | 0 | 500 | 20 | 2000 |
| Cobalt | | 1000 | | 500 | 750 | 0 | 500 | 20 | 2000 |
| Copper | | 1000 | | 500 | 750 | 0 | 500 | 10 | 2000 |
| Iron | | | 10000 | 5000 | 7500 | 200000 | 200000 | 1000 | 20000 |
| Lead | | 1000 | | 500 | 750 | 0 | 1000 | 10 | 1 |
| Lithium | | | 1000 | 500 | 750 | 0 | 500 | 100 | 10000 |
| Magnesium | | | 100000 | 50000 | 75000 | 500000 | 500000 | 1000 | 100000 |
| Manganese | | 1000 | | 500 | 750 | 0 | 500 | 5 | 0.5 |
| Molybdenum | 1000 | | | 500 | 750 | 0 | 1000 | 100 | 5000 |
| Nickel | | 1000 | | 500 | 750 | 0 | 1000 | 20 | 2000 |
| Potassium | | | 100000 | 50000 | 75000 | 0 | 75000 | 5000 | 500000 |
| Selenium | 1000 | | | 500 | 750 | 0 | 1000 | 10 | 1 |
| Silver | | 1000 | | 500 | 750 | 0 | 1000 | 10 | 1 |
| Sodium | | | 100000 | 50000 | 75000 | 0 | 75000 | 1000 | 10000 |

| ELEMENT | S2 (µg/L) | S5 (µg/L) | S9 (µg/L) | ICV /CCV (µg/L) | CCV (µg/L) | ICSA (µg/L) | ICSAB (µg/L) | RL (Water) (µg/L) | RL (Soil) (µg/Kg) |
|-----------|--------------|--------------|--------------|-----------------------|---------------|----------------|-----------------|-------------------------|-------------------------|
| Strontium | 1000 | | | 500 | 750 | 0 | 500 | 20 | 1000 |
| Thallium | 1000 | | | 500 | 750 | 0 | 1000 | 10 | 1 |
| Tin | 1000 | | | 500 | 750 | 0 | 1000 | 100 | 5000 |
| Uranium | | | 10000 | 5000 | 7500 | 0 | 1000 | 1000 | 50000 |
| Vanadium | 1000 | | | 500 | 750 | 0 | 500 | 10 | 2000 |
| Zinc | 1000 | | | 500 | 750 | 0 | 1000 | 20 | 1000 |

TABLE 7
ICP ANALYTICAL SEQUENCE FOR 6010B METHOD

| RUN ID LABEL | | SAMPLE DESCRIPTION | SOLUTION ID LABEL |
|-----------------|---------------------|---|-------------------|
| ICP | TRACE | | |
| S0 | S0 | Calibration Standard 1 (blank) | S0 |
| S3, S6 | S2;S5 or S2, S5, S9 | ICP-61E Calibration Standard 3/ TRACE-61E Calibration Standard 2,5 | S3, S2, S5,S6, S9 |
| ICV | ICV | Initial Calibration Verification | ICV |
| ICB | ICB | Initial Calibration Blank | ICB |
| ICSA | ICSA | Initial Interference Solution A | ICSA |
| ICSAB | ICSAB | Initial Interference Solution A and B | ICSAB |
| CCV1 | CCV1 | Continuing Calibration Verification #1 | CCV |
| CCB1 | CCB1 | Continuing Calibration Blank #1 | S0 |
| ICMSSSX B | ICMSSSX B | Preparation Blank | |
| ICMSSXL/C | ICMSSXL/C | Lab Control Sample | |
| Sample 1 | Sample 1 | Sample 1 | |
| Sample 1M | Sample 1M | Sample 1 MS Duplicate | |
| Sample 1S | Sample 1S | Sample 1 Matrix Spike | |
| Sample 1T | Sample 1T | Sample 1 Serial Dilution(5 x dilution sample 1) only if sample is unusual | |
| Sample 1A | Sample 1A | Sample 1 Post Digestion spike only if sample is unusual | |
| Sample 2 to 4 | Sample 2 to 4 | Sample 2 to Sample 5 | |
| CCV2 | CCV2 | Continuing Calibration Verification #2 | CCV |
| CCB2 | CCB2 | Continuing Calibration Blank #2 | S0 |
| Sample 5 to 14 | Sample 5 to 14 | Maximum of 10 Samples | |
| CCV3 | CCV3 | Continuing Calibration Verification #3 | CCV |
| CCB3 | CCB3 | Continuing Calibration Blank #3 | S0 |
| Sample 15 to 20 | Sample 15 to 20 | Sample 15 to 20 or a maximum of 10 samples (sample 15 to 24) | |
| CCV4 | CCV4 | Continuing Calibration Verification #4 | CCV |
| CCB4 | CCB4 | Continuing Calibration Blank #4 | S0 |

TABLE 8
MDL CONCENTRATION LEVELS

| ELEMENT | WATER (µg/L) | SOIL (µg/kg) |
|------------|--------------|--------------|
| Aluminum | 100 | 10 |
| Antimony | 150 | 15 |
| Arsenic | 150 | 15 |
| Barium | 5 | 0.5 |
| Beryllium | 5 | 0.5 |
| Boron | 20 | 2.0 |
| Cadmium | 20 | 2.0 |
| Calcium | 500 | 50 |
| Chromium | 20 | 20 |
| Cobalt | 50 | 5.0 |
| Copper | 10 | 1.0 |
| Iron | 50 | 5.0 |
| Lead | 100 | 10 |
| Lithium | 20 | 2.0 |
| Magnesium | 500 | 5.0 |
| Manganese | 50 | 5.0 |
| Molybdenum | 50 | 5.0 |
| Nickel | 50 | 5.0 |
| Potassium | 2000 | 200 |
| Selenium | 300 | 30 |
| Silver | 40 | 4.0 |
| Sodium | 500 | 50 |
| Strontium | 5 | 0.5 |
| Thallium | 300 | 30 |
| Tin | 300 | 30 |
| Titanium | 10 | 1.0 |
| Uranium | 500 | 50 |
| Vanadium | 20 | 2.0 |
| Zinc | 20 | 2.0 |

TYPICAL SAMPLE REPORT

METHOD 3010A/6010B
 METALS BY ICP

```

=====
Client       : XYZ, INC.                Date Collected: 03/08/06
Project      : CLEAN LAND PROJECT      Date Received: 03/09/06
SDG NO.     : 06C091                  Date Extracted: 03/20/06 11:30
Sample ID   : V8JSW1AS                 Date Analyzed: 03/23/06 16:42
Lab Samp ID : C091-01A                 Dilution Factor: 1
Lab File ID : I07C054017              Matrix          : WATER
Ext Btch ID : IPC040W                  % Moisture      : NA
Calib. Ref.: I07C054010              Instrument ID   : EMAXTI07
=====
  
```

| PARAMETERS | RESULTS (ug/L) | RL (ug/L) | MDL (ug/L) |
|------------|-------------------|--------------|---------------|
| Aluminum | 10400 | 200 | 60 |
| Antimony | 4590 | 100 | 40 |
| Barium | 1020 | 5 | 2 |
| Beryllium | 1030 | 5 | 1.0 |
| Cadmium | 991 | 5 | 2 |
| Calcium | 68100 | 500 | 100 |
| Chromium | 997 | 10 | 5 |
| Cobalt | 981 | 15 | 5 |
| Copper | 984 | 10 | 5 |
| Iron | 11300 | 100 | 40 |
| Magnesium | 79500 | 200 | 100 |
| Manganese | 1200 | 5 | 3 |
| Molybdenum | 978 | 15 | 10 |
| Nickel | 966 | 20 | 10 |
| Potassium | 53800 | 1000 | 830 |
| Silver | 962 | 10 | 5 |
| Sodium | 276000 | 500 | 250 |
| Vanadium | 998 | 10 | 5 |
| Zinc | 1050 | 20 | 5 |

TYPICAL LCS/LCSD SUMMARY

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
SDG NO.: 06C091
METHOD: METHOD 3010A/6010B

=====

MATRIX: WATER % MOISTURE: NA
DILTN FACTR: 1 1 1
SAMPLE ID: MBLK1W
CONTROL NO.: IPC040WB IPC040WL IPC040WC
LAB FILE ID: I07C054012 I07C054013 I07C054014
DATIME EXTRCTD: 03/20/0611:30 03/20/0611:30 03/20/0611:30 DATE COLLECTED: NA
DATIME ANALYZD: 03/23/0616:21 03/23/0616:25 03/23/0616:30 DATE RECEIVED: 03/20/06
PREP. BATCH: IPC040W IPC040W IPC040W
CALIB. REF: I07C054010 I07C054010 I07C054010

ACCESSION:

| PARAMETER | BLNK RSLT ug/L | SPIKE AMT ug/L | BS RSLT ug/L | BS % REC | SPIKE AMT ug/L | BSD RSLT ug/L | BSD % REC | RPD % | QC LIMIT % | MAX RPD % |
|------------|-------------------|-------------------|-----------------|-------------|-------------------|------------------|--------------|----------|---------------|--------------|
| Aluminum | ND | 10000 | 10700 | 107 | 10000 | 10600 | 106 | 2 | 80-120 | 20 |
| Antimony | ND | 5000 | 4950 | 99 | 5000 | 4900 | 98 | 1 | 80-120 | 20 |
| Barium | ND | 1000 | 1000 | 100 | 1000 | 977 | 98 | 2 | 80-120 | 20 |
| Beryllium | ND | 1000 | 1050 | 105 | 1000 | 1030 | 103 | 2 | 80-120 | 20 |
| Cadmium | ND | 1000 | 1030 | 103 | 1000 | 1020 | 102 | 1 | 80-120 | 20 |
| Calcium | ND | 50000 | 54600 | 109 | 50000 | 54000 | 108 | 1 | 80-120 | 20 |
| Chromium | ND | 1000 | 1040 | 104 | 1000 | 1020 | 102 | 1 | 80-120 | 20 |
| Cobalt | ND | 1000 | 1020 | 102 | 1000 | 1000 | 100 | 1 | 80-120 | 20 |
| Copper | ND | 1000 | 1020 | 102 | 1000 | 998 | 100 | 2 | 80-120 | 20 |
| Iron | ND | 10000 | 10900 | 109 | 10000 | 10700 | 107 | 1 | 80-120 | 20 |
| Magnesium | ND | 50000 | 54700 | 109 | 50000 | 53200 | 106 | 3 | 80-120 | 20 |
| Manganese | ND | 1000 | 1030 | 103 | 1000 | 1020 | 102 | 2 | 80-120 | 20 |
| Molybdenum | ND | 1000 | 1050 | 105 | 1000 | 1020 | 102 | 2 | 80-120 | 20 |
| Nickel | ND | 1000 | 1010 | 101 | 1000 | 1010 | 101 | 1 | 80-120 | 20 |
| Potassium | ND | 50000 | 52500 | 105 | 50000 | 53000 | 106 | 1 | 80-120 | 20 |
| Silver | ND | 1000 | 993 | 99 | 1000 | 637 | 64* | 44* | 80-120 | 20 |
| Sodium | ND | 50000 | 53200 | 106 | 50000 | 52100 | 104 | 2 | 80-120 | 20 |
| Vanadium | ND | 1000 | 1030 | 103 | 1000 | 1010 | 101 | 2 | 80-120 | 20 |
| Zinc | ND | 1000 | 1070 | 107 | 1000 | 1050 | 105 | 2 | 80-120 | 20 |

TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
SDG NO.: 06C091
METHOD: METHOD 3010A/6010B

MATRIX: WATER % MOISTURE: NA
DILT/N FACTR: 1 1 1
SAMPLE ID: V8JSW1
CONTROL NO.: C091-01 C091-01M C091-01S
LAB FILE ID: I07C054018 I07C054015 I07C054016
DATIME EXTRCTD: 03/20/0611:30 03/20/0611:30 03/20/0611:30 DATE COLLECTED: 03/08/06
DATIME ANALYZD: 03/23/0616:47 03/23/0616:34 03/23/0616:38 DATE RECEIVED: 03/09/06
PREP. BATCH: IPC040W IPC040W IPC040W
CALIB. REF: I07C054010 I07C054010 I07C054010

ACCESSION:

| PARAMETER | SMPL RSLT ug/L | SPIKE AMT ug/L | MS RSLT ug/L | MS % REC | SPIKE AMT ug/L | MSD RSLT ug/L | MSD % REC | RPD % | QC LIMIT % | MAX RPD % |
|------------|-------------------|-------------------|-----------------|-------------|-------------------|------------------|--------------|----------|---------------|--------------|
| Aluminum | ND | 10000 | 10500 | 105 | 10000 | 10500 | 104 | 0 | 80-120 | 20 |
| Antimony | ND | 5000 | 4890 | 98 | 5000 | 4920 | 98 | 0 | 80-120 | 20 |
| Barium | 60.2 | 1000 | 1020 | 96 | 1000 | 1020 | 96 | 1 | 80-120 | 20 |
| Beryllium | ND | 1000 | 1030 | 103 | 1000 | 1030 | 103 | 0 | 80-120 | 20 |
| Cadmium | ND | 1000 | 1010 | 101 | 1000 | 1010 | 101 | 0 | 80-120 | 20 |
| Calcium | 18100 | 50000 | 70200 | 104 | 50000 | 70800 | 105 | 1 | 80-120 | 20 |
| Chromium | ND | 1000 | 1010 | 101 | 1000 | 1010 | 101 | 0 | 80-120 | 20 |
| Cobalt | ND | 1000 | 995 | 99 | 1000 | 1000 | 100 | 1 | 80-120 | 20 |
| Copper | ND | 1000 | 994 | 99 | 1000 | 989 | 99 | 0 | 80-120 | 20 |
| Iron | 866 | 10000 | 11400 | 106 | 10000 | 11500 | 106 | 1 | 80-120 | 20 |
| Magnesium | 30600 | 50000 | 81300 | 101 | 50000 | 82100 | 103 | 1 | 80-120 | 20 |
| Manganese | 211 | 1000 | 1210 | 99 | 1000 | 1210 | 100 | 0 | 80-120 | 20 |
| Molybdenum | ND | 1000 | 1010 | 101 | 1000 | 1000 | 100 | 1 | 80-120 | 20 |
| Nickel | ND | 1000 | 991 | 99 | 1000 | 984 | 98 | 1 | 80-120 | 20 |
| Potassium | 3900 | 50000 | 57300 | 107 | 50000 | 57200 | 107 | 0 | 80-120 | 20 |
| Silver | ND | 1000 | 965 | 97 | 1000 | 953 | 95 | 1 | 80-120 | 20 |
| Sodium | 234000 | 50000 | 275000 | 81 | 50000 | 274000 | 78* | 1 | 80-120 | 20 |
| Vanadium | ND | 1000 | 1010 | 101 | 1000 | 1010 | 101 | 0 | 80-120 | 20 |
| Zinc | 29.4 | 1000 | 1070 | 104 | 1000 | 1060 | 104 | 0 | 80-120 | 20 |

EMAX QUALITY CONTROL DATA

SERIAL DILUTION ANALYSIS

CLIENT: XYZ, INC.
 PROJECT: CLEAN LAND PROJECT
 BATCH NO.: 06C091
 METHOD: METHOD 3010A/6010B

=====

MATRIX: WATER % MOISTURE: NA
 DILUTION FACTOR: 1 5
 SAMPLE ID: V8JSW1 V8JSW1DL
 EMAX SAMP ID: C091-01 C091-01J
 LAB FILE ID: I07C054018 I07C054019
 DATE EXTRACTED: 03/20/0611:30 03/20/0611:30 DATE COLLECTED: 03/08/06
 DATE ANALYZED: 03/23/0616:47 03/23/0616:51 DATE RECEIVED: 03/09/06
 PREP. BATCH: IPC040W IPC040W
 CALIB. REF: I07C054010 I07C054010

ACCESSION:

| PARAMETER | SMPL RSLT (ug/L) | SERIAL DIL RSLT (ug/L) | DIF RSLT % | QC LIMIT (%) |
|------------|---------------------|---------------------------|---------------|-------------------|
| Aluminum | ND | ND | NA | 10 |
| Antimony | ND | ND | 0 | 10 |
| Barium | 60.2 | 58.4 | 3 | 10 |
| Beryllium | ND | ND | 0 | 10 |
| Cadmium | ND | ND | 0 | 10 |
| Calcium | 18100 | 17900 | 1 | 10 |
| Chromium | ND | ND | 0 | 10 |
| Cobalt | ND | ND | 0 | 10 |
| Copper | ND | ND | 0 | 10 |
| Iron | 866 | 857 | 1 | 10 |
| Magnesium | 30600 | 30100 | 2 | 10 |
| Manganese | 211 | 210 | 0 | 10 |
| Molybdenum | ND | ND | 0 | 10 |
| Nickel | ND | ND | 0 | 10 |
| Potassium | 3900 | ND | NA | 10 |
| Silver | ND | ND | 0 | 10 |
| Sodium | 234000 | 224000 | 4 | 10 |
| Vanadium | ND | ND | 0 | 10 |
| Zinc | 29.4 | .041J | NA | 10 |

TYPICAL ANALYTICAL SPIKE REPORT

EMAX QUALITY CONTROL DATA
 ANALYTICAL SPIKE ANALYSIS

CLIENT: XYZ, INC.
 PROJECT: CLEAN LAND PROJECT
 SDG NO.: 06C091
 METHOD: METHOD 3010A/6010B

=====

MATRIX: WATER % MOISTURE: NA
 DILTN FACTR: 1 1
 SAMPLE ID: V8JSW1
 CONTROL NO.: C091-01 C091-01A
 LAB FILE ID: I07C054018 I07C054017
 DATIME EXTRCTD: 03/20/0611:30 03/20/0611:30 DATE COLLECTED: 03/08/06
 DATIME ANALYZD: 03/23/0616:47 03/23/0616:42 DATE RECEIVED: 03/09/06
 PREP. BATCH: IPC040W IPC040W
 CALIB. REF: I07C054010 I07C054010

ACCESSION:

| PARAMETER | SMPL RSLT (ug/L) | SPIKE AMT (ug/L) | AS RSLT (ug/L) | AS % REC | QC LIMIT (%) |
|------------|---------------------|---------------------|-------------------|-------------|-------------------|
| Aluminum | ND | 10000 | 10400 | 103 | 75-125 |
| Antimony | ND | 5000 | 4590 | 92 | 75-125 |
| Barium | 60.2 | 1000 | 1020 | 96 | 75-125 |
| Beryllium | ND | 1000 | 1030 | 103 | 75-125 |
| Cadmium | ND | 1000 | 991 | 99 | 75-125 |
| Calcium | 18100 | 50000 | 68100 | 100 | 75-125 |
| Chromium | ND | 1000 | 997 | 100 | 75-125 |
| Cobalt | ND | 1000 | 981 | 98 | 75-125 |
| Copper | ND | 1000 | 984 | 98 | 75-125 |
| Iron | 866 | 10000 | 11300 | 104 | 75-125 |
| Magnesium | 30600 | 50000 | 79500 | 98 | 75-125 |
| Manganese | 211 | 1000 | 1200 | 99 | 75-125 |
| Molybdenum | ND | 1000 | 978 | 98 | 75-125 |
| Nickel | ND | 1000 | 966 | 97 | 75-125 |
| Potassium | 3900 | 50000 | 53800 | 100 | 75-125 |
| Silver | ND | 1000 | 962 | 96 | 75-125 |
| Sodium | 234000 | 50000 | 276000 | 82 | 75-125 |
| Vanadium | ND | 1000 | 998 | 100 | 75-125 |
| Zinc | 29.4 | 1000 | 1050 | 102 | 75-125 |

TYPICAL CASE NARRATIVE

CLIENT: XYZ, INC.

PROJECT: CLEAN LAND PROJECT

SDG: 06C091

**METHOD 3010A/6010B
METALS BY ICP**

Seven (7) water samples were received on 03/09/06 for Metals analysis by Method 3010A/6010B in accordance with “Test Methods for Evaluating Solid Waste, Physical/Chemical Methods”, SW846, 3rd edition and DOD QSM (2002).

1. Holding Time

All samples were analyzed within holding time.

2. Calibration

Initial calibration was analyzed at a frequency specified by the project and verified by initial and continuing calibration verifications (ICV/ICB and CCV/CCB). All target analytes met calibration requirements.

3. Method Blank

Method blank was digested and analyzed with the samples at a frequency specified by the project and that results are compliant to project requirement.

4. Lab Control Sample/Lab Control Sample Duplicate

Lab control samples were analyzed at a frequency specified by the project and recoveries met the project QC limits.

5. Matrix Spike/Matrix Spike Duplicate

MS/MSD are digested with the samples and analyzed at a frequency specified by the project. All analytes were within the project QC limits except Sodium which was spiked at a concentration level much lower than the sodium concentration found in the sample. Non-compliant result was indicated by an asterisk “*” (refer to MS/MSD Report Summary).

6. Dilution Test and Post Digestion Spike

Dilution test and post digestion spike were analyzed for every preparation batch. Positive results of dilution test met method requirements.

Post Digestion Spike Summary reported Sodium below QC limits confirming the result of MS/MSD. No action is further taken because Dilution Test for Sodium met the required limit.

7. Sample Analysis

Samples were analyzed in conformance to the method and project requirements. Positive results were qualitatively identified in accordance to the method and applied quantitation is based on the initial calibration.

SUMMARY OF IN-HOUSE QUALITY CONTROL PROCEDURES

| QC PROCEDURES | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1 st Rvw | 2 nd Rvw |
|--|--|--|--|--|---------------------|---------------------|
| Initial Calibration (min. 1 standard and a blank) | Daily initial calibration prior to sample analysis | Acceptable ICV | Correct the problem and repeat initial calibration. | | | |
| Initial Calibration Verifications (ICV) Second Source | Daily after initial calibration. | All analytes within $\pm 10\%$ of expected value RSD of Replicate integrations: $< 5\%$ | Correct the problem and repeat initial calibration. | | | |
| Calibration Verifications (CCV) | Daily before sample analysis, every 10 samples and at the end of the analysis sequence | All analytes within $\pm 10\%$ of expected value RSD of replicate integrations $< 5\%$ | Repeat calibration and reanalyze all samples since last successful calibration. | | | |
| Calibration Blanks (ICB/CCB) | After every calibration verification | No analytes detected $\geq 3X$ IDL | Correct problem then reanalyze calibration blank and previous samples. | | | |
| Interference Check Sample (ICSA/ICSAB) | Analyzed at the beginning of each analytical run | Within $\pm 20\%$ of expected value | Terminate analysis, correct the problem, reanalyze ICS, and reanalyze all affected samples | | | |
| Method Blank | One per preparation batch | No analytes detected $\geq RL$ | Reprep and reanalyze method blank and all samples processed with the contaminated blank. | Apply B to specific analyte(s) on all associated samples | | |
| Laboratory Control Sample (LCS) | One per preparation batch | % Recovery : 80 - 120% | Reprep and reanalyze LCS and all associated samples | | | |
| Matrix Spikes (MS/MSD) | One MS/MSD every 20 project samples per matrix | % Recovery: 75 – 125% RPD $\pm 20\%$ | Discuss in case narrative | | | |
| Dilution Test (5X) | Each new sample matrix | 1:5 dilution must agree within $\pm 10\%$ of the original determination | Perform post digestion spike addition | | | |
| Post Digestion Spike Addition | When dilution test fails | Recovery within 75-125% of expected value | Refer to project requirement. | | | |
| Results reported between MDL and RL | N/A | N/A | N/A | Refer to PSR | | |
| Comments: | | | | Reviewed By: | | |
| | | | | Date: | | |

**DEMONSTRATION OF CAPABILITY
METHOD 3010-6010B**

Conc. Unit: mg/L
Sample Amount(ml): 50
Extract Volume(ml): 50

Date Digested: 01/05/06
Data Analyzed: 01/05/06
Analyzed by: Karen Hirakawa

| PARAMETER | Found Concentration (ng/L) | | | | True Value (mg/L) | Ave. Conc. (mg/L) | Ave. Rec. (%) | SD | RSD (%) | QC Criteria %Rec. | Comments |
|------------|----------------------------|------------|------------|------------|-------------------|-------------------|---------------|------|---------|-------------------|----------|
| | IPA007WC | IPA007WC1 | IPA007WL | IPA007WL1 | | | | | | | |
| | I73A004_12 | I73A004_13 | I73A004_14 | I73A004_15 | | | | | | | |
| Aluminum | 10.3 | 9.96 | 10.3 | 9.98 | 10.0 | 10.1 | 101 | 0.19 | 1.9 | 80-120 | PASSED |
| Antimony | 5.23 | 5.24 | 5.17 | 5.13 | 5.0 | 5.19 | 104 | 0.05 | 1.0 | 80-120 | PASSED |
| Arsenic | 1.03 | 1 | 1.03 | 1 | 1.0 | 1.02 | 102 | 0.02 | 1.7 | 80-120 | PASSED |
| Barium | 0.97 | 0.94 | 0.97 | 0.94 | 1.0 | 0.96 | 96 | 0.02 | 1.8 | 80-120 | PASSED |
| Beryllium | 1.02 | 1 | 1.02 | 1 | 1.0 | 1.01 | 101 | 0.01 | 1.1 | 80-120 | PASSED |
| Boron | 1.09 | 1.09 | 1.08 | 1.07 | 1.0 | 1.08 | 108 | 0.01 | 0.9 | 80-120 | PASSED |
| Cadmium | 0.98 | 0.95 | 0.97 | 0.95 | 1.0 | 0.96 | 96 | 0.02 | 1.6 | 80-120 | PASSED |
| Calcium | 49.4 | 48 | 50.7 | 47.7 | 50.0 | 49 | 98 | 1.38 | 2.8 | 80-120 | PASSED |
| Chromium | 1 | 0.97 | 1 | 0.97 | 1.0 | 0.99 | 99 | 0.02 | 1.7 | 80-120 | PASSED |
| Cobalt | 0.97 | 0.94 | 0.97 | 0.95 | 1.0 | 0.96 | 96 | 0.01 | 1.6 | 80-120 | PASSED |
| Copper | 1.02 | 1 | 1.03 | 0.99 | 1.0 | 1.01 | 101 | 0.02 | 1.8 | 80-120 | PASSED |
| Iron | 10.9 | 10.6 | 10.9 | 10.6 | 10.0 | 10.8 | 108 | 0.17 | 1.6 | 80-120 | PASSED |
| Lead | 0.99 | 0.96 | 0.99 | 0.97 | 1.0 | 0.98 | 98 | 0.01 | 1.5 | 80-120 | PASSED |
| Magnesium | 49.6 | 48.1 | 50.8 | 48 | 50.0 | 49.1 | 98 | 1.34 | 2.7 | 80-120 | PASSED |
| Manganese | 0.99 | 0.96 | 0.99 | 0.96 | 1.0 | 0.98 | 98 | 0.02 | 1.8 | 80-120 | PASSED |
| Molybdenum | 1 | 1 | 0.99 | 0.98 | 1.0 | 0.99 | 99 | 0.01 | 1.0 | 80-120 | PASSED |
| Nickel | 0.98 | 0.95 | 0.97 | 0.95 | 1.0 | 0.96 | 96 | 0.02 | 1.6 | 80-120 | PASSED |
| Potassium | 50.5 | 46.4 | 53.3 | 48 | 50.0 | 49.6 | 99 | 3.02 | 6.1 | 80-120 | PASSED |
| Selenium | 1.01 | 0.96 | 1 | 0.97 | 1.0 | 0.99 | 99 | 0.02 | 2.4 | 80-120 | PASSED |
| Silver | 1.01 | 0.98 | 1.01 | 0.98 | 1.0 | 1 | 100 | 0.02 | 1.7 | 80-120 | PASSED |
| Sodium | 49.2 | 47.7 | 50.7 | 47.3 | 50.0 | 48.7 | 97 | 1.55 | 3.2 | 80-120 | PASSED |
| Strontium | 1.04 | 1.04 | 1.04 | 1.02 | 1.0 | 1.04 | 104 | 0.01 | 1.0 | 80-120 | PASSED |
| Thallium | 1.03 | 0.98 | 1.02 | 0.98 | 1.0 | 1 | 100 | 0.03 | 2.6 | 80-120 | PASSED |
| Tin | 1.01 | 1.01 | 1.01 | 1 | 1.0 | 1.01 | 101 | 0.00 | 0.5 | 80-120 | PASSED |
| Titanium | 1.04 | 1.05 | 1.04 | 1.02 | 1.0 | 1.04 | 104 | 0.01 | 1.2 | 80-120 | PASSED |
| Vanadium | 1.01 | 0.98 | 1.01 | 0.98 | 1.0 | 1 | 100 | 0.02 | 1.7 | 80-120 | PASSED |
| Zinc | 1.01 | 0.98 | 1.01 | 0.98 | 1.0 | 1 | 100 | 0.02 | 1.7 | 80-120 | PASSED |

**DEMONSTRATION OF CAPABILITY
METHOD 3050B-6010B**

Conc. Unit: mg/L
Sample Amt(ml): 50
Extract Vol.(ml): 50

Date Digested: 01/05/06
Data Analyzed: 01/06/06
Analyzed by: Karen Hirakawa

| PARAMETER | Found Concentration (mg/kg) | | | | True Value (mg/kg) | Ave. Conc. (mg/L) | Ave. Rec. (%) | SD | RSD (%) | QC Criteria % Rec. | Comments |
|------------|-----------------------------|------------|------------|------------|--------------------|-------------------|---------------|--------|---------|--------------------|----------|
| | IPA008 SC | IPA008 SC1 | IPA008 SL | IPA008 SL1 | | | | | | | |
| | I07A008_11 | I07A008_12 | I07A008_13 | I07A008_14 | | | | | | | |
| Aluminum | 990 | 951 | 965 | 952 | 1000 | 965 | 96 | 18.16 | 1.9 | 80 - 120 | PASSED |
| Antimony | 454 | 436 | 445 | 443 | 500 | 445 | 89 | 7.42 | 1.7 | 80 - 120 | PASSED |
| Arsenic | 98.4 | 93.8 | 95 | 94 | 100 | 95.3 | 95 | 2.13 | 2.2 | 80 - 120 | PASSED |
| Barium | 94.4 | 93.4 | 92.9 | 106 | 100 | 96.7 | 97 | 6.25 | 6.5 | 80 - 120 | PASSED |
| Beryllium | 101 | 96.4 | 98.2 | 96.7 | 100 | 98.1 | 98 | 2.10 | 2.1 | 80 - 120 | PASSED |
| Boron | 98.9 | 94.6 | 96.1 | 94.8 | 100 | 96.1 | 96 | 1.98 | 2.1 | 80 - 120 | PASSED |
| Cadmium | 95.3 | 91.8 | 92.8 | 92.3 | 100 | 93.1 | 93 | 1.55 | 1.7 | 80 - 120 | PASSED |
| Calcium | 4810 | 4580 | 4760 | 4630 | 5000 | 4700 | 94 | 107.86 | 2.3 | 80 - 120 | PASSED |
| Chromium | 96.7 | 92.3 | 94.3 | 92.9 | 100 | 94.1 | 94 | 1.96 | 2.1 | 80 - 120 | PASSED |
| Cobalt | 95 | 90.6 | 92.4 | 91 | 100 | 92.3 | 92 | 1.99 | 2.2 | 80 - 120 | PASSED |
| Copper | 95.9 | 92.3 | 93.7 | 92.5 | 100 | 93.6 | 94 | 1.65 | 1.8 | 80 - 120 | PASSED |
| Iron | 998 | 960 | 974 | 964 | 1000 | 974 | 97 | 17.05 | 1.8 | 80 - 120 | PASSED |
| Lead | 95 | 91.6 | 92.9 | 91.6 | 100 | 92.8 | 93 | 1.60 | 1.7 | 80 - 120 | PASSED |
| Magnesium | 4730 | 4540 | 4680 | 4560 | 5000 | 4630 | 93 | 92.15 | 2.0 | 80 - 120 | PASSED |
| Manganese | 95.9 | 91.8 | 93.6 | 92.2 | 100 | 93.4 | 93 | 1.85 | 2.0 | 80 - 120 | PASSED |
| Molybdenum | 94.4 | 91.2 | 93.4 | 91.1 | 100 | 92.5 | 93 | 1.64 | 1.8 | 80 - 120 | PASSED |
| Nickel | 94.4 | 92 | 92.7 | 91.9 | 100 | 92.8 | 93 | 1.16 | 1.2 | 80 - 120 | PASSED |
| Potassium | 4760 | 4510 | 4640 | 4580 | 5000 | 4620 | 92 | 105.95 | 2.3 | 80 - 120 | PASSED |
| Selenium | 93.1 | 93.6 | 88.4 | 88.2 | 100 | 90.8 | 91 | 2.92 | 3.2 | 80 - 120 | PASSED |
| Silver | 96 | 89.4 | 93.7 | 92.9 | 100 | 93 | 93 | 2.74 | 2.9 | 80 - 120 | PASSED |
| Sodium | 4830 | 4620 | 4760 | 4630 | 5000 | 4710 | 94 | 102.31 | 2.2 | 80 - 120 | PASSED |
| Strontium | 99 | 95.4 | 96.8 | 95.5 | 100 | 96.7 | 97 | 1.68 | 1.7 | 80 - 120 | PASSED |
| Thallium | 96 | 90.7 | 96.5 | 96.8 | 100 | 95 | 95 | 2.89 | 3.0 | 80 - 120 | PASSED |
| Tin | 96.5 | 94.6 | 95.4 | 93.5 | 100 | 95 | 95 | 1.27 | 1.3 | 80 - 120 | PASSED |
| Titanium | 98.9 | 95.1 | 96.9 | 95.3 | 100 | 96.6 | 97 | 1.76 | 1.8 | 80 - 120 | PASSED |
| Vanadium | 96.3 | 92.4 | 94.3 | 92.6 | 100 | 93.9 | 94 | 1.81 | 1.9 | 80 - 120 | PASSED |
| Zinc | 96.8 | 93 | 94.9 | 94 | 100 | 94.7 | 95 | 1.62 | 1.7 | 80 - 120 | PASSED |

ANALYSIS RUN LOG

ANALYSIS RUN LOG FOR ICP

SOP → EMAX-6010-Rev. 3 → EMAX-CLP-TAL → Method File: Autosampler Table:

| Matrix: | | Start Date: | | Time: | | End Date: | | Time: | | Book# A31 -034 | | | |
|----------------|-------------|---------------|----|--------|-------|----------------|-------------|---------------|----|----------------|-------|----------------|--|
| Data File Name | Prep. Batch | Lab Sample ID | DF | Matrix | Notes | Data File Name | Prep. Batch | Lab Sample ID | DF | Matrix | Notes | Instrument No. | |
| | | | | | | | | | | | | 31 | |
| | | | | | | | | | | | | Std. | ID |
| | 01 | | | | | | 26 | | | | | S ₀ | |
| | 02 | | | | | | 27 | | | | | S ₁ | |
| | 03 | | | | | | 28 | | | | | S ₂ | |
| | 04 | | | | | | 29 | | | | | S ₃ | |
| | 05 | | | | | | 30 | | | | | S ₄ | |
| | 06 | | | | | | 31 | | | | | S ₅ | |
| | 07 | | | | | | 32 | | | | | S ₆ | |
| | 08 | | | | | | 33 | | | | | ICV | |
| | 09 | | | | | | 34 | | | | | ICVH1 | |
| | 10 | | | | | | 35 | | | | | ICVH2 | |
| | 11 | | | | | | 36 | | | | | CCV | |
| | 12 | | | | | | 37 | | | | | ICSA | |
| | 13 | | | | | | 38 | | | | | ICSAB | |
| | 14 | | | | | | 39 | | | | | MRL | |
| | 15 | | | | | | 40 | | | | | | |
| | 16 | | | | | | 41 | | | | | | Comments: |
| | 17 | | | | | | 42 | | | | | | |
| | 18 | | | | | | 43 | | | | | | |
| | 19 | | | | | | 44 | | | | | | |
| | 20 | | | | | | 45 | | | | | | |
| | 21 | | | | | | 46 | | | | | | |
| | 22 | | | | | | 47 | | | | | | |
| | 23 | | | | | | 48 | | | | | | Analyzed By: _____ |
| | 24 | | | | | | 49 | | | | | | Date Disposed: _____ |
| | 25 | | | | | | 50 | | | | | | This page is checked during data review. |


EMAX LABORATORIES, INC. 1835 W. 20th St. Torrance, CA 90501

ANALYTICAL BATCH*

ANALYTICAL BATCH *

SAMPLE PREPARATION LOG

DIGESTION LOG FOR ICP METALS

SOP o EMAX-3005 Rev. No. 3 o EMAX-3010 Rev. No. 2 o EMAX-3050 Rev. No. 2 o EMAX-CLP-TAL o Book # EIP-046

| Matrix: | Start Date: | Time: | Temp: | °C | Ending Date: | Time: | Temp.: | °C | | | | | | |
|----------------|---------------|--------------------|-------------------|-----------|------------------|------------------------|--------|---------------------|-----------------------|---------|---|--------------------|-------------------|-----------------|
| Sample Prep ID | Lab Sample ID | Matrix Description | | | Turbidity <1 NTU | Sample Amount (g ml) | pH | Extract Volume (ml) | Digestate Description | | Standards | ID | Amount Added (ml) | |
| | | Color | Texture / Clarity | Artifacts | | | | | Color | Clarity | | | | |
| 01 | | | | | | | | | | | LCS -1 | | | |
| 02 | | | | | | | | | | | LCS -2 | | | |
| 03 | | | | | | | | | | | LCS -3 | | | |
| 04 | | | | | | | | | | | MS Reagent | Lot# / ID | Amount Added (ml) | |
| 05 | | | | | | | | | | | HNO ₃ | | | |
| 06 | | | | | | | | | | | HCl | | | |
| 07 | | | | | | | | | | | H ₂ O ₂ | | | |
| 08 | | | | | | | | | | | HNO ₃ (1:1) | | | |
| 09 | | | | | | | | | | | Digestate Location | | | |
| 10 | | | | | | | | | | | Extract Location | | | |
| 11 | | | | | | | | | | | Legend: | | | |
| 12 | | | | | | | | | | | Texture | Cs = Coarse | Md = Medium | Fn = Fine |
| 13 | | | | | | | | | | | Clarity | Cr = Clear | Cy = Cloudy | Td = Turbid |
| 14 | | | | | | | | | | | Artifacts | Rk = rocks | Sl = Shale | Vg = Vegetation |
| 15 | | | | | | | | | | | Color | Bu = blue | Bk = Black | Bn = Brown |
| 16 | | | | | | | | | | | | Gn = Green | Og = Orange | Rd = Red |
| 17 | | | | | | | | | | | | Yw = Yellow | Cl = Colorless | |
| 18 | | | | | | | | | | | Comments: Samples for Methods 200.7 or 200.8 Analyses | | | |
| 19 | | | | | | | | | | | If turbidity ≤ 1 NTU no digestion is required unless otherwise required by th | | | |
| 20 | | | | | | | | | | | | | | |
| 21 | | | | | | | | | | | | | | |
| 22 | | | | | | | | | | | Prepared By: | Standard Added By: | | |
| 23 | | | | | | | | | | | Witnessed By: | Extracts Rcvd. By: | | |
| 24 | | | | | | | | | | | Checked By: | | | |
| 25 | | | | | | | | | | | Date Disposed: | Disposed by: | | |


EMAX LABORATORIES, INC. 1835 W. 20th St. Torrance, CA 90501

BATCH: _____

STANDARD OPERATING PROCEDURES

TRACE METALS BY ICP-MS

SOP No.: EMAX-6020 Revision No. 4 Effective Date: 09-Jun-09

Prepared By: Mary Jane Mendoza Date: 09-06-09

Approved By: Kenette Pimentel *[Signature]* Date: 06-09-09
QA Manager

Approved By: Caspar Pang *[Signature]* Date: 07-06-09
Laboratory Director

Control Number: 6020-04

1.0 SCOPE AND APPLICATION

- 1.1. This procedure is applicable for the determination of sub- $\mu\text{g/L}$ concentrations of a large number of elements in drinking water, wastewater, groundwater, aqueous, extract, soil, sludge, and sediment samples using the Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) method. All matrices require proper sample preparation prior to analysis.
- 1.2. The elements and their corresponding isotopes are listed in Table 1.
- 1.3. This SOP is an adaptation of the SW846 Method 6020A.

2.0 SUMMARY OF METHOD

- 2.1. Metal analytes in water are acid digested from a pre-measured sample. Nitric acid and hydrochloric acid are added to the sample and heated without boiling until the volume is substantially reduced. The digestate is diluted back to its original sample volume using reagent water.
- 2.2. Metal analytes in soil are acid digested from a pre-measured sample. Nitric acid is added to the sample and heated to initialize digestion. It is further oxidized with 30% hydrogen peroxide and the acid used for final reflux is hydrochloric acid.
- 2.3. Digestates are introduced by pneumatic nebulization resulting aerosol into a high temperature argon plasma, where they are decomposed, atomized and ionized. The ions produced are extracted from the plasma via the sample and skimmer orifices in the interface region of the mass spectrometer. The extracted ions are guided by an off-axis Lens System to reduce background noise, passes through an Octopole Reaction System (ORS) where some ions require a simple reaction with H_2 or He to eliminate matrix interference prior to entering the Quadrupole Mass Filter (QMF). The QMF separates ions based on their mass-to-charge ratios and ions are counted by electron multiplier detector.
- 2.4. Quantitation is accomplished by comparing the response of a major ion relative to an internal standard using a calibration curve.
- 2.5. **Interference**
 - 2.5.1. **Isobaric Elemental Interference.** Are caused by isotopes of different elements which form singly or doubly charge ions of the same nominal mass-to-charge ratio. The signal of an isotope of an interfering element is determined and subtracted from the analyte isotope signal.
 - 2.5.2. **Isobaric Polyatomic Ion Interference.** Are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest. To correct for isobaric polyatomic ion interferences, optimize the cell gas pressure on each analyte so that when the ORS employs simple reaction gases (H_2 and He) side reactions create new and unpredictable interferences. The ORS is equipped with notch filters and by using scanning voltages the created interferences are prevented from reaching the analyzer.

STANDARD OPERATING PROCEDURES

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- 2.5.3. **Physical Interference.** Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. An internal standard can be used to correct physical interference if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. When the intensity level of the internal standard is less than 70% of the intensity of the first standard used during calibration, the sample must be diluted and re-analyzed.
- 2.5.4. **Memory Interference.** Contamination by carry-over can occur whenever high concentrations are analyzed in sequence with a low concentration sample. To reduce potential carry-over the rinse period between samples must be long enough to eliminate significant memory effect.

3.0 REPORTING LIMITS**3.1. Method Detection Limit (MDL)**

- 3.1.1. Prepare MDL standard. Refer to Table 5 for MDL concentration levels.
- 3.1.2. Digest the prepared MDL as described in Section 10.1.
- 3.1.3. Analyzed as described in Section 10.5.
- 3.1.4. Refer to EMAX QA04 for MDL evaluation and verification.

3.2. Reporting Limits (RLs)

- 3.2.1. Target analytes with respective reporting limits are listed in Table 3.
- 3.2.2. RL verification or lower limit quantitation check (LLQC) should undergo an entire preparation and analytical process.
- 3.2.3. Verify reporting limits every three months.
- 3.2.4. Reporting limits are verified when all the analytes are within $\pm 30\%$ of the true value.

3.3. Instrument Detection Limit (IDL)

- 3.3.1. Establish IDL as described in Section 10.4.9.
- 3.3.2. Determine IDL at least every three months.

4.0 DYNAMIC RANGE

- 4.1. Linear Dynamic Range (LDR) is the concentration over which the instrument response remains linear.
- 4.2. Establish LDR as described in Section 10.4. Established LDR is listed in Table 5.
- 4.3. Verify the established LDR every six months or when there is a significant change in the instrument signal, whichever comes first.

5.0 SAMPLE HOLDING TIME AND PRESERVATION**5.1. Water Samples**

- 5.1.1. Collect water samples in polyethylene or glass bottles.
- 5.1.2. For total recoverable metal analysis, preserve sample at the time of collection to pH < 2 with nitric acid.

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5.1.3. For dissolved metal analysis, filter sample through a 0.45- μ m filter at the time of collection and preserve to pH < 2. Preserve samples requested properly in the lab and observe at least 24 hours from the time preservative is added before sample digestion.

5.1.4. Store samples in the same condition as received unless specified in the project requirement.

5.2. Soil Samples

5.2.1. Collect soil samples in glass jars or brass tubes.

5.2.2. Store soil samples at $\leq 6^{\circ}\text{C}$ until analysis.

5.3. Holding Time

5.3.1. Analyze all samples within 180 days from the collection date.

6.0 ASSOCIATED SOPs

- 6.1. EMAX-QA04 - IDL/MDL/RL
- 6.2. EMAX-QA08 - Corrective Action
- 6.3. EMAX-QC01 - Quality Control for Chemicals
- 6.4. EMAX-QC02 - Analytical Standard Preparation
- 6.5. EMAX-QC06 - Calibration of Micropipette
- 6.6. EMAX-QC07 - Glassware Cleaning
- 6.7. EMAX-SM04 - Analytical and QC Labeling
- 6.8. EMAX-3005 - Acid Digestion, Total Recoverable Metals and Dissolved
- 6.9. EMAX-3010 - Acid Digestion, Total Metals for Aqueous
- 6.10. EMAX-3050 - Acid Digestion, Total Metals for Solids

7.0 SAFETY

- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. During operation or maintenance of the instrument take the following precautions:
 - Close the instruments hoods and panels prior to operation.
 - Check the exhaust system for a positive extraction at the exhaust duct.
 - Handle the solvents correctly.
 - Check the drain vessels frequently.
 - Make sure that the argon tank is chained.
 - Wait for the instrument interface region to cool down prior to instrument maintenance.
 - Observe all cautions and warnings stipulated in the Agilent 7500 ICP/MS Manuals.
- 7.3. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, gloves, at all times when performing this procedure. Observe all chemical hygiene procedures as mentioned in the Chemical Hygiene Plan.

STANDARD OPERATING PROCEDURES

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- 7.4. Place all wastes generated during analytical process in the waste containers. Endorse these wastes to waste disposal section for proper disposal.
- 7.5. Place all wastes generated during digestion, i.e., filter papers, paper towels, etc., and/or any solid material contaminated with acid, in the wastes containers under the fume hoods. Label these wastes acidic wastes and give to the waste management unit for proper disposal. DO NOT DISPOSE ACIDIC WASTE IN THE TRASH CAN.
- 7.6. Label all acidic rinsate ($\text{pH} \leq 2$) acidic water and give to the waste management unit for proper disposal. DO NOT DISPOSE ACID WASTE IN THE SINK.
- 7.7. If for any reason, solvent and/or other reagents get in contact with the skin or any other part of the body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND REAGENTS**8.1. Instruments and Supplies**

- 8.1.1. ICP-MS: Agilent 7500CE Octopole Reaction System
- 8.1.2. Autosampler: CETAC ASX-520
- 8.1.3. Computer: IBM Compatible
- 8.1.4. RF Generator: Agilent RF Generators
- 8.1.5. Data Acquisition: Agilent Chemstation (G1834B)
- 8.1.6. Autosampler rack(s): 17-mm, 60 positions
- 8.1.7. Culture tubes: 17-mm, polypropylene
- 8.1.8. Volumetric Flask: 100-mL
- 8.1.9. Micropipets: 100-, 1000- μL
- 8.1.10. Pipet Tips: 100-, 1000- μL
- 8.1.11. Polyethylene bottles: 250-, 500-, 1000-mL
- 8.1.12. Liquid argon
- 8.1.13. Hydrogen, Compressed: Ultra-high purity
- 8.1.14. Helium, compressed: Ultra-high purity
- 8.1.15. Balance Sartorius LC 620 S or equivalent
- 8.1.16. Spatula Stainless steel or equivalent
- 8.1.17. Digestion vessel 50 ml, 100 ml snap seal
- 8.1.18. Pipette 1ml, 0.100 ml, 5 ml
- 8.1.19. Digestion block Aluminum blocks or equivalent
- 8.1.20. Thermometer Range 0 - 110°C
- 8.1.21. Filter Whatman #41 or equivalent
- 8.1.22. Digestate Container 50 ml polyethylene vessel, 100 ml Corning snap seal or equivalent
- 8.1.23. Disposable watch glass

STANDARD OPERATING PROCEDURES

TRACE METALS BY ICP-MSSOP No.: EMAX-6020 Revision No. 4 Effective Date: 09-Jun-09**8.2. Chemicals and Reagents**

- 8.2.1. DI water, ASTM Type II or equivalent
- 8.2.2. Nitric Acid, Trace high purity grade, concentrated
- 8.2.3. Hydrochloric acid, Trace high purity grade, concentrated
- 8.2.4. Hydrogen Peroxide, VW3690-5 VWR or equivalent

9.0 STANDARDS**9.1. Tune Check Standard**

| STANDARD | SOURCE | ELEMENTS | CONC. (mg/L) | MATRIX |
|-----------------------|----------------------|-------------------|-----------------|---------------------|
| Tuning Solution | Agilent | Li, Y, Ce, Tl, Co | 0.01 | 2% HNO ₃ |
| Tuning Check Standard | High Purity Standard | Co, In, Li, Tl | 10 | 2% HNO ₃ |

- 9.1.1. Prepare tuning check standard at concentration level suggested below.

| STANDARD | Aliquot, (mL) | Final volume (mL) | Final Concentration (mg/L) |
|------------------------------------|---------------|----------------------|-------------------------------|
| Intermediate tuning check standard | 0.5 | 50 | 0.100 |

9.2. Internal Standard (IS)

- 9.2.1. Purchase stock internal standard as certified standard at concentration listed below or equivalent.

| STANDARD | SOURCE | ELEMENTS | CONC. (mg/L) | MATRIX |
|----------|---------|--------------------------------|-----------------|----------------------|
| IS | Agilent | Li, Sc, Ge, Rh, In, Tb, Bi, Lu | 100 | 10% HNO ₃ |

9.3. Calibration Standards**9.3.1. Calibration Stock Standard**

- 9.3.1.1. Purchase custom-made certified mixed stock standards as listed in the table below or equivalent.

| STANDARD | SOURCE | ELEMENTS | CONC. (µg/mL) | MATRIX |
|-------------|-------------|---|------------------|---------------------------------------|
| SM-2208-001 | High Purity | As, B, Se, Sr, Tl, Ti, V, Zn, Sb, Mo, Sn | 10 | 2% HNO ₃ and Trace HF Acid |
| SM-2208-002 | High Purity | Ba, Be, Cd, Cr, Co, Cu, Pb, Li, Mn, Ni, Ag, U | 10 | 2% HNO ₃ |
| SM-2208-003 | High Purity | Al, Fe, K, Ca, Mg, Na | 1000 | 4% HNO ₃ |

- 9.3.2. **Matrix Acid Blank (S0)**

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9.3.2.1 Prepare matrix acid solution by mixing 3% by volume nitric acid and 2% by volume hydrochloric acid in reagent water. Transfer into a clean HDPE bottle and identify the solution as S0.

9.3.2.2 Use this solution for standards or digestate dilutions.

9.3.3. Initial Calibration Standard

9.3.3.1 The initial calibration consists of a blank (S0) and three standards (S3, S4 and S5). Prepare the standards as suggested below. Refer to Table 3 for final concentrations for each analyte. Please note: More standard points may be added at the discretion of the analyst.

| | SM-2208-001 (mL) | SM-2208-002 (mL) | SM-2208-003 (mL) | Final volume (mL) | Final Concentration (mg/L) |
|----|---------------------|---------------------|---------------------|----------------------|-------------------------------|
| S3 | 0.050 | .050 | 0.050 | 50 | 0.01/1 |
| S4 | 0.250 | 0.250 | 0.250 | | 0.05/5 |
| S5 | 0.500 | 0.500 | 0.500 | | 0.1/10 |

9.3.4. Continuing Calibration Verification (CCV) Standard

9.3.4.1 Prepare CCV using the stock standards and S0 as suggested below. Refer to Table 3 for final concentrations for each analyte.

| From Calibration Stock Standard | Aliquot (mL) | Final volume (mL) | CCV Final Concentration (mg/L) |
|---------------------------------|-----------------|----------------------|--------------------------------|
| SM-2208-001 | 0.250 | 50 | 0.05 |
| SM-2208-002 | 0.250 | | 0.05 |
| SM-2208-003 | 0.250 | | 5.0 |

9.3.4.2 Prepare intermediate solution for Low Level CCV (LLCCV) using the stock standards and S0 as suggested below. Refer to Table 3 for final concentrations of each analyte.

| | From Calibration Stock Standard | Aliquot (mL) | Final volume (mL) | LLCCV Final Concentration (mg/L) |
|-------|---------------------------------|-----------------|----------------------|----------------------------------|
| MIX 7 | SM-2208-001 | 1.0 | 10 | 1.0 |
| | SM-2208-002 | 1.0 | | 1.0 |
| | SM-2208-003 | 1.0 | | 100 |
| Zn | Zn | 0.5 | | 10 |

9.4. Secondary Source Standard

9.4.1. Purchase secondary stock standard from a different source as certified standards or equivalent. Refer to list below.

| SOURCE | STANDARD | ELEMENTS | CONC. (mg/L) | MATRIX |
|--------|------------|---|-----------------|-----------------------------|
| CPI | EMAX MIX 2 | As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Sr, Tl, Ti, V, Zn, Ag, Sb, Mo, Li, U, Sn | 10 | 5% HNO ₃ + Tr HF |
| | EMAX MIX 3 | Al, Fe, Ca, Mg, K, Na | 1000 | 5% HNO ₃ |

9.4.2. Refer to EMAX-QC02 for detailed procedure of standard preparation and labeling.

STANDARD OPERATING PROCEDURES

TRACE METALS BY ICP-MSSOP No.: EMAX-6020 Revision No. 4 Effective Date: 09-Jun-09**9.5. Initial Calibration Verification (ICV)**

9.5.1. Prepare ICV using the secondary stock standards and S0 as suggested below. Refer to Table 3 for final concentrations for each analyte.

| From Stock Standard | Aliquot (mL) | Final Volume (mL) | ICV Final Concentration (mg/L) |
|---------------------|--------------|-------------------|--------------------------------|
| EMAX MIX 2 | 0.3 | 50 | 0.06 |
| EMAX MIX 3 | 0.3 | | 6 |

9.6. Interference Standards (ICSA/ICSAB)

9.6.1. Purchase ICS stock standard as mix certified standards at concentration levels listed below.

| STANDARD | SOURCE | ELEMENTS | CONC. (mg/L) | MATRIX |
|---------------------------------|--------------------|---|--------------|-----------------------------|
| 6020ICS-0A (ICSA) | Inorganic Ventures | Al, Ca, Fe, Mg, Na, P, K, S | 1000 | 1.4% HNO ₃ |
| | | C | 2000 | |
| | | Cl | 10000 | |
| | | Mo, Ti | 20 | |
| 6020ICS-0A + EMAX MIX 2 (ICSAB) | 6020ICS-0A | Al, Ca, Fe, Mg, Na, P, K, S | 1000 | 1.4% HNO ₃ |
| | | C | 2000 | |
| | | Cl | 10000 | |
| | | Mo, Ti | 20 | |
| | EMAX MIX 2 | As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Sr, Tl, Ti, V, Zn, Ag, Sb, Mo, Li, U, Sn | 10 | 5% HNO ₃ + Tr HF |

9.6.2. Prepare Intermediate ICSA and ICSAB standards at concentration levels suggested below. Refer to Table 3 final concentrations.

| Standard | Parent Standard | Aliquot (mL) | Final Volume (mL) | Final Concentration (mg/L) |
|--------------------|-----------------|--------------|-------------------|----------------------------|
| Intermediate ICSA | 6020ICS-0A | 5 | 50 | Varied |
| Intermediate ICSAB | 6020ICS-0A | 5 | 50 | Varied |
| | EMAX MIX 2 | 0.10 | | |

9.7. LCS/MS Spike Standard

9.7.1. Purchase LCS/MS standards as certified custom-mixed.

| STANDARD | SOURCE | ELEMENTS | CONC. (mg/L) | MATRIX |
|------------|--------|---|--------------|-----------------------------|
| EMAX MIX 2 | CPI | B, Sr, As, Ba, Be, Ag, Cd, Cr, Co, Cu, Tl, Pb, Mn, Ni, Se, V, Zn, Ti, Sb, Mo, Li, U, Sn | 10 | 5% HNO ₃ + Tr HF |
| EMAX MIX 3 | | Al, Fe, Ca, Mg, K, Na | 1000 | 5% HNO ₃ |

9.8. P/A Tuning Standard

STANDARD OPERATING PROCEDURES

TRACE METALS BY ICP-MSSOP No.: EMAX-6020 Revision No. 4 Effective Date: 09-Jun-09

- 9.8.1. Using the calibration stock standard from 9.3.1, prepare a 50 µg/L and 100 µg/L mixed standard. These standards should also include 50 µg/L and 100 µg/L of internal standard, which can be prepared by using the internal standard stock from 9.2.1.

10.0 PROCEDURES**10.1. Sample Preparation for Water Samples**

- 10.1.1. Based from the work order, determine the samples to form a preparative batch (not to exceed 20 samples per preparative batch). Withdraw the sample(s) from the sample control room and bring them to the preparation area. Allow the samples to equilibrate at room temperature.
- 10.1.2. Shake the sample container. Pour a small amount of sample into the sample cap and trickle just enough to wet the pH indicator strip. Compare the color of the wet strip to the indicator chart displayed in the pH indicator box. Record the pH in the digestion log. If the pH value is <2, proceed to 10.1.3. If the pH value is ≥2, check if special instruction is written on the analysis folder or in the COC. Otherwise, fill out an NCR and inform the supervisor immediately. **DO NOT PROCEED WITH THE DIGESTION. WAIT FOR FURTHER INSTRUCTION.**
- 10.1.3. Line up the samples chronologically under the hood. Check and record the lot number of the digestion vessels if it has been verified for accuracy. Take digestion vessels and label each one corresponding to the samples withdrawn and place them in front of each sample making sure that their labels agree. Take four more vessels and label them as preparation blank, LCS, matrix spike and matrix spike duplicate¹.
- 10.1.4. Mix the sample thoroughly to achieve homogeneity. Fill each digestion vessel up to the 50-ml mark. (The reduction of the volume is due to waste minimization).
- 10.1.5. Record the volume in the digestion log. Use reagent water for blank and LCS.
- 10.1.6. Take another digestion vessel; fill it with tap water to 50-ml mark. Put a thermometer inside and let it sit on the digestion block. Turn the thermostat to a pre-determined mark to deliver heat at 90°C - 95°C. Record the temperature reading in the digestion log.
- 10.1.7. **Standard Addition**
- 10.1.7.1. Call for a witness for standard addition. Have the witness verify the setting of the micropipette and the expiration dates of the spike standards.
- 10.1.7.2. Add 0.25 ml of each from EMAX MIX 2 and 3 (see Section 9.4.1) solutions to matrix spike samples and LCS.
- 10.1.8. **Acid Digestion for Dissolved Metals**
- 10.1.8.1. Add 0.5 ml of concentrated HNO₃ and 0.25 ml concentrated HCl to each of the digestion vessels.
- 10.1.8.2. Cap the digestion vessels with disposable watch glass.
- 10.1.8.3. Check that the temperature of the digestion block is 85°C, adjust if necessary. If temperature happens to be less than 85°C, adjust the thermostat and wait until temperature falls at 85°C.
- 10.1.8.4. Place the digestion vessels on the digestion block and reduce volume of sample to about 10 ml. Do not boil. This step takes about 1 hour for 50 ml aliquot.

¹ *Note that SW3005A requires MS and sample duplicate. However, the analytical methods SW6010B and SW7000A require MS/MSD. To satisfy for both preparation and analytical methods, MSD is prepared because it can be regarded as duplicate sample as well.*

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- 10.1.8.5. Reflux gently for another 15 minutes. Remove the digestion vessels from the digestion plate and allow the vessels to cool down.
- 10.1.8.6. Using a reagent water wash bottle, rinse the disposable watch glass collecting the rinsate on the same digestion vessel that it covered.
- 10.1.8.7. Dilute the digestate with reagent water to the 50-ml mark of the digestion vessel. Seal the vessel and shake. If the digestate appears to be turbid, pass it through Whatman #41 filter and collect it in a new polyethylene container.
- 10.1.9. **Acid Digestion for Total Recoverable Metals**
- 10.1.9.1. Add 0.5 ml of concentrated HNO₃ and 0.25 ml concentrated HCl to each of the digestion vessels.
- 10.1.9.2. Cap the digestion vessels with disposable watch glass.
- 10.1.9.3. Check that the temperature of the digestion block is 85°C, adjust if necessary. If temperature happens to be less than 85°C, adjust the thermostat and wait until temperature falls at 85°C.
- 10.1.9.4. Place the digestion vessels on the digestion block and reduce volume of sample to about 10 ml. Do not boil. This step takes about 1 hour for 50 ml aliquot.
- 10.1.9.5. Reflux gently for another 15 minutes. Remove the digestion vessels from the digestion block and allow the vessels to cool down.
- 10.1.9.6. Using a reagent water wash bottle, rinse the disposable watch glass collecting the rinsate on the same digestion vessel that it covered.
- 10.1.9.7. Dilute the digestate with reagent water to the 50-ml mark of the digestion vessel. Seal the vessel and shake. If the digestate appears to be turbid, pass it through Whatman #41 filter and collect it in a new polyethylene container.
- 10.2. **Sample Preparation for Soil Samples**
- 10.2.1. **Sample Handling**
- 10.2.1.1. Based from the work order, determine the samples to form a preparative batch (not to exceed 20 field samples). Withdraw the sample(s) from the sample control room and bring them to the weighing area. Allow the samples to equilibrate at room temperature.
- 10.2.1.2. Take digestion vessels and label each one corresponding to the samples withdrawn. Take four more vessels and label them as preparation blank, LCS, matrix spike and matrix spike duplicate.
- 10.2.1.3. Mix the sample thoroughly to achieve homogeneity.
- 10.2.1.4. Take the digestion vessel to the nearest 0.001g. Using a stainless spatula, scoop 1g of the homogenized sample into the digestion vessel. Record the weight to the nearest 0.001-g. Cover the vessel with conical watch glass and place it in a digestion block.
- 10.2.2. **Pre-heating the Hot Plate**
- 10.2.2.1. Put a thermometer inside a digestion vessel, fill it with tap water halfway, and insert it in a digestion block. Turn the thermostat to a pre-determined mark to deliver heat at approximately 95°C ($\pm 5^\circ\text{C}$).
- 10.2.3. **Standard Addition.**
- 10.2.3.1. Call for a witness for standard addition. Have the witness verify the setting of the

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micropipette and the expiration dates of the spike standards.

10.2.3.2. Add 5 ml of each EMAX MIX 2 and 3 (see Section 9.4.1) solutions to matrix spike samples and LCS.

10.2.4. Acid Digestion

10.2.4.1. Add 10 ml of 1:1 (v/v) nitric acid (HNO_3) into the vessels, swirl the vessels to mix the acid and the sample. Add same amount of acid into a clean and empty vessel and designate it as blank. Insert the vessels in the digestion block(s). Cap the digestion vessels with disposable watch glass.

10.2.4.2. Check that the temperature of the digestion block is $95^\circ\text{C} \pm 5^\circ\text{C}$, adjust if necessary. If temperature happens to be $\geq 100^\circ\text{C}$, adjust the thermostat and wait until temperature falls within $95^\circ\text{C} \pm 5^\circ\text{C}$. Record the temperature reading in the digestion log.

10.2.4.3. Reflux for 10 to 15 minutes without boiling.

10.2.4.4. Allow the vessels to cool down for at least 5 minutes. Lift the cap and add 5 ml of concentrated HNO_3 . Place the disposable watch glass back before working on the next vessel.

10.2.4.5. Return the vessels to the digestion blocks and reflux for another 30 minutes.

10.2.4.6. Repeat steps 10.2.4.4 and 10.2.4.5 until no brown fumes are visible in the digestion vessels, indicating complete reaction with HNO_3 . Then continue to reflux at $95^\circ\text{C} \pm 5^\circ\text{C}$ without boiling for two (2) hours or until the volume is reduced to approximately 5-ml.

10.2.4.7. Allow the vessels to cool down for at least 5 minutes.

10.2.4.8. Add 2 ml of reagent water. Then add 3 ml of 30% hydrogen peroxide (H_2O_2) to each vessel, swirling each one of them after every addition to initiate peroxide reaction.

10.2.4.9. Return the vessels to the heated digestion blocks. Care must be taken to ensure that losses do not occur due to excessive effervescence.

10.2.4.10. Continue to add 30 % H_2O_2 in 1 ml aliquot at a time (not to exceed 10 ml) with warming, until effervescence is minimal or until the general appearance of the digestate is unchanged.

10.2.4.11. Continue to reflux the mixture at $95^\circ\text{C} \pm 5^\circ\text{C}$ for 2 hours or until volume is reduced to approximately 5 ml, whichever comes first. Remove the digestion vessels from the digestion block.

10.2.4.12. Lift the disposable watch glass, add 10 ml of concentrated HCl. Swirl the vessel until added reagents are properly mixed with the solution. Place the disposable watch glass back before working on the next vessel. Return the vessels into the heated digestion block. Reflux for additional 15 minutes. Subsequently, remove the digestion vessels from the hot plate, allow the vessels to cool down and proceed to 10.1.5.

10.2.5. Digestate Filtration

10.2.5.1. Place Whatman #41 filter papers into each funnel resting on holders. Rinse the filter papers with reagent water.

10.2.5.2. Place a pre-labeled digestate container under each funnel making sure that the labels are visible.

10.2.5.3. Check the labels to make sure that they agree. Pour the digestate into the filter.

10.2.5.4. Filter and collect the digestate in the labeled container. Dilute the digestate with reagent

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water to the 100-ml mark of the digestion vessel. Cover the vessel and invert it several times.

10.3. Instrument Parameters

10.3.1. Set instrument parameters as suggested below.

10.3.2. Plasma Condition

- RF Power: 1550 W
- RF Matching: 1.68 V
- Sample Depth: 8.0 mm
- Torch Height: -0.4 mm
- Torch Vertical: 0 mm
- Carrier Gas: 0.9 L/min
- Make-up Gas: 0.2 L/min Note: Total Carrier and Make-up gas not to exceed 1.1 L/min.
- Peristaltic pump: 0.1 rps
- Spray Chamber (S/C) Temp: 2°C

10.3.3. Ion Lenses

- Extract 1: 0 V
- Extract 2 : -130V
- Omega Bias-ce: -20 V
- Omega Lens-ce: -2 V
- Cell Entrance: -30 V
- QP Focus: 3 V
- Cell Exit: -30 V

10.3.4. Octopole Parameters

- Octopole RF: 190 V
- Octopole Bias: -9 V

10.3.5. Q-Pole Parameters

- AMU Gain: 127
- AMU Offset: 125
- Axis Gain: 0.9996
- Axis Offset: 0.04
- QP Bias: -3 V

10.3.6. Detector Parameters

- Discriminator: 8 mV
- Analog HV: 1630 V

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- Pulse HV: 990 V

10.3.7. Reaction Cell

- H₂ Gas: 3.0 mL/min
- He Gas: 4 mL/min

10.3.8. Adjust the instrument parameters to optimize the instrument performance in conformance to the tuning requirement.

10.3.9. Print the most current instrument parameters and place in the appropriate binder for easy reference. Replaced instrument parameter set-up should be archived chronologically for future reference and historical record.

10.4. **Calibration**10.4.1. Instrument Set-Up

10.4.1.1. Set up the ICP-MS with proper operating parameters. Refer to Section 10.3.

10.4.1.2. Ignite the plasma and allow the instrument to become thermally stable for at least 30 minutes.

10.4.1.3. Check the peristaltic pump to deliver a steady flow.

10.4.2. Tuning the Instrument

10.4.2.1. Tune the instrument according to Normal Mode, Hydrogen Mode and Helium Mode without the internal standard. Refer to Section 10.3 for parameters. On the ICP-MS main Menu, go to Instrument and click Tune and run the tuning solution without the internal standard. After about 60 seconds (making sure the solution is in the system) click start and evaluate the counts of the isotopes according to the table below.

| Mode | Range | | |
|-------------|-------------------|--------------------|---------------------|
| Normal | Li6 ≥ 6400 counts | Y89 ≥ 16000 counts | Tl205 ≥ 9606 counts |
| Hydrogen | Ar/Ar78 < 10 | Co59 ≥ 3000 counts | |
| Helium | V51/Co59 < 0.6 | Co59 ≥ 7000 counts | ArCl-75 < 10 counts |

10.4.2.2. If non-compliant, adjust parameters (e.g. Torch height, Torch vertical, Octopole Bias and QP Bias) and repeat the tune process until the required range is met.

10.4.2.3. Click Generate report for a full scan of the tune. Save all tune values to the current method.

10.4.3. Perform a P/A Factor Evaluation

10.4.3.1. Analyze the 50 µ/L P/A Tuning Standard ,

10.4.3.2. Under “Tune,” access “P/A Factor.”

10.4.3.3. Select “Load masses from acquired method” and then select calcium “Ca” from the list of elements. Select “delete” to remove Ca from the P/A Factor analysis.

10.4.3.4. Select “run.”

10.4.3.5. When complete accept the changes.

10.4.3.6. Print out P/A Factor and store the printout with the tuning data.

10.4.3.7. Accept the new P/A Factors.

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- 10.4.3.8. Under “file” select “copy tune parameters” and copy the P/A Factors to both the H2 and He modes.
- 10.4.3.9. Save file as “norm.u”.
- 10.4.4. **Perform Tune Check**
 - 10.4.4.1. Analyze the intermediate tune check solution. (9.1.1) using 4 independent runs.
 - 10.4.4.2. Evaluate the tune check so that the mass calibration differs no more than 0.1 AMU of the true value and the resolution to be less than 0.9 AMU full width.
- 10.4.5. **Initial Calibration (ICAL)**
 - 10.4.5.1. Analyze a calibration blank (S0) and a multi-point calibration standard (Section 9.3.3.1).
 - 10.4.5.2. Set the instrument rinse time to 90 seconds between each standard solution.
 - 10.4.5.3. Refer to Appendix 1 for ICAL acceptance criteria and /or corrective action.
- 10.4.6. **Initial Calibration Verification (ICV)/ Instrument Calibration Blank (ICB)**
 - 10.4.6.1. Analyze the ICV (Section 9.5.1) from a second source to verify the concentration of the ICAL.
 - 10.4.6.2. Analyze a low-level ICV (LLICV) from the same source as the calibration standard to verify the lower limit of quantitation (RL).
 - 10.4.6.3. Analyze an ICB after LLICV to demonstrate absence of instrument contamination.
 - 10.4.6.4. Refer to Appendix 1 for ICV, LLICV and ICB acceptance criteria and /or corrective action.
- 10.4.7. **Continuing Calibration Verification (CCV)/ Continuing Calibration Blank (CB)**
 - 10.4.7.1. Analyze CCV to check the validity of the ICAL every 10 samples and at the end of the analytical sequence.
 - 10.4.7.2. Analyze low-level CCV (LLCCV) to check the system stability at low end of ICAL at the end of the analytical sequence.
 - 10.4.7.3. Analyze a CCB every after LLCCV to demonstrate the absence of instrument contamination.
 - 10.4.7.4. Refer to Appendix 1 for CCV, LLCCV and CCB acceptance criteria and/or corrective action.
- 10.4.8. **ICSA and ICSAB**
 - 10.4.8.1. Analyze ICSA and ICSAB at the beginning of each analytical run and every 12 hours thereafter.
- 10.4.9. **Establishing Instrument Detection Limit (IDL)**
 - 10.4.9.1. Analyze a minimum of seven consecutive method blanks.
 - 10.4.9.2. Repeat the process within three non-consecutive days.
 - 10.4.9.3. Calculate the standard deviation of each run.
 - 10.4.9.4. The average of the standard deviation of the three runs determines the IDL for each analyte.
- 10.4.10. **Establishing Linear Dynamic Range (LDR)**
 - 10.4.10.1. The upper limit of the linear range is an observed signal no more than 10% below the level extrapolated from lower standards.

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10.4.10.2. Prepare a standard at the determined LDR, analyze and quantitate against the normal calibration curve to verify the LDR. Percent recovery must be within $\pm 10\%$ of the expected value.

10.4.10.3. If non-compliant, re-establish the LDR.

10.5. Analysis**10.5.1. Analytical Sequence**

10.5.1.1. From the main menu, go to Sequence and input the analytical sequence into the instrument using data acquisition software. Refer to Table 4.

10.5.1.2 Set QC limits on QC samples for easy verification while analytical samples are running.

10.5.1.3 Using the analytical sequence, arrange the standards and the digestates to be analyzed chronologically.

10.5.1.4 Transfer about 10 mL of its content into the autosampler tubes placing them on the autosampler rack in the same order as the analytical sequence. A dilution of x10 for soil samples is required due to the high acid content of the digestate.

10.5.1.5 Dilution Test sample is prepared at 5 times dilution. Seal the tube with Parafilm and invert the tube several times to ensure adequate mixing.

10.5.1.6 Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.

10.5.1.7 A 100 $\mu\text{g/L}$ internal standard shall be spiked into each sample.

10.5.1.8 Set the prepared analytical samples into the auto-sampler and start the analytical run.

10.5.2. Sample Result Evaluation

10.5.2.1 Check QC parameters as soon as the data is available.

- Check the initial calibration verification (ICV, LLICV and ICB) against Appendix 1.
- Check MB, LCS against Appendix 1. Perform specified corrective action if necessary.
- Check the MS, duplicate sample, serial dilution and post digestion spike results. If matrix interference is indicated, dilute the sample and re-analyzed or check the PSR if MSA is waived, otherwise refer 10.5.3
- Check intensity of internal standard on each sample.
- If any of the above checkpoints is non-compliant, perform the specified corrective action in /Appendix 1. If results indicate digestion problem, fill-up an NCR and order re-digestion for the affected sample(s). If unresolved, consult the Supervisor for further action.

10.5.2.2 Check the sample rack to ensure that the Autosampler did not skip any sample.

10.5.2.3 Check concentration of target analytes. If the response exceeds LDR, dilute and reanalyze the sample at a concentration within the LDR.

10.5.2.4 Check other QC requirements like ICSA, ICSAB, CCV, LLCCV, CCB against Appendix 1.

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10.5.3.1 Perform MSA for all EP extracts, samples for delisting petition and whenever a new matrix is encountered.

10.5.3.2 Prepare two sample solutions (Ms1 and Ms2) with identical sample aliquot (V_x). Add reagent water to Ms1 and spike standard to Ms2. Choose a concentration and volume of spike standard so that the response of Ms2 is twice of Ms1 avoiding excessive dilution of the sample.

- $Ms1 = V_x + V_w$
- $Ms2 = V_x + V_s$

where:

V_x - volume of sample

V_s - volume of spike

V_w - volume of reagent water

Example: Sample concentration is tentatively determined at 5 µg/L.

Ms1 – take 5 mL of sample then add 5-mL of reagent water. This solution will have an approximate concentration of 2.5 µg/L.

Ms2 – take 5 mL of sample then add 5-mL of 5 µg/L spike standard. This solution will have an approximate concentration of 5 µg/L.

10.5.3.3 Analyze Ms1 and Ms2 and calculate the results using Eq-10.6.6.

10.5.3.4 For TCLP extracts, perform MSA* if:

- ✓ The recovery of the matrix spike is $\leq 50\%$ and the concentration of the contaminant does not exceed the regulatory level.
- ✓ When the measured concentration of the analyte in the extract is within 20% of the appropriate regulatory level.

Note: *Matrix spike standards must be added after filtration (pre-digestion) of the TCLP extract and prior to preservation.

10.6. **Calculations**

10.6.1. The computer software is designed to calculate the concentrations in the digestates, based on the assumption that the initial calibration is linear through the origin. Thus, for aqueous samples, the computer-produced results represent the concentration of the sample.

10.6.2. For water samples, if the initial sample taken was V_s , different from 50 mL, then calculate the concentration using the following equation:

$$C_s = C_i \left(\frac{V_e}{V_s} \right) DF \quad \text{Eq-10.6.2}$$

where:

- C_s - concentration in the sample, µg/L
- C_i - concentration in the digestate, µg/L

² Adapted from Section 8.7.1 USEPA SW846 Method 7000

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V_s - volume of sample taken, mL
 V_e - volume of digestate, mL
 DF - dilution factor

10.6.3. For solids, use the following equation to calculate the concentration.

$$C_s = C_i \left(\frac{V_e}{W_s} \right) \left(\frac{100}{100 - \%H_2O} \right) DF \quad \text{Eq-10.6.3}$$

Where:

C_s - concentration in the sample, $\mu\text{g/L}$
 C_i - concentration in the digestate (computer generated), $\mu\text{g/L}$
 W_s - sample amount taken, g
 V_e - volume of digestate, mL
 $\%H_2O$ - percent moisture of the sample
 DF - dilution factor

10.6.4. Calculate the percent recovery (%R)

$$\% \text{ Recovery} = \frac{C_f - C}{C_s} * 100 \quad \text{Eq-10.6.4}$$

where:

C_f - concentration found, $\mu\text{g/L}$
 C - concentration of sample, $\mu\text{g/L}$
 C_s - concentration of spike, $\mu\text{g/L}$

10.6.5. Relative Percent Difference (%RPD)

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2} \right)} \times 100 \quad \text{Eq-10.6.5}$$

Where:

RPD = Relative Percent Difference
 C_1 = Measured concentration of the first sample aliquot
 C_2 = Measured concentration of the second sample aliquot

10.6.6. Calculate for sample result determined from MSA

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x} \quad \text{Eq-10.6.6}$$

Where:

C_x - concentration of the sample
 C_s - concentration of the spike
 S_1 - analytical signal of Ms1
 S_2 - analytical signal of Ms2

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- V_x – volume of sample aliquot
 V_s – volume of spike/reagent water

10.7. Report Generation

- 10.7.1. Print the summary of the analytical run, perform a data transfer into a disk, and convert the instrument electronic output file into an ASCII file format.
- 10.7.2. Run the ICPCHK.exe program for calibration check.
- 10.7.3. Identify samples that need to be re-analyzed, if any, and report all samples that met the analytical requirements.
- 10.7.4. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated and re-analyses.
- Sample Results
 - LCS Summary
 - MS Summary
 - Duplicate Summary
 - CCV Summary
 - ICV Summary
 - ICAL Summary
- 10.7.5. Generate the case narrative to include discussion of the following as found in the review process:
- Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples were analyzed within holding time. For non-compliance, state the number of days/hours that the sample(s) were off from holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
 - Initial Calibration Verification (ICV) – That all target analytes met calibration requirements For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
 - Method Blank – That MB was analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte affected and the associated sample results were flagged with “B”.
 - Lab Control Sample – That LCS was analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Furthermore, if corrective action is not possible (e.g., no more samples to re-analyze) state that results were qualified.
 - Matrix Spike/Matrix Spike Duplicate – That MS/MSD is extracted with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC

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limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.

- Dilution Test and Post Digestion Spike – That dilution test and post digestion spike are analyzed for every preparation batch. That positive results of dilution test are evaluated to meet the method requirement and that Post Digestion Spike is evaluated for analytes that are not detected. For non-compliant analytes, reference the associated form, (Serial Dilution Test or Analytical Spike) and that non-compliant results were indicated by “*” or refer to MSA result.
- Sample Analysis – That samples were analyzed in conformance to the method and project requirements. That positive results were qualitatively identified in accordance to the method and applied quantitation is based on the initial calibration.
- Other Anomalies (if any) – shall be discussed on a case to case basis concurred by the Supervisor or the Lab Director. Include the NCR in the data package if required by the project, otherwise archive the NCR with the analytical folder.

10.7.6. Submit the analysis package for secondary review.

10.8. Data Review

10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.

- Review the ICPCHK.exe output file to ensure that it agrees with the instrument output. Check Project Specific Requirement (PSR) or Appendix 1 for acceptance criteria.
- Check frequency of calibration verification. Verify results to be within acceptance limits.
- Check of target analytes concentration to be within linear range.
- Verify interference check results to be within acceptance limits.

10.8.2. If any of the above checkpoints is non-compliant, re-analysis is required.

10.9. Preventive Maintenance

10.9.1. Instruments shall receive routine preventive maintenance that is properly recorded in the instrument-specific maintenance logs. The list of maintenance is summarized in Form 6020FM. The practice ensures optimum operating condition of the equipment thus reducing the possibility of frequent instrument malfunction.

11.0 QUALITY CONTROL**11.1. Sample Preparation QC**

- 11.1.1. A preparative batch consists of 20 or fewer samples of the same matrix that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.
- 11.1.2. Every preparative batch shall have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. These QC samples shall be digested together with the field samples.
- 11.1.3. All reagents shall be subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2. Sample Analysis QC

11.2.1. Perform a tune check before every analytical run, an initial calibration and initial calibration verification (ICV / LLICV). Obtain the ICV standard from a different source from that of the initial

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- calibration and LLICV from the same source as the ICAL. Analyze an instrument calibration blank (ICB) after the LLICV. No further analysis shall be valid unless acceptance criteria are met.
- 11.2.2. Monitor the intensities of all internal standards for every analysis. Refer to Appendix 1 for acceptance criteria.
- 11.2.3. Verify inter-element and background correction factors with ICSA and ICSAB standards after ICB every 12 hours.
- 11.2.4. Verify calibration with continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the analytical run. Also verify LLCCV at the end of the analytical run.
- 11.2.5. Evaluate results of MS/MSD to document matrix interference.
- 11.2.6. Perform Post Digestion Spike whenever recoveries for MS/MSD failed.
- 11.2.7. Evaluate Dilution Test result if post digestion spike result failed to meet the acceptance criteria. Failure typically happens when analyte concentrations are high.
- 11.2.8. Refer to Appendix 1 for acceptance criteria.
- 11.3. **Method QC**
- 11.3.1. Establish a Method Detection Limit Study before the analytical procedure can be used.
- 11.3.2. Establish Method proficiency before the analytical procedure can be used.
- 11.3.3. Establish demonstration of proficiency for all analysts conducting this analysis.
- 11.3.4. Perform dynamic range study at least every six months or whenever there is a significant change in instrument response unless otherwise specified by the project.

12.0 CORRECTIVE ACTION

- 12.1. Quality control procedures and corresponding corrective actions are summarized in Appendix 1.
- 12.2. If tune is non-compliant, consider the following suggestions to correct the problem:
- Check the instrument settings and make sure that the instrument parameters are properly set up.
 - Check argon gas flow.
 - Perform auto tune or visual optimization
 - If the problem persists, inform the Supervisor.
- 12.3. If correlation coefficient (R) of ICAL is non-compliant, consider the following suggestions to help you correct the problem:
- Check the calibration points for possible presence of out-lier. If out-lier is present, prepare a fresh standard and repeat the calibration.
 - Check the connections and make sure that they are air-tight. Perform maintenance as needed.
 - Presence of bubbles is indicative of poor connection between the sipper and the nebulizer.
 - Poor precision to inability to light the plasma is a symptom of a poor drain tube connection
 - Poor precision and carry-over problems are indicative of a dirty spray chamber.

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- Relative increase in the sensitivity ratio of the higher: lower atomic number elements are indicative of stretched pump tubing. The sample flow rate decreases as the tubing stretches.
 - Check the argon gas flow. Loss of signal is indicative of low or no argon gas flow.
 - Poor precision and a gradual loss of signal is indicative of “salting-out” in the nebulizer and/or spray chamber due to samples with high dissolved or suspended solids. This problem will necessitate nebulizer and spray chamber cleaning.
 - If the problem persists, inform the Supervisor.
- 12.4. If ICV is non-compliant, consider the following suggestions to help you correct the problem:
- If the RSD is high it is indicative that the carry-over might be present in the spray chamber.
 - If result is bias high, prepare a fresh standards and repeat calibration.
 - If the problem persists, inform the Supervisor.
- 12.5. If ICB/CCB is non-compliant, consider the following suggestions to correct the problem:
- Prepare a fresh calibration blank solution. Perform instrument rinsing and repeat the ICB/CCB prior to re-analysis of associated sample(s).
 - Carry-over problem is indicative of dirty spray chamber, nebulizer and/or torch. Perform instrument maintenance and repeat the calibration.
 - If the problem persists, inform the Supervisor.
- 12.6. If CCV or LLICV or LLCCV is non-compliant, consider the following suggestions to correct the problem:
- Check the connections prior to re-running the ICAL. Refer to Section 12.3.
 - Prepare a new standard and repeat the ICAL.
- 12.7. If the intensity of the Internal Standard is non-compliant, consider the following suggestions to correct the problem:
- Check for drift occurrence by observing the internal standard intensities in the calibration blank.
 - If drift has occurred, terminate the analysis, recalibrate, verify the new calibration and reanalyze the affected samples.
 - If drift has not occurred, dilute affected samples five fold and reanalyze with the addition of appropriate amounts of internal standards.
- 12.8. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
- Rule-out instrument contamination by checking the CCBs. Refer to Section 12.5.
 - Rule-out reagent contamination by testing each reagent as described in EMAX-QC01.
 - Rule-out digestate vessel contamination by adding verified reagents heating the vessels prior to testing.
 - Common environmental contaminants – Ca, Si, Fe, Na, Mg, K, Tl, Cu, Mn, can be minimized by maintaining the lab clean.
 - Other sources of contamination:
 - Sweat contains Ca, Mg, Pb, K, NH_4^+ , SO_4^{2-} , PO_4^{3-} , and Cd (for those who smoke).
 - Cosmetics can contain high concentrations of Al, Be, Ca, Cu, Cr, K, Fe, Mn, Ti, and Zn.
 - Some hair dyes contain $\text{Pb}(\text{OAC})_2$.

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- Dandruff shampoo can contain significant levels of Se.
- Eye make-up may contain Hg as a preservative.
- Calamine lotion is almost pure ZnO.
- Watches and jewelry contain an assortment of elements and should not be worn in the laboratory.
- Re-digest MB and the associated samples with reagents free of contamination or with newly opened reagents.
- If the problem persists, inform the Supervisor.

12.9. If LCS is non-compliant, consider the following suggestions to correct the problem:

- If result is bias-high, check the LCS standard by analyzing at the spike level.
If the LCS check is within 80-120 % of the expected value, check the calibration of the micropipette use for spiking. Re-digest and re-analyze the LCS and the associated samples.
If the LCS check is not within 80-120%, prepare a fresh LCS standard, re-digest and re-analyze LCS and the associated samples.
- Common Problems with Ag, As, Ba, Pb, and Cr, indicating stock standard degradation, are as follows:
Low Silver (Ag) recovery is indicative of Chloride contamination causing AgCl precipitation
Low Arsenic (As) recovery is indicative of loss during sample preparation as volatile oxides (AsO₃) or precipitation as AsCl₃
Low Barium (Ba) recovery is indicative of SO₄or CrO₄ contamination. Barium will form precipitates with HF and H₂SO₄.
High Lead (Pb) recovery is indicative of environmental contamination.

12.10. Execute a Non-Conformance Report (NCR) when the following circumstances occur:

- 12.10.1. Corrective action needs the function of other department; e.g., the sample needs to be re-digested. Refer to EMAX-QA08 for details of completing an NCR.
- 12.10.2. Corrective action needs the assistance of the Project Manager; e.g., sample passed the holding time, insufficient amount of sample, etc.
- 12.10.3. Corrective action prescribed in the IQC Summary does not correct the problem.

12.11. For other problems encountered, inform the supervisor immediately for further instruction.

13.0 POLLUTION PREVENTION

- 13.1. Unused samples, digestates and instrument wastes are very acidic and are very corrosive. Endorse the wastes to the Waste Management Unit for proper treatment or disposal.

14.0 WASTE MANAGEMENT

- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES

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15.1. Definition of Terms

- 15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

Preparation batch is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.

Analytical batch is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.

- 15.1.2. Calibration – is defined as an instrument response per unit measure. It is an experimental value by measuring the response of an instrument per unit target analyte under the method specific condition. A determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.3. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.4. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.5. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.6. Matrix – is a physical state of a sample. Most of environmental samples are classified as water, soil or air.
- 15.1.7. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.8. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.9. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical procedure to monitor contamination.
- 15.1.10. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.11. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.12. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.2. Application of EMAX QC Procedures

- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing metals analysis. In the event that project-specific requirement exists, the quality assurance project plan shall take precedence over this SOP.

15.3. Department of Defense (DoD) Projects

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15.3.1. When samples from DoD sponsored projects are analyzed for trace metals, the calibration, QC, corrective action, and data flagging requirements shall follow the quality assurance project plan (QAPP). In the absence of QAPP, the DoD Quality Systems Manual latest version shall be applied.

15.4. Department of Energy (DoE) Projects

15.4.1. When samples from DoE sponsored projects are analyzed for trace metals, the calibration, QC, corrective action, and data flagging requirements shall follow the quality assurance project plan (QAPP). In the absence of QAPP, the DoE Quality Systems for Analytical Services, latest version shall be applied.

16.0 REFERENCES

- 16.1. "Test Methods for Evaluating Solid Waste, Physical / Chemical Methods", EPA Publication SW-846 Update IV, Method 6020A.
- 16.2. EMAX Quality Systems Manual, as updated.

17.0 APPENDICES**17.1. Tables**

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17.3. Appendices

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TABLE 1. ICP-MS ELEMENTS & ISOTOPES

| ELEMENT | SYMBOL | MASS | Tune Mode | Internal Standard |
|------------|--------|------|-----------|-------------------|
| Aluminum | Al | 27 | 3 | Sc45 |
| Antimony | Sb | 121 | 3 | In115 |
| Arsenic | As | 75 | 2 | Ge72 |
| Barium | Ba | 137 | 3 | In115 |
| Beryllium | Be | 9 | 3 | Li6 |
| Boron | B | 11 | 3 | Li6 |
| Cadmium | Cd | 111 | 3 | In115 |
| Calcium | Ca | 43 | 1 | Sc45 |
| Chromium | Cr | 53 | 2 | Sc45 |
| Cobalt | Co | 59 | 3 | Sc45 |
| Copper | Cu | 63 | 2 | Sc45 |
| Iron | Fe | 57 | 1 | Sc45 |
| Lead | Pb | 208 | 3 | Bi209 |
| Lithium | Li | 7 | 3 | Li6 |
| Magnesium | Mg | 24 | 3 | Sc45 |
| Manganese | Mn | 55 | 3 | Sc45 |
| Molybdenum | Mo | 95 | 3 | In115 |
| Nickel | Ni | 60 | 2 | Sc45 |
| Potassium | K | 39 | 3 | Sc45 |
| Selenium | Se | 82 | 1 | Ge72 |
| Silicon | Si | 28 | 1 | Sc45 |
| Silver | Ag | 107 | 3 | In115 |
| Sodium | Na | 23 | 1 | Sc45 |
| Strontium | Sr | 88 | 3 | Y89 |
| Thallium | Tl | 205 | 3 | Bi209 |
| Tin | Sn | 118 | 3 | In115 |
| Titanium | Ti | 47 | 3 | Sc45 |
| Uranium | U | 238 | 3 | Bi209 |
| Vanadium | V | 50 | 2 | Sc45 |
| Zinc | Zn | 66 | 3 | Ge72 |

Tune Mode: 1=Reaction H₂ Mode; 2=Collision He Mode; 3= Normal Mode

TABLE 2. CALIBRATION STANDARD AND VERIFICATION PREPARATION for METHOD 6020A (ICP-MS)

| Standard # | Mixed Standard Name | Conc. (µg/mL) | Source | Preparation | | |
|-----------------------|---------------------|---------------|-------------|--------------|-----------------|---------------------|
| | | | | Aliquot (mL) | Final Vol. (mL) | Final Conc. (µg/mL) |
| S3 | SM-2208-01 | 10 | High Purity | 0.05 | 50 | 0.01 |
| | SM-2208-02 | 10 | | 0.05 | | 0.01 |
| | SM-2208-03 | 1000 | | 0.05 | | 1.0 |
| S4 | SM-2208-01 | 10 | High Purity | 0.25 | 50 | 0.05 |
| | SM-2208-02 | 10 | | 0.25 | | 0.05 |
| | SM-2208-03 | 1000 | | 0.25 | | 5.0 |
| S5 | SM-2208-01 | 10 | High Purity | 0.50 | 50 | 0.01 |
| | SM-2208-02 | 10 | | 0.50 | | 0.01 |
| | SM-2208-03 | 1000 | | 0.50 | | 10.0 |
| ICV | EMAX MIX 2 | 10 | CPI | 0.3 | 50 | 0.06 |
| | EMAX MIX 3 | 1000 | | 0.3 | | 6 |
| CCV | SM-2208-01 | 10 | High Purity | 0.25 | 50 | 0.05 |
| | SM-2208-02 | 10 | | 0.25 | | 0.05 |
| | SM-2208-03 | 1000 | | 0.25 | | 5.0 |
| LLICV / LLCCV (water) | Mix 7 | 1/100 | High Purity | 0.05 | 50 | 0.001 / 0.1 |
| | Zn | 10 | | 0.045 | | 0.01 |
| LLICV / LLCCV (soil) | Mix 7 | 1/100 | High Purity | 0.025 | 50 | 0.0005/ 0.05 |
| | Zn | 10 | | 0.0025 | | 0.001 |

TABLE 3. CALIBRATION STANDARDS CONCENTRATION AND REPORTING LIMIT FOR ICP-MS

| ELEMENT | ICAL (mg/L) | | | ICV / CCV (mg/L) | ICSA (mg/L) | ICSAB (mg/L) | LLICV,LLCCV, RL (Water) (mg/L) | LLICV,LLCCV, RL (Soil) (mg/Kg) |
|------------|-------------|------|-----|------------------------|----------------|-----------------|--------------------------------------|--------------------------------------|
| | S3 | S4 | S5 | | | | | |
| Aluminum | 1 | 5 | 10 | 6 | 100 | 100 | 0.100 | 100 |
| Antimony | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Arsenic | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Barium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Beryllium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Boron | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.2 | 0.010 | 10 |
| Cadmium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Calcium | 1 | 5 | 10 | 6 | 100 | 100 | 0.100 | 100 |
| Chromium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Cobalt | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Copper | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Iron | 1 | 5 | 10 | 6 | 100 | 100 | 0.100 | 100 |
| Lead | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Lithium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.002 | 0.5 |
| Magnesium | 1 | 5 | 10 | 6 | 100 | 100 | 0.100 | 100 |
| Manganese | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Molybdenum | 0.01 | 0.05 | 0.1 | 0.06 | 2 | 2 | 0.002 | 0.5 |
| Nickel | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Potassium | 1 | 5 | 10 | 6 | 100 | 100 | 0.100 | 100 |
| Selenium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Silver | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Sodium | 1 | 5 | 10 | 6 | 100 | 100 | 0.100 | 100 |
| Strontium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Thallium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Tin | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 10 |
| Titanium | 0.01 | 0.05 | 0.1 | 0.06 | 2 | 2 | 0.002 | 0.5 |
| Uranium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Vanadium | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.001 | 0.5 |
| Zinc | 0.01 | 0.05 | 0.1 | 0.06 | 0 | 0.02 | 0.010 | 1.0 |

TABLE 4. ICP-MS ALYTICAL SEQUENCE

| RUN ID LABEL | SAMPLE DESCRIPTION | SOLUTION ID LABEL |
|----------------------|--|--------------------------|
| S0 | Calibration Standard 1 (blank) | S0 |
| S3, S4, S5 | ICAL Standards | S3, S4, S5 |
| ICV | Initial Calibration Verification | ICV |
| LLICV | Low Level Initial Calibration Verification | LLICV |
| ICB | Initial Calibration Blank | ICB |
| ICSA | Initial Interference Solution A | ICSA |
| ICSAB | Initial Interference Solution A and B | ICSAB |
| CCV1 | Continuing Calibration Verification #1 | CCV |
| CCB1 | Continuing Calibration Blank #1 | S0 |
| IMSSSSB ³ | Preparation Blank | |
| IMSSSSL/C | Lab Control Sample | |
| Sample 1 | Sample 1 | |
| Sample 1M | Sample 1 Matrix Spike | |
| Sample 1S | Sample 1 Matrix Spike Duplicate | |
| Sample 1J | Sample 1 Serial Dilution(5x dilution sample 1) | |
| Sample 1A | Sample 1 Post Digestion spike | |
| Samples 2 to 4 | Sample 2 to Sample 5 | |
| CCV2 | Continuing Calibration Verification #2 | CCV |
| CCB2 | Continuing Calibration Blank #2 | S0 |
| Samples 5 to 14 | Maximum of 10 Samples | |
| CCV3 | Continuing Calibration Verification #3 | CCV |
| CCB3 | Continuing Calibration Blank #3 | S0 |
| Samples 15 to 20 | Sample 15 to 20 or a maximum of 10 samples (sample 15 to 24) | |
| ICSA | Initial Interference Solution A | ICSA |
| ICSAB | Initial Interference Solution B | ICSAB |
| CCV4 | Continuing Calibration Verification #4 | CCV |
| LLCCV | Low Level Continuing Calibration Verification | LLCCV |
| CCB4 | Continuing Calibration Blank #4 | S0 |

³ where IMSSSS is the digestion batch reference.

TABLE 5. MDL AND LINEAR RANGE CONCENTRATION LEVELS

| ELEMENT | WATER (µg/L) | SOIL (mg/kg) | LINEAR RANGE µg/L |
|------------|--------------|--------------|----------------------|
| Aluminum | 50 | 20 | 200000 |
| Antimony | 0.5 | 0.1 | 3000 |
| Arsenic | 0.5 | 0.1 | 3000 |
| Barium | 0.5 | 0.1 | 3000 |
| Beryllium | 0.5 | 0.1 | 500 |
| Boron | 5 | 5.0 | 500 |
| Cadmium | 0.5 | 0.1 | 3000 |
| Calcium | 50 | 20 | 300000 |
| Chromium | 0.5 | 0.1 | 3000 |
| Cobalt | 0.5 | 0.1 | 3000 |
| Copper | 0.5 | 0.2 | 3000 |
| Iron | 50 | 20 | 200000 |
| Lead | 0.5 | 0.1 | 3000 |
| Lithium | 0.5 | 0.2 | 500 |
| Magnesium | 50 | 20 | 300000 |
| Manganese | 0.5 | 0.1 | 3000 |
| Molybdenum | 1.0 | 0.1 | 3000 |
| Nickel | 0.5 | 0.1 | 3000 |
| Potassium | 50 | 20 | 400000 |
| Selenium | 0.5 | 0.1 | 3000 |
| Silver | 0.5 | 0.1 | 250 |
| Sodium | 50 | 20 | 400000 |
| Strontium | 0.5 | 0.1 | 3000 |
| Thallium | 0.5 | 0.1 | 3000 |
| Tin | 0.5 | 5.0 | 3000 |
| Titanium | 1 | 0.1 | 3000 |
| Vanadium | 0.5 | 0.1 | 3000 |
| Zinc | 5 | 0.5 | 3000 |

FIGURE 1. TYPICAL SAMPLE REPORT

METHOD 6020A
 DISSOLVED METALS BY ICP-MS

```

=====
Client       : XYZ, INC.                Date Collected: 05/04/09
Project      : CLEAN WATER PROJECT     Date Received: 05/04/09
SDG NO.     : 09E036                  Date Extracted: 05/06/09 09:00
Sample ID   : 5-4-09-MW-5             Date Analyzed: 05/06/09 22:54
Lab Samp ID : E036-07                 Dilution Factor: 1
Lab File ID : 98E04059                Matrix          : WATER
Ext Btch ID : IME005W                 % Moisture     : NA
Calib. Ref. : 98E04050                Instrument ID   : EMAXTI98
=====
  
```

| PARAMETERS | RESULTS (ug/L) | RL (ug/L) | MDL (ug/L) |
|------------|-------------------|--------------|---------------|
| Aluminum | ND | 100 | 50.0 |
| Antimony | ND | 1.00 | 0.500 |
| Arsenic | 1.46 | 1.00 | 0.500 |
| Barium | 73.0 | 1.00 | 0.500 |
| Beryllium | ND | 1.00 | 0.500 |
| Cadmium | ND | 1.00 | 0.500 |
| Calcium | 51800 | 100 | 50.0 |
| Chromium | 1.41 | 1.00 | 0.500 |
| Cobalt | 0.618J | 1.00 | 0.500 |
| Copper | 1.16 | 1.00 | 0.500 |
| Iron | ND | 100 | 50.0 |
| Lead | ND | 1.00 | 0.500 |
| Magnesium | 21600 | 100 | 50.0 |
| Manganese | 9.40 | 1.00 | 0.500 |
| Molybdenum | 5.59 | 2.00 | 1.00 |
| Nickel | 158 | 1.00 | 0.500 |
| Potassium | 1640 | 100 | 50.0 |
| Selenium | 0.823J | 1.00 | 0.500 |
| Silver | ND | 1.00 | 0.500 |
| Sodium | 132000 | 100 | 50.0 |
| Thallium | ND | 1.00 | 0.500 |
| Vanadium | 27.1 | 1.00 | 0.500 |
| Zinc | ND | 10.0 | 5.00 |

FIGURE 2.

TYPICAL LCS/LCD SUMMARY

| EMAX QUALITY CONTROL DATA LCS/LCD ANALYSIS | | | | | | | | | | | |
|---|---------------------|-------------------|-----------------|-----------------|-------------------|------------------|--------------|----------|---------|------------|--------------|
| CLIENT: | XYZ, INC. | | | | | | | | | | |
| PROJECT: | CLEAN WATER PROJECT | | | | | | | | | | |
| SDG NO.: | 09E036 | | | | | | | | | | |
| METHOD: | METHOD 6020A | | | | | | | | | | |
| ===== | | | | | | | | | | | |
| MATRIX: | WATER | | | % MOISTURE: | | | NA | | | | |
| DILTN FACTR: | 1 | 1 | 1 | | | | | | | | |
| SAMPLE ID: | MBLK1W | | | | | | | | | | |
| CONTROL NO.: | IME005WB | IME005WL | IME005WC | | | | | | | | |
| LAB FILE ID: | 98E04029 | 98E04030 | 98E04031 | | | | | | | | |
| DATIME EXTRCTD: | 05/06/0909:00 | 05/06/0909:00 | 05/06/0909:00 | DATE COLLECTED: | NA | | | | | | |
| DATIME ANALYZD: | 05/06/0919:46 | 05/06/0919:52 | 05/06/0919:59 | DATE RECEIVED: | 05/06/09 | | | | | | |
| PREP. BATCH: | IME005W | IME005W | IME005W | | | | | | | | |
| CALIB. REF: | 98E04026 | 98E04026 | 98E04026 | | | | | | | | |
| ACCESSION: | | | | | | | | | | | |
| PARAMETER | BLNK RSLT ug/L | SPIKE AMT ug/L | BS RSLT ug/L | BS % REC | SPIKE AMT ug/L | BSD RSLT ug/L | BSD % REC | RPD % | QC % | LIMIT % | MAX RPD % |
| Aluminum | ND | 5000 | 4940 | 99 | 5000 | 4910 | 98 | 0 | 80-120 | 20 | |
| Antimony | ND | 50.0 | 48.3 | 97 | 50.0 | 48.2 | 96 | 0 | 80-120 | 20 | |
| Arsenic | ND | 50.0 | 49.6 | 99 | 50.0 | 49.9 | 100 | 1 | 80-120 | 20 | |
| Barium | ND | 50.0 | 50.0 | 100 | 50.0 | 49.6 | 99 | 1 | 80-120 | 20 | |
| Beryllium | ND | 50.0 | 48.5 | 97 | 50.0 | 48.8 | 98 | 1 | 80-120 | 20 | |
| Cadmium | ND | 50.0 | 49.6 | 99 | 50.0 | 49.6 | 99 | 0 | 80-120 | 20 | |
| Calcium | ND | 5000 | 4970 | 99 | 5000 | 4990 | 100 | 0 | 80-120 | 20 | |
| Chromium | ND | 50.0 | 48.8 | 98 | 50.0 | 48.6 | 97 | 1 | 80-120 | 20 | |
| Cobalt | ND | 50.0 | 50.0 | 100 | 50.0 | 49.5 | 99 | 1 | 80-120 | 20 | |
| Copper | ND | 50.0 | 47.8 | 96 | 50.0 | 48.5 | 97 | 1 | 80-120 | 20 | |
| Iron | ND | 5000 | 4910 | 98 | 5000 | 4950 | 99 | 1 | 80-120 | 20 | |
| Lead | ND | 50.0 | 49.5 | 99 | 50.0 | 49.3 | 99 | 0 | 80-120 | 20 | |
| Magnesium | ND | 5000 | 4920 | 98 | 5000 | 4900 | 98 | 0 | 80-120 | 20 | |
| Manganese | ND | 50.0 | 51.4 | 103 | 50.0 | 50.8 | 102 | 1 | 80-120 | 20 | |
| Molybdenum | ND | 50.0 | 49.7 | 99 | 50.0 | 50.1 | 100 | 1 | 80-120 | 20 | |
| Nickel | ND | 50.0 | 48.1 | 96 | 50.0 | 47.8 | 96 | 1 | 80-120 | 20 | |
| Potassium | ND | 5000 | 4990 | 100 | 5000 | 4980 | 100 | 0 | 80-120 | 20 | |
| Selenium | ND | 50.0 | 49.7 | 99 | 50.0 | 50.2 | 100 | 1 | 80-120 | 20 | |
| Silver | ND | 50.0 | 49.0 | 98 | 50.0 | 49.1 | 98 | 0 | 80-120 | 20 | |
| Sodium | ND | 5000 | 4930 | 99 | 5000 | 4980 | 100 | 1 | 80-120 | 20 | |
| Thallium | ND | 50.0 | 49.0 | 98 | 50.0 | 49.6 | 99 | 1 | 80-120 | 20 | |
| Vanadium | ND | 50.0 | 48.1 | 96 | 50.0 | 48.2 | 96 | 0 | 80-120 | 20 | |
| Zinc | ND | 50.0 | 50.5 | 101 | 50.0 | 51.6 | 103 | 2 | 80-120 | 20 | |

FIGURE 3.

TYPICAL MS/MSD SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN WATER PROJECT
SDG NO.: 09E036
METHOD: METHOD 6020A

MATRIX: WATER
DILT N FACTR: 1 1 % MOISTURE: NA
SAMPLE ID: 5-4-09-MW-5
CONTROL NO.: E036-07 E036-07M E036-07S
LAB FILE ID: 98E04059 98E04057 98E04058
DATIME EXTRACTD: 05/06/0909:00 05/06/0909:00 05/06/0909:00 DATE COLLECTED: 05/04/09
DATIME ANALYZD: 05/06/0922:54 05/06/0922:42 05/06/0922:48 DATE RECEIVED: 05/04/09
PREP. BATCH: IME005W IME005W IME005W
CALIB. REF: 98E04050 98E04050 98E04050

ACCESSION:

| PARAMETER | SAMPL RSLT ug/L | SPIKE AMT ug/L | MS RSLT ug/L | MS % REC | SPIKE AMT ug/L | MSD RSLT ug/L | MSD % REC | RPD % | QC LIMIT % | MAX RPD % |
|------------|--------------------|-------------------|-----------------|-------------|-------------------|------------------|--------------|----------|---------------|--------------|
| Aluminum | ND | 5000 | 4740 | 95 | 5000 | 4770 | 95 | 1 | 80-120 | 20 |
| Antimony | ND | 50 | 50.7 | 101 | 50 | 50.2 | 100 | 1 | 80-120 | 20 |
| Arsenic | 1.46 | 50 | 52.6 | 102 | 50 | 52.8 | 103 | 0 | 80-120 | 20 |
| Barium | 73 | 50 | 126 | 105 | 50 | 125 | 104 | 1 | 80-120 | 20 |
| Beryllium | ND | 50 | 49.7 | 99 | 50 | 49.2 | 98 | 1 | 80-120 | 20 |
| Cadmium | ND | 50 | 50.6 | 101 | 50 | 49.6 | 99 | 2 | 80-120 | 20 |
| Calcium | 51800 | 5000 | 56300 | 90 | 5000 | 55900 | 82 | 1 | 80-120 | 20 |
| Chromium | 1.41 | 50 | 49.1 | 95 | 50 | 48.9 | 95 | 0 | 80-120 | 20 |
| Cobalt | .618J | 50 | 44 | 87 | 50 | 44.1 | 87 | 0 | 80-120 | 20 |
| Copper | 1.16 | 50 | 46.6 | 91 | 50 | 46.6 | 91 | 0 | 80-120 | 20 |
| Iron | ND | 5000 | 4720 | 94 | 5000 | 4690 | 94 | 1 | 80-120 | 20 |
| Lead | ND | 50 | 49.6 | 99 | 50 | 49.1 | 98 | 1 | 80-120 | 20 |
| Magnesium | 21600 | 5000 | 26000 | 89 | 5000 | 26200 | 93 | 1 | 80-120 | 20 |
| Manganese | 9.4 | 50 | 55.6 | 92 | 50 | 56.4 | 94 | 1 | 80-120 | 20 |
| Molybdenum | 5.59 | 50 | 55.7 | 100 | 50 | 55.7 | 100 | 0 | 80-120 | 20 |
| Nickel | 158 | 50 | 203 | 91 | 50 | 202 | 89 | 0 | 80-120 | 20 |
| Potassium | 1640 | 5000 | 6790 | 103 | 5000 | 6730 | 102 | 1 | 80-120 | 20 |
| Selenium | .823J | 50 | 52.9 | 104 | 50 | 52.6 | 104 | 1 | 80-120 | 20 |
| Silver | ND | 50 | 48.4 | 97 | 50 | 47.9 | 96 | 1 | 80-120 | 20 |
| Sodium | 132000 | 5000 | 137000 | 88 | 5000 | 136000 | 82 | 0 | 80-120 | 20 |
| Thallium | ND | 50 | 49.3 | 99 | 50 | 49.5 | 99 | 0 | 80-120 | 20 |
| Vanadium | 27.1 | 50 | 76.2 | 98 | 50 | 76 | 98 | 0 | 80-120 | 20 |
| Zinc | ND | 50 | 52.7 | 105 | 50 | 52.6 | 105 | 0 | 80-120 | 20 |

FIGURE 4.

TYPICAL CASE NARRATIVE

CASE NARRATIVE

Client : XYZ, INC.

Project : CLEAN WATER PROJECT

SDG : 09E036

**METHOD 6020A
DISSOLVED METALS BY ICP-MS**

A total of six (6) water samples were received on 05/04/09 for Dissolved Metals by ICP-MS analysis, Method 6020A in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods & DoD QSM, Version 3.

Holding Time

Samples were analyzed within the prescribed holding time.

Calibration

Initial Calibration was established as prescribed by the method and was verified using a secondary source. Interference checks were performed and results were within required limits. Continuing calibration verifications and continuing calibration blanks were carried out at a frequency specified by the project. All calibration requirements were within acceptance criteria. MRL was analyzed as required by the project. Refer to MRL summary form for details.

Method Blank

Method blank was analyzed at the frequency required by the project. For this SDG, one (1) method blank was analyzed with the samples. Result was compliant to project requirement.

Lab Control Sample

A set of LCS/LCD was analyzed with the samples in this SDG. Percent recoveries for IME005WL/C were all within QC limits.

Matrix QC Sample

Matrix QC sample was analyzed at a frequency prescribed by the project. Percent recoveries for E036-07M/S were within project QC limits. In addition, analytical spike and serial dilution were analyzed for matrix interference evaluation. Results were within method acceptance criteria.

Sample Analysis

Samples were analyzed according to prescribed analytical procedures. All project requirements were met otherwise anomalies were discussed within the associated QC parameter.

SUMMARY OF IN-HOUSE QUALITY CONTROL PROCEDURES

| QC PROCEDURES | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1 st Rvw | 2 nd Rvw |
|--|--|--|---|--|---------------------|---------------------|
| Tune Check (Mass calibration and resolution check) | Daily before ICAL. | ±0.10 AMU (Mass of Isotope) <0.9 AMU full width resolution RSD of 4 replicates : ≤5% | Correct problem and repeat tune check. | | | |
| Initial Calibration (multi-point) | Daily initial calibration prior to sample analysis. | $r \geq 0.998$ | Correct the problem and repeat the initial calibration. | | | |
| Initial Calibration Verifications (ICV) Second Source | Daily after the initial calibration. | All analytes within ±10% of expected value RSD of Replicate integrations: < 5% | Correct the problem and repeat the initial calibration. | | | |
| Low Level Calibration Verification (LLICV / LLCCV) | LLICV: Daily after initial calibration. LLCCV: At the end of the analysis sequence | All analytes with ± 30% of expected value. | Correct the problem and repeat the initial calibration. | | | |
| Calibration Verifications (CCV) | Daily before sample analysis, after every 10 samples and at the end of the analysis sequence. | All analytes within ±10% of expected value. RSD of replicate integrations < 5%. | Repeat calibration and reanalyze all samples since last successful calibration. | | | |
| Calibration Blanks (ICB/CCB) | After every calibration verification | All target analytes < RL. | Correct problem then reanalyze calibration blank and previous samples. | | | |
| Interference Check Sample (ICSA/ICSAB) | Analyze at the beginning of each analytical run or once every 12 hours , whichever is more frequent. | Within ±20% of expected value | Terminate analysis, correct the problem, reanalyze ICS, and reanalyze all affected samples | | | |
| Internal Standard (IS) | ICV, LLICV, CCV, LLCCV, CCBs, MB, LCS, every sample | IS Intensities > 70% from Initial Calibration Blank IS Intensity | Correct problem then re-analyze | | | |
| Method Blank | One per preparation batch | All target analytes < ½ RL. | Re-digest and reanalyze method blank and all samples processed with the contaminated blank. | Apply B to specific analyte(s) on all associated samples | | |
| Laboratory Control Sample (LCS) | One per preparation batch | % Recovery: 80% - 120% | Re-digest and reanalyze LCS and all associated samples | | | |
| Matrix Spikes (MS/MSD) | One MS/MSD every 20 project samples per matrix | % Recovery: 75% - 125% RPD ≤20% | Evaluate post spike and dilution test: • If parent sample result is "ND", evaluate post spike. • If parent sample result is high (i.e., 4x of spike concentration) and post spike failed, evaluate dilution test. | | | |
| Post Digestion Spike Addition | When MS fails. | Recovery within 80-120% of expected value | Correct the problem then reanalyze post digestion spike addition | | | |
| Dilution Test (5X) | When MS fails. | 1:5 dilution must agree within ±10% of the original determination | Evaluate. Discuss in case narrative. | | | |
| Instrument Detection Limit (IDL) | Every three months | | Correct the problem and repeat the IDL determination. | | | |
| Comments: | | | | Reviewed By: | | |
| | | | | Date: | | |

DEMONSTRATION OF CAPABILITY



6390 Joyce Drive # 100 Golden, CO 80403
 Phone 303-940-0033 Fax 866-283-0269
 www.wibby.com

Final Report - Water Pollution Proficiency Testing

Study: WP0109

Opening Date: January 5, 2009 - Closing Date: February 19, 2009

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Kenette Pimentel, Quality Assurance Manager
 310-618-8889 ext. 205

EPA Lab ID: CA00291

| Trace Metals (PT-TM-WP) | | | | | | | | Lot #: 8082-04 | |
|-------------------------|-----------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 1000 | Aluminum | 10158000 | SW 8020 A | µg/L | 341 | 373 | 283 - 402 | 253 - 432 | Acceptable |
| 1005 | Antimony | 10158000 | SW 8020 A | µg/L | 526 | 497 | 412 - 589 | 388 - 633 | Acceptable |
| 1010 | Arsenic | 10158000 | SW 8020 A | µg/L | 76.3 | 73.5 | 65.1 - 86.9 | 59.7 - 92.4 | Acceptable |
| 1015 | Barium | 10158000 | SW 8020 A | µg/L | 239 | 243 | 215 - 263 | 207 - 269 | Acceptable |
| 1020 | Beryllium | 10158000 | SW 8020 A | µg/L | 29.9 | 27.8 | 25.7 - 32.9 | 24.1 - 34.0 | Acceptable |
| 1025 | Boron | 10158000 | SW 8020 A | µg/L | 882 | 877 | 780 - 979 | 731 - 1030 | Acceptable |
| 1030 | Cadmium | 10158000 | SW 8020 A | µg/L | 67.1 | 67 | 59.9 - 73.9 | 56.4 - 77.4 | Acceptable |

| Trace Metals (PT-TM-WP) cont'd | | | | | | | | Lot #: 8082-04 | |
|--------------------------------|------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 1040 | Chromium | 10158000 | SW 8020 A | µg/L | 59.5 | 61 | 52.7 - 65.9 | 49.4 - 69.2 | Acceptable |
| 1050 | Cobalt | 10158000 | SW 8020 A | µg/L | 49.2 | 50.9 | 44.1 - 54.1 | 41.6 - 56.2 | Acceptable |
| 1055 | Copper | 10158000 | SW 8020 A | µg/L | 101 | 102 | 90.9 - 111 | 88.5 - 114 | Acceptable |
| 1070 | Iron | 10158000 | SW 8020 A | µg/L | 561 | 577 | 505 - 617 | 494 - 637 | Acceptable |
| 1075 | Lead | 10158000 | SW 8020 A | µg/L | 71.9 | 72.4 | 61.5 - 82.5 | 56.3 - 87.7 | Acceptable |
| 1100 | Molybdenum | 10158000 | SW 8020 A | µg/L | 74.5 | 71.3 | 63.2 - 85.0 | 57.8 - 90.5 | Acceptable |
| 1105 | Nickel | 10158000 | SW 8020 A | µg/L | 316 | 313 | 284 - 348 | 291 - 356 | Acceptable |

DEMONSTRATION OF CAPABILITY



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Final Report - Water Pollution Proficiency Testing

Study: WP0109

Opening Date: January 5, 2009 - Closing Date: February 19, 2009

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Kenette Pimentel, Quality Assurance Manager
 310-618-8889 ext. 205

EPA Lab ID: CA00291

| Trace Metals (PT-TM-WP) cont'd | | | | | | | | | |
|---------------------------------------|-----------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|-----------------------|
| | | | | | | | | | Lot #: 8082-04 |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 1140 | Selenium | 10158000 | SW 8020 A | µg/L | 267 | 269 | 226 - 294 | 209 - 310 | Acceptable |
| 1150 | Silver | 10158000 | SW 8020 A | µg/L | 29.0 | 28.9 | 25.7 - 31.9 | 24.2 - 33.5 | Acceptable |
| 1160 | Strontium | 10158000 | SW 8020 A | µg/L | 276 | 267 | 248 - 304 | 241 - 312 | Acceptable |
| 1165 | Thallium | 10158000 | SW 8020 A | µg/L | 90.5 | 87.3 | 61.1 - 114 | 48.0 - 127 | Acceptable |
| 1185 | Vanadium | 10158000 | SW 8020 A | µg/L | 76.7 | 75.2 | 69.0 - 84.4 | 66.4 - 86.8 | Acceptable |
| 1150 | Silver | 10014809 | EPA 200.8 | µg/L | 29.0 | 28.9 | 25.7 - 31.9 | 24.2 - 33.5 | Acceptable |
| 1190 | Zinc | 10158000 | SW 8020 A | µg/L | 134 | 135 | 121 - 152 | 113 - 160 | Acceptable |

DEMONSTRATION OF CAPABILITY



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Final Report - Soil / Hazardous Waste PT

Study: HW0109

Opening Date: January 19, 2009 - Closing Date: March 5, 2009

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Kenette Pimentel, Quality Assurance Manager
 310-618-8889 ext. 205

EPA Lab ID: CA00291

| Metals (PT-MET-SOIL) | | | | | | | | Lot #: 7035-04 |
|----------------------|-----------|-------------|--------------------|-------|----------------|--------|-------------------|----------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 1000 | Aluminum | 10158000 | SW 8020 A | mg/kg | 7390 | 10700 | 5440 - 17400 | Acceptable |
| 1005 | Antimony | 10158000 | SW 8020 A | mg/kg | 115 | 48.7 | 11.5 - 127 | Acceptable |
| 1010 | Arsenic | 10158000 | SW 8020 A | mg/kg | 98.0 | 72.3 | 52.2 - 108 | Acceptable |
| 1015 | Barium | 10158000 | SW 8020 A | mg/kg | 182 | 205 | 146 - 253 | Acceptable |
| 1020 | Beryllium | 10158000 | SW 8020 A | mg/kg | 103 | 80.9 | 63.0 - 113 | Acceptable |
| 1030 | Cadmium | 10158000 | SW 8020 A | mg/kg | 170 | 148 | 111 - 191 | Acceptable |
| 1035 | Calcium | 10158000 | SW 8020 A | mg/kg | 10100 | 10000 | 8360 - 13700 | Acceptable |
| 1040 | Chromium | 10158000 | SW 8020 A | mg/kg | 193 | 167 | 125 - 230 | Acceptable |
| 1050 | Cobalt | 10158000 | SW 8020 A | mg/kg | 82.0 | 78.1 | 60.0 - 101 | Acceptable |
| 1055 | Copper | 10158000 | SW 8020 A | mg/kg | 258 | 229 | 185 - 303 | Acceptable |
| 1070 | Iron | 10158000 | SW 8020 A | mg/kg | 23300 | 24700 | 12500 - 38200 | Acceptable |
| 1075 | Lead | 10158000 | SW 8020 A | mg/kg | 154 | 127 | 94.1 - 189 | Acceptable |
| 1085 | Magnesium | 10158000 | SW 8020 A | mg/kg | 7450 | 7520 | 6010 - 10100 | Acceptable |
| 1090 | Manganese | 10158000 | SW 8020 A | mg/kg | 445 | 568 | 401 - 746 | Acceptable |

DEMONSTRATION OF CAPABILITY



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Study: HW0109

Opening Date: January 19, 2009 - Closing Date: March 5, 2009

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EPA Lab ID: CA00291

| Metals (PT-MET-SOIL) cont'd | | | | | | | | Lot #: 7035-04 |
|------------------------------------|------------|-------------|--------------------|-------|----------------|--------|-------------------|----------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 1100 | Molybdenum | 10156000 | SW 6020 A | mg/kg | 73.0 | 68.3 | 46.8 - 90.3 | Acceptable |
| 1106 | Nickel | 10156000 | SW 6020 A | mg/kg | 98.2 | 78.9 | 60.8 - 109 | Acceptable |
| 1125 | Potassium | 10156000 | SW 6020 A | mg/kg | 3800 | 3780 | 2570 - 5350 | Acceptable |
| 1140 | Selenium | 10156000 | SW 6020 A | mg/kg | 101 | 73.3 | 47.9 - 111 | Acceptable |
| 1150 | Silver | 10156000 | SW 6020 A | mg/kg | 37.4 | 22.8 | 15.8 - 41.1 | Acceptable |
| 1155 | Sodium | 10156000 | SW 6020 A | mg/kg | 2120 | 272 | 212 - 2330 | Acceptable |
| 1160 | Strontium | 10156000 | SW 6020 A | mg/kg | 110 | 100 | 73.5 - 135 | Acceptable |
| 1165 | Thallium | 10156000 | SW 6020 A | mg/kg | 98.3 | 81.7 | 56.8 - 115 | Acceptable |
| 1175 | Tin | 10156000 | SW 6020 A | mg/kg | 310 | 108 | 64.8 - 341 | Acceptable |
| 1185 | Vanadium | 10156000 | SW 6020 A | mg/kg | 52.8 | 81.6 | 47.3 - 117 | Acceptable |
| 1190 | Zinc | 10156000 | SW 6020 A | mg/kg | 197 | 198 | 146 - 272 | Acceptable |
| NELAC Experimental Analytes | | | | | | | | |
| 1025 | Boron | 10156000 | SW 6020 A | mg/kg | 109 | 85.2 | 47.8 - 127 | Acceptable |

6020FS.

SAMPLE PREPARATION LOG

DIGESTION LOG FOR ICP-MS METALS

SOP EMAX-3005 Rev. No. 4 EMAX-3010 Rev. No. 3 EMAX-3050 Rev. No. 3 EMAX-CLP-TAL EMAX-200.8 Rev. 1 EMAX-6020 Rev. 4

Book # EIM-020

| Matrix: | | Start Date: | Time: | Temp: | °C | Ending Date: | Time: | Temp.: | °C | | | | | |
|--------------|----------------|---------------|--------------------|-------------------|-----------|------------------------|-------|---------------------|-----------------------|----------------|---|--------------|--------------------|---------------|
| BATCH: _____ | Sample Prep ID | Lab Sample ID | Matrix Description | | | Sample Amount (g / ml) | pH | Extract Volume (ml) | Digestate Description | | Standards | ID | Amount Added (ml) | |
| | | | Color | Texture / Clarity | Artifacts | | | | Color | Clarity | | | | |
| | 01 | | | | | | | | | | LCS -1 | | | |
| | 02 | | | | | | | | | | LCS -2 | | | |
| | 03 | | | | | | | | | | LCS -3 | | | |
| | 04 | | | | | | | | | | MS | | | |
| | 05 | | | | | | | | | | Reagent | Lot# / ID | Amount Added (ml) | |
| | 06 | | | | | | | | | | HNO ₃ | | | |
| | 07 | | | | | | | | | | HCl | | | |
| | 08 | | | | | | | | | | H ₂ O ₂ | | | |
| | 09 | | | | | | | | | | HNO ₃ (1:1) | | | |
| | 10 | | | | | | | | | | Digestate Location | | | |
| | 11 | | | | | | | | | | Extract Location | | | |
| | 12 | | | | | | | | | | Legend: | | | |
| | 13 | | | | | | | | | | Texture | Cs = Coarse | Md = Medium | Fu = Fine |
| | 14 | | | | | | | | | | Clarity | Cr = Clear | Cy = Cloudy | Td = Turbid |
| | 15 | | | | | | | | | | Artifacts | Rk = rocks | Sl = Shale | Vg=Vegetation |
| | 16 | | | | | | | | | | Color | Bu = blue | Bk = Black | Bu = Brown |
| | 17 | | | | | | | | | | | Gn = Green | Og = Orange | Rd = Red |
| | 18 | | | | | | | | | | | Yw = Yellow | Cl = Colorless | |
| | 19 | | | | | | | | | | Comments: | | | |
| | 20 | | | | | | | | | | <input type="checkbox"/> Soil Samples - diluted 1-ml of digestate to 10-ml reagent water (DF=10X) | | | |
| | 21 | | | | | | | | | | | | | |
| | 22 | | | | | | | | | | | | | |
| | 23 | | | | | | | | | | | | | |
| | 24 | | | | | | | | | | Prepared By: | | Standard Added By: | |
| | 25 | | | | | | | | | | Witnessed By: | | Extracts Rcvd By: | |
| 26 | | | | | | | | | | Checked By: | | | | |
| 27 | | | | | | | | | | Date Disposed: | | Disposed by: | | |

STANDARD OPERATING PROCEDURE
MERCURY IN LIQUID WASTE

SOP No.: EMAX-7470 Revision No. 4 Effective Date: 16-May-08
 Prepared By: Mary Jane Mendoza *[Signature]* Date: 05.05.08
 Approved By: Kenette Pimentel *[Signature]* Date: 05.05.08
 QA Manager
 Approved By: Caspar Pang *[Signature]* Date: 5/5/08
 Laboratory Director

Control Number: 7470-04-

1.0 SCOPE AND APPLICATION

- 1.1. This procedure applies to the measurement of Mercury in aqueous wastes, leachates, and wastewater samples by Cold Vapor Absorption Technique. This SOP is an adaptation of Methods 7470A.

2.0 SUMMARY OF METHOD

- 2.1. A representative amount of sample is digested in nitric and sulfuric acids, followed by oxidation with potassium permanganate and potassium persulfate.
- 2.2. Organic mercurial are broken down and converted into mercuric ions in order to respond to the cold vapor atomic absorption technique. Persulfate oxidation step, followed by addition of permanganate ensures that organo-mercury compounds are oxidized.
- 2.3. Absorption of radiation by mercury vapor at 253.7 nm is then measured in the digested samples.
- 2.4. **Interferences**
- 2.4.1. Sulfides, as sodium sulfide, Copper and Chloride at high concentrations are known to interfere with the recovery of mercury. Samples containing such interference may require additional permanganate (about 12.5 mL).
- 2.4.2. Care must be taken to ensure that free chlorine is absent before the mercury is swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent. In addition, the dead air space in the Digestion vessel must be purged before adding stannous sulfate.
- 2.4.3. Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

3.0 REPORTING LIMITS

3.1. Method Detection Limit (MDL)

- 3.1.1. Prepare one method blank, one LCS and a minimum of 7 samples spiked with MDL standard using reagent water. Digest and analyze them as described in Section 10.
- 3.1.2. Refer to EMAX-QA04 for acceptance criteria.

3.2. Method Reporting Limit (RL)

- 3.2.1. Reporting limit is equal to the concentration of the lowest calibration point, unless otherwise specified by the project.

STANDARD OPERATING PROCEDURE**MERCURY IN LIQUID WASTE**

SOP No.: EMAX-7470

Revision No. 4Effective Date: 16-May-08

4.0 DYNAMIC RANGE

- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (see Section 9.6). All samples analyzed above this range shall be considered “over range” and shall require dilution to properly quantitate.
- 4.2. The lowest quantifiable range of diluted samples is equal to the concentration of the lowest calibration point. All diluted samples analyzed below this range shall be considered as “under range” and shall require lower dilution factor to properly quantitate.

5.0 PRESERVATION AND HOLDING TIME

- 5.1. Check that samples received are contained in HDPE pre-cleaned containers preserved with HNO₃ and cooled at 4 °C (± 2 °C).
- 5.2. Digest all samples within 28 days from date of collection.

6.0 ASSOCIATED SOPs

- | | | |
|------|-----------|---------------------------------|
| 6.1. | EMAX-QA04 | Method Detection Limit Study |
| 6.2. | EMAX-QA08 | Corrective Action |
| 6.3. | EMAX-QC01 | Quality Control of Chemicals |
| 6.4. | EMAX-QC02 | Analytical Standard Preparation |
| 6.5. | EMAX-QC07 | Glassware Cleaning |

7.0 SAFETY

- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves, at all times when performing this procedure. Perform preparation and analysis of mercury performed in a fume hood equipped with an exhaust fan or blower.
- 7.3. Place all wastes generated during analytical process placed in the waste containers. Endorse these wastes to waste disposal section for proper disposal.
- 7.4. If for any reason, sample and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist inform your supervisor immediately so that proper action can be taken.
- 7.5. Do not look directly at the Mercury Lamp while lit. The radiation may cause damage to your eyes.
- 7.6. Perform all reagent additions under a fume hood.
- 7.7. Mercury Analyzers are to be used by trained personnel only.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES

STANDARD OPERATING PROCEDURE

MERCURY IN LIQUID WASTESOP No.: EMAX-7470 Revision No. 4 Effective Date: 16-May-08

- 9.3.3. Prepare calibration standards from the primary standard.
- 9.3.4. Prepare initial calibration verification standards and spiking standards from the secondary standard.

| Stock Std | Name | Source | CAT # | CONC | NOTES |
|----------------|---------|--------|-----------|-----------|---------------|
| Primary | Mercury | Leeman | 602-00064 | 100 mg/L | Or equivalent |
| ICV/CCV/LCS/MS | Mercury | ERA | 027 | 1000 mg/L | Or equivalent |

9.4. **Intermediate Standard Solution**

- 9.4.1. From 100 mg/L stock solution take a 1 ml aliquot and dilute to 100 ml using reagent water. The solution shall have a final concentration of 1.0 mg/L.
- 9.4.2. Prepare secondary dilution from 1000 µg/L stock solution take a 1 ml aliquot and dilute to 100 ml using reagent water. This solution shall have a final concentration of 10 mg/L.

9.5. **Working Standard**

- 9.5.1. From the secondary dilution of intermediate standard prepare the working standard solution to have a final concentration of 50 µg/L

9.6. **Initial Calibration Standards**

- 9.6.1. From the working solution prepare the following *Leeman* standards in 100 ml volumetric flasks.

| Level | Aliquot(ml) | Final Volume(ml) | Concentration (µg/L) |
|-------|-------------|------------------|----------------------|
| S1 | 0 | 50 | 0 |
| S2 | 0.2 | 50 | 0.2 |
| S3 | 1.0 | 50 | 1.0 |
| S4 | 2.0 | 50 | 2.0 |
| S5 | 5.0 | 50 | 5.0 |
| S6 | 10.0 | 50 | 10.0 |

9.7. **ICV/CCV/LCS/MS**

- 9.7.1. From the working standard, prepare ICV/CCV/LCS/MS solutions using *ERA* Standards.

| Name | Aliquot(ml) | Final Volume(ml) | Concentration (µg/L) |
|--------|-------------|------------------|----------------------|
| ICV | 2.0 | 50 | 2.0 |
| CCV | 5.0 | 50 | 5.0 |
| LCS/MS | 5.0 | 50 | 5.0 |

STANDARD OPERATING PROCEDURE

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Revision No. 4Effective Date: 16-May-08**10.0 PROCEDURES****10.1. Sample Preparation**

- 10.1.1. Transfer 50 ml of sample into 100 ml digestion vessel. Use reagent water for method blank, LCS, and calibration standards. For STLC and TCLP extracts, use 5 ml sample volume diluted with reagent water to 50 ml. (The reduction of the volume is due to waste minimization.)
- 10.1.2. Add spike standards to LCS/MS. Add appropriate standards for calibration standards. Subsequently perform the following steps for each of the prepared analytical samples.
- 10.1.3. Add 2.5 ml of concentrated H₂SO₄ and 1.25 ml concentrated HNO₃ with mixing after each addition.
- 10.1.4. Add 7.5 ml of 5% KMnO₄ solution to each vessel.
- 10.1.5. Swirl each vessel to mix and let it stand by for 15 min. Check each vessel if purple color persist. If not, add permanganate solution at 2.5 ml increments swirling the Digestion vessel at every addition, until purple color persists.
- Add the maximum amount of permanganate solution added to a sample, to the method blank, LCS, calibration standards and calibration verification standards.*
- 10.1.6. Add 4 ml of 5% potassium persulfate. Heat for 2 hours in hot block maintained at 95°C.
- 10.1.7. Allow the samples to cool.
- 10.1.8. For Leeman-PS200 and HYDRA AA Mercury Analyzer, add 3 ml hydroxylamine hydrochloride solution and dilute to 75 ml using reagent water. Proceed to 10.2.

10.2. Instrument Parameters**10.2.1. PROTOCOL****10.2.1.1. Set Values**

| | | |
|-----------------------|----------|----------|
| Instrument ID: | PS200 | HYDRA AA |
| Number of Integration | 1 | |
| Uptake time | 20 sec. | 18 sec. |
| Weight | N | N |
| Dilution | N | N |
| On/Off, times, gains | | |
| On | Y | Y |
| Time | 10 | 10 |
| INSTRUMENT | PS200 | HYDRA AA |
| Gas | 0.35 LPM | 0.15 LPM |
| Pump Rate | 5 mL/min | 7 mL/min |
| AUTOSAMPLER – Setup | | |

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-
- CCB1
 - Method Blank
 - LCS
 - QC Sample
 - Post Analytical Spike
 - Serial Dilution
 - Matrix Spike
 - Maximum of 5 sample
 - CCV
 - CCB
 - Maximum of 10 sample
 - CCV
 - CCB
- 10.4.2. Prepare a Dilution Test sample at 5 times dilution. Pipette 1.4- ml of sample , add 5.6-ml of S₀ into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.
- 10.4.3. Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.
- 10.4.4. Check QC criteria as soon as the data is available.
- 10.4.4.1. Check the LCS recoveries against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.
 - 10.4.4.2. Check the matrix spike recovery against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.
 - 10.4.4.3. Check sample result concentrations are within the calibration range.
- 10.4.5. **Dealing with Carryover**
- 10.4.5.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
 - 10.4.5.2. If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
 - 10.4.5.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carryover. If carryover is confirmed, proceed with data reduction and report the data from re-analysis.
- 10.4.6. **Method of Standard Addition (MSA)**
- 10.4.6.1. Perform MSA for all EP extracts, samples for de-listing petition, whenever a new matrix is encountered and/or as indicated above.

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Revision No. 4 Effective Date: 16-May-08*where:**r(x, y) - correlation coefficient**N - number of measurements**x_i - found value of the ith measurement*

$$r(x, y) = \frac{\frac{1}{N} \sum_{i=1}^N (x_i - \bar{x})(y_i - \bar{y})}{(SD_x)(SD_y)}$$

*x̄ - mean of found values**y_i - true value of the ith measurement**ȳ - mean of true values**SD_x - standard deviation of the found values**SD_y - standard deviation of the true values*10.6.4. **Sample Result** -

Eq.-10.6.4

*where:**C - sample concentration in ug / L or ug / Kg**CF - calibration factor*

$$C = (R_s)(CF)(DF) \frac{V_e}{S_a}$$

*DF - dilution factor**R_s - sample absorbance**V_e - extract volume in L**S_a - sample amount (ml or g)*10.6.5. **Accuracy** -

Eq.-10.6.5

*where :**%R - percent recovery*

$$\%R = \frac{C_r - C}{C_s} * 100\%$$

*C_r - concentration recovered**C - sample concentration**C_s - known concentration of spike*10.6.6. **Precision** -

Eq.-10.6.6

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where:

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

RPD □ - relative percent difference

 C_1 □ - Measured Concentration of the first sample aliquot C_2 □ - Measured Concentration of the second sample aliquot**10.6.7. Calculation for MSA - Eq-10.6.7**

where:

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x}$$

 C_x □ - concentration of the sample C_s □ - concentration of the spike S_1 □ - analytical signal of MS1 S_2 □ - analytical signal of MS2 V_x □ - volume of sample aliquot V_s □ - volume of spike/reagent water**10.7. Report Generation**

10.7.1. Generate the report using the following in-house reporting program:

| Executable Files | Required Support Files | Output |
|------------------------|---|--|
| WDBX ¹ .exe | Login File (requires network) Seq_name.sq; Gcints.txt | Method.txt [this file integrates the login sample information and the analytical sample information] |
| IF1VX.exe | Method.txt; Method.met; Method.crf; Project.pln; Qcell.txt | Sample Results (Form1) |
| IQCVX.exe | Method.txt; Method.crf; Method.qc; Project.pln; Qcell.txt | QC Summary for LCS and MS |
| QCX | Method.txt; Method.crf; Method.qc; Project.pln; Qcell.txt | Summary for Dilution Test |

10.8. Data Review

10.8.1. Check the data entry against the analytical log.

10.8.2. Check Method Blank against reporting limit.

10.8.3. Check LCS, MS/MSD and Dilution test against QC limits.

10.8.4. Evaluate analytical spike test if dilution test failed.

10.8.5. Check for possible carry-over and if confirmation is performed.

10.8.6. Generate the case narrative to include discussion of the following as found in the review process:

¹ X – latest program version

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- Number of samples analyzed
- Analytical method(s) applied
- Holding Time – That samples were analyzed within holding time. For non-compliance, state the number of days/hours that the sample(s) were off from holding time.
- Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
- Initial Calibration Verification (ICV) – That all target analytes met calibration requirements For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
- Method Blank – That MB was analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte affected and the associated sample results were flagged with “B”.
- Lab Control Sample – That LCS was analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Furthermore, if corrective action is not possible (e.g., no more samples to re-analyze) state that results were qualified.
- Matrix Spike/Matrix Spike Duplicate – That MS/MSD is extracted with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Dilution Test and Post Digestion Spike – That dilution test and post digestion spike are analyzed for every preparation batch. That positive results of dilution test are evaluated to meet the method requirement and that Post Digestion Spike is evaluated for analytes that are not detected. For non-compliant analytes, reference the associated form, (Serial Dilution Test or Analytical Spike) and that non-compliant results were indicated by “*” or refer to MSA result.
- Sample Analysis – That samples were analyzed in conformance to the method and project requirements. That positive results were qualitatively identified in accordance to the method and applied quantitation is based on the initial calibration.
- Other Anomalies (if any) – shall be discussed on a case to case basis concurred by the Supervisor or the Lab Director. Include the NCR in the data package if required by the project, otherwise archive the NCR with the analytical folder.

10.7.4. Submit the analysis package for secondary review.

10.9. Preventive Maintenance

10.9.1. Daily routine maintenance shall be observed religiously. Observe manufacturer’s notes regarding DOs and DON’Ts:

- System preparation is a **MUST** before instrument startup.
- Make certain that drying tube has been packed loosely. If drying tube is blocked, liquid may backflow into the optical cell; this will require disassembly and leaning.
- Do not shutdown the instrument when operational, abort the run first if interruption is needed.

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- 10.9.2. Daily routine maintenance, trouble shooting and major repairs shall be recorded in the maintenance log.
- 10.9.3. Maintain the instrument clean at all times.
- 10.9.4. For trouble shooting, consult the Operations Manual, Section 4.

11.0 QUALITY CONTROL**11.1. Sample Preparation QC**

- 11.1.1. A preparative batch consists of 20 or fewer samples of the same matrix, that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.
- 11.1.2. Every preparative batch shall have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. These QC samples shall be digested together with the field samples.
- 11.1.3. All reagents shall be subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2. Sample Analysis QC

- 11.2.1. Every analytical run shall be preceded with an initial calibration and initial calibration verification (ICV). The ICV standard should be obtained from a different source from that of the initial calibration. Analyze an instrument calibration blank (ICB) after the ICV. No further analysis shall be valid unless acceptance criteria are met.
- 11.2.2. Verify calibration with continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the analytical run.
 - 11.2.2.1. Dilution Test shall be performed whenever a new or unusual sample matrix is encountered.
 - 11.2.2.2. Evaluate Post Digestion Spike result if the dilution test failed to meet the acceptance criteria.
- 11.2.3. Use method of Standard Addition (MSA) technique for analysis of all EP extracts and whenever a new sample matrix is being analyzed.
- 11.2.4. Refer to Appendix 1 for acceptance criteria.

11.3. Method QC

- 11.3.1. Method Detection Limit Study must be established before the analytical procedure can be used.
- 11.3.2. Method proficiency must be established before the analytical procedure can be used.
- 11.3.3. All analysts conducting this analysis must have established demonstration of proficiency.
- 11.3.4. Perform dynamic range study at least every six months or whenever there is a significant change in instrument response. The analytically determined concentration of this standard must be within 10% of the expected value.

12.0 CORRECTIVE ACTION**12.1. Calibration**

- 12.1.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

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- Replace the sample tubing, prepare fresh rinsate and re-prepare fresh SnCl₂. Rinse the system for at least 15 minutes prior to calibration.
- If problem persist, run the latest calibration standard that passed to check for possible instrumentation problem. If it passes, this is an indication that no instrumentation problem exist, re-digest the calibration standards. If it failed, clean the lamp, prior to re-calibration.
- If problem persist, inform the supervisor for further action

12.1.2. If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest ICV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the ICV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

12.1.3. If CCV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest CCV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the CCV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

12.2. Sample Prep QCs

12.2.1. If method blank is non-compliant, consider the following suggestions to correct the problem:

- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM, otherwise perform the corrective action as specified in the PSR.

12.2.2. If LCS is non-compliant, consider the following suggestions to correct the problem:

- Check for errors in calculation and concentration of the analyte solution
- Check instrument performance to determine if it is within acceptable guidelines
- Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.
- If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate digestion problem, fill-up an NCR and order re-digestion to include the associated sample(s).

12.2.3. If MS is non-compliant, consider the following suggestions to correct the problem:

- If recovery failed to meet the acceptance criteria, and sample result is > 5X the MRL, and the spike amount is >4X of the parent sample concentration, evaluate the serial dilution sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.
- If recovery failed to meet the acceptance criteria, and sample result is ≤ 5X the MRL, and the spike amount is >4X the parent sample concentration, evaluate the post digestion spike sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.

13.0 POLLUTION PREVENTION

13.1. Mercury is a very volatile element, dangerous levels are readily attained in air. Mercury vapour should not exceed 0.1 mg/m⁻³ in air. Air saturated with the vapor at 20°C contains mercury in a concentration far greater than that limit. The danger increases at higher temperatures. It is therefore important that mercury

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be handled with care. Containers of mercury should be securely covered and spillage should be avoided. Mercury should only be handled under in a well-ventilated area. Prepare all standards in the fume hoods.

- 13.2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. A bypass must be included on the system to vent the mercury vapor into an exhaust hood.
- 13.3. Small amounts of mercury spillage can be cleaned up by addition of sulphur powder. The resulting mixture should be properly labeled and turned over to the waste disposal unit for proper disposal.

14.0 WASTE MANAGEMENT

- 14.1. Collect all waste generated, and properly turn them over to the waste disposal unit.
- 14.2. All expired standards and highly concentrated samples must be separated and properly labeled before endorsement to the waste disposal unit.

15.0 SUPPLEMENTARY NOTES

- 15.1. For soil and other solid samples, a single replicate is analyzed instead of triplicate analyses as indicated in SW-846.
- 15.2. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing Mercury analysis by cold vapor. In the instance where there is project or program specific quality control, the requirements given in the project shall take precedence over this SOP.
- 15.3. **Definition of Terms**
 - 15.3.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
 - 15.3.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
 - 15.3.2.1. **Preparation batch** is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
 - 15.3.2.2. **Analytical batch** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
 - 15.3.3. Calibration – is defined as an instrument response per unit measure. It is an experimental value by measuring the response of an instrument per unit target analyte under the method specific condition. A determinant measured from a standard to obtain the correct value of an instrument output.
 - 15.3.4. Corrective Action - Action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
 - 15.3.5. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
 - 15.3.6. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.

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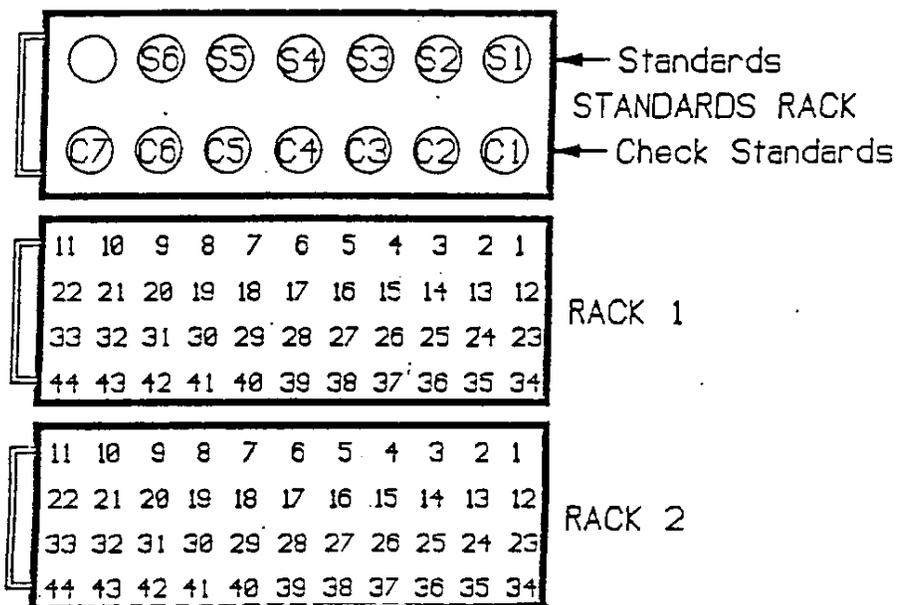
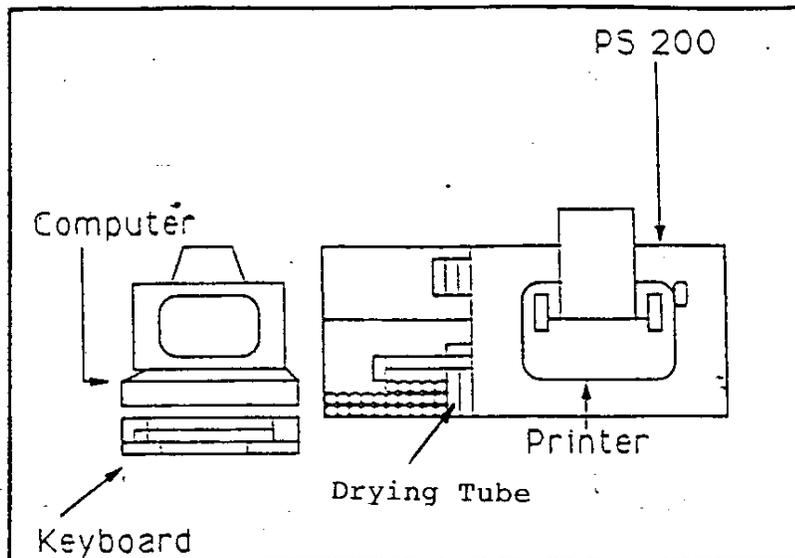
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| LEEMAN PS 200 MERCURY ANALYZER | |
|---|-------------------------|
| Description | Part Number |
| Sample Pump Tubing (blue or gray) | 309-00030 |
| Reductant Pump Tubing (red or orange) | 309-00029 |
| Drain Assembly Pump Tubing (black or gray) | 120-00050 |
| Hg Dying Tube | 318-00016 |
| Glass wool, 1 oz | 0240-1118 or equivalent |
| Fuse 2A for 110-125 VAC operation | 213-00002 |
| Fuse 1A for 208-240 VAC operation | 213-00003 |
| Fuse 3A for power supply chassis | 213-0003 |
| Mercury Lamp | 217-00003 |

Figure 1:

AUTOSAMPLER LAYOUT



Autosampler layout

Figure 2: TYPICAL CALIBRATION CURVE

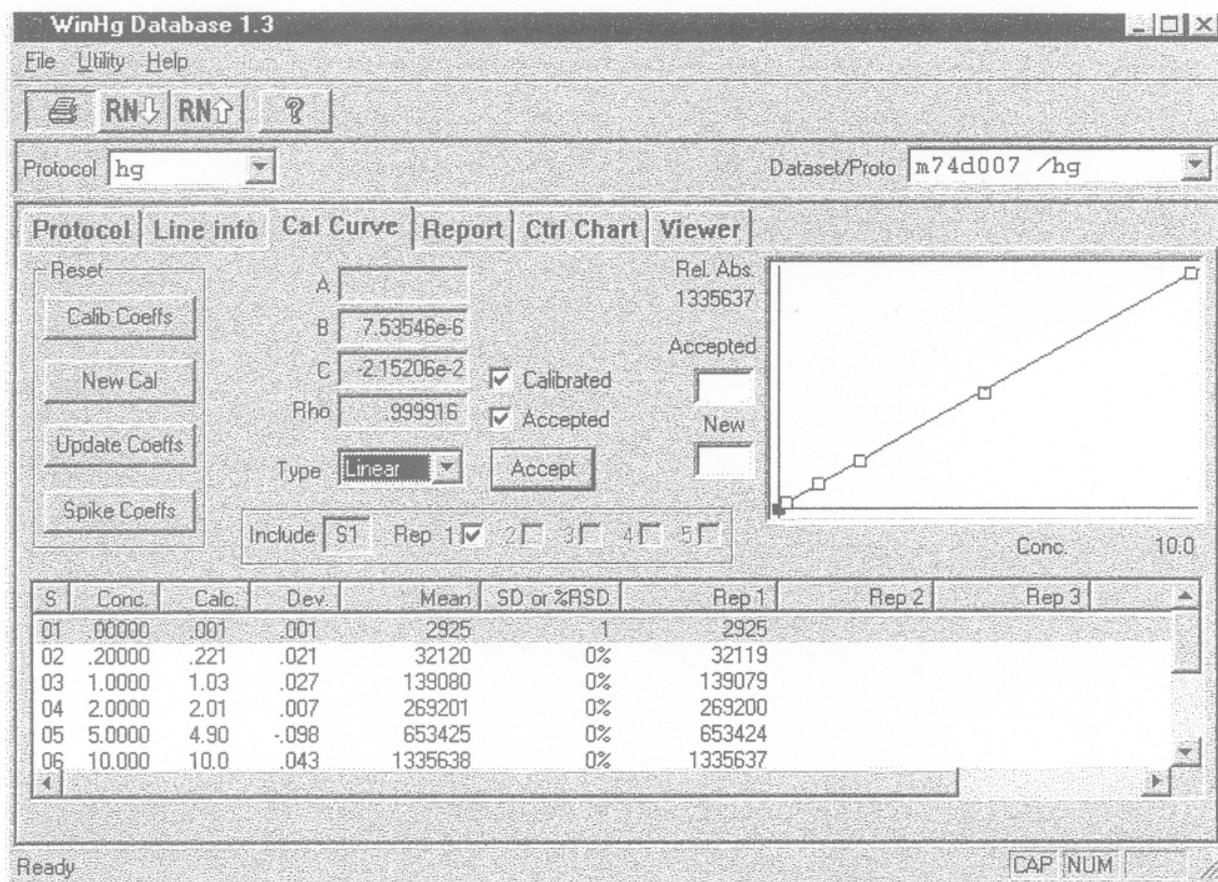


Figure 3:

TYPICAL SAMPLE REPORT SUMMARY

| METHOD 7470A MERCURY BY COLD VAPOR | | | | | | | | | | | | |
|---------------------------------------|---------------|---------------|---------------|-----------------|---------------|-----------------|-----------------|------------------|---------------|-----------------------|---------------|---------------|
| Client : XYZ INC. | | | | | | | | | | Matrix : WATER | | |
| Project : DOWN THE DRAIN PROJECT | | | | | | | | | | Instrument ID : TI047 | | |
| Batch No. : 08D131 | | | | | | | | | | | | |
| Received | EMAX | RESULTS | RL | MDL | Analysis | Extraction | Collection | | | | | |
| SAMPLE ID | SAMPLE ID | (mg/L) | DLF MOIST | (mg/L) | (mg/L) | DATETIME | DATETIME | LFID | CAL REF | PREP BATCH | DATETIME | |
| ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | |
| MBLK1W | HGD022WB | ND | 1 | NA | 0.000500 | 0.000100 | 04/24/0819:38 | 04/24/0812:30 | M47D021065 | M47D021056 | HGD022W | NA |
| 04/24/08 | LCS1W | HGD022WL | 0.00509 | 1 | NA | 0.000500 | 0.000100 | 04/24/0817:47 | 04/24/0812:30 | M47D021011 | M47D021008 | HGD022W |
| NA | 04/24/08 | LCD1W | HGD022WC | 0.00505 | 1 | NA | 0.000500 | 0.000100 | 04/24/0817:49 | 04/24/0812:30 | M47D021012 | M47D021008 |
| HGD022W | NA | 04/24/08 | MBLK1S | TXD004SB | ND | 1 | NA | 0.00500 | 0.00100 | 04/24/0818:01 | 04/24/0812:30 | M47D021018 |
| M47D021008 | HGD022W | NA | 04/24/08 | BALSAM SLUDGEAS | D131-01A | 0.0208 | 1 | NA | 0.00500 | 0.00100 | 04/24/0818:03 | 04/24/0812:30 |
| M47D021019 | M47D021008 | HGD022W | 04/09/08 | 04/14/08 | BALSAM SLUDGE | D131-01 | ND | 1 | NA | 0.00500 | 0.00100 | 04/24/0818:09 |
| 04/24/0812:30 | M47D021022 | M47D021020 | HGD022W | 04/09/08 | 04/14/08 | BALSAM SLUDGEDL | D131-01J | ND | 1 | NA | 0.0250 | 0.00500 |
| 04/24/0818:11 | 04/24/0812:30 | M47D021023 | M47D021020 | HGD022W | 04/09/08 | 04/14/08 | BALSAM SLUDGEMS | D131-01M | 0.0515 | 1 | NA | 0.00500 |
| 0.00100 | 04/24/0818:14 | 04/24/0812:30 | M47D021024 | M47D021020 | HGD022W | 04/09/08 | 04/14/08 | BALSAM SLUDGEMSD | D131-01S | 0.0501 | 1 | NA |
| 0.00500 | 0.00100 | 04/24/0818:16 | 04/24/0812:30 | M47D021025 | M47D021020 | HGD022W | 04/09/08 | 04/14/08 | | | | |

Figure 4:

TYPICAL LAB CONTROL SAMPLE REPORT SUMMARY

| EMAX QUALITY CONTROL DATA LCS/LCD ANALYSIS | | | | | | | | | | | |
|---|------------------------|---------------|---------------|-----------------|-----------|----------|-------|-------------|----------|---------|--|
| CLIENT: | XYZ INC. | | | | | | | | | | |
| PROJECT: | DOWN THE DRAIN PROJECT | | | | | | | | | | |
| SDG NO.: | 08D131 | | | | | | | | | | |
| METHOD: | METHOD 7470A | | | | | | | | | | |
| ===== | | | | | | | | | | | |
| MATRIX: | WATER | | | | | | | % MOISTURE: | NA | | |
| DILTN FACTR: | 1 | 1 | 1 | | | | | | | | |
| SAMPLE ID: | MBLK1W | | | | | | | | | | |
| CONTROL NO.: | HGD022WB | HGD022WL | HGD022WC | | | | | | | | |
| LAB FILE ID: | M47D021065 | M47D021011 | M47D021012 | | | | | | | | |
| DATIME EXTRCTD: | 04/24/0812:30 | 04/24/0812:30 | 04/24/0812:30 | DATE COLLECTED: | NA | | | | | | |
| DATIME ANALYZD: | 04/24/0819:38 | 04/24/0817:47 | 04/24/0817:49 | DATE RECEIVED: | 04/24/08 | | | | | | |
| PREP. BATCH: | HGD022W | | | | | | | | | | |
| CALIB. REF: | M47D021056 | M47D021008 | M47D021008 | | | | | | | | |
| ACCESSION: | | | | | | | | | | | |
| PARAMETER | BLNK RSLT | SPIKE AMT | BS RSLT | BS | SPIKE AMT | BSD RSLT | BSD | RPD | QC LIMIT | MAX RPD | |
| | mg/L | mg/L | mg/L | % REC | mg/L | mg/L | % REC | % | % | % | |
| ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | |
| Mercury | ND | 0.00500 | 0.00509 | 102 | 0.00500 | 0.00505 | 101 | 1 | 80-120 | 20 | |

Figure 5:

TYPICAL MATRIX SPIKE REPORT SUMMARY

| EMAX QUALITY CONTROL DATA MS/MSD ANALYSIS | | | | | | | | | | | |
|--|------------------------|---------------|---------------|-----------------|-----------|----------|-------|-------------|----------|---------|--|
| CLIENT: | XYZ INC. | | | | | | | | | | |
| PROJECT: | DOWN THE DRAIN PROJECT | | | | | | | | | | |
| SDG NO.: | 08D131 | | | | | | | | | | |
| METHOD: | METHOD 7470A | | | | | | | | | | |
| ===== | | | | | | | | | | | |
| MATRIX: | WATER | | | | | | | % MOISTURE: | NA | | |
| DILTN FACTR: | 1 | 1 | 1 | | | | | | | | |
| SAMPLE ID: | BALSAM SLUDGE | | | | | | | | | | |
| CONTROL NO.: | D131-01 | D131-01M | D131-01S | | | | | | | | |
| LAB FILE ID: | M47D021022 | M47D021024 | M47D021025 | | | | | | | | |
| DATIME EXTRCTD: | 04/24/0812:30 | 04/24/0812:30 | 04/24/0812:30 | DATE COLLECTED: | 04/09/08 | | | | | | |
| DATIME ANALYZD: | 04/24/0818:09 | 04/24/0818:14 | 04/24/0818:16 | DATE RECEIVED: | 04/14/08 | | | | | | |
| PREP. BATCH: | HGD022W | | | | | | | | | | |
| CALIB. REF: | M47D021020 | M47D021020 | M47D021020 | | | | | | | | |
| ACCESSION: | | | | | | | | | | | |
| PARAMETER | SMPL RSLT | SPIKE AMT | MS RSLT | MS | SPIKE AMT | MSD RSLT | MSD | RPD | QC LIMIT | MAX RPD | |
| | mg/L | mg/L | mg/L | % REC | mg/L | mg/L | % REC | % | % | % | |
| ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | |
| Mercury | ND | 0.05000 | 0.0515 | 103 | 0.05000 | 0.0501 | 100 | 3 | 75-125 | 20 | |

Figure 6-TYPICAL CASE NARRATIVE

Figure 6:

TYPICAL DILUTION TEST REPORT SUMMARY

| EMAX QUALITY CONTROL DATA | | | | |
|---------------------------|---------------------|---------------------------|-----------------|-----------------|
| SERIAL DILUTION ANALYSIS | | | | |
| CLIENT: | MWH LABORATORIES | | | |
| PROJECT: | 237095 | | | |
| BATCH NO.: | 08D131 | | | |
| METHOD: | METHOD 7470A | | | |
| ===== | | | | |
| MATRIX: | WATER | | % MOISTURE: | NA |
| DILUTION FACTOR: | 1 | 1 | | |
| SAMPLE ID: | BALSAM SLUDGE | BALSAM SLUDGEDL | | |
| EMAX SAMP ID: | D131-01 | D131-01J | | |
| LAB FILE ID: | M47D021022 | M47D021023 | | |
| DATE EXTRACTED: | 04/24/0812:30 | 04/24/0812:30 | DATE COLLECTED: | 04/09/08 |
| DATE ANALYZED: | 04/24/0818:09 | 04/24/0818:11 | DATE RECEIVED: | 04/14/08 |
| PREP. BATCH: | HGD022W | HGD022W | | |
| CALIB. REF: | M47D021020 | M47D021020 | | |
| ACCESSION: | | | | |
| PARAMETER | SMPL RSLT (mg/L) | SERIAL DIL RSLT (mg/L) | DIF RSLT (%) | QC LIMIT (%) |
| ----- | ----- | ----- | ----- | ----- |
| Mercury | ND | ND | 0 | 10 |

Figure 7:

TYPICAL ANALYTICAL SPIKE REPORT SUMMARY

| EMAX QUALITY CONTROL DATA | | | | | |
|---------------------------|---------------------|---------------------|-------------------|-------------|-----------------|
| ANALYTICAL SPIKE ANALYSIS | | | | | |
| CLIENT: | MWH LABORATORIES | | | | |
| PROJECT: | 237095 | | | | |
| SDG NO.: | 08D131 | | | | |
| METHOD: | METHOD 7470A | | | | |
| ===== | | | | | |
| MATRIX: | WATER | | % MOISTURE: | NA | |
| DILTN FACTR: | 1 | 1 | | | |
| SAMPLE ID: | BALSAM SLUDGE | | | | |
| CONTROL NO.: | D131-01 | D131-01A | | | |
| LAB FILE ID: | M47D021022 | M47D021019 | | | |
| DATIME EXTRCTD: | 04/24/0812:30 | 04/24/0812:30 | DATE COLLECTED: | 04/09/08 | |
| DATIME ANALYZD: | 04/24/0818:09 | 04/24/0818:03 | DATE RECEIVED: | 04/14/08 | |
| PREP. BATCH: | HGD022W | HGD022W | | | |
| CALIB. REF: | M47D021020 | M47D021008 | | | |
| ACCESSION: | | | | | |
| PARAMETER | SMPL RSLT (mg/L) | SPIKE AMT (mg/L) | AS RSLT (mg/L) | AS % REC | QC LIMIT (%) |
| ----- | ----- | ----- | ----- | ----- | ----- |
| Mercury | ND | 0.0200 | 0.0208 | 104 | 85-115 |

Figure 6-TYPICAL CASE NARRATIVE

Figure 8:

TYPICAL CASE NARRATIVE

CASE NARRATIVE

CLIENT: XYZ INC.

PROJECT: DOWN THE DRAIN PROJECT

SDG: 08D131

Method SW7470A

Mercury

A total of 1 water sample was received on 4/14/2008 for Mercury to be analyzed by Method SW7471A in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

HOLDING TIME

Samples were analyzed within holding time as prescribed by the method.

CALIBRATION

Initial calibration was analyzed specified by method and verified by a second source. Continuing calibration verification (CCB/CCV) was carried out at a frequency specified by the project. All calibration requirements were met.

METHOD BLANK

Method Blank was analyzed at a frequency specified by the project. Results are compliant to project requirements.

LAB CONTROL SAMPLE

LCS/LCD were analyzed at a frequency specified by the project. All percent recoveries met the project QC limits.

MATRIX QC SAMPLE

MS/MSD were analyzed as specified by the project. All QC results were within the project specified QC limits.

SAMPLE DUPLICATE

Duplicate samples were analyzed as specified by the project. Results were within the project specified QC limits.

SAMPLE ANALYSIS

Samples were analyzed according to the prescribed QC procedures. All criteria were met.

SUMMARY OF QUALITY CONTROL PROCEDURES

| PARAMETER | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1 ST Rvw | 2 nd Rvw |
|--|---|--|---|-----------------------------------|------------------------|------------------------|
| Initial multipoint calibration | Daily initial calibration prior to sample analysis | Correlation coefficient $R \geq 0.995$ for linear regression | Correct the problem then repeat initial calibration | | | |
| Initial calibration verification (second source) | Daily after initial calibration | Analyte within $\pm 10\%$ of expected value | Correct the problem then repeat initial calibration | | | |
| Calibration blank | After every calibration verification | No analyte detected $\geq RL$ | Correct the problem then re-analyze calibration blank and previous samples | | | |
| Calibration verification | Daily, before sample analysis, every 10 samples and at the end of analysis sequence | The analyte within $\pm 20\%$ of expected value | Repeat calibration and re-analyze all samples since last successful calibration | | | |
| Method blank | One per preparation batch | No analytes detected $\geq RL$ | Re-prep and re-analyze method blank and all samples processed with the contaminated blank | Apply B on all associated samples | | |
| LCS | One LCS per preparation batch | %Rec.: 80-120% | Re-prep and re-analyze the LCS and all associated samples | | | |
| Dilution Test | Each preparatory batch | Within $\pm 10\%$ of the undiluted sample result | Perform recovery test | | | |
| Recovery test (Analytical Spike) | When dilution test fails | Recovery within 85-115% of expected results | Run all samples by the method of standard addition | | | |
| MS/MSD | One MS/MSD per every 20 project samples per matrix | %Rec. 75-125%, RPD=20% | If all other QC samples are in control, discuss in the case narrative, otherwise check the source of the problem and re-analyze the parent sample and the MS/MSD. | | | |
| Comments: RL = lowest calibration point | | | | Reviewed by | | |
| | | | | Date | | |

APPENDIX 2:

DEMONSTRATION OF CAPABILITY



6390 Joyce Drive Phone 303-940-0033
 # 100 Fax 866-283-0269
 Golden, CO 80403 www.wibby.com

Final Report - Water Pollution Proficiency Testing

Study: WP0108

Opening Date: January 7, 2008 - Closing Date: February 21, 2008

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-618-8889 ext. 205

EPA Lab ID: CA00291

| Mercury (PT-HG-WP) | | | | | | | | | Lot #: 8070-05 |
|--------------------|---------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|----------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 1095 | Mercury | 10037204 | EPA 245.2 | µg/L | 21.6 | 21.5 | 16.3 - 26.9 | 13.7 - 29.5 | Acceptable |
| 1095 | Mercury | 10165807 | SW7470A | µg/L | 21.6 | 21.5 | 16.3 - 26.9 | 13.7 - 29.5 | Acceptable |

7470FS:

SAMPLE PREPARATION LOG

DIGESTION LOG FOR MERCURY

SOP EMAX-7470 Rev. No.3 EMAX-7471 Rev. No.3 EMAX-CLP-245.5 EMAX-CLP-245.1

Matrix: Start Date: Time: Ending Date: Time: Book # E47-040

| Sample Prep ID | Lab Sample ID | Matrix Description | | | Sample Amount (g ml) | Dry Weight | pH | Extract Volume (ml) | Matrix Description | | Standards | ID | Conc. (µg/L) | Amount Added (ml) |
|----------------|---------------|--------------------|-------------------|-----------|------------------------|------------|----|---------------------|--------------------|---------|--|-------------|--------------|-------------------|
| | | Color | Texture / Clarity | Artifacts | | | | | Color | Clarity | | | | |
| 01 | | | | | | | | | | | ICAL | | | |
| 02 | | | | | | | | | | | ICV | | | |
| 03 | | | | | | | | | | | CCV/MS | | | |
| 04 | | | | | | | | | | | LCS | | | |
| 05 | | | | | | | | | | | Reagent | Lot# / ID | | |
| 06 | | | | | | | | | | | HNO ₃ | | | |
| 07 | | | | | | | | | | | HCl | | | |
| 08 | | | | | | | | | | | H ₂ SO ₄ | | | |
| 09 | | | | | | | | | | | KMnO ₄ | | | |
| 10 | | | | | | | | | | | K ₂ S ₂ O ₈ | | | |
| 11 | | | | | | | | | | | NH ₂ OH.HCl | | | |
| 12 | | | | | | | | | | | SnCl ₂ | | | |
| 13 | | | | | | | | | | | Temp of Digestion Bath: °C | | | |
| 14 | | | | | | | | | | | Legend | | | |
| 15 | | | | | | | | | | | Color | | | |
| 16 | | | | | | | | | | | Bu = blue | Cs = Coarse | Cr = Clear | Rk = rocks |
| 17 | | | | | | | | | | | Bk = black | Md = Medium | Cy = Cloudy | Sl = Shale |
| 18 | | | | | | | | | | | Bn = Brown | Fn = Fine | Td = Turbid | Vg=Vegetation |
| 19 | | | | | | | | | | | Gn = Green | | | |
| 20 | | | | | | | | | | | Og = Orange | | | |
| 21 | | | | | | | | | | | Rd = Red | | | |
| 22 | | | | | | | | | | | Yw = Yellow | | | |
| 23 | | | | | | | | | | | Comments: _____ | | | |
| 24 | | | | | | | | | | | Prepared By: _____ | | | |
| 25 | | | | | | | | | | | Standard Added By: _____ Witnessed By: _____ | | | |
| 26 | | | | | | | | | | | Checked By: _____ | | | |
| 27 | | | | | | | | | | | Disposed By: _____ Date Disposed: _____ | | | |
| 28 | | | | | | | | | | | | | | |

EMAX LABORATORIES, INC. 1835 W. 20th St. Torrance, CA 90501

BOOK

STANDARD OPERATING PROCEDURE
MERCURY IN SOLID OR SEMISOLID WASTE

SOP No.: EMAX-7471 Revision No. 4 Effective Date: 16-May-08

Prepared By: Mary Jane Mendoza *MJM* Date: 05.05.08

Approved By: Kenette Pimentel *KP* Date: 05.05.08
 QA Manager

Approved By: Caspar Pang *CP* Date: 5/5/08
 Laboratory Director

Control Number: 7471-04-

1.0 SCOPE AND APPLICATION

- 1.1. This procedure applies to the measurement of Mercury in domestic and industrial wastes, soils, sediments, extracts and sludge samples by Cold Vapor Absorption Technique. This SOP is an adaptation of Methods 7471A.

2.0 SUMMARY OF METHOD

- 2.1. A representative amount of sample is digested in nitric and sulfuric acids, followed by oxidation with potassium permanganate and potassium persulfate.
- 2.2. Organic mercurial are broken down and converted into mercuric ions in order to respond to the cold vapor atomic absorption technique. Persulfate oxidation step, followed by addition of permanganate ensures that organo-mercury compounds are oxidized.
- 2.3. Absorption of radiation by mercury vapor at 253.7 nm is then measured in the digested samples.
- 2.4. **Interferences**
- 2.4.1. Sulfides, as sodium sulfide, Copper and Chloride at high concentrations are known to interfere with the recovery of mercury. Samples containing such interference may require additional permanganate (about 12.5 mL).
- 2.4.2. Care must be taken to ensure that free chlorine is absent before the mercury is swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent. In addition, the dead air space in the digestion vessel must be purged before adding stannous sulfate.
- 2.4.3. Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

3.0 REPORTING LIMITS

3.1. Method Detection Limit (MDL)

- 3.1.1. Prepare one method blank, one LCS and a minimum of 7 samples spiked with MDL standard using reagent water. Digest and analyze them as described in Section 10.
- 3.1.2. Refer to EMAX-QA04 for acceptance criteria.

3.2. Method Reporting Limit (RL)

- 3.2.1. Reporting limit is equal to the concentration of the lowest calibration point, unless otherwise specified by the project.

4.0 DYNAMIC RANGE

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- 4.1. The highest quantifiable range requiring no dilution is equal to the concentration of the highest calibration point (see Section 9.6). All samples analyzed above this range shall be considered “over range” and shall require dilution to properly quantitate.
- 4.2. The lowest quantifiable range of diluted samples is equal to the concentration of the lowest calibration point. All diluted samples analyzed below this range shall be considered as “under range” and shall require lower dilution factor to properly quantitate.

5.0 PRESERVATION AND HOLDING TIME

- 5.1. Check that samples received are contained in HDPE pre-cleaned containers preserved with HNO₃ and cooled at 4 °C (± 2 °C).
- 5.2. Digest all samples within 28 days from date of collection.

6.0 ASSOCIATED SOPs

- | | | |
|------|-----------|---------------------------------|
| 6.1. | EMAX-QA04 | Method Detection Limit Study |
| 6.2. | EMAX-QA08 | Corrective Action |
| 6.3. | EMAX-QC01 | Quality Control of Chemicals |
| 6.4. | EMAX-QC02 | Analytical Standard Preparation |
| 6.5. | EMAX-QC07 | Glassware Cleaning |

7.0 SAFETY

- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. Treat all reagents, standards, and samples as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves, at all times when performing this procedure. Perform preparation and analysis of mercury performed in a fume hood equipped with an exhaust fan or blower.
- 7.3. Place all wastes generated during analytical process placed in the waste containers. Endorse these wastes to waste disposal section for proper disposal.
- 7.4. If for any reason, sample and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist inform your supervisor immediately so that proper action can be taken.
- 7.5. Do not look directly at the Mercury Lamp while lit. The radiation may cause damage to your eyes.
- 7.6. Perform all reagent additions under a fume hood.
- 7.7. Mercury Analyzers are to be used by trained personnel only.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES

- 8.1. **Instruments and Supplies**
 - 8.1.1. Mercury Analyzers

STANDARD OPERATING PROCEDURE
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| Stock Std | Name | Source | CAT # | CONC | NOTES |
|----------------|---------|--------|-----------|-----------|---------------|
| Primary | Mercury | Leeman | 602-00064 | 100 mg/L | Or equivalent |
| ICV/CCV/LCS/MS | Mercury | ERA | 027 | 1000 mg/L | Or equivalent |

9.4. Intermediate Standard Solution

9.4.1. From 100 mg/L stock solution take a 1 ml aliquot and dilute to 100 ml using reagent water. The solution shall have a final concentration of 1.0 mg/L.

9.4.2. Prepare secondary dilution from 1000 µg/L stock solution take a 1 ml aliquot and dilute to 100 ml using reagent water. This solution shall have a final concentration of 10 mg/L.

9.5. Working Standard

9.5.1. From the secondary dilution of intermediate standard prepare the working standard solution to have a final concentration of 50 µg/L

9.6. Initial Calibration Standards

9.6.1. From the working solution prepare the following *Leeman* standards in 100 ml volumetric flasks.

| Level | Aliquot(ml) | Final Volume(ml) | Concentration (µg/L) |
|-------|-------------|------------------|----------------------|
| S1 | 0 | 50 | 0 |
| S2 | 0.2 | 50 | 0.2 |
| S3 | 1.0 | 50 | 1.0 |
| S4 | 2.0 | 50 | 2.0 |
| S5 | 5.0 | 50 | 5.0 |
| S6 | 10.0 | 50 | 10.0 |

9.7. ICV/CCV/LCS/MS

9.7.1. From the working standard, prepare ICV/CCV/LCS/MS solutions using *ERA* Standards.

| Name | Aliquot(ml) | Final Volume(ml) | Concentration (µg/L) |
|--------|-------------|------------------|----------------------|
| ICV | 2.0 | 50 | 2.0 |
| CCV | 5.0 | 50 | 5.0 |
| LCS/MS | 5.0 | 50 | 5.0 |

10.0 PROCEDURES

10.1. Sample Preparation

10.1.1. Weigh a representative 0.6 gm portion of wet sample into Digestion vessels.

10.1.2. Add spike standards to LCS/MS. Add appropriate standards for ICV and CCVs in a clean Digestion vessel.

10.1.3. Add 5 ml of Reagent water and 5 ml of aqua regia with mixing after each addition.

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10.2.2. DATA OUTPUT – Specify Report

| Data Output | Real Time | Post Run |
|---|-----------|----------|
| Samples | Y | Y |
| Standards | Y | Y |
| Updates | Y | Y |
| Peaks | N | N |
| IEC Stds. | N | N |
| Check Stds. | Y | Y |
| Dups and % Diff. | Y | Y |
| Wavelength | N | N |
| Rel. Absorbances | N | N |
| % RSD | Y | Y |
| Scans to PRN | | N |
| Detail | | Y |
| Summary | | N |
| Post Run Copies | | 1 |
| Post Run Report Order [1-sorted; 2- sequential] | 2 | |

10.3. **Calibration**

10.3.1. **Initial Calibration (ICAL)**

- 10.3.1.1. Prepare initial calibration solution as described in Section 9.6.1
- 10.3.1.2. Perform the same procedural steps used for analytical samples as described in Section 10.1.
- 10.3.1.3. Analyze them as described in Section 10.4.
- 10.3.1.4. Refer to Section 10.5 for calculation.
- 10.3.1.5. Initiate initial calibration as described in the instrument operations manual and acquire the calibration data for review after calibration is completed.
- 10.3.1.6. Refer to Appendix 1 for acceptance criteria.
- 10.3.1.7. Verify the initial calibration by a secondary source standard.

10.3.2. **Initial Calibration Verification (ICV)**

- 10.3.2.1. Prepare ICV as described in Section 9.7
- 10.3.2.2. Perform the same procedural steps used for analytical samples as described in Section 10.1.
- 10.3.2.3. Analyze the ICV sample to verify the concentration of the ICAL
- 10.3.2.4. Refer to Appendix 1 for acceptance criteria.

10.3.3. **Continuing Calibration Verification (CCV)**

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- 10.3.3.1. Prepare CCV as described in Section 9.7.
- 10.3.3.2. Perform the same procedural steps used for analytical samples as described in Section 10.1.
- 10.3.3.3. Analyze CCV sample to verify the validity of ICAL.
- 10.3.3.4. Refer to Appendix 1 for acceptance criteria.

10.4. Analysis

10.4.1. Calibration and Analytical Sequence

10.4.1.1. Refer to the instrument operations manual for proper calibration and analytical sequence setup (auto sampler setup).

10.4.1.2. Analytical batch ID naming convention : MIIMSSS

where:

- M – is for Mercury and always is the first character
- II – is the instrument number
- M – is the month code (A to L = January to December)
- SSS – is a sequential number (resets to 001 for the first folder created each month)

10.4.1.3. Typical Calibration Sequence

- S1 – 0.00000
- S2 – 0.20000
- S3 – 1.0000
- S4 – 2.0000
- S5 – 5.0000
- S6 – 10.000

10.4.1.4. Typical Analytical Sequence

- ICV
- ICB
- CCV1
- CCB1
- Method Blank
- LCS
- QC Sample
- Post Analytical Spike
- Serial Dilution
- Matrix Spike
- Maximum of 5 sample
- CCV

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- CCB
- Maximum of 10 sample
- CCV
- CCB

10.4.2. Prepare a Dilution Test sample at 5 times dilution. Pipette 1.4- ml of sample , add 5.6-ml of S_0 into a sample tube. Seal the tube with parafilm and invert the tube several times to ensure adequate mixing.

10.4.3. Prepare a Post Digestion Spike test sample. Using the un-spiked sample digestate (preferably the QC sample), add MS standard to maintain the same spike level as the MS sample digestate.

10.4.4. Check QC criteria as soon as the data is available.

10.4.4.1. Check the LCS recoveries against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.

10.4.4.2. Check the matrix spike recovery against project specific requirement (PSR). In the absence of PSR, default to Appendix 1 for QC limits.

10.4.4.3. Check sample result concentrations are within the calibration range.

10.4.5. Dealing with Carryover

10.4.5.1. Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.

10.4.5.2. If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.

10.4.5.3. If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carryover. If carryover is confirmed, proceed with data reduction and report the data from re-analysis.

10.4.6. Method of Standard Addition (MSA)

10.4.6.1. Perform MSA for all EP extracts, samples for de-listing petition, whenever a new matrix is encountered and/or as indicated above.

10.4.6.2. Prepare three sample solutions (Ms1, Ms2, Ms3) to objectively produce equal increments of concentration in the final solution without diluting the sample more than 50% of its original volume and expected concentrations falls within the linear range.

Example: Sample concentration is tentatively determined at 2 $\mu\text{g/L}$.

Ms1 – take 10-ml of digestate and add 0.2-ml of 100- $\mu\text{g/L}$ spike standard ($\approx 6\text{-}\mu\text{g/L}$)

Ms2 – take 10-ml of digestate and add 0.4-ml of 100- $\mu\text{g/L}$ spike standard ($\approx 7\text{-}\mu\text{g/L}$)

Ms 3 – take 10-ml of digestate and add 0.6-ml of 100- $\mu\text{g/L}$ spike standard ($\approx 8\text{-}\mu\text{g/L}$)

10.4.6.3. Analyze Ms1, Ms2 and Ms3 and calculate the results using Eq-10.6.7.

10.4.6.4. Download the electronic data to the network.

10.4.6.5. Complete the analytical log and make a copy. Highlight the data to be reported and draft the case narrative.

10.4.6.6. Submit the analytical folder to data processing.

10.5. Data Reduction

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- 10.5.1. Check that the analytical data generated indicating positive results are quantitatively correct.
- 10.5.2. Check that analytical results are generated by the prescribed calibration schedule of method.
- 10.5.3. Check for possible carry-over. Re-analyze samples having trace level concentration preceded by a sample having a concentration over the calibration range.
- 10.5.4. Check the MS/MSD, serial dilution and the post digestion spike results. If matrix interference is indicated, check the PSR if MSA is waived, otherwise refer to 10.4.6.

10.6. Calculations

10.6.1. **Calibration Factor** - Eq.-10.6.1

where:

$$CF = \frac{R_a}{C_a}$$

CF - calibration factor
R_a - Response for analyte measured in absorbance
C_a - known concentration of analyte measured in ug / L

10.6.2. **Average Calibration Factor** - Eq.-10.6.2

where:

$$ACF = \frac{\sum CF}{n}$$

ACF - average response factor
∑ CF - sum of calibration factors
n - number of calibration points

10.6.3. **Correlation Coefficient** - Eq.10.6.3

where:

r(x, y) - correlation coefficient

N - number of measurements

x_i - found value of the ith measurement

$$r(x, y) = \frac{\frac{1}{N} \sum_{i=1}^N (x_i - \bar{x})(y_i - \bar{y})}{(SD_x)(SD_y)}$$

y_i - true value of the ith measurement

̄x - mean of found values

̄y - mean of true values

SD_x - standard deviation of the found values

SD_y - standard deviation of the true values

10.6.4. **Sample Result** - Eq.-10.6.4

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where:

 C - sample concentration in ug / L or ug / Kg CF - calibration factor

$$C = (R_s)(CF)(DF) \frac{V_e}{S_a}$$

 DF - dilution factor R_s - sample absorbance V_e - extract volume in L S_a - sample amount (ml or g)

10.6.5. Accuracy -

Eq.-10.6.5

where :

%R - percent recovery

$$\%R = \frac{C_r - C}{C_s} * 100\%$$

 C_r - concentration recovered C - sample concentration C_s - known concentration of spike

10.6.6. Precision -

Eq.-10.6.6

where:

RPD - relative percent difference

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

 C_1 - Measured Concentration of the first sample aliquot C_2 - Measured Concentration of the second sample aliquot

10.6.7. Calculation for MSA -

Eq.-10.6.7

where:

 C_x - concentration of the sample C_s - concentration of the spike

$$C_x = \frac{(S_2)(V_s)(C_s)}{(S_2 - S_1)V_x}$$

 S_1 - analytical signal of MS1 S_2 - analytical signal of MS2 V_x - volume of sample aliquot V_s - volume of spike/reagent water**10.7. Report Generation**

10.7.1. Generate the report using the following in-house reporting program:

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MERCURY IN SOLID OR SEMISOLID WASTE

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| Executable Files | Required Support Files | Output |
|------------------------|---|--|
| WDBX ¹ .exe | Login File (requires network) Seq_name.sq; Gcints.txt | Method.txt [this file integrates the login sample information and the analytical sample information] |
| IF1VX.exe | Method.txt; Method.met; Method.crf; Project.pln; Qcell.txt | Sample Results (Form1) |
| IQCVX.exe | Method.txt; Method.crf; Method.qc; Project.pln; Qcell.txt | QC Summary for LCS and MS |
| QCX | Method.txt; Method.crf; Method.qc; Project.pln; Qcell.txt | Summary for Dilution Test |

10.8. Data Review

- 10.8.1. Check the data entry against the analytical log.
- 10.8.2. Check Method Blank against reporting limit.
- 10.8.3. Check LCS, MS/MSD and Dilution test against QC limits.
- 10.8.4. Evaluate analytical spike test if dilution test failed.
- 10.8.5. Check for possible carry-over and if confirmation is performed.
- 10.8.6. Generate the case narrative to include discussion of the following as found in the review process:
 - Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples were analyzed within holding time. For non-compliance, state the number of days/hours that the sample(s) were off from holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
 - Initial Calibration Verification (ICV) – That all target analytes met calibration requirements For non-compliance, state the analyte(s) that is (are) non-compliant and the data qualified.
 - Method Blank – That MB was analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte affected and the associated sample results were flagged with “B”.
 - Lab Control Sample – That LCS was analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Furthermore, if corrective action is not possible (e.g., no more samples to re-analyze) state that results were qualified.
 - Matrix Spike/Matrix Spike Duplicate – That MS/MSD is extracted with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.

¹ X – latest program version

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- Dilution Test and Post Digestion Spike – That dilution test and post digestion spike are analyzed for every preparation batch. That positive results of dilution test are evaluated to meet the method requirement and that Post Digestion Spike is evaluated for analytes that are not detected. For non-compliant analytes, reference the associated form, (Serial Dilution Test or Analytical Spike) and that non-compliant results were indicated by “*” or refer to MSA result.
- Sample Analysis – That samples were analyzed in conformance to the method and project requirements. That positive results were qualitatively identified in accordance to the method and applied quantitation is based on the initial calibration.
- Other Anomalies (if any) – shall be discussed on a case to case basis concurred by the Supervisor or the Lab Director. Include the NCR in the data package if required by the project, otherwise archive the NCR with the analytical folder.

10.7.4. Submit the analysis package for secondary review.

10.9. **Preventive Maintenance**

10.9.1. Daily routine maintenance shall be observed religiously. Observe manufacturer’s notes regarding DOs and DON’Ts:

- System preparation is a **MUST** before instrument startup.
- Make certain that drying tube has been packed loosely. If drying tube is blocked, liquid may backflow into the optical cell; this will require disassembly and leaning.
- Do not shutdown the instrument when operational, abort the run first if interruption is needed.

10.9.2. Daily routine maintenance, trouble shooting and major repairs shall be recorded in the maintenance log.

10.9.3. Maintain the instrument clean at all times.

10.9.4. For trouble shooting, consult the Operations Manual, Section 4.

11.0 **QUALITY CONTROL**

11.1. **Sample Preparation QC**

11.1.1. A preparative batch consists of 20 or fewer samples of the same matrix, that are prepared for analysis simultaneously or sequentially, using the same lots of all reagents.

11.1.2. Every preparative batch shall have at least one method blank, one LCS and a set of MS/MSD unless otherwise specified by the project. These QC samples shall be digested together with the field samples.

11.1.3. All reagents shall be subjected to QC check prior to its use. Refer to EMAX-QC01 for details.

11.2. **Sample Analysis QC**

11.2.1. Every analytical run shall be preceded with an initial calibration and initial calibration verification (ICV). The ICV standard should be obtained from a different source from that of the initial calibration. Analyze an instrument calibration blank (ICB) after the ICV. No further analysis shall be valid unless acceptance criteria are met.

11.2.2. Verify calibration with continuing calibration verification (CCV) standard and continuing calibration blank (CCB) after every ten samples and at the end of the analytical run.

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11.2.2.1. Dilution Test shall be performed whenever a new or unusual sample matrix is encountered.

11.2.2.2. Evaluate Post Digestion Spike result if the dilution test failed to meet the acceptance criteria.

11.2.3. Use method of Standard Addition (MSA) technique for analysis of all EP extracts and whenever a new sample matrix is being analyzed.

11.2.4. Refer to Appendix 1 for acceptance criteria.

11.3. Method QC

11.3.1. Method Detection Limit Study must be established before the analytical procedure can be used.

11.3.2. Method proficiency must be established before the analytical procedure can be used.

11.3.3. All analysts conducting this analysis must have established demonstration of proficiency.

11.3.4. Perform dynamic range study at least every six months or whenever there is a significant change in instrument response. The analytically determined concentration of this standard must be within 10% of the expected value.

12.0 CORRECTIVE ACTION

12.1. Calibration

12.1.1. If initial calibration is non-compliant, consider the following suggestions to correct the problem:

- Replace the sample tubing, prepare fresh rinsate and re-prepare fresh SnCl₂. Rinse the system for at least 15 minutes prior to calibration.
- If problem persist, run the latest calibration standard that passed to check for possible instrumentation problem. If it passes, this is an indication that no instrumentation problem exist, re-digest the calibration standards. If it failed, clean the lamp, prior to re-calibration.
- If problem persist, inform the supervisor for further action

12.1.2. If the ICV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest ICV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the ICV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

12.1.3. If CCV is non-compliant, consider the following suggestions to correct the problem:

- Run the latest CCV standard that passed to check for possible standards preparation error. If it passes, this is an indication of standards preparation error. Re-digest the CCV and re-analyze. If it fails, refer to 12.1.1 prior to re-calibration.

12.2. Sample Prep QCs

12.2.1. If method blank is non-compliant, consider the following suggestions to correct the problem:

- Check the sample results. If sample results are non-detected, you may report the result upon concurring with the PM, otherwise perform the corrective action as specified in the PSR.

12.2.2. If LCS is non-compliant, consider the following suggestions to correct the problem:

- Check for errors in calculation and concentration of the analyte solution
- Check instrument performance to determine if it is within acceptable guidelines

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- Recalculate the data and/or reanalyze the extract if any of the above checks reveals a problem.
- If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate digestion problem, fill-up an NCR and order re-digestion to include the associated sample(s).

12.2.3. If MS is non-compliant, consider the following suggestions to correct the problem:

- If recovery failed to meet the acceptance criteria, and sample result is $> 5X$ the MRL, and the spike amount is $>4X$ of the parent sample concentration, evaluate the serial dilution sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.
- If recovery failed to meet the acceptance criteria, and sample result is $\leq 5X$ the MRL, and the spike amount is $>4X$ the parent sample concentration, evaluate the post digestion spike sample result. Refer to Appendix 1 for acceptance criteria. If it fails to meet the acceptance criteria, perform MSA.

13.0 POLLUTION PREVENTION

- 13.1. Mercury is a very volatile element, dangerous levels are readily attained in air. Mercury vapour should not exceed 0.1 mg/m^3 in air. Air saturated with the vapor at 20°C contains mercury in a concentration far greater than that limit. The danger increases at higher temperatures. It is therefore important that mercury be handled with care. Containers of mercury should be securely covered and spillage should be avoided. Mercury should only be handled under in a well-ventilated area. Prepare all standards in the fume hoods.
- 13.2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. A bypass must be included on the system to vent the mercury vapor into an exhaust hood.
- 13.3. Small amounts of mercury spillage can be cleaned up by addition of sulphur powder. The resulting mixture should be properly labeled and turned over to the waste disposal unit for proper disposal.

14.0 WASTE MANAGEMENT

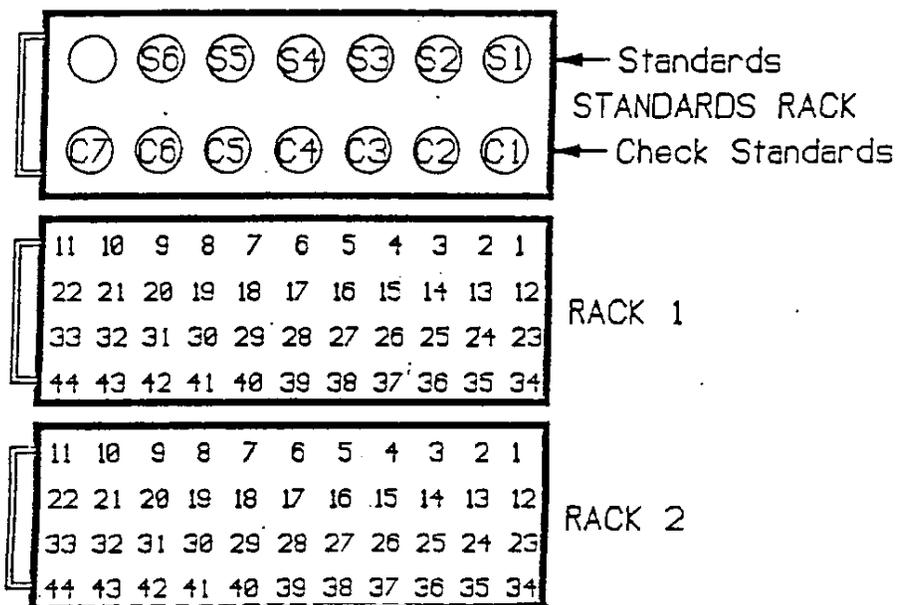
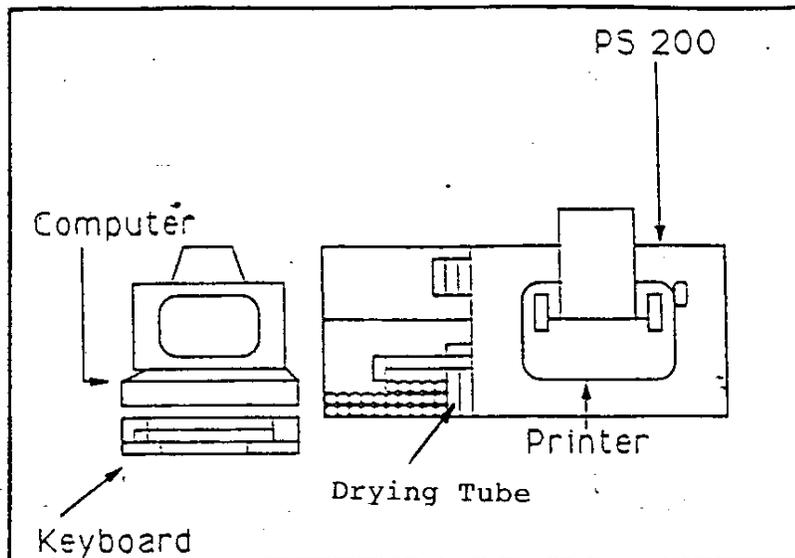
- 14.1. Collect all waste generated, and properly turn them over to the waste disposal unit.
- 14.2. All expired standards and highly concentrated samples must be separated and properly labeled before endorsement to the waste disposal unit.

15.0 SUPPLEMENTARY NOTES

- 15.1. For soil and other solid samples, a single replicate is analyzed instead of triplicate analyses as indicated in SW-846.
- 15.2. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing Mercury analysis by cold vapor. In the instance where there is project or program specific quality control, the requirements given in the project shall take precedence over this SOP.
- 15.3. **Definition of Terms**
- 15.3.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
- 15.3.2. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.

Figure 1:

AUTOSAMPLER LAYOUT



Autosampler layout

Figure 2: TYPICAL CALIBRATION CURVE

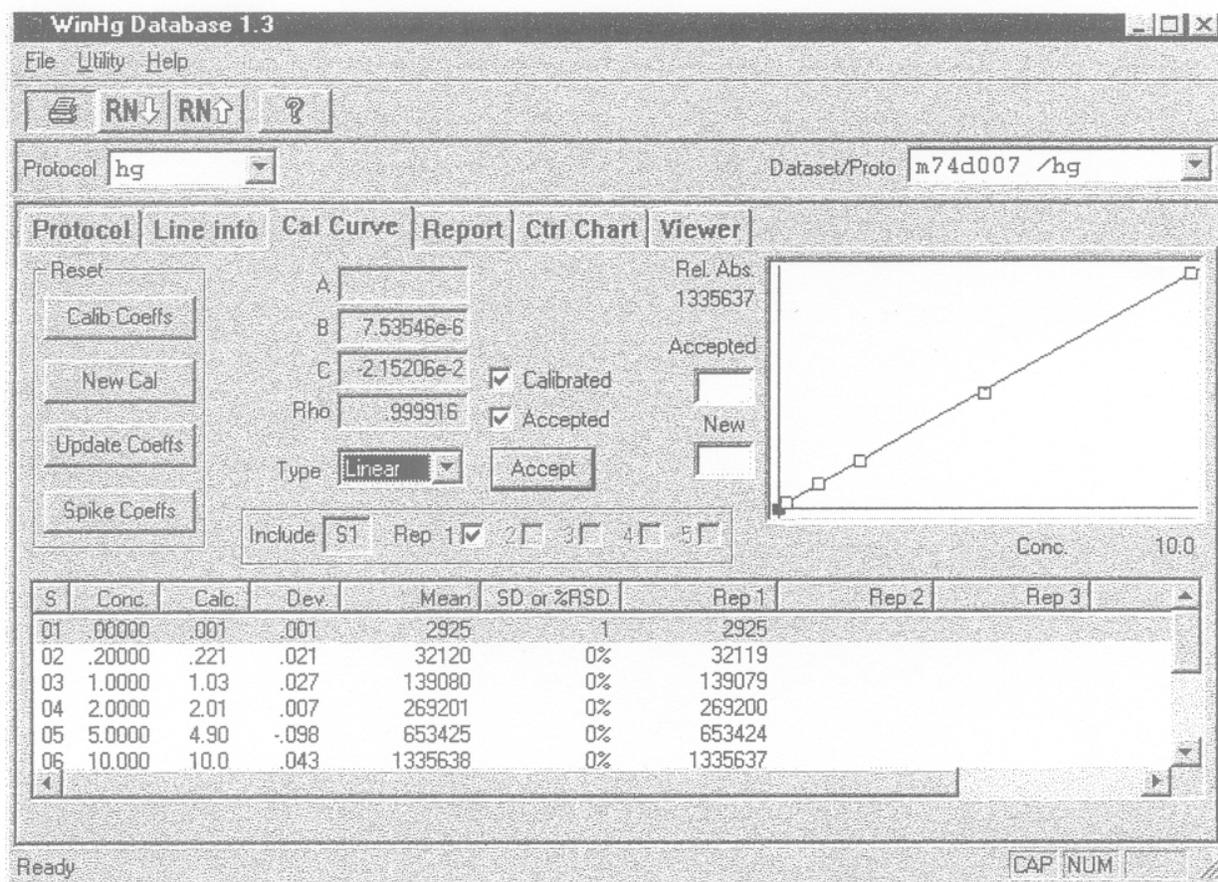


Figure 3:

TYPICAL SAMPLE REPORT SUMMARY

| METHOD 7471A MERCURY BY COLD VAPOR | | | | | | | | | | | | | |
|---------------------------------------|---------------------------|----------|-------|-------|---------|----------|------------|---------------|---------------|------------|-----------------------|---------|----------|
| Client : XYZ INC. | | | | | | | | | | | Matrix : SOIL | | |
| Project : CLEAN LAND PROJECT | | | | | | | | | | | Instrument ID : TI047 | | |
| Batch No. : 08D125 | | | | | | | | | | | | | |
| Received | EMAX | RESULTS | RL | | MDL | Analysis | Extraction | | | Collection | | | |
| SAMPLE ID | SAMPLE ID | (mg/kg) | DLF | MOIST | (mg/kg) | DATETIME | DATETIME | LFID | CAL REF | PREP BATCH | DATETIME | | |
| 04/15/08 | MBLK1S | HGD017SB | ND | 1 | NA | 0.100 | 0.0330 | 04/15/0817:57 | 04/15/0815:30 | M47D016034 | M47D016032 | HGD017S | NA |
| 04/15/08 | LCS1S | HGD017SL | 0.843 | 1 | NA | 0.100 | 0.0330 | 04/15/0817:59 | 04/15/0815:30 | M47D016035 | M47D016032 | HGD017S | NA |
| 04/15/08 | LCD1S | HGD017SC | 0.827 | 1 | NA | 0.100 | 0.0330 | 04/15/0818:01 | 04/15/0815:30 | M47D016036 | M47D016032 | HGD017S | NA |
| 04/11/08 | INSITU G9-I-1 | D125-08 | ND | 1 | NA | 0.100 | 0.0330 | 04/15/0818:05 | 04/15/0815:30 | M47D016038 | M47D016032 | HGD017S | 04/10/08 |
| 04/11/08 | INSITU G9-I-1DL | D125-08J | ND | 5 | NA | 0.500 | 0.165 | 04/15/0818:07 | 04/15/0815:30 | M47D016039 | M47D016032 | HGD017S | 04/10/08 |
| 04/11/08 | INSITU G9-I-1MS | D125-08M | 0.863 | 1 | NA | 0.100 | 0.0330 | 04/15/0818:09 | 04/15/0815:30 | M47D016040 | M47D016032 | HGD017S | 04/10/08 |
| 04/11/08 | INSITU G9-I-1MSD | D125-08S | 0.853 | 1 | NA | 0.100 | 0.0330 | 04/15/0818:11 | 04/15/0815:30 | M47D016041 | M47D016032 | HGD017S | 04/10/08 |
| 04/11/08 | DRUM G9-1 | D125-01 | ND | 1 | NA | 0.100 | 0.0330 | 04/15/0818:13 | 04/15/0815:30 | M47D016042 | M47D016032 | HGD017S | 04/07/08 |
| 04/11/08 | DRUM G9-5 | D125-02 | ND | 1 | NA | 0.100 | 0.0330 | 04/15/0818:15 | 04/15/0815:30 | M47D016043 | M47D016032 | HGD017S | 04/07/08 |
| 04/11/08 | DRUM G9-6 | D125-03 | ND | 1 | NA | 0.100 | 0.0330 | 04/15/0818:22 | 04/15/0815:30 | M47D016046 | M47D016044 | HGD017S | 04/07/08 |
| 04/09/08 | ROLL-OFF B1-1 04/11/08 | D125-04 | ND | 1 | NA | 0.100 | 0.0330 | 04/15/0818:24 | 04/15/0815:30 | M47D016047 | M47D016044 | HGD017S | |
| 04/11/08 | ROLL-OFF B1-2 | D125-05 | 0.263 | 1 | NA | 0.100 | 0.0330 | 04/15/0818:26 | 04/15/0815:30 | M47D016048 | M47D016044 | HGD017S | 04/09/08 |
| 04/11/08 | ROLL-OFF B1-3 | D125-06 | ND | 1 | NA | 0.100 | 0.0330 | 04/15/0818:28 | 04/15/0815:30 | M47D016049 | M47D016044 | HGD017S | 04/09/08 |
| 04/11/08 | ROLL-OFF B1-4 | D125-07 | 2.83 | 10 | NA | 1.00 | 0.330 | 04/16/0811:31 | 04/15/0815:30 | M47D017011 | M47D017009 | HGD017S | 04/09/08 |

Figure 4:

TYPICAL LAB CONTROL SAMPLE REPORT SUMMARY

| EMAX QUALITY CONTROL DATA LCS/LCD ANALYSIS | | | | | | | | | | | |
|---|--------------------|---------------|---------------|-----------------|-----------|----------|-------|-------------|----------|---------|--|
| CLIENT: | XYZ INC. | | | | | | | | | | |
| PROJECT: | CLEAN LAND PROJECT | | | | | | | | | | |
| SDG NO.: | 08D125 | | | | | | | | | | |
| METHOD: | METHOD 7471A | | | | | | | | | | |
| ===== | | | | | | | | | | | |
| MATRIX: | SOIL | | | | | | | % MOISTURE: | | NA | |
| DILTN FACTR: | 1 | | 1 | | | | | | | | |
| SAMPLE ID: | MBLK1S | | | | | | | | | | |
| CONTROL NO.: | HGD017SB | HGD017SL | HGD017SC | | | | | | | | |
| LAB FILE ID: | M47D016034 | M47D016035 | M47D016036 | | | | | | | | |
| DATIME EXTRCTD: | 04/15/0815:30 | 04/15/0815:30 | 04/15/0815:30 | DATE COLLECTED: | | NA | | | | | |
| DATIME ANALYZD: | 04/15/0817:57 | 04/15/0817:59 | 04/15/0818:01 | DATE RECEIVED: | | 04/15/08 | | | | | |
| PREP. BATCH: | HGD017S | | | | | | | | | | |
| CALIB. REF: | M47D016032 | M47D016032 | M47D016032 | | | | | | | | |
| ACCESSION: | | | | | | | | | | | |
| PARAMETER | BLNK RSLT | SPIKE AMT | BS RSLT | BS | SPIKE AMT | BSD RSLT | BSD | RPD | QC LIMIT | MAX RPD | |
| | mg/kg | mg/kg | mg/kg | % REC | mg/kg | mg/kg | % REC | % | % | % | |
| ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | |
| Mercury | ND | 0.833 | 0.843 | 101 | 0.833 | 0.827 | 99 | 2 | 80-120 | 20 | |

Figure 5:

TYPICAL MATRIX SPIKE REPORT SUMMARY

| EMAX QUALITY CONTROL DATA MS/MSD ANALYSIS | | | | | | | | | | | |
|--|--------------------|---------------|---------------|-----------------|-----------|----------|-------|-------------|----------|---------|--|
| CLIENT: | XYZ INC. | | | | | | | | | | |
| PROJECT: | CLEAN LAND PROJECT | | | | | | | | | | |
| SDG NO.: | 08D125 | | | | | | | | | | |
| METHOD: | METHOD 7471A | | | | | | | | | | |
| ===== | | | | | | | | | | | |
| MATRIX: | SOIL | | | | | | | % MOISTURE: | | NA | |
| DILTN FACTR: | 1 | 1 | 1 | | | | | | | | |
| SAMPLE ID: | INSITU G9-I-1 | | | | | | | | | | |
| CONTROL NO.: | D125-08 | D125-08M | D125-08S | | | | | | | | |
| LAB FILE ID: | M47D016038 | M47D016040 | M47D016041 | | | | | | | | |
| DATIME EXTRCTD: | 04/15/0815:30 | 04/15/0815:30 | 04/15/0815:30 | DATE COLLECTED: | 04/10/08 | | | | | | |
| DATIME ANALYZD: | 04/15/0818:05 | 04/15/0818:09 | 04/15/0818:11 | DATE RECEIVED: | 04/11/08 | | | | | | |
| PREP. BATCH: | HGD017S | | | | | | | | | | |
| CALIB. REF: | M47D016032 | M47D016032 | M47D016032 | | | | | | | | |
| ACCESSION: | | | | | | | | | | | |
| PARAMETER | SMPL RSLT | SPIKE AMT | MS RSLT | MS | SPIKE AMT | MSD RSLT | MSD | RPD | QC LIMIT | MAX RPD | |
| | mg/kg | mg/kg | mg/kg | % REC | mg/kg | mg/kg | % REC | % | % | % | |
| ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | |
| Mercury | ND | 0.833 | 0.863 | 104 | 0.833 | 0.853 | 102 | 1 | 75-125 | 20 | |

Figure 6-TYPICAL CASE NARRATIVE

Figure 6:

TYPICAL DILUTION TEST REPORT SUMMARY

| EMAX QUALITY CONTROL DATA | | | | |
|---------------------------|--------------------|-----------------|-----------------|----------|
| SERIAL DILUTION ANALYSIS | | | | |
| CLIENT: | XYZ INC. | | | |
| PROJECT: | CLEAN LAND PROJECT | | | |
| BATCH NO.: | 08D125 | | | |
| METHOD: | METHOD 7471A | | | |
| ===== | | | | |
| MATRIX: | SOIL | | % MOISTURE: | NA |
| DILUTION FACTOR: | 1 | 5 | | |
| SAMPLE ID: | INSITU G9-I-1 | INSITU G9-I-1DL | | |
| EMAX SAMP ID: | D125-08 | D125-08J | | |
| LAB FILE ID: | M47D016038 | M47D016039 | | |
| DATE EXTRACTED: | 04/15/0815:30 | 04/15/0815:30 | DATE COLLECTED: | 04/10/08 |
| DATE ANALYZED: | 04/15/0818:05 | 04/15/0818:07 | DATE RECEIVED: | 04/11/08 |
| PREP. BATCH: | HGD017S | HGD017S | | |
| CALIB. REF: | M47D016032 | M47D016032 | | |
| ACCESSION: | | | | |
| | | | | |
| PARAMETER | SMPL RSLT | SERIAL DIL RSLT | DIF RSLT | QC LIMIT |
| | (mg/kg) | (mg/kg) | % | (%) |
| ----- | ----- | ----- | ----- | ----- |
| Mercury | ND | ND | 0 | 10 |

Figure 6-TYPICAL CASE NARRATIVE

Figure 8:

TYPICAL CASE NARRATIVE

CASE NARRATIVE

CLIENT: XYZ INC.

PROJECT: CLEAN LAND PROJECT

SDG: 08D125

Method SW7471A

Mercury

A total of 8 soil samples were received on 4/11/2008 for Mercury to be analyzed by Method SW7471A in accordance with USEPA SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.

HOLDING TIME

Samples were analyzed within holding time as prescribed by the method.

CALIBRATION

Initial calibration was analyzed specified by method and verified by a second source. Continuing calibration verification (CCB/CCV) was carried out at a frequency specified by the project. All calibration requirements were met.

METHOD BLANK

Method Blank was analyzed at a frequency specified by the project. Results are compliant to project requirements.

LAB CONTROL SAMPLE

LCS/LCD were analyzed at a frequency specified by the project. All percent recoveries met the project QC limits.

MATRIX QC SAMPLE

MS/MSD were analyzed as specified by the project. All QC results were within the project specified QC limits.

SAMPLE ANALYSIS

Samples were analyzed according to the prescribed QC procedures. All analytical requirements were met.

SUMMARY OF QUALITY CONTROL PROCEDURES

| PARAMETER | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1 ST Rvw | 2 nd Rvw |
|--|---|--|---|-----------------------------------|------------------------|------------------------|
| Initial multipoint calibration | Daily initial calibration prior to sample analysis | Correlation coefficient $R \geq 0.995$ for linear regression | Correct the problem then repeat initial calibration | | | |
| Initial calibration verification (second source) | Daily after initial calibration | Analyte within $\pm 10\%$ of expected value | Correct the problem then repeat initial calibration | | | |
| Calibration blank | After every calibration verification | No analyte detected $\geq RL$ | Correct the problem then re-analyze calibration blank and previous samples | | | |
| Calibration verification | Daily, before sample analysis, every 10 samples and at the end of analysis sequence | The analyte within $\pm 20\%$ of expected value | Repeat calibration and re-analyze all samples since last successful calibration | | | |
| Method blank | One per preparation batch | No analytes detected $\geq RL$ | Re-prep and re-analyze method blank and all samples processed with the contaminated blank | Apply B on all associated samples | | |
| LCS | One LCS per preparation batch | %Rec.: 80-120% | Re-prep and re-analyze the LCS and all associated samples | | | |
| Dilution Test | Each preparatory batch | Within $\pm 10\%$ of the undiluted sample result | Perform recovery test | | | |
| Recovery test (Analytical Spike) | When dilution test fails | Recovery within 85-115% of expected results | Run all samples by the method of standard addition | | | |
| MS/MSD | One MS/MSD per every 20 project samples per matrix | %Rec. 75-125%, RPD=20% | If all other QC samples are in control, discuss in the case narrative, otherwise check the source of the problem and re-analyze the parent sample and the MS/MSD. | | | |
| Comments: RL = lowest calibration point | | | | Reviewed by | | |
| | | | | Date | | |

APPENDIX 2:

DEMONSTRATION OF CAPABILITY



6390 Joyce Drive Phone 303-940-0033
 # 100 Fax 866-283-0269
 Golden, CO 80403 www.wibby.com

Final Report - Soil / Hazardous Waste PT

Study: HW0707

Opening Date: July 23, 2007 - Closing Date: September 6, 2007

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-618-8889 ext. 205

EPA Lab ID: CA00291

Metals (PT-MET-SOIL) Lot #: 7029-04

| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
|------------|---------|-------------|--------------------|-------|----------------|--------|-------------------|------------|
| 1095 | Mercury | 10166206 | SW7417A | mg/kg | 20.1 | 9.07 | 4.53 - 22.1 | Acceptable |

7470FS:

SAMPLE PREPARATION LOG

DIGESTION LOG FOR MERCURY

SOP EMAX-7470 Rev. No.3 EMAX-7471 Rev. No.3 EMAX-CLP-245.5 EMAX-CLP-245.1

Matrix: Start Date: Time: Ending Date: Time: Book # E47-040

| Sample Prep ID | Lab Sample ID | Matrix Description | | | Sample Amount (g ml) | Dry Weight | pH | Extract Volume (ml) | Matrix Description | | Standards | ID | Conc. (µg/L) | Amount Added (ml) |
|----------------|---------------|--------------------|-------------------|-----------|------------------------|------------|----|---------------------|--------------------|---------|--|-------------|--------------|-------------------|
| | | Color | Texture / Clarity | Artifacts | | | | | Color | Clarity | | | | |
| 01 | | | | | | | | | | | ICAL | | | |
| 02 | | | | | | | | | | | ICV | | | |
| 03 | | | | | | | | | | | CCV/MS | | | |
| 04 | | | | | | | | | | | LCS | | | |
| 05 | | | | | | | | | | | Reagent | Lot# / ID | | |
| 06 | | | | | | | | | | | HNO ₃ | | | |
| 07 | | | | | | | | | | | HCl | | | |
| 08 | | | | | | | | | | | H ₂ SO ₄ | | | |
| 09 | | | | | | | | | | | KMnO ₄ | | | |
| 10 | | | | | | | | | | | K ₂ S ₂ O ₈ | | | |
| 11 | | | | | | | | | | | NH ₂ OH.HCl | | | |
| 12 | | | | | | | | | | | SnCl ₂ | | | |
| 13 | | | | | | | | | | | Temp of Digestion Bath: °C | | | |
| 14 | | | | | | | | | | | Legend | | | |
| 15 | | | | | | | | | | | Color | | | |
| 16 | | | | | | | | | | | Bu = blue | Cs = Coarse | Cr = Clear | Rk = rocks |
| 17 | | | | | | | | | | | Bk = black | Md = Medium | Cy = Cloudy | Sl = Shale |
| 18 | | | | | | | | | | | Bn = Brown | Fn = Fine | Td = Turbid | Vg=Vegetation |
| 19 | | | | | | | | | | | Gn = Green | | | |
| 20 | | | | | | | | | | | Og = Orange | | | |
| 21 | | | | | | | | | | | Rd = Red | | | |
| 22 | | | | | | | | | | | Yw = Yellow | | | |
| 23 | | | | | | | | | | | Comments: _____ | | | |
| 24 | | | | | | | | | | | Prepared By: _____ | | | |
| 25 | | | | | | | | | | | Standard Added By: _____ Witnessed By: _____ | | | |
| 26 | | | | | | | | | | | Checked By: _____ | | | |
| 27 | | | | | | | | | | | Disposed By: _____ Date Disposed: _____ | | | |
| 28 | | | | | | | | | | | | | | |

EMAX LABORATORIES, INC. 1835 W. 20th St. Torrance, CA 90501

BOOK

STANDARD OPERATING PROCEDURES

ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY

SOP No.: EMAX-8081 Revision No. 6 Effective Date: 03-Sept-07

Prepared By: Tu Nisamaneepong  Date: 08-17-07

Approved By: Kenette Pimentel 
QA Manager Date: 08-17-07

Approved By: Kam Pang 
Laboratory Director Date: 8/17/07

Control Number: **8081-06-****1.0 SCOPE AND APPLICATION**

- 1.1. This procedure is used to determine the concentration of various Organochlorine Pesticides in soil, sediment, sludge, and wastewater samples by gas chromatography method. This SOP is an adaptation of Method 8081A.

2.0 SUMMARY OF METHOD

- 2.1. This method provides gas chromatographic conditions for the identification and quantitation of organochlorine pesticides with dual Electron Capture Detectors (ECD). The samples are extracted in methylene chloride and exchanged to hexane before GC analysis.
- 2.2. **Interferences**
- 2.2.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations. Avoiding contact with any plastic materials and checking all solvents for phthalate contamination can minimize interferences. Glassware must be scrupulously cleaned.
- 2.2.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination is most likely present in sediment samples. The copper cleanup, GPC or other cleanup technique can be used for sulfur removal.

3.0 QUANTITATION LIMITS**3.1. Method Detection Limit**

- 3.1.1. Prepare a minimum of seven samples for each matrix. Add MDL spike standard (Refer to EMAX-QA04 for suggested spike levels). Prepare a method blank and LCS as described in Section 10.1 using standards as described in Section 9.8.
- 3.1.2. Analyze the samples as described in Section 10.4 and calculate the results as described in Section 10.6.
- 3.1.3. Perform MDL evaluation and verification as described in EMAX-QA04.

3.2. Reporting Limit

- 3.2.1. The lowest calibration point shall define reporting limit unless otherwise specified by the project.
- 3.2.2. Reporting limits as determined by this SOP are as listed in the table below.

| Analytes | Water (µg/L) | Soil (µg/Kg) |
|--|--------------|--------------|
| alpha-BHC, Endosulfan I, gamma-BHC, Heptachlor, Aldrin, alpha-Chlordane, beta-BHC, delta-BHC, gamma- | 0.05 | 2 |

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| Analytes | Water (µg/L) | Soil (µg/Kg) |
|--|--------------|--------------|
| Chlordane, Heptachlor Epoxide | | |
| DDD, DDE, DDT, Dieldrin, Endrin, Endosulfan II, Endosulfan Sulfate, Endrin Aldehyde, Endrin Ketone | 0.1 | 4 |
| Methoxychlor | 0.5 | 20 |

4.0 DYNAMIC RANGE

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point (see Sec. 9.4). All samples analyzed above this concentration are considered "over-range" and shall require dilution for proper quantitation.
- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required for proper quantitation.
- 4.3. The linear dynamic range for this method as determined in this SOP is listed on the table below.

| Analytes | Water (µg/L) | Soil (µg/Kg) |
|---|--------------|--------------|
| alpha-BHC, Endosulfan I, gamma-BHC, Heptachlor, Aldrin, alpha-Chlordane, beta-BHC, delta-BHC, gamma-Chlordane, Heptachlor Epoxide | 0.05 – 0.6 | 2 – 20 |
| DDD, DDE, DDT, Dieldrin, Endrin, Endosulfan II, Endosulfan Sulfate, Endrin Aldehyde, Endrin Ketone | 0.1 – 1.2 | 4 – 40 |
| Methoxychlor | 0.5 – 6 | 20 - 200 |

5.0 HOLDING TIME AND PRESERVATION**5.1. Holding Time**

- 5.1.1. Extract water and soil samples within 7 and 14 days respectively from the date of collection.
- 5.1.2. Analyze extracts within 40 days after extraction completion date.

5.2. Preservation

- 5.2.1. Store samples and extract at 4°C (± 2°C).

6.0 ASSOCIATED SOPs

- 6.1. EMAX-DM01 Data Flow and Review
- 6.2. EMAX-QA04 Method Detection Limit Study
- 6.3. EMAX-QA08 Corrective Action
- 6.4. EMAX-QC02 Analytical Standard Preparation
- 6.5. EMAX-SM04 Analytical and QC Sample Labeling
- 6.6. EMAX-QA08 Corrective Action
- 6.7. EMAX-3510 Extraction, Separatory Funnel
- 6.8. EMAX-3520 Extraction, Continuous Liquid/Liquid

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- 6.9. EMAX-3550 Extraction, Pulse Sonication
 6.10. EMAX-3540 Extraction, Soxhlet
 6.11. EMAX-3620 Cleanup, Florisil
 6.12. EMAX-3640 Cleanup, GPC
 6.13. EMAX-3660 Cleanup, Sulfur

7.0 SAFETY

- 7.1. Read all MSDS of chemicals listed in this SOP.
 7.2. Treat all reagents, standards, and samples as potential hazards.
 7.3. ECD contains minute quantity of Radioactive Ni (63), conduct a wipe test (experienced personnel or manufacturer respectively only) semi-annually or sooner if potential problem is suspected.
 7.4. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure.
 7.5. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES**8.1. Instruments and Supplies**

| | |
|--------------------|---|
| Gas Chromatography | HP 5890 Series II |
| Detector | Dual Electron Capture Detectors |
| Column | RTX CLPEST I (30 m x 0.32 mm x 0.5 µm) RTX CLPEST II (30 m x 0.32 mm x 0.25 µm) (Alternate columns may be used after verification of performance) |
| Data System | EZ Chrom |
| Auto Sampler | HP Model 7673A or equivalent |
| Gas | ultra-high purity nitrogen ultra-high purity hydrogen |
| Microsyringes | 10, 25, 100 and 500 µl with a 0.006 mm ID needle (Hamilton 702N or equivalent) for dilution purposes |
| Automatic Pipettes | Pipetman, 1000 and 200 µl |
| Transfer Pipette | Pasteur |

8.2. Chemicals and Reagents

| | |
|--------------------|----------------------------|
| Solvent [GC-grade] | Methylene Chloride, Hexane |
|--------------------|----------------------------|

9.0 STANDARDS

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ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHYSOP No.: EMAX-8081 Revision No. 6 Effective Date: 03-Sept-07**9.1. Standard Preparation**

- 9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX SM04, respectively.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.2. Stock Standard

- 9.2.1. Purchase Primary Calibration stock standards as certified solutions in two mixtures (mixture A and B). After opening, transfer the stock standard to an inert vial and store with a minimum of headspace.
- 9.2.2. Purchase a Secondary set of stock standards from a different source to verify the concentration of the first set of standard.
- 9.2.3. Purchase LCS/MS, surrogate and performance evaluation standards as certified solutions from various suppliers.
- 9.2.4. All standards shall be stored at 4°C ($\pm 2^{\circ}\text{C}$).

9.3. Intermediate Standard

- 9.3.1. Prepare intermediate standards as suggested in Tables 2 and 2A
- 9.3.2. Store all prepared standards in an inert vial with minimum headspace at 4°C ($\pm 2^{\circ}\text{C}$).

9.4. Initial Calibration Standard (ICAL)

- 9.4.1. Prepare five calibration standards as suggested in Table 1A – ICAL Standard Preparation from primary intermediate standard (refer to Table 2)

9.5. Initial Calibration Verification (ICV)

- 9.5.1. Prepare an intermediate standard from the second source stock standard (refer to Table 2A) and use this to prepare the ICV at concentration levels suggested in Table 3.

9.6. Daily Calibration Check Standard (DCC)

- 9.6.1. Prepare DCC from the same source as the ICAL standard (refer to Table 2) as suggested in Table 3.

9.7. Surrogate Standard

- 9.7.1. Prepare surrogate standard as suggested in Table 4 – Surrogate Standard Preparation

9.8. LCS/MS Spike Standard

- 9.8.1. Prepare LCS solution as suggested in Table 2A.
- 9.8.2. Prepare MS spike standard as suggested in Table 5.

9.9. Performance Evaluation Mixture (PEM)

- 9.9.1. Prepare PEM as suggested in Table 6 – Performance Evaluation Mixture Preparation

10.0 PROCEDURES**10.1. Sample Preparation**

- 10.1.1. Prepare aqueous samples as described in EMAX-3520 or EMAX-3510.
- 10.1.2. Prepare solid samples as described in EMAX-3550, EMAX-3540 or EMAX-3545.
- 10.1.3. Perform extract clean up (if necessary) as described in EMAX-3620, EMAX-3640 or EMAX-3660, whichever is appropriate.

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10.2.1. Method 8081A requires an analytical system complete with a temperature programmable gas chromatograph equipped with an autosampler suitable for on column injection of 1 to 5 μL .

10.2.2. Gas Pressure

| | |
|---------------------|--------|
| N ₂ Tank | 40 psi |
| H ₂ Tank | 80 psi |

10.2.3. Temperature Program

| | |
|-------------------|---------------------------|
| Initial Temp | 120°C, hold for 2 minutes |
| Rate 1 | 20°C/min. |
| Temperature 1 | 220°C, hold for 2 minutes |
| Rate 2 | 2°C/min |
| Temperature 2 | 230°C, hold for 0 minutes |
| Rate 3 | 25°C/ min |
| Final Temperature | 300°C, hold for 5 minutes |
| Injector | 280°C |
| Detector | 300°C |
| Injection Volume | 1 μL |

10.3. Calibration**10.3.1. Performance Evaluation Check**

10.3.1.1. Analyze instrument blank and a PEM containing DDT and Endrin to monitor the system performance at 12-hour interval prior to performing any calibration.

10.3.1.2. Calculate the breakdown by using Equations 10.6.6 (%B_T) for DDT and 10.6.7 (%B_E) for Endrin.

10.3.1.3. Check Appendix 1 for acceptance criteria before proceeding with sample analysis.

10.3.1.4. If system failed to meet the acceptance criteria, refer to Section 12 for corrective action.

10.3.2. Initial Calibration (ICAL)

10.3.2.1. Perform ICAL if instrument is new, ICV or DCC failed to meet acceptance criteria or after a major instrument repair.

10.3.2.2. A minimum of five calibration standards over the concentration range of interest are sequentially injected into the GC. Refer to Table 1 for ICAL concentrations. Peak areas are obtained from each analyte.

10.3.2.3. Calculate the calibration factor (CF) using the peak areas and their respective concentration according to Eq-10.6.1.

10.3.2.4. Calculate the Average Calibration Factor (ACF) and the RSD according to Eq. 10.6.4 and 10.6.3 respectively.

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10.3.2.5. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.3.3. **Initial Calibration Verification (ICV)**

10.3.3.1. Analyze ICV prepared from another source as described in Section 9.5 to verify the concentrations of the ICAL.

10.3.3.2. Calculate the CF and the percent difference (%D) according to Eq-10.6.1 and 10.6.5 respectively. Refer to Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.3.4. **Multicomponent Target Analyses**

10.3.4.1. For multicomponent target analytes (Toxaphene), a five-point calibration standard shall be included in initial calibration for pattern recognition and quantitation.

10.3.4.2. Integrate the total response of the chromatogram to obtain the total area. Calculate the calibration factor (CF) by using Equation 10.6.1.

10.3.5. **Daily Calibration Check (DCC)**

10.3.5.1. Analyze DCC at the start of the 12-hour shift prior to sample analysis and close the analytical run with an ending DCC.

10.3.5.2. Calculate the %D by using Equation 10.6.5. Check Appendix 1 for acceptance criteria. If acceptance criteria are not met refer to Section 12 for corrective action.

10.4. **Analysis**

10.4.1. **Analytical Sequence**

10.4.1.1. Following the instrument data acquisition software, prepare the analytical sequence file as follows:

10.4.1.2. IB – instruction blank

10.4.1.3. PEM – performance evaluation mixture

10.4.1.4. ICAL – initial calibration standards followed by Initial Calibration Verification or DCC1 – continuing calibration standard

10.4.1.5. MB – method blank

10.4.1.6. LCS – lab control sample

10.4.1.7. Samples – up to 12 hours

10.4.1.8. DCC2 – continuing calibration standard or ending DCC

10.4.2. **Sample Analysis**

10.4.2.1. Transfer approximately 0.5 ml of extract to a 2-ml amber auto sampler vial (or equivalent) using a Pasteur pipette. Seal the vial with a Teflon lined septum and aluminum rim cap. Similarly, prepare the analytical standards and QC samples.

10.4.2.2. Introduce sample extract into the GC using direct injection technique (1 to 5 µl) after all system quality control criteria have been met.

10.4.2.3. If the responses exceed the linear range of the system, dilute the sample and re-analyze.

10.4.3. **Sample Result Evaluation**

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- 10.4.3.1. Check QC parameters as soon as the data is available.
- ✓ Check surrogate recoveries against Appendix 1.
 - ✓ Check concentration of target analytes. If the response exceeds the calibration range, dilute and re-analyze the sample until the response falls within the calibration range.
 - ✓ If any of the above checkpoints indicate a problem, re-analysis is required. If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate extraction problem, fill-up an NCR and order re-extraction for the affected sample(s).
- 10.4.3.2. Positive identification is made when a peak falls within the retention time window of a target analyte on both columns established by the standard reference compound.
- 10.4.3.3. If one column meets the retention time criteria and a retention time shift is suspected on the other column, use the following guideline in reporting the data:
- ✓ Check that the expanded window does not exceed the RTW of the column in control or the established RTW or the CLP RTW (refer to table 7) which ever is greater.
 - ✓ If the above condition is met, report the data and include a description of the observation in the case narrative.

10.4.4. **Retention Time Windows**

10.4.4.1. Establishing RTW

- 10.4.4.1.1. Collect at least three Daily Calibration Standards analyzed over a period of 72 hours.
- 10.4.4.1.2. Calculate the Standard Deviation (SD) of absolute retention time obtained for each analyte.
- 10.4.4.1.3. The width of RTW is defined by $\pm 3X$ SD obtained from 10.4.4.1.2.

10.4.4.2. Evaluating RTW

- 10.4.4.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained or use the CLP¹ retention time window (refer to Table 7) which ever is narrower, until RTW is obtained for the instrument.
- 10.4.4.2.2. For new instruments, in the interim use the CLP retention time window (refer to Table 7) until RTW is obtained for the new instrument parameters condition.

10.4.4.3. Application of RTW

- 10.4.4.3.1. Establish the center of absolute retention time for each analyte to include the surrogate(s) from the daily calibration check at the beginning of the analytical shift then apply the established RTW.
- 10.4.4.3.2. Whenever the observe retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.

10.4.4.4. Updating RTW

¹ CLP-OLM4.2 Table 1 D-79/PEST

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10.4.4.4.1. Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur:

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of RTW)
- Major instrument maintenance (e.g. replacements of detector or column; temperature program change, etc.)

10.4.5. **Manual Integration**

10.4.5.1. Refer to EMAX-DM01 for details of manual integration.

10.4.6. **Dealing with Carryover**

- ✓ Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- ✓ If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
- ✓ If there was a target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.5. **Data Reduction**

10.5.1. Check the chromatogram of positively identified peaks.

- ✓ Peaks fall within the established retention time window on both columns
- ✓ Peaks are sharp and not saturated
- ✓ Peaks are properly integrated (refer to Figure-1 for Peak Evaluation Techniques)
- ✓ Target analyte peak is present in both columns to confirm positive identification.

10.5.2. Positive identification is confirmed when the identified analyte is present in both columns. The agreement between the quantitative results should be evaluated after the identification is made. Calculate the relative percent difference (RPD) between the two results according to Equation 10.6.10.2.

- 10.5.2.1. If the RPD is less than 40% and the peaks do not indicate any anomalies, report the higher result.
- 10.5.2.2. If the RPD is less than 40% and one of the peaks indicate an anomaly, report the result from the better peak.
- 10.5.2.3. If the RPD is greater than 40%, use professional judgment to select the most appropriate result. If no evidence of any chromatographic interference, report the higher result.

10.6. **Calculations**

10.6.1. Calculate for Calibration Factor (CF).

$$CF = \frac{R_a}{C_k} \quad \text{Eq. 10.6.1}$$

where:

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ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHYSOP No.: EMAX-8081 Revision No. 6 Effective Date: 03-Sept-07*CF* - is the calibration factor*R_a* - is the analyte response measured in peak area*C_k* - is the known concentration of the analyte in µg/L

10.6.2. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}} \quad \text{Eq. 10.6.2}$$

where:

SD - is the standard deviation*x_i* - is the result at the *i*th measurement \bar{x} - is the mean*N* - is the number of measurements

10.6.3. Calculate for Percent Relative Standard Deviation (%RSD).

$$\%RSD = \left[\frac{SD}{ACF} \right] 100 \quad \text{Eq. 10.6.3}$$

where:

%RSD - is the percent relative standard deviation*SD* - is the standard deviation*ACF* - is the average calibration factor

10.6.4. Calculate for Average Calibration Factor (ACF)

$$ACF = \frac{\sum CF}{N} \quad \text{Eq. 10.6.4}$$

where:

ACF - is the average calibration factor $\sum CF$ - is the summation of the calibration factors*N* - is the number of calibration points

10.6.5. Calculate for Percent Difference for DCC from ACF

$$\%D = \left[\frac{ACF - CF}{ACF} \right] 100 \quad \text{Eq. 10.6.5}$$

where:

ACF - is the average calibration factor*CF* - is the calibration factor of the DCC10.6.6. Calculate for % Breakdown for DDT (%B_i).

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$$\%B_T = \frac{A_D + A_E}{A_T + A_D + A_E} \quad \text{Eq-10.6.6}$$

where:

- $\%B_T$ - % DDT Breakdown
 A_D - Total area of DDD
 A_E - Total area of DDE
 A_T - Total area of DDT

10.6.7. Calculate for % Breakdown for Endrin ($\%B_E$).

$$\%B_E = \frac{A_A + A_K}{A_E + A_A + A_K} \quad \text{Eq-10.6.7}$$

where:

- $\%B_E$ - % Endrin Breakdown
 A_A - Total area of Endrin Aldehyde
 A_K - Total area of Endrin Ketone
 A_E - Total area of Endrin

10.6.8. Sample Results

10.6.8.1. Water Samples

$$C = \left(\frac{R_a}{AFC} \right) \left(\frac{V_e}{S_a} \right) DF \quad \text{Eq-10.6.8.1}$$

where:

- C - Concentration of sample measured in $\mu\text{g/L}$
 R_a - Total response of analyte in peak area
 AFC - Average response factor measure in ICAL
 V_e - Volume of extract in ml
 S_a - Sample amount in ml
 DF - Dilution factor of sample extract

10.6.8.2. Soil Samples

$$C = \left(\frac{R_a}{AFC} \right) \left(\frac{V_e}{S_a (\% \text{Solid})} \right) DF \quad \text{Eq.10.6.8.2}$$

where

- C - Concentration of analyte to be measured ($\mu\text{g/kg}$)
 R_a - Total response of analyte in peak area
 AFC - Average response factor

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- V_e - Volume of extract in ml
 S_a - Sample Amount in g
 $\% \text{ Solid}$ - $\frac{100 - \% \text{ moisture}}{100}$
 D - Dilution factor of the sample extract

10.6.9. Multi-peak Compound in Sample (Toxaphene)

10.6.9.1. Total area is integrated and concentration is determined by equation 10.6.8.1 or 10.6.8.2.

10.6.10. Accuracy and Precision

10.6.10.1. Percent Recovery

$$\%R = \frac{C_f - C}{C_s} * 100 \quad \text{Eq. 10.6.5.1}$$

where:

- $\%R$ - percent recovery
 C_f - concentration found
 C_s - concentration of surrogate spike

10.6.10.2. Relative Percent Difference (RPD)

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq. 10.6.5.2}$$

where:

- $\%RPD$ - Relative Percent Difference
 $C1$ - Measured concentration of the first sample aliquot
 $C2$ - Measured concentration of the second sample aliquot

10.6.11. Method Detection Limit

$$MDL = T_7SD \quad \text{Eq.-10.6.11}$$

where:

- MDL - method detection limit
 T_7 - degrees of freedom for 7 measurements, which is 3.14
 SD - standard deviation (see Eq-10.6.1.3) from 7 measurements.

10.7. Report Generation

- 10.7.1. Generate the method.txt file using WBDX¹.exe
 10.7.2. Generate Lab Chronicle using Labchron.exe

¹ X – version number

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- 10.7.3. Generate sample results using F1VX¹.exe
- 10.7.4. Generate the QC Summary file using QCVX¹.exe
- 10.7.5. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.
 - Sample Results
 - LCS Summary
 - MS/MSD Summary
 - DCC Summary
 - ICAL Summary
 - ICV Summary
 - Copy of Analysis Log
 - Copy of Preparation Log

10.8. Data Review

- 10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
 - ✓ Check the chromatogram of all positively identified peak(s) to determine the final results according to Section 10.5.2.
 - ✓ Check surrogate recoveries against project specific criteria (PSR). In the absence of PSR, default to in-house QC limits.
 - ✓ Check concentration of target analytes if calibration range is exceeded.
 - ✓ If any of the above checkpoints indicate a problem, re-analysis is required.
- 10.8.2. Generate the case narrative to include discussion of the following as found in the review process:
 - Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples extracted and analyzed within the holding time. For non-compliance, state the number of days that the sample(s) was out of holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Method Blank – That MB was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s) affected and the associated sample results were flagged with “B”.
 - Lab Control Samples (if applicable) – That LCS was prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”.

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Further more, if corrective action is not possible (e.g. no more samples to re-extract) state that results were qualified.

- Matrix Spike Samples – That MS/MSD (if applicable) were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Sample Analysis – That samples were analyzed in conformance to the method and project requirements.
- Other Anomalies (if any) – Discussed on a case by case basis concurred by the Supervisor or the Lab Director.

10.8.3. Submit the analysis package for secondary review.

10.9. Preventive Maintenance

10.9.1. Refer to form 8081FM for daily routine maintenance check points.

10.9.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back to control.

10.9.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality.

11.0 QUALITY CONTROL**11.1. Preparative Batch**

11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD and a maximum of 20 field samples of similar matrix.

11.1.2. In the absence of MS/MSD, LCS/LCD is prepared.

11.2. Analytical Batch QC

11.2.1. Instrument Performance Evaluation Check must be analyzed daily. Acceptance criteria and corrective action are discussed in Section 10.3.1.4 and Appendix 1.

11.2.2. A continuing calibration shall be performed before any other analysis is done. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.5 and Appendix 1.

11.3. Method QC

11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.

11.3.2. A valid MDL must exist prior to sample analysis.

11.3.3. A valid ICAL must exist prior to sample analysis.

11.3.4. Instrument performance must be checked prior to sample analysis.

11.3.5. Check Appendix 1 for acceptance criteria.

11.3.6. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.

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- 11.3.7. Surrogate standard shall be added to all samples, including method blank LCS/LCD and MS/MSD. Check PSR for QC Control Limits.
 - 11.3.8. Perform QC check prior to utilizing the surrogate and LCS/MS spike standards by analyzing the prepared standard at the spiking level. Results should be within $\pm 20\%$ of the expected value.

12.0 CORRECTIVE ACTIONS

- 12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures in Appendix 1. Document out-of-control event and corrective action in the analytical logbook. If the problem persists, consult the supervisor.
- 12.2. If PEM failed to meet the DDT and Endrin breakdown acceptance criteria, consider the following suggestions to correct the problem:
 - 12.2.1. Deactivate or replace the injection liner.
 - 12.2.2. Check that the injector nut is leak free.
 - 12.2.3. If problem persist inform the Supervisor.
- 12.3. If Initial calibration is non-compliant, consider the following suggestions to correct the problem:
 - 12.3.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.
 - 12.3.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and reanalyze.
 - 12.3.3. If the highest ICAL point appears to be saturated, drop the highest point.
 - 12.3.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.
Note: The lowest calibration point identifies the reporting limit (RL). Therefore, check that the RL is in conformance to the current projects where the ICAL will be used.
 - 12.3.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:
 - 12.3.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.
 - 12.3.5.2. Check the gas flow
 - 12.3.5.3. Prepare a fresh standard and repeat calibration
 - 12.3.6. If the problem persists, inform the supervisor.
- 12.4. If the ICV is non-compliant, consider the following suggestions to correct the problem:
 - 12.4.1. Re-analyze ICV (to rule out poor injection)
 - 12.4.2. If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error
 - 12.4.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration
 - 12.4.4. If the problem persists, inform the supervisor
- 12.5. If the instrument blank is non-compliant, consider the following suggestions to correct problem:
 - 12.5.1. Rule out instrument contamination by performing the instrument daily maintenance, such as

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- changing septum, cleaning liner, cleaning or using new auto sampler syringe.
- 12.5.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.
- 12.5.3. Rule out preparation contamination by preparing a new instrument blank
- 12.5.4. If the problem persists, inform the supervisor.
- 12.6. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:
- 12.6.1. Change the liner
- 12.6.2. Clean injection port
- 12.6.3. Prepare new standard
- 12.6.4. Cut or replace column
- 12.6.5. Clean the detector
- 12.6.6. Rule out leaks by checking all connections
- 12.6.7. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL
- 12.7. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
- 12.7.1. Rule out instrument contamination by checking instrument blank
- 12.7.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01
- 12.7.3. Rule out glassware contamination used for extraction as described in EMAX-QC07
- 12.7.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents
- 12.7.5. If the problem persists, inform the supervisor
- 12.8. If LCS is non-compliant, perform the following suggestions to correct the problem:
- 12.8.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level
- 12.8.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.
- 12.8.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.
- 12.9. Execute a Non-Conformance Report (NCR) when the following circumstances occur:
- 12.9.1. If corrective action needs the function of other department; e.g., if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.
- 12.9.2. If corrective action needs the assistance of the project manager; e.g. If the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.
- 12.10. For other problems encountered, inform the supervisor immediately for further instructions.

13.0 POLLUTION PREVENTION

- 13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.

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- 13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 WASTE MANAGEMENT

- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES**15.1. Definition of Terms**

- 15.1.1. Analyte – The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together.
- 15.1.2. Batch – A group of samples that are prepared and/or analyzed at the same time using the same lot of reagents.
- 15.1.2.1. **Preparation batch** is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate.
- 15.1.2.2. **Analytical batch** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.3. Calibration – A determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.4. Corrective Action - Action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
- 15.1.5. Instrument Method – A file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.6. Instrument Blank – A target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.7. Lab Control Sample (LCS) – A target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.8. Matrix – A component or form of a sample.
- 15.1.9. Matrix Spike (MS) – A sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.10. Matrix Spike Duplicate (MSD) – A replicate of MS analyzed to monitor precision or recovery.
- 15.1.11. Method Blank – A target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.12. Method Detection Limit - The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

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- 15.1.13. Nonconformance - An indication or judgment that a product or service has not met the requirements of the relevant specifications, contract or regulation; also the state of failing to meet the requirements.
- 15.1.14. Raw Data - Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.
- 15.1.15. Sample – A specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.16. Sample Duplicate – A replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.17. Sub-sample – An aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.
- 15.1.18. Surrogate - A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.
- 15.2. **Application of EMAX QC Procedures**
- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing pesticides analysis by GC. In instances where there is a project or program QAPP, the requirements given in the project shall take precedence over this SOP.
- 15.3. **Air Force Center for Environmental Excellence (AFCEE) projects**
- 15.3.1. When samples from AFCEE sponsored projects are analyzed for pesticides by GC, shall follow project specific requirements as specified by the QAPP. In the absence of a QAPP, the calibration, QC, corrective action, and data flagging requirements the AFCEE QAPP latest version shall be applied.
- 15.4. **U.S. Army Corps of Engineers (USACE) Projects**
- 15.4.1. When samples from USACE sponsored projects are analyzed for pesticides by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, DoD QSM latest version shall be applied.
- 15.5. **Naval Facilities Engineering Service Center (NFESC) Projects**
- 15.5.1. When samples from NFESC sponsored projects are analyzed for pesticides by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, DoD QSM latest version shall be applied.
- 15.6. **Department of Energy (DOE) Projects**
- 15.6.1. When samples from DOE sponsored projects are analyzed for pesticides by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, DOE QSAS shall be applied.

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16.0 REFERENCES

- 16.1. Method 8081A, Test Methods for Evaluating Solid Wastes, USEPA SW846
 16.2. EMAX Quality Systems Manual, EMAX-QS00, latest revision.

17.0 FIGURES, TABLES AND APPENDICES**17.1. Figures**

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17.3. Appendices

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17.4. Forms

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Figure 1 - Peak Evaluation Technique

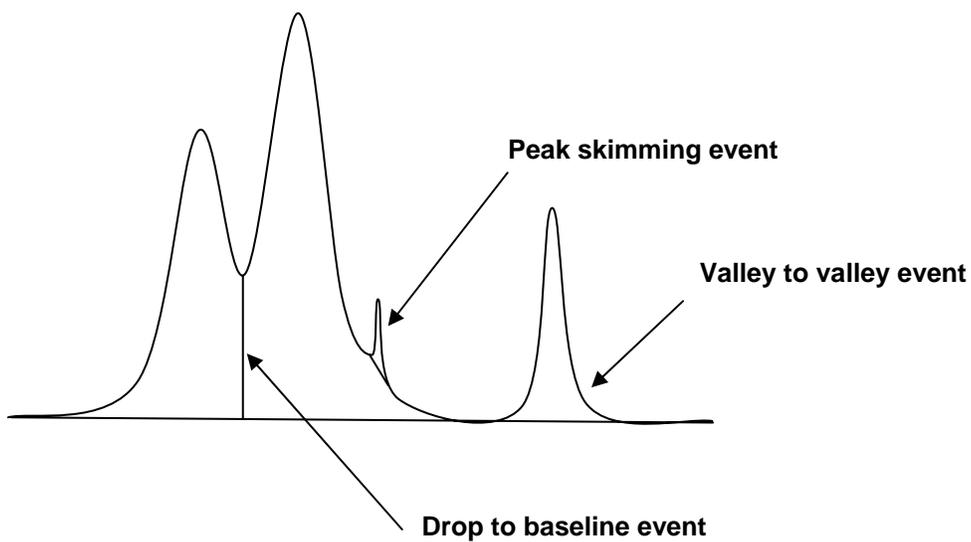


FIGURE 2
 TYPICAL CHROMATOGRAM

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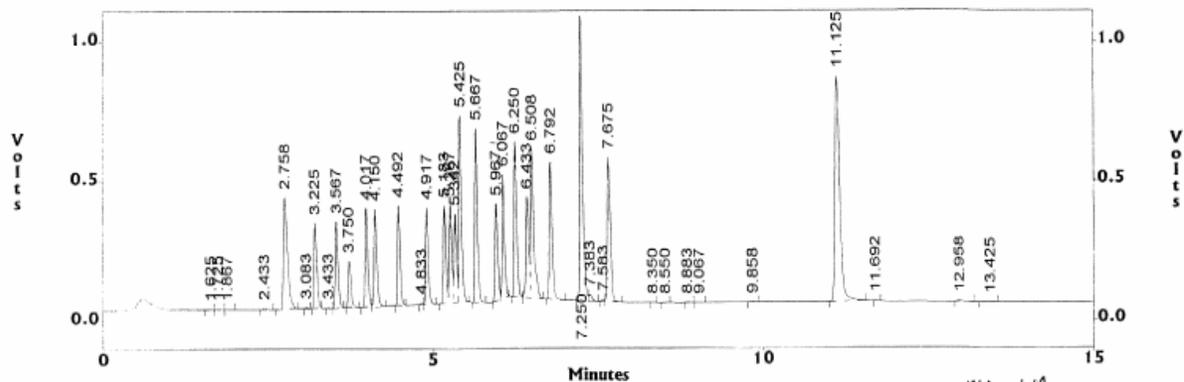
EPA 8081 by GC/ECD
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\kg10\kg10.008
 Method : c:\ezchrom\methods\cp71g10.met
 Sample ID : CP71G1006
 Acquired : Jul 10, 2007 15:12:20
 Printed : Jul 10, 2007 17:56:31
 User : Jay

Channel A Results

| # | Peak Name | Ret.Time(min) | Area | Ave. CF | ESTD Conc. (ppb) |
|----|--------------------|---------------|---------|---------|------------------|
| 5 | TCX | 2.758 | 1695669 | 11134.1 | 160.0 |
| 7 | alpha-BHC | 3.225 | 938046 | 8940.8 | 80.0 |
| 9 | gamma-BHC | 3.567 | 908695 | 9093.8 | 80.0 |
| 10 | beta-BHC | 3.750 | 493562 | 6074.4 | 80.0 |
| 11 | delta-BHC | 4.017 | 970629 | 9538.4 | 80.0 |
| 12 | Heptachlor | 4.150 | 976549 | 12320.7 | 80.0 |
| 13 | Aldrin | 4.492 | 908548 | 10515.2 | 80.0 |
| 15 | Heptachlor Epoxide | 4.917 | 903951 | 11872.0 | 80.0 |
| 16 | gamma-Chlordane | 5.183 | 882008 | 11086.4 | 80.0 |
| 17 | alpha-Chlordane | 5.267 | 902001 | 11331.0 | 80.0 |
| 18 | Endosulfan I | 5.342 | 854734 | 10560.1 | 80.0 |
| 19 | DDE | 5.425 | 1793811 | 11531.6 | 160.0 |
| 20 | Dieldrin | 5.667 | 1643132 | 10090.0 | 160.0 |
| 21 | Endrin | 5.967 | 934684 | 5861.1 | 160.0 |
| 22 | DDD | 6.067 | 1135469 | 7034.3 | 160.0 |
| 23 | Endosulfan II | 6.250 | 1573137 | 10459.7 | 160.0 |
| 24 | Endrin Aldehyde | 6.433 | 1075641 | 7545.5 | 160.0 |
| 25 | DDT | 6.508 | 1731934 | 10902.2 | 160.0 |
| 26 | Endosulfan Sulfate | 6.792 | 1510399 | 10022.8 | 160.0 |
| 27 | Methoxychlor | 7.250 | 3338542 | 4999.4 | 800.0 |
| 30 | Endrin Ketone | 7.675 | 1706755 | 10990.4 | 160.0 |
| 36 | DCB | 11.125 | 4086751 | 16156.0 | 320.0 |

c:\ezchrom\chrom\kg10\kg10.008 -- Channel A



EPA 8081 by GC/ECD
 EMAX Analytical Laboratories, Inc.

File : c:\ezchrom\chrom\kg10\kg10.008
 Method : c:\ezchrom\methods\cp71g10.met
 Sample ID : CP71G1006
 Acquired : Jul 10, 2007 15:12:20
 Printed : Jul 10, 2007 17:56:31
 User : Jay

Channel B Results

| # | Peak Name | Ret. Time (min) | Area | Ave. CF | ESTD Conc. (ppb) |
|----|--------------------|-----------------|---------|---------|------------------|
| 2 | TCX | 1.825 | 1890877 | 11389.0 | 160.0 |
| 4 | alpha-BHC | 2.317 | 1025632 | 9834.3 | 80.0 |
| 5 | gamma-BHC | 2.650 | 1027022 | 9908.2 | 80.0 |
| 6 | beta-BHC | 2.933 | 405118 | 5604.4 | 80.0 |
| 7 | Heptachlor | 2.992 | 1352936 | 17002.2 | 80.0 |
| 8 | delta-BHC | 3.175 | 1092235 | 10951.8 | 80.0 |
| 9 | Aldrin | 3.283 | 1076991 | 13448.5 | 80.0 |
| 11 | Heptachlor Epoxide | 3.775 | 1000966 | 12507.4 | 80.0 |
| 12 | gamma-Chlordane | 4.008 | 903880 | 11225.0 | 80.0 |
| 13 | alpha-Chlordane | 4.075 | 1149995 | 13370.1 | 80.0 |
| 14 | Endosulfan I | 4.142 | 1070017 | 12316.5 | 80.0 |
| 15 | DDE | 4.233 | 2042508 | 11601.5 | 160.0 |
| 16 | Dieldrin | 4.392 | 1848422 | 10737.7 | 160.0 |
| 17 | Endrin | 4.667 | 1339927 | 8168.2 | 160.0 |
| 18 | DDD | 4.767 | 1357459 | 7695.7 | 160.0 |
| 19 | Endosulfan II | 4.950 | 1581659 | 10335.8 | 160.0 |
| 20 | DDT | 5.017 | 1969009 | 10852.3 | 160.0 |
| 21 | Endrin Aldehyde | 5.133 | 1148121 | 7773.7 | 160.0 |
| 23 | Endosulfan Sulfate | 5.375 | 1596811 | 10109.0 | 160.0 |
| 24 | Methoxychlor | 5.633 | 3411605 | 5242.9 | 800.0 |
| 25 | Endrin Ketone | 6.008 | 1836158 | 11516.9 | 160.0 |
| 26 | DCB | 7.700 | 4138731 | 16797.7 | 320.0 |

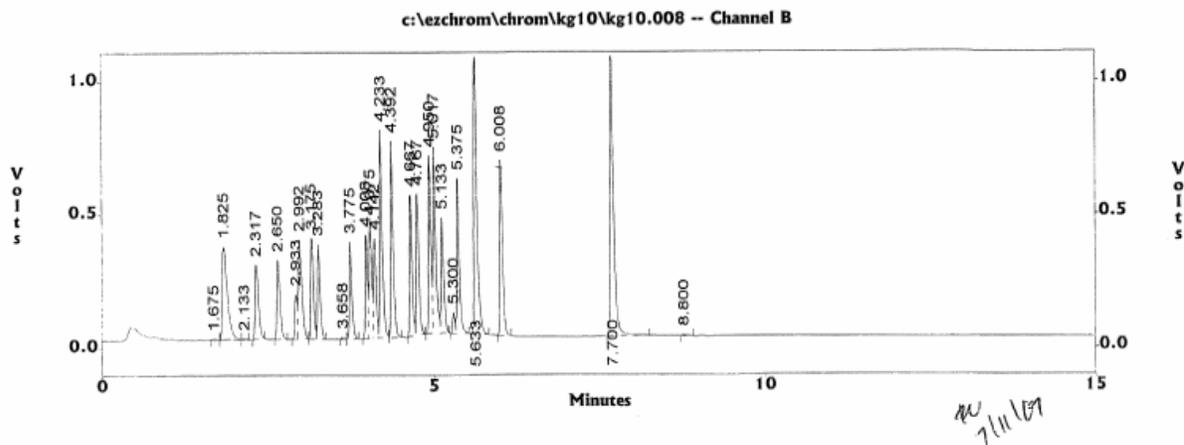


Figure 3 – TYPICAL ICAL SUMMARY
INITIAL CALIBRATION

METHOD 8081A

Lab Name : EMAX Inc
Instrument ID : GCTO71 HP-5890
GC Column : ZB-MULTIRESIDUE-1
Column size ID : .32MMX30M
LFID & Datetime: KG10003A 07/10/07 13:22
LFID & Datetime: KG10004A 07/10/07 13:44
LFID & Datetime: KG10005A 07/10/07 14:06
LFID & Datetime: KG10006A 07/10/07 14:28
LFID & Datetime: KG10007A 07/10/07 14:50
LFID & Datetime: KG10008A 07/10/07 15:12
LFID & Datetime: KG10009A 07/10/07 16:27
CONC UNIT: ppb

| COMPOUND | CONC X | CALIBRATION FACTORS (AREA or HEIGHT)/UNIT | | | | | | | MEAN | %RSD |
|--------------------|--------|---|---------|---------|---------|----------|----------|---------|----------|------|
| | | 1.00X | 2.00X | 4.00X | 8.00X | 16.00X | 32.00X | 64.00X | | |
| alpha-BHC | 2.50 | 7326.80 | 7652.60 | 7970.00 | 8673.95 | 10295.88 | 11725.58 | NA | 8940.8 | 19.3 |
| gamma-BHC | 2.50 | 7554.80 | 7941.20 | 8308.70 | 9004.40 | 10395.17 | 11358.69 | NA | 9093.83 | 16.4 |
| beta-BHC | 2.50 | 5985.60 | 5683.40 | 5823.00 | 5962.05 | 6139.60 | 6169.52 | 6757.91 | 6074.441 | 5.7 |
| delta-BHC | 2.50 | 7894 | 8413 | 8811 | 8851 | 11129 | 12133 | NA | 9538.42 | 17.7 |
| Heptachlor | 2.50 | 12924 | 12687 | 12624 | 11159 | 12315 | 12207 | 12328 | 12320.7 | 4.6 |
| Aldrin | 2.50 | 9285 | 9880 | 10215 | 10289 | 11058 | 11357 | 11523 | 10515.2 | 7.8 |
| Heptachlor Epoxide | 2.50 | 12860 | 12754 | 11912 | 11543 | 11817 | 11299 | 10919 | 11872.1 | 6.1 |
| gamma-Chlordane | 2.50 | 10354 | 11443 | 11415 | 11364 | 11177 | 11025 | 10826 | 11086.4 | 3.5 |
| alpha-Chlordane | 2.50 | 10066 | 11486 | 12253 | 11672 | 11554 | 11275 | 11010 | 11331.0 | 6.0 |
| Endosulfan I | 2.50 | 9693 | 10674 | 10441 | 10830 | 10813 | 10684 | 10786 | 10560.1 | 3.8 |
| DDE | 5.00 | 10703 | 12055 | 12639 | 12202 | 11798 | 11211 | 10113 | 11531.6 | 7.8 |
| Dieldrin | 5.00 | 9593 | 10130 | 10263 | 10307 | 10492 | 10270 | 9574 | 10090.0 | 3.6 |
| Endrin | 5.00 | 5856.60 | 5792.50 | 5753.50 | 5912.38 | 5993.16 | 5841.77 | 5877.81 | 5861.103 | 1.3 |
| DDD | 5.00 | 7525.80 | 6184.00 | 6277.95 | 7452.15 | 7271.23 | 7096.68 | 7432.44 | 7034.321 | 8.1 |
| Endosulfan II | 5.00 | 11266 | 10798 | 10677 | 11165 | 10345 | 9832 | 9136 | 10459.7 | 7.3 |
| Endrin Aldehyde | 5.00 | 8567.60 | 7966.40 | 7451.70 | 7584.13 | 6980.35 | 6722.76 | NA | 7545.49 | 8.8 |
| DDT | 5.00 | 11896 | 11088 | 10574 | 10796 | 10983 | 10825 | 10153 | 10902.2 | 4.9 |
| Endosulfan Sulfate | 5.00 | 10943 | 10889 | 10228 | 9886 | 9937 | 9440 | 8835 | 10022.8 | 7.5 |
| Methoxychlor | 25.00 | 5139.44 | 5737.98 | 5349.78 | 4939.28 | 4656.76 | 4173.18 | NA | 4999.4 | 10.9 |
| Endrin Ketone | 5.00 | 10215 | 11746 | 11463 | 11206 | 11358 | 10667 | 10278 | 10990.4 | 5.5 |
| SURROGATE | X | 1.00X | 2.00X | 4.00X | 8.00X | 16.00X | 32.00X | 64.00X | MEAN | %RSD |
| TCX | 5.00 | 13210 | 10907 | 10961 | 10959 | 10960 | 10598 | 10344 | 11134.1 | 8.5 |
| DCB | 10.00 | 17904 | 18528 | 17762 | 15472 | 14499 | 12771 | NA | 16156.0 | 14.1 |

INITIAL CALIBRATION
METHOD 8081B

Lab Name : EMAX Inc
Instrument ID : GCTO71 HP-5890
GC Column : ZB-MULTIRESIDUE-2
Column size ID : .32MMX30M
LFID & Datetime: KG10003B 07/10/07 13:22
LFID & Datetime: KG10004B 07/10/07 13:44
LFID & Datetime: KG10005B 07/10/07 14:06
LFID & Datetime: KG10006B 07/10/07 14:28
LFID & Datetime: KG10007B 07/10/07 14:50
LFID & Datetime: KG10008B 07/10/07 15:12
LFID & Datetime: KG10009B 07/10/07 16:27
CONC UNIT: ppb

| COMPOUND | CONC X | CALIBRATION FACTORS (AREA or HEIGHT)/UNIT | | | | | | | MEAN | %RSD |
|--------------------|--------|---|---------|---------|---------|---------|---------|---------|----------|------|
| | | 1.00X | 2.00X | 4.00X | 8.00X | 16.00X | 32.00X | 64.00X | | |
| alpha-BHC | 2.50 | 7985 | 9009 | 8901 | 9553 | 10737 | 12820 | NA | 9834.27 | 17.5 |
| gamma-BHC | 2.50 | 7826 | 8338 | 9101 | 9886 | 11461 | 12838 | NA | 9908.21 | 19.4 |
| beta-BHC | 2.50 | 6722.00 | 6032.20 | 5659.60 | 5027.25 | 5075.60 | 5063.98 | 5650.41 | 5604.434 | 11.1 |
| delta-BHC | 2.50 | 10937 | 9553 | 9289 | 10196 | 12082 | 13653 | NA | 10951.8 | 15.2 |
| Heptachlor | 2.50 | 19942 | 16520 | 15736 | 16078 | 16486 | 16912 | 17342 | 17002.2 | 8.2 |
| Aldrin | 2.50 | 15483 | 14228 | 12244 | 11984 | 12514 | 13462 | 14225 | 13448.5 | 9.5 |
| Heptachlor Epoxide | 2.50 | 12366 | 12802 | 12374 | 12444 | 12725 | 12512 | 12328 | 12507.4 | 1.5 |
| gamma-Chlordane | 2.50 | 10662 | 11423 | 11547 | 10889 | 11378 | 11299 | 11378 | 11225.0 | 2.9 |
| alpha-Chlordane | 2.50 | 11330 | 12195 | 13468 | 13716 | 14316 | 14375 | 14190 | 13370.1 | 8.8 |
| Endosulfan I | 2.50 | 10805 | 11613 | 11562 | 12316 | 13179 | 13375 | 13366 | 12316.5 | 8.3 |
| DDE | 5.00 | 9622 | 11211 | 11985 | 12455 | 12243 | 12766 | 10930 | 11601.5 | 9.4 |
| Dieldrin | 5.00 | 9429 | 10319 | 10791 | 11117 | 11689 | 11553 | 10266 | 10737.7 | 7.4 |
| Endrin | 5.00 | 7408.80 | 7985.70 | 8169.60 | 8236.45 | 8643.08 | 8374.54 | 8359.40 | 8168.224 | 4.8 |
| DDD | 5.00 | 6054.80 | 6772.70 | 7498.60 | 7772.70 | 8597.56 | 8484.12 | 8689.29 | 7695.683 | 13.0 |
| Endosulfan II | 5.00 | 9799 | 10804 | 11316 | 11306 | 10517 | 9885 | 8724 | 10335.8 | 9.0 |
| Endrin Aldehyde | 5.00 | 7849.00 | 8251.00 | 8101.85 | 7737.63 | 7526.70 | 7175.76 | NA | 7773.66 | 5.0 |
| DDT | 5.00 | 8729 | 9917 | 10842 | 11205 | 11833 | 12306 | 11135 | 10852.3 | 11.1 |
| Endosulfan Sulfate | 5.00 | 9674 | 10588 | 10337 | 10324 | 10468 | 9980 | 9393 | 10109.0 | 4.4 |
| Methoxychlor | 25.00 | 5497.64 | 5910.22 | 5618.58 | 5167.48 | 4998.89 | 4264.51 | NA | 5242.89 | 11.0 |
| Endrin Ketone | 5.00 | 11023 | 11820 | 12022 | 11965 | 12117 | 11476 | 10195 | 11516.9 | 6.0 |
| SURROGATE | X | 1.00X | 2.00X | 4.00X | 8.00X | 16.00X | 32.00X | 64.00X | MEAN | %RSD |
| TCX | 5.00 | 10109 | 11131 | 11388 | 11533 | 11934 | 11818 | 11810 | 11389.0 | 5.5 |
| DCB | 10.00 | 18871 | 19508 | 18101 | 16638 | 14735 | 12934 | NA | 16797.7 | 15.2 |

Figure 4 – TYPICAL ICAL RTW SUMMARY
INITIAL CALIBRATION RETENTION TIME STUDY
METHOD 8081A

Lab Name : EMAX Inc
Instrument ID : GCTO71 HP-5890
GC Column : ZB-MULTIRESIDUE-1
Column size ID : .32MMX30M
LFID & Datetime: KG10003A 07/10/07 13:22
LFID & Datetime: KG10004A 07/10/07 13:44

LFID & Datetime: KG10005A 07/10/07 14:06
 LFID & Datetime: KG10006A 07/10/07 14:28
 LFID & Datetime: KG10007A 07/10/07 14:50
 LFID & Datetime: KG10008A 07/10/07 15:12
 LFID & Datetime: KG10009A 07/10/07 16:27

| COMPOUND | RT OF STANDARDS (MIN) | | | | | | | MEAN RT | RT WINDOW | | RTWINDOW WIDTH |
|--------------------|-----------------------|--------|--------|--------|--------|--------|--------|---------|-----------|--------|----------------|
| | 1.0X | 2.0X | 4.0X | 8.0X | 16.0X | 32.0X | 64.0X | | FROM | TO | |
| alpha-BHC | 3.225 | 3.225 | 3.225 | 3.225 | 3.225 | 3.225 | 3.233 | 3.226 | 3.188 | 3.264 | 0.038 |
| gamma-BHC | 3.567 | 3.567 | 3.567 | 3.567 | 3.567 | 3.567 | 3.567 | 3.567 | 3.529 | 3.605 | 0.038 |
| beta-BHC | 3.758 | 3.750 | 3.758 | 3.750 | 3.750 | 3.750 | 3.758 | 3.753 | 3.715 | 3.791 | 0.038 |
| delta-BHC | 4.008 | 4.008 | 4.017 | 4.017 | 4.017 | 4.017 | 4.017 | 4.014 | 3.976 | 4.052 | 0.038 |
| Heptachlor | 4.150 | 4.150 | 4.150 | 4.150 | 4.150 | 4.150 | 4.150 | 4.150 | 4.112 | 4.188 | 0.038 |
| Aldrin | 4.492 | 4.492 | 4.492 | 4.492 | 4.492 | 4.492 | 4.500 | 4.493 | 4.455 | 4.531 | 0.038 |
| Heptachlor Epoxide | 4.917 | 4.917 | 4.917 | 4.917 | 4.917 | 4.917 | 4.917 | 4.917 | 4.867 | 4.967 | 0.050 |
| gamma-Chlordane | 5.192 | 5.183 | 5.192 | 5.183 | 5.183 | 5.183 | 5.192 | 5.187 | 5.137 | 5.237 | 0.050 |
| alpha-Chlordane | 5.275 | 5.275 | 5.275 | 5.275 | 5.275 | 5.275 | 5.267 | 5.275 | 5.274 | 5.212 | 0.062 |
| Endosulfan I | 5.350 | 5.350 | 5.350 | 5.350 | 5.342 | 5.342 | 5.350 | 5.348 | 5.285 | 5.411 | 0.063 |
| DDE | 5.425 | 5.425 | 5.425 | 5.425 | 5.425 | 5.425 | 5.433 | 5.426 | 5.363 | 5.489 | 0.063 |
| Dieldrin | 5.667 | 5.667 | 5.667 | 5.667 | 5.667 | 5.667 | 5.675 | 5.668 | 5.593 | 5.743 | 0.075 |
| Endrin | 5.967 | 5.967 | 5.967 | 5.967 | 5.967 | 5.967 | 5.967 | 5.967 | 5.880 | 6.054 | 0.087 |
| DDD | 6.067 | 6.067 | 6.067 | 6.067 | 6.067 | 6.067 | 6.075 | 6.068 | 5.969 | 6.167 | 0.099 |
| Endosulfan II | 6.258 | 6.258 | 6.258 | 6.250 | 6.250 | 6.250 | 6.258 | 6.255 | 6.166 | 6.344 | 0.089 |
| Endrin Aldehyde | 6.442 | 6.442 | 6.442 | 6.442 | 6.442 | 6.433 | 6.442 | 6.441 | 6.339 | 6.543 | 0.102 |
| DDT | 6.517 | 6.508 | 6.517 | 6.508 | 6.508 | 6.508 | 6.517 | 6.512 | 6.411 | 6.613 | 0.101 |
| Endosulfan Sulfate | 6.792 | 6.792 | 6.792 | 6.792 | 6.792 | 6.792 | 6.800 | 6.793 | 6.668 | 6.918 | 0.125 |
| Methoxychlor | 7.258 | 7.258 | 7.258 | 7.258 | 7.250 | 7.250 | 7.275 | 7.258 | 7.121 | 7.395 | 0.137 |
| Endrin Ketone | 7.683 | 7.675 | 7.683 | 7.675 | 7.675 | 7.675 | 7.683 | 7.678 | 7.540 | 7.816 | 0.138 |
| SURROGATE | 1.0X | 2.0X | 4.0X | 8.0X | 16.0X | 32.0X | 64.0X | RT | FROM | TO | WIDTH |
| TCX | 2.758 | 2.758 | 2.758 | 2.758 | 2.758 | 2.758 | 2.758 | 2.758 | 2.720 | 2.796 | 0.038 |
| DCB | 11.133 | 11.133 | 11.133 | 11.125 | 11.125 | 11.125 | 11.158 | 11.133 | 11.058 | 11.208 | 0.075 |

INITIAL CALIBRATION RETENTION TIME STUDY
METHOD 8081A

Lab Name : EMAX Inc
 Instrument ID : GCTO71 HP-5890
 GC Column : ZB-MULTIRESIDUE-2
 Column size ID : .32MMX30M
 LFID & Datetime: KG10003B 07/10/07 13:22
 LFID & Datetime: KG10004B 07/10/07 13:44
 LFID & Datetime: KG10005B 07/10/07 14:06
 LFID & Datetime: KG10006B 07/10/07 14:28
 LFID & Datetime: KG10007B 07/10/07 14:50
 LFID & Datetime: KG10008B 07/10/07 15:12
 LFID & Datetime: KG10009B 07/10/07 16:27

| COMPOUND | RT OF STANDARDS (MIN) | | | | | | | MEAN RT | RT WINDOW | | RTWINDOW WIDTH |
|--------------------|-----------------------|-------|-------|-------|-------|-------|-------|---------|-----------|-------|----------------|
| | 1.0X | 2.0X | 4.0X | 8.0X | 16.0X | 32.0X | 64.0X | | FROM | TO | |
| alpha-BHC | 2.317 | 2.317 | 2.325 | 2.317 | 2.317 | 2.317 | 2.317 | 2.318 | 2.292 | 2.344 | 0.026 |
| gamma-BHC | 2.650 | 2.650 | 2.658 | 2.650 | 2.650 | 2.650 | 2.650 | 2.651 | 2.613 | 2.689 | 0.038 |
| beta-BHC | 2.933 | 2.942 | 2.942 | 2.933 | 2.933 | 2.933 | 2.933 | 2.936 | 2.898 | 2.974 | 0.038 |
| delta-BHC | 3.175 | 3.175 | 3.183 | 3.175 | 3.175 | 3.175 | 3.175 | 3.176 | 3.138 | 3.214 | 0.038 |
| Heptachlor | 3.000 | 3.000 | 3.000 | 3.000 | 2.992 | 2.992 | 2.992 | 2.997 | 2.969 | 3.025 | 0.028 |
| Aldrin | 3.283 | 3.283 | 3.283 | 3.283 | 3.283 | 3.283 | 3.283 | 3.283 | 3.257 | 3.309 | 0.026 |
| Heptachlor Epoxide | 3.775 | 3.775 | 3.775 | 3.775 | 3.775 | 3.775 | 3.767 | 3.774 | 3.736 | 3.812 | 0.038 |
| gamma-Chlordane | 4.017 | 4.017 | 4.017 | 4.008 | 4.008 | 4.008 | 4.008 | 4.012 | 3.974 | 4.050 | 0.038 |
| alpha-Chlordane | 4.075 | 4.075 | 4.075 | 4.075 | 4.075 | 4.075 | 4.067 | 4.074 | 4.036 | 4.112 | 0.038 |
| Endosulfan I | 4.142 | 4.142 | 4.142 | 4.142 | 4.142 | 4.142 | 4.133 | 4.141 | 4.103 | 4.179 | 0.038 |
| DDE | 4.233 | 4.233 | 4.233 | 4.233 | 4.233 | 4.233 | 4.233 | 4.233 | 4.183 | 4.283 | 0.050 |
| Dieldrin | 4.392 | 4.392 | 4.392 | 4.392 | 4.392 | 4.392 | 4.392 | 4.392 | 4.342 | 4.442 | 0.050 |
| Endrin | 4.667 | 4.667 | 4.667 | 4.667 | 4.667 | 4.667 | 4.658 | 4.666 | 4.604 | 4.728 | 0.062 |
| DDD | 4.767 | 4.767 | 4.767 | 4.767 | 4.767 | 4.767 | 4.758 | 4.766 | 4.691 | 4.841 | 0.075 |
| Endosulfan II | 4.950 | 4.950 | 4.950 | 4.950 | 4.950 | 4.950 | 4.950 | 4.950 | 4.887 | 5.013 | 0.063 |
| Endrin Aldehyde | 5.142 | 5.133 | 5.142 | 5.133 | 5.133 | 5.133 | 5.133 | 5.136 | 5.049 | 5.223 | 0.087 |
| DDT | 5.017 | 5.017 | 5.025 | 5.017 | 5.017 | 5.017 | 5.017 | 5.018 | 4.943 | 5.093 | 0.075 |
| Endosulfan Sulfate | 5.375 | 5.375 | 5.375 | 5.375 | 5.375 | 5.375 | 5.375 | 5.375 | 5.286 | 5.464 | 0.089 |
| Methoxychlor | 5.642 | 5.633 | 5.633 | 5.633 | 5.633 | 5.633 | 5.650 | 5.637 | 5.524 | 5.750 | 0.113 |
| Endrin Ketone | 6.017 | 6.017 | 6.017 | 6.008 | 6.008 | 6.008 | 6.008 | 6.012 | 5.899 | 6.125 | 0.113 |
| SURROGATE | 1.0X | 2.0X | 4.0X | 8.0X | 16.0X | 32.0X | 64.0X | RT | FROM | TO | WIDTH |
| TCX | 1.825 | 1.825 | 1.833 | 1.825 | 1.825 | 1.825 | 1.825 | 1.826 | 1.800 | 1.852 | 0.026 |
| DCB | 7.708 | 7.700 | 7.708 | 7.700 | 7.700 | 7.700 | 7.717 | 7.705 | 7.667 | 7.743 | 0.038 |

Figure 5 – TYPICAL PEM BREAKDOWN CALCULATION SUMMARY

PEM PEST BREAKDOWN CALCULATION
 METHOD 8081A

Lab Name : EMAX
 Instrument ID : GCT071 HP-5890
 GC Column : ZB-MULTIRESIDUE-1 ZB-MULTIRESIDUE-2
 Column size ID : .32MMX30M .32MMX30M
 PEM LFID & Datetime : KG10002A KG10002B 07/10/07 13:01

Base on AREA

| LFID | DDD | AREA | | | TOTAL | % Breakdown | | | QL | QCLIMIT |
|----------|----------|-----------------|---------------|----------|-----------------|---------------|-------|-------|---------|---------|
| | | DDE | DDT | TOTAL | | DDD | DDE | TOTAL | | |
| KG10002A | 0.0 | 36005.0 | 900034.0 | 936039.0 | 0.00 | 3.85 | 3.85 | | 15 | |
| KG10002B | 0.0 | 5468.0 | 986678.0 | 992146.0 | 0.00 | 0.55 | 0.55 | | 15 | |
| LFID | ENDRIN | ENDRIN ALDEHYDE | ENDRIN KETONE | TOTAL | ENDRIN ALDEHYDE | ENDRIN KETONE | TOTAL | QL | QCLIMIT | |
| KG10002A | 610357.0 | 16234.0 | 47818.0 | 674409.0 | 2.41 | 7.09 | 9.50 | | 15 | |
| KG10002B | 897002.0 | 31273.0 | 55696.0 | 983971.0 | 3.18 | 5.66 | 8.84 | | 15 | |

Figure 6 – TYPICAL SAMPLE RESULT REPORT
 SW3550B/8081A
 PESTICIDES

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=====
Client      : XYZ, INC.                      Date Collected: 06/19/07
Project    : CLEAN LAND PROJECT             Date Received: 06/20/07
Batch No.  : 07F235                          Date Extracted: 06/22/07 11:30
Sample ID  : CDDSB1                          Date Analyzed: 06/27/07 12:47
Lab Samp ID: F235-01                          Dilution Factor: 1
Lab File ID: SF27009A                         Matrix          : SOIL
Ext Btch ID: CPF036S                          % Moisture     : 15.2
Calib. Ref.: SF27003A                         Instrument ID   : GCT008
=====
  
```

| PARAMETERS | RESULTS (ug/kg) | RL (ug/kg) | MDL (ug/kg) |
|----------------------|--------------------|---------------|----------------|
| ALPHA-BHC | (ND) ND | 2.4 | 0.71 0.71 |
| GAMMA-BHC (LINDANE) | (ND) ND | 2.4 | 0.71 0.71 |
| BETA-BHC | (ND) ND | 2.4 | 0.71 0.71 |
| HEPTACHLOR | 1.2J (1.7J) | 2.4 | 0.71 0.71 |
| DELTA-BHC | (ND) ND | 2.4 | 0.71 0.71 |
| ALDRIN | (ND) 1.0J | 2.4 | 0.71 0.71 |
| HEPTACHLOR EPOXIDE | 1.5J (ND) | 2.4 | 0.71 0.71 |
| GAMMA-CHLORDANE | (ND) 6.2 | 2.4 | 0.71 0.71 |
| ALPHA-CHLORDANE | (ND) ND | 2.4 | 0.71 0.71 |
| ENDOSULFAN I | (ND) ND | 2.4 | 0.71 0.71 |
| 4,4'-DDE | (ND) ND | 4.7 | 1.4 1.4 |
| DIELDRIN | (ND) ND | 4.7 | 1.4 1.4 |
| ENDRIN | (ND) ND | 4.7 | 1.4 1.4 |
| 4,4'-DDD | (ND) ND | 4.7 | 1.4 1.4 |
| ENDOSULFAN II | (ND) ND | 4.7 | 1.4 1.4 |
| 4,4'-DDT | (ND) ND | 4.7 | 1.4 1.4 |
| ENDRIN ALDEHYDE | (ND) ND | 4.7 | 1.4 1.4 |
| ENDOSULFAN SULFATE | (ND) 4.7 | 4.7 | 1.8 1.8 |
| ENDRIN KETONE | (ND) ND | 4.7 | 1.4 1.4 |
| METHOXYCHLOR | (ND) ND | 24 | 4.7 4.7 |
| TOXAPHENE | (ND) ND | 59 | 12 12 |
| ----- | | | |
| SURROGATE PARAMETERS | % RECOVERY | QC LIMIT | |
| TETRACHLORO-M-XYLENE | 86 (109) | 10-160 | |
| DECACHLOROBIPHENYL | (143) 127 | 30-150 | |

Figure 7- TYPICAL LCS REPORT SUMMARY
EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
BATCH NO.: 07F235
METHOD: SW3520C/8081A

MATRIX: WATER % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: MBLK1W
LAB SAMP ID: CPF033WB CPF033WL CPF033WC
LAB FILE ID: SF29047A SF29048A SF29049A
DATE EXTRACTED: 06/21/0712:00 06/21/0712:00 06/21/0712:00 DATE COLLECTED: NA
DATE ANALYZED: 06/30/0701:11 06/30/0701:27 06/30/0701:42 DATE RECEIVED: 06/21/07
PREP. BATCH: CPF033W CPF033W CPF033W
CALIB. REF: SF29043A SF29043A SF29043A

ACCESSION:

| PARAMETER | BLNK RSLT (ug/L) | SPIKE AMT (ug/L) | BS RSLT (ug/L) | BS % REC | SPIKE AMT (ug/L) | BSD RSLT (ug/L) | BSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|---------------------|---------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|------------|-----------------|----------------|
| alpha-BHC | (ND) | ND | 0.200 (0.253) | 0.251 (126) | 125 | 0.200 (0.227) | 0.231 (114) | 116 (11) | 8 | 30-150 |
| gamma-BHC (Lindane) | (ND) | ND | 0.200 (0.238) | 0.232 (119) | 116 | 0.200 (0.222) | 0.216 (111) | 108 (7) | 7 | 40-130 |
| beta-BHC | (ND) | ND | 0.200 (0.233) | 0.214 (116) | 107 | 0.200 (0.218) | 0.199 (109) | 100 (7) | 7 | 60-130 |
| Heptachlor | (ND) | ND | 0.200 (0.225) | 0.214 (112) | 107 | 0.200 (0.208) | 0.198 (104) | 99 (8) | 8 | 30-140 |
| delta-BHC | (ND) | ND | 0.200 (0.217) | 0.209 (108) | 104 | 0.200 (0.203) | 0.194 (101) | 97 (7) | 7 | 30-150 |
| Aldrin | (ND) | ND | 0.200 (0.220) | 0.216 (110) | 108 | 0.200 (0.206) | 0.202 (103) | 101 (7) | 7 | 40-130 |
| Heptachlor Epoxide | (ND) | ND | 0.200 (0.211) | 0.209 (105) | 104 | 0.200 (0.199) | 0.197 (100) | 98 (6) | 6 | 50-140 |
| gamma-Chlordane | (ND) | ND | 0.200 (0.217) | 0.215 (108) | 108 | 0.200 (0.205) | 0.214 (102) | 107 (6) | 0 | 60-140 |
| alpha-Chlordane | (ND) | ND | 0.200 (0.213) | 0.212 (106) | 106 | 0.200 (0.201) | 0.200 (100) | 100 (6) | 6 | 50-140 |
| Endosulfan I | (ND) | ND | 0.200 (0.219) | 0.215 (110) | 108 | 0.200 (0.207) | 0.204 (104) | 102 (6) | 5 | 60-140 |
| 4,4'-DDE | (ND) | ND | 0.400 (0.421) | 0.418 (105) | 104 | 0.400 (0.399) | 0.395 (100) | 99 (5) | 6 | 50-140 |
| Dieldrin | (ND) | ND | 0.400 (0.460) | 0.453 (115) | 113 | 0.400 (0.437) | 0.430 (109) | 108 (5) | 5 | 60-140 |
| Endrin | (ND) | ND | 0.400 (0.448) | 0.453 (112) | 113 | 0.400 (0.365) | 0.372 (91) | 93 (20) | 20 | 50-140 |
| 4,4'-DDD | (ND) | ND | 0.400 (0.473) | 0.471 (118) | 118 | 0.400 (0.448) | 0.447 (112) | 112 (5) | 5 | 50-160 |
| Endosulfan II | (ND) | ND | 0.400 (0.437) | 0.425 (109) | 106 | 0.400 (0.409) | 0.407 (102) | 102 (7) | 4 | 60-150 |
| 4,4'-DDT | (ND) | ND | 0.400 (0.410) | 0.429 (107) | 102 | 0.400 (0.388) | 0.407 (97) | 102 (6) | 5 | 60-140 |
| Endrin aldehyde | (ND) | ND | 0.400 (0.528) | 0.459 (132) | 115 | 0.400 (0.544) | 0.459 (136) | 115 (3) | 0 | 60-160 |
| Endosulfan Sulfate | (ND) | ND | 0.400 (0.421) | 0.425 (105) | 106 | 0.400 (0.406) | 0.398 (101) | 100 (4) | 7 | 70-140 |
| Endrin Ketone | (ND) | ND | 0.400 (0.520) | 0.459 (130) | 115 | 0.400 (0.485) | 0.466 (121) | 116 (7) | 2 | 30-150 |
| Methoxychlor | (ND) | ND | 2.00 (2.22) | 2.37 (111) | 118 | 2.00 (2.15) | 2.21 (108) | 110 (3) | 7 | 70-150 |

| SURROGATE PARAMETER | SPIKE AMT (ug/L) | BS RSLT (ug/L) | BS % REC | SPIKE AMT (ug/L) | BSD RSLT (ug/L) | BSD % REC | QC LIMIT (%) |
|----------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|-----------------|
| Tetrachloro-m-xylene | 0.400 | (0.392) 0.372 | (98) 93 | 0.400 | (0.350) 0.341 | (88) 85 | 30-130 |
| Decachlorobiphenyl | 0.800 | (0.827) 0.805 | (103) 101 | 0.800 | (0.799) 0.777 | (100) 97 | 40-150 |

Figure 8- TYPICAL MS/MSD REPORT SUMMARY
EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, ICN.
PROJECT: CLEAN LAND PROJECT

BATCH NO.: 07F235
METHOD: SW3550B/8081A

MATRIX: SOIL % MOISTURE: 11.5
DILUTION FACTOR: 1 1 1
SAMPLE ID: CDDSB9
LAB SAMP ID: F235-09 F235-09M F235-09S
LAB FILE ID: SF27017A SF27018A SF27019A
DATE EXTRACTED: 06/22/0711:30 06/22/0711:30 06/22/0711:30 DATE COLLECTED: 06/19/07
DATE ANALYZED: 06/27/0714:50 06/27/0715:06 06/27/0715:21 DATE RECEIVED: 06/20/07
PREP. BATCH: CPF036S CPF036S CPF036S
CALIB. REF: SF27003A SF27003A SF27003A
ACCESSION:

| PARAMETER | SMP L | RSLT | SPIKE AMT | MS RSLT | MS | SPIKE AMT | MSD RSLT | MSD | RPD | QC LIMIT | MAX RPD |
|---------------------|-----------|---------|-----------|-------------|-------------|-----------|-------------|-------------|-----------|----------|---------|
| | (ug/kg) | (ug/kg) | (ug/kg) | (ug/kg) | % REC | (ug/kg) | (ug/kg) | % REC | (%) | (%) | (%) |
| alpha-BHC | (ND) | ND | 7.54 | 10.0 (10.2) | 133 (135) | 7.54 | 9.17 (9.56) | 122 (127) | 9 (6) | 30-150 | 50 |
| gamma-BHC (Lindane) | (ND) | ND | 7.54 | (9.61) 10.3 | (127) 137 | 7.54 | (11.3) 11.8 | (150) 157* | (16) 14 | 20-150 | 50 |
| beta-BHC | (ND) | ND | 7.54 | 10.9 (10.2) | 145 (135) | 7.54 | 11.8 (11.0) | 147* (146*) | 8 (8) | 50-140 | 50 |
| Heptachlor | (ND) | ND | 7.54 | (10.0) 8.99 | (133) 119 | 7.54 | (9.41) 8.51 | (125) 113 | (6) 5 | 20-140 | 50 |
| delta-BHC | (ND) | ND | 7.54 | 7.97 (9.43) | 106 (125) | 7.54 | 7.63 (9.67) | 101 (128) | 4 (3) | 30-150 | 50 |
| Aldrin | (ND) | ND | 7.54 | 7.85 (9.12) | 104 (121) | 7.54 | 7.76 (8.45) | 103 (112) | 1 (8) | 20-160 | 50 |
| Heptachlor Epoxide | (ND) | ND | 7.54 | (8.96) 8.10 | (119) 107 | 7.54 | (7.86) 8.13 | (104) 108 | (13) 0 | 40-140 | 50 |
| gamma-Chlordane | (ND) | ND | 7.54 | (9.45) 18.3 | (125) 243* | 7.54 | (8.14) 38.1 | (108) 505* | (15) 70* | 60-160 | 50 |
| alpha-Chlordane | (ND) | ND | 7.54 | (8.74) 8.13 | (116) 108 | 7.54 | (7.88) 7.84 | (105) 104 | (10) 4 | 50-140 | 50 |
| Endosulfan I | (ND) | ND | 7.54 | 7.69 (8.11) | 102 (108) | 7.54 | 7.31 (7.63) | 97 (101) | 5 (6) | 50-160 | 50 |
| 4,4'-DDE | (ND) | ND | 15.1 | (27.9) 26.6 | (185*) 177* | 15.1 | (17.6) 16.5 | (117) 110 | (45) 47 | 50-150 | 50 |
| Dieldrin | (ND) | ND | 15.1 | 17.7 (20.0) | 118 (132) | 15.1 | 16.9 (18.2) | 112 (121) | 5 (9) | 10-160 | 50 |
| Endrin | (ND) | ND | 15.1 | 19.4 (20.0) | 128 (133) | 15.1 | 18.6 (19.2) | 123 (127) | 4 (4) | 20-160 | 50 |
| 4,4'-DDD | (ND) | ND | 15.1 | 24.2 (23.9) | 161* (158) | 15.1 | 20.8 (19.7) | 138 (130) | 15 (19) | 50-160 | 50 |
| Endosulfan II | (ND) | ND | 15.1 | 15.8 (17.5) | 105 (116) | 15.1 | 14.7 (16.4) | 98 (109) | 7 (6) | 40-160 | 50 |
| 4,4'-DDT | 5.7 (6.8) | | 15.1 | 66.8 (77.5) | 406* (469*) | 15.1 | 27.7 (28.0) | 146 (141) | 83* (94*) | 30-160 | 50 |
| Endrin aldehyde | (ND) | ND | 15.1 | (20.4) 13.8 | (135) 92 | 15.1 | (19.0) 12.7 | (126) 84 | (7) 8 | 50-140 | 50 |
| Endosulfan Sulfate | (ND) | ND | 15.1 | (18.4) 17.2 | (122) 114 | 15.1 | (17.3) 15.8 | (114) 105 | (6) 8 | 40-160 | 50 |
| Endrin Ketone | (ND) | ND | 15.1 | (20.8) 18.0 | (138) 120 | 15.1 | (18.9) 17.9 | (125) 119 | (10) 1 | 50-160 | 50 |
| Methoxychlor | (ND) | ND | 75.3 | (148) 147 | (196*) 195* | 75.3 | (141) 138 | (187*) 183* | (5) 6 | 60-160 | 50 |

| SURROGATE PARAMETER | SPIKE AMT | MS RSLT | MS | SPIKE AMT | MSD RSLT | MSD | QC LIMIT |
|----------------------|-----------|-------------|-----------|-----------|-------------|-----------|----------|
| | (ug/kg) | (ug/kg) | % REC | (ug/kg) | (ug/kg) | % REC | (%) |
| Tetrachloro-m-xylene | 15.1 | (16.6) 13.6 | (110) 90 | 15.1 | (15.5) 13.0 | (103) 87 | 10-160 |
| Decachlorobiphenyl | 30.2 | (34.0) 32.2 | (113) 107 | 30.2 | (32.8) 30.6 | (109) 101 | 30-150 |

* : Out side of QC Limit.

Figure 9 – TYPICAL CASE NARRATIVE

CASE NARRATIVE

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
SDG: 07F235

**Method SW8081A
Pesticides Organochlorine**

A total of 1 water and 11 soil samples were received on 6/20/2007 for Pesticides Organochlorine Method SW8081A in accordance with USEPA SW846, 3rd Edition.

HOLDING TIME

Samples were analyzed within holding time as prescribed by the project.

INITIAL CALIBRATION

Multi-calibration points were analyzed to establish ICAL. All project calibrations requirements were satisfied. Refer to Initial Calibration summary form.

Initial Calibration was verified using a secondary source. All analytes met the project requirement. Refer to the ICV following the ICAL report.

INSTRUMENT PERFORMANCE EVALUATION CHECK

Instrument blank and PEM standard containing DDT and Endrin were analyzed prior to continuing calibration verification. The Instrument blank was free from contamination and the DDT and Endrin breakdown check were within the acceptance criteria as specified by the method. Refer to PEM Pest Breakdown Calculation Summary Form.

CONTINUING CALIBRATION CHECK

Continuing calibration check was performed at a frequency specified by the project. Results are within the project specified acceptance criteria.

METHOD BLANK

Method Blank was analyzed at a frequency specified by the project. Results are compliant to project requirement.

SURROGATE

Surrogates were added to MB, LCS/LCD, MS/MSD and every sample prior to analysis. All percent recoveries met the project QC limits. Surrogate results are included in the report forms.

LAB CONTROL SAMPLES

LCS/LCD were analyzed at a frequency specified by the project. All percent recoveries met the project QC limits.

MATRIX SPIKE

MS/MSD were analyzed as specified by the project. All QC results were within the project specified QC limits.

SAMPLE ANALYSIS

Samples were analyzed according to the prescribed QC procedures. All analytical specifications were met.

When sample results are confirmed by a second column, the relative percentage difference (RPD) between the two results is calculated. If RPD is less than 40%, and no evidence of chromatographic problems, the higher result is reported. If RPD is greater than 40%, the chromatogram is checked for anomalies and results are selected based on the best professional judgment. Where no evidence of any chromatographic interference is observed, the higher result is reported.

TABLE 1
ICAL Concentration of Individual Analytes

| Target Analyte Mix A | ICAL STANDARD CONCENTRATION (ug/L) | | | | |
|----------------------------------|------------------------------------|-----|----------------|-----|-----|
| | 1 | 2 | 3 ¹ | 4 | 5 |
| alpha-BHC | 5 | 10 | 20 | 40 | 60 |
| DDD | 10 | 20 | 40 | 80 | 120 |
| DDT | 10 | 20 | 40 | 80 | 120 |
| Dieldrin | 10 | 20 | 40 | 80 | 120 |
| Endosulfan I | 5 | 10 | 20 | 40 | 60 |
| Endrin | 10 | 20 | 40 | 80 | 120 |
| gamma-BHC | 5 | 10 | 20 | 40 | 60 |
| Heptachlor | 5 | 10 | 20 | 40 | 60 |
| Methoxychlor | 50 | 100 | 200 | 400 | 600 |
| Tetrachloro-m-xylene (Surrogate) | 5 | 10 | 20 | 40 | 60 |
| Decachlorobiphenyl (Surogate) | 10 | 20 | 40 | 60 | 120 |
| Target Analyte Mix B | | | | | |
| Aldrin | 5 | 10 | 20 | 40 | 60 |
| alpha-Chlordane | 5 | 10 | 20 | 40 | 60 |
| beta-BHC | 5 | 10 | 20 | 40 | 60 |
| DDE | 10 | 20 | 40 | 80 | 120 |
| delta-BHC | 5 | 10 | 20 | 40 | 60 |
| Endosulfan II | 10 | 20 | 40 | 80 | 120 |
| Endosulfan Sulfate | 10 | 20 | 40 | 80 | 120 |
| Endrin Aldehyde | 10 | 20 | 40 | 80 | 120 |
| Endrin Ketone | 10 | 20 | 40 | 80 | 120 |
| gamma-Chlordane | 5 | 10 | 20 | 40 | 60 |
| Heptachlor Epoxide | 5 | 10 | 20 | 40 | 60 |

Table 1A
ICAL Standard Preparation

| Standard # | Compound Name | Stock/Intermediate Soln. Conc. (ug/L) | Preparation | | | Final Conc. (ug/L) |
|------------|---------------|---------------------------------------|--------------|---------|-------------------|--------------------|
| | | | Aliquot (µL) | Solvent | Final Volume (µL) | |
| 1 | Mix A | 80-800 | 25 | Hexane | 400 | 5-50 |
| | Mix B | 80-160 | 25 | Hexane | 400 | 5-10 |
| 2 | Mix A | 80-800 | 50 | Hexane | 400 | 10-100 |
| | Mix B | 80-160 | 50 | Hexane | 400 | 10-20 |
| 3 | Mix A | 80-800 | 100 | Hexane | 400 | 20-200 |
| | Mix B | 80-160 | 100 | Hexane | 400 | 20-40 |
| 4 | Mix A | 80-800 | 200 | Hexane | 400 | 40-400 |
| | Mix B | 80-160 | 200 | Hexane | 400 | 40-80 |
| 5 | Mix A | 80-800 | 300 | Hexane | 400 | 60-600 |
| | Mix B | 80-160 | 300 | Hexane | 400 | 60-120 |

¹ Used as initial calibration verification and continuing calibration.

TABLE 2
Intermediate Primary Standard Preparation

Source:

| AccuStandard Mix A | Stock Std. (µg/L) | Final Conc. (ug/L) | Preparation | |
|----------------------------------|-------------------|--------------------|--|--|
| alpha-BHC | 8,000 | 80 | 100 µL is measured from Accustandard Mix A and diluted to 10 ml of hexane. | |
| gamma-BHC | 8,000 | 80 | | |
| DDD | 16,000 | 160 | | |
| DDT | 16,000 | 160 | | |
| Dieldrin | 16,000 | 160 | | |
| Endosulfan I | 8,000 | 80 | | |
| Endrin | 16,000 | 160 | | |
| Heptachlor | 8,000 | 80 | | |
| Methoxychlor | 80,000 | 800 | | |
| Tetrachloro-m-xylene (Surrogate) | 8,000 | 80 | | |
| Decachlorobiphenyl (Surogate) | 16,000 | 160 | | |
| AccuStandard Mix B | | | | 100 µL is measured from Accustandard Mix B and diluted to 10 ml of hexane. |
| Aldrin | 8,000 | 80 | | |
| alpha-Chlordane | 8,000 | 80 | | |
| beta-BHC | 8,000 | 80 | | |
| DDE | 16,000 | 160 | | |
| delta-BHC | 8,000 | 80 | | |
| Endosulfan II | 16,000 | 160 | | |
| Endosulfan Sulfate | 16,000 | 160 | | |
| Endrin Aldehyde | 16,000 | 160 | | |
| Endrin Ketone | 16,000 | 160 | | |
| gamma-Chlordane | 8,000 | 80 | | |
| Heptachlor Epoxide | 8,000 | 80 | | |
| Tetrachloro-m-xylene (Surrogate) | 8,000 | 80 | | |
| Decachlorobiphenyl (Surogate) | 16,000 | 160 | | |

Table 2A
Intermediate Secondary Standard Preparation

| ULTRASstandard Mix A | Stock Std. (µg/L) | Final Conc. (ug/L) | Preparation | |
|----------------------------------|-------------------|--------------------|--|--|
| alpha-BHC | 5,000 | 80 | 400 µL is measured from ULTRASstandard Mix A and diluted to 25 ml of hexane. | |
| gamma-BHC | 5,000 | 80 | | |
| DDD | 10,000 | 160 | | |
| DDT | 10,000 | 160 | | |
| Dieldrin | 10,000 | 160 | | |
| Endosulfan I | 5,000 | 80 | | |
| Endrin | 10,000 | 160 | | |
| Heptachlor | 5,000 | 80 | | |
| Methoxychlor | 50,000 | 800 | | |
| Tetrachloro-m-xylene (Surrogate) | 5,000 | 80 | | |
| Decachlorobiphenyl (Surrogate) | 10,000 | 160 | | |
| ULTRASstandard Mix B | | | | 400 µL is measured from ULTRASstandard Mix B and diluted to 25 ml of hexane. |
| Aldrin | 5,000 | 80 | | |
| alpha-Chlordane | 5,000 | 80 | | |
| beta-BHC | 5,000 | 80 | | |
| DDE | 10,000 | 160 | | |
| delta-BHC | 5,000 | 80 | | |
| Endosulfan II | 10,000 | 160 | | |
| Endosulfan Sulfate | 10,000 | 160 | | |
| Endrin Aldehyde | 10,000 | 160 | | |
| Endrin Ketone | 10,000 | 160 | | |
| gamma-Chlordane | 5,000 | 80 | | |
| Heptachlor Epoxide | 5,000 | 80 | | |

Note: Table 2 and Table 2A may be interchanged, however the source of LCS shall follow the source of the secondary standard or from a third vendor.

TABLE 3
Check Standard Preparation (DCC)

| Standard # | Compound Name | Intermediate Soln. Conc. (ug/L) | Source | Preparation | | | Final |
|------------|---------------|---------------------------------|--------------|--------------|---------------------|---------------|--------------|
| | | | | Aliquot (ul) | Dil.Soln./ Modifier | Dil.Vol. (ul) | Conc. (ug/L) |
| DCC | Mix A | 80-800 | AccuStandard | 100 | Hexane | 400 | 20-200 |
| | Mix B | 80-160 | AccuStandard | 100 | Hexane | 400 | 20-40 |

TABLE 4
Surrogate Standard Preparation

| Compound Name | Stock/Intermediate Soln. Conc. (ug/L) | Source | Preparation | | | Final |
|---|---------------------------------------|------------------|--------------|---------------------|----------------|--------------|
| | | | Aliquot (ml) | Dil.Soln./ Modifier | Dil. Vol. (ml) | Conc. (ug/L) |
| Pesticide Surrogate Mix (TCX) Dichlorobiphenyl | 200,000 | Ultra Scientific | 0.50 | Hexane | 500 | 200 |

TABLE 5
Spike Standard Preparation

| Compound Name | Stock/Intermediate Soln. Conc. (ug/L) | Source | Preparation | | | Final |
|----------------------------|---------------------------------------|------------------|--------------|---------------------|----------------|--------------|
| | | | Aliquot (ml) | Dil.Soln./ Modifier | Dil. Vol. (ml) | Conc. (ug/L) |
| Pesticide Matrix Spike Mix | 50,000-100,000 | Ultra Scientific | 0.50 | Hexane | 50 | 500-1000 |

TABLE 6
Performance Evaluation Mixture Preparation

| Compound Name | Stock/Intermediate Soln. Conc. (ug/L) | Source | Preparation | | | Final |
|---------------|---------------------------------------|------------------|--------------|---------------------|----------------|-------------|
| | | | Aliquot (ml) | Dil.Soln./ Modifier | Dil. Vol. (ml) | Conc (ug/L) |
| PEM | 1000-25000 | Ultra Scientific | 0.1 | Hexane | 100 | 10-250 |

TABLE 7
CLP Retention Time Windows for Pesticides
(CLP OLM04.2 D-79/PEST)

| Compound | Retention Time Window(minutes) | Compound | Retention Time Window(minutes) |
|--------------------|--------------------------------|----------------------|--------------------------------|
| alpha-BHC | ± 0.05 | Endrin Ketone | ± 0.07 |
| beta-BHC | ± 0.05 | 4,4'-DDD | ± 0.07 |
| gamma-BHC(Lindane) | ± 0.05 | 4,4'-DDE | ± 0.07 |
| delta-BHC | ± 0.05 | 4,4'-DDT | ± 0.07 |
| Heptachlor | ± 0.05 | Endosulfan II | ± 0.07 |
| Aldrin | ± 0.05 | Endosulfan Sulfate | ± 0.07 |
| alpha-Chlordane | ± 0.07 | Methoxychlor | ± 0.07 |
| gamma-Chlordane | ± 0.07 | Aroclors | ± 0.07 |
| Heptachlor Epoxide | ± 0.07 | Toxaphene | ± 0.07 |
| Dieldrin | ± 0.07 | | |
| Endrin | ± 0.07 | Tetrachloro-m-xylene | ± 0.05 |
| Endrin Aldehyde | ± 0.07 | Decachlorobiphenyl | ± 0.10 |

SUMMARY OF IN-HOUSE QUALITY CONTROL PROCEDURES

| QC PROCEDURE | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1 ST Rvw | 2 ND Rvw |
|---|---|---|---|--|---------------------|---------------------|
| 5 point Initial Calibration for analytes | Initially, as needed | 1) RSD for all analytes ≤20% 2) linear – least squares regression $r > 0.995$ 3) non-linear – COD > 0.990 (6 points shall be used for second order, 7 points shall be used for third order) | Correct then problem then repeat initial calibration | | | |
| Second-source calibration verification for all analytes | Once per 5-point initial calibration | All analytes within ±15% of expected value | Repeat injection of ICV. If the problem persists then perform troubleshooting and repeat the ICAL. | | | |
| Initial calibration verification | Daily, before sample analysis | Mean D% of all analytes within ±15% | Correct the problem then repeat initial calibration | | | |
| Calibration verification | Every 12 hours of analysis time and at the end of the analysis sequence | Mean D% of all analytes within ±15% | Correct the problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification | | | |
| Breakdown check (Endrin and DDT) | Every 12-hours | Degradation ≤15% of each analyte. | Repeat breakdown check | | | |
| Method Blank | One per preparation batch | No analytes detected ≥RL | Re-prep and re-analyze method blank and all samples processed with the contaminated blank | Apply B to specific analyte(s) on all associated samples | | |
| LCS | One LCS per preparation batch | Within EMAX QC limits | Re-prep and re-analyze the LCS and all associated samples | | | |
| Surrogate spike | Every sample, spiked sample, standard and method blank | Within EMAX QC limits | Correct the problem then re-extract and analyze sample | | | |
| MS/MSD | One MS/MSD per every 20 project samples per matrix | Within EMAX QC limits | None | | | |
| Confirmation | 100% for al positive results | Same as primary column | If quantitation criteria are not met, use confirmation for qualitative identification only. | | | |
| Results reported between MDL and RL | None | None | None | Apply J to all values between MDL and RL | | |
| Comments: RL = lowest calibration point | | | | Reviewed By: | | |
| | | | | Date | | |

DEMONSTRATION OF CAPABILITY

Final Report - Water Pollution Proficiency Testing

Study: WP0107

Opening Date: January 8, 2007 - Closing Date: February 22, 2007

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-619-8899 ext. 205

EPA Lab ID: CA00291

| Pesticides (PT-PEST-WP) | | | | | | | | | Lot #: 8058-29 |
|-------------------------|---------------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|-----------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 7025 | Aldrin | 10103603 | EPA808 | µg/L | 13.1 | 14.8 | 6.06 - 15.6 | 3.67 - 18.0 | Acceptable |
| 7025 | Aldrin | 10178606 | SW3520C/SW8081A | µg/L | 13.1 | 14.8 | 6.06 - 15.6 | 3.67 - 18.0 | Acceptable |
| 7110 | alpha-BHC | 10103603 | EPA808 | µg/L | 10.0 | 12.6 | 6.01 - 12.1 | 4.49 - 13.6 | Check for Error |
| 7110 | alpha-BHC | 10178606 | SW3520C/SW8081A | µg/L | 10.0 | 12.6 | 6.01 - 12.1 | 4.49 - 13.6 | Check for Error |
| 7115 | beta-BHC | 10103603 | EPA808 | µg/L | 8.70 | 9.65 | 4.98 - 10.4 | 3.62 - 11.8 | Acceptable |
| 7115 | beta-BHC | 10178606 | SW3520C/SW8081A | µg/L | 8.70 | 9.65 | 4.98 - 10.4 | 3.62 - 11.8 | Acceptable |
| 7105 | delta-BHC | 10103603 | EPA808 | µg/L | 2.62 | 3.01 | 1.30 - 3.21 | 0.826 - 3.68 | Acceptable |
| 7105 | delta-BHC | 10178606 | SW3520C/SW8081A | µg/L | 2.62 | 3.01 | 1.30 - 3.21 | 0.826 - 3.68 | Acceptable |
| 7120 | gamma-BHC (Lindane) | 10103603 | EPA808 | µg/L | 5.78 | 6.44 | 3.29 - 7.06 | 2.34 - 8.01 | Acceptable |
| 7120 | gamma-BHC (Lindane) | 10178606 | SW3520C/SW8081A | µg/L | 5.78 | 6.44 | 3.29 - 7.06 | 2.34 - 8.01 | Acceptable |
| 7240 | alpha-Chlordane | 10103603 | EPA808 | µg/L | 6.89 | 7.42 | 4.13 - 8.25 | 3.10 - 9.28 | Acceptable |
| 7240 | alpha-Chlordane | 10178606 | SW3520C/SW8081A | µg/L | 6.89 | 7.42 | 4.13 - 8.25 | 3.10 - 9.28 | Acceptable |
| 7245 | gamma-Chlordane | 10103603 | EPA808 | µg/L | 6.42 | 7.11 | 3.65 - 7.70 | 2.63 - 8.72 | Acceptable |
| 7245 | gamma-Chlordane | 10178606 | SW3520C/SW8081A | µg/L | 6.42 | 7.11 | 3.65 - 7.70 | 2.63 - 8.72 | Acceptable |
| 7355 | DDD (4,4) | 10103603 | EPA808 | µg/L | 4.76 | 5.91 | 2.63 - 6.01 | 1.79 - 6.86 | Acceptable |
| 7355 | DDD (4,4) | 10178606 | SW3520C/SW8081A | µg/L | 4.76 | 5.91 | 2.63 - 6.01 | 1.79 - 6.86 | Acceptable |
| 7360 | DDE (4,4) | 10103603 | EPA808 | µg/L | 8.70 | 8.28 | 5.10 - 9.98 | 3.88 - 11.2 | Acceptable |
| 7360 | DDE (4,4) | 10178606 | SW3520C/SW8081A | µg/L | 8.70 | 8.28 | 5.10 - 9.98 | 3.88 - 11.2 | Acceptable |
| 7365 | DDT (4,4) | 10103603 | EPA808 | µg/L | 8.39 | 8.12 | 4.58 - 10.4 | 3.13 - 11.8 | Acceptable |
| 7365 | DDT (4,4) | 10178606 | SW3520C/SW8081A | µg/L | 8.39 | 8.12 | 4.58 - 10.4 | 3.13 - 11.8 | Acceptable |
| 7470 | Dieldrin | 10103603 | EPA808 | µg/L | 10.5 | 11.7 | 6.66 - 12.7 | 5.15 - 14.2 | Acceptable |
| 7470 | Dieldrin | 10178606 | SW3520C/SW8081A | µg/L | 10.5 | 11.7 | 6.66 - 12.7 | 5.15 - 14.2 | Acceptable |
| 7510 | Endosulfan I | 10103603 | EPA808 | µg/L | 4.90 | 4.35 | 2.09 - 5.87 | 1.15 - 6.81 | Acceptable |
| 7510 | Endosulfan I | 10178606 | SW3520C/SW8081A | µg/L | 4.90 | 4.35 | 2.09 - 5.87 | 1.15 - 6.81 | Acceptable |
| 7515 | Endosulfan II | 10103603 | EPA808 | µg/L | 16.2 | 17.8 | 7.67 - 18.7 | 4.92 - 21.4 | Acceptable |
| 7515 | Endosulfan II | 10178606 | SW3520C/SW8081A | µg/L | 16.2 | 17.8 | 7.67 - 18.7 | 4.92 - 21.4 | Acceptable |
| 7520 | Endosulfan sulfate | 10103603 | EPA808 | µg/L | 12.1 | 12.4 | 6.74 - 15.4 | 4.56 - 17.6 | Acceptable |
| 7520 | Endosulfan sulfate | 10178606 | SW3520C/SW8081A | µg/L | 12.1 | 12.4 | 6.74 - 15.4 | 4.56 - 17.6 | Acceptable |
| 7540 | Endrin | 10103603 | EPA808 | µg/L | 2.05 | 2.72 | 1.26 - 2.82 | 0.870 - 3.21 | Acceptable |
| 7540 | Endrin | 10178606 | SW3520C/SW8081A | µg/L | 2.05 | 2.72 | 1.26 - 2.82 | 0.870 - 3.21 | Acceptable |

DEMONSTRATION OF CAPABILITY

Final Report - Water Pollution Proficiency Testing

Study: WP0107

Opening Date: January 8, 2007 - Closing Date: February 22, 2007

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-618-8889 ext. 205

EPA Lab ID: CA00291

| Pesticides (PT-PEST-WP) cont'd | | | | | | | | | Lot #: 8058-29 |
|---------------------------------------|---------------------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|-----------------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 7530 | Endrin aldehyde | 10103603 | EPA808 | µg/L | 11.8 | 13.4 | 5.91 - 15.3 | 3.55 - 17.7 | Acceptable |
| 7530 | Endrin aldehyde | 10178606 | SW3520C/SW8081A | µg/L | 11.8 | 13.4 | 5.91 - 15.3 | 3.55 - 17.7 | Acceptable |
| 7685 | Heptachlor | 10103603 | EPA808 | µg/L | 9.38 | 10 | 4.66 - 11.1 | 3.03 - 12.8 | Acceptable |
| 7685 | Heptachlor | 10178606 | SW3520C/SW8081A | µg/L | 9.38 | 10 | 4.66 - 11.1 | 3.03 - 12.8 | Acceptable |
| 7690 | Heptachlor Epoxide (beta) | 10103603 | EPA808 | µg/L | 5.66 | 5.83 | 3.66 - 7.07 | 2.80 - 7.93 | Acceptable |
| 7690 | Heptachlor Epoxide (beta) | 10178606 | SW3520C/SW8081A | µg/L | 5.66 | 5.83 | 3.66 - 7.07 | 2.80 - 7.93 | Acceptable |
| 7810 | Methoxychlor | 10103603 | EPA808 | µg/L | 5.63 | 7.11 | 2.70 - 7.78 | 1.43 - 9.05 | Acceptable |
| 7810 | Methoxychlor | 10178606 | SW3520C/SW8081A | µg/L | 5.63 | 7.11 | 2.70 - 7.78 | 1.43 - 9.05 | Acceptable |
| NELAC Experimental Analytes | | | | | | | | | |
| 7535 | Endrin ketone | 10103603 | EPA808 | µg/L | 0.00 | 0 | | | Acceptable |
| 7535 | Endrin ketone | 10178606 | SW3520C/SW8081A | µg/L | 0.00 | 0 | | | Acceptable |
| Chlordane (PT-CHLOR-WP) | | | | | | | | | Lot #: 8058-30 |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 7250 | Chlordane (total) | 10103603 | EPA808 | µg/L | 7.86 | 8.92 | 4.35 - 9.98 | 2.95 - 11.4 | Acceptable |
| 7250 | Chlordane (total) | 10178606 | SW3520C/SW8081A | µg/L | 7.86 | 8.92 | 4.35 - 9.98 | 2.95 - 11.4 | Acceptable |
| Toxaphene (PT-TXP-WP) | | | | | | | | | Lot #: 8058-31 |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
| 8250 | Toxaphene (total) | 10103603 | EPA808 | µg/L | 70.9 | 52.3 | 15.2 - 106 | 7.09 - 128 | Acceptable |
| 8250 | Toxaphene (total) | 10178606 | SW3520C/SW8081A | µg/L | 70.9 | 52.3 | 15.2 - 106 | 7.09 - 128 | Acceptable |

DEMONSTRATION OF CAPABILITY

Final Report - Soil / Hazardous Waste PT

Study: HW0107

Opening Date: January 22, 2007 - Closing Date: March 8, 2007

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

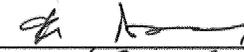
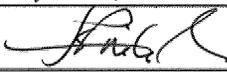
Contact: Ms. Kenette Pimentel
 310-618-8889 ext. 205

EPA Lab ID: CA00291

| Pesticides (PT-PEST-SOIL) | | | | | | | | Lot #: 7027-14 |
|---|---------------------|-------------|--------------------|-------|----------------|--------|-------------------|-----------------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 7025 | Aldrin | 10178606 | SW3550B/SW8081A | µg/kg | 178 | 182 | 48.3 - 308 | Acceptable |
| 7110 | alpha-BHC | 10178606 | SW3550B/SW8081A | µg/kg | 430 | 474 | 86.5 - 773 | Acceptable |
| 7115 | beta-BHC | 10178606 | SW3550B/SW8081A | µg/kg | 440 | 430 | 0.00 - 903 | Acceptable |
| 7105 | delta-BHC | 10178606 | SW3550B/SW8081A | µg/kg | 247 | 236 | 75.6 - 418 | Acceptable |
| 7120 | gamma-BHC (Lindane) | 10178606 | SW3550B/SW8081A | µg/kg | 137 | 140 | 45.1 - 229 | Acceptable |
| 7355 | DDD (4,4) | 10178606 | SW3550B/SW8081A | µg/kg | 110 | 97.8 | 0.00 - 241 | Acceptable |
| 7360 | DDE (4,4) | 10178606 | SW3550B/SW8081A | µg/kg | 90.4 | 70.9 | 0.00 - 182 | Acceptable |
| 7365 | DDT (4,4) | 10178606 | SW3550B/SW8081A | µg/kg | 210 | 208 | 72.5 - 348 | Acceptable |
| 7470 | Dieldrin | 10178606 | SW3550B/SW8081A | µg/kg | 136 | 129 | 29.4 - 242 | Acceptable |
| 7510 | Endosulfan I | 10178606 | SW3550B/SW8081A | µg/kg | 25.5 | 21.8 | 0.00 - 51.5 | Acceptable |
| 7515 | Endosulfan II | 10178606 | SW3550B/SW8081A | µg/kg | 325 | 319 | 61.5 - 589 | Acceptable |
| 7520 | Endosulfan sulfate | 10178606 | SW3550B/SW8081A | µg/kg | 343 | 359 | 0.00 - 726 | Acceptable |
| 7540 | Endrin | 10178606 | SW3550B/SW8081A | µg/kg | 145 | 139 | 58.5 - 232 | Acceptable |
| 7530 | Endrin aldehyde | 10178606 | SW3550B/SW8081A | µg/kg | 13.0 | 12.8 | 0.00 - 35.1 | Acceptable |
| 7685 | Heptachlor | 10178606 | SW3550B/SW8081A | µg/kg | 157 | 139 | 30.0 - 283 | Acceptable |
| 7690 | Heptachlor epoxide | 10178606 | SW3550B/SW8081A | µg/kg | 51.7 | 42.6 | 19.1 - 84.2 | Acceptable |
| 7810 | Methoxychlor | 10178606 | SW3550B/SW8081A | µg/kg | 362 | 449 | 0.00 - 897 | Acceptable |
| Additional State Specific Analytes | | | | | | | | |
| 7240 | alpha-Chlordane | 10178606 | SW3550B/SW8081A | µg/kg | <5 | 0 | | Acceptable |
| 7245 | gamma-Chlordane | 10178606 | SW3550B/SW8081A | µg/kg | <5 | 0 | | Acceptable |
| Toxaphene (PT-TXP-SOIL) | | | | | | | | Lot #: 7027-16 |
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 8250 | Toxaphene | 10178606 | SW3550B/SW8081A | µg/kg | 201 | 191 | 0.00 - 511 | Acceptable |

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POLYCHLORINATED BIPHENYLS (PCB) BY GAS CHROMATOGRAPHY

SOP No.: EMAX-8082 Revision No. 2 Date: 03-Sep-07
 Prepared By: Tu Nisamanepong  Date: 08-17-07
 Approved By: Kenette Pimentel  Date: 08.17.07
 QA Manager
 Approved By: Kam Pang  Date: 8/17/07
 Laboratory Director

Control Number: **8082-01-**

1.0 SCOPE AND APPLICATION

- 1.1. This procedure is used to determine the concentration of Polychlorinated Biphenyls as Aroclors in soil, sediment, sludge, and wastewater samples by gas chromatography method. This SOP is an adaptation of Method 8082.

2.0 SUMMARY OF METHOD

- 2.1. This method provides gas chromatographic conditions for the detection of polychlorinated biphenyls compounds with dual Electron Capture Detector (ECD). The samples are extracted in methylene chloride, exchanged to hexane and clean up by appropriate method before GC analysis.
- 2.2. **Interferences**
- 2.2.1. Interferences by phthalate esters co-extracted from the sample or introduced during sample preparation can pose a major problem in PCB determinations. Interferences can be minimized by avoiding contact with any plastic materials and checking all solvents for phthalate contamination. Glassware must be scrupulously cleaned. Sulfuric acid/permanganate cleanup technique can be used for Phthalate esters removal from the extract.
- 2.2.2. The presence of elemental sulfur will result in broad peaks that may cause chromatographic interfere with the determination of PCBs. Sulfur contamination is most likely present in sediment samples. The copper cleanup, GPC or other cleanup technique can be used for sulfur removal from the extract.

3.0 QUANTITATION LIMITS**3.1. Method Detection Limit**

- 3.1.1. Prepare a minimum of seven samples for each matrix. Add MDL spike standard (Refer to EMAX-QA04 for suggested spike levels). Prepare a method blank and LCS as described in Section 10.1 using standards as described in Section 9.8.
- 3.1.2. Analyze the samples as described in Section 10.4 and calculate the results as described in Section 10.6.
- 3.1.1. Perform MDL evaluation and verification as described in EMAX-QA04.

3.2. Reporting Limit

- 3.2.1. The lowest calibration point shall define reporting limit unless otherwise specified by the project.

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4.0 DYNAMIC RANGE

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point (see Sec. 9.4). All samples analyzed above this concentration are considered "over-range" and shall require dilution to properly quantitate.
- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required to properly quantitate.
- 4.3. Typical dynamic ranges are:
 - 4.3.1. Water – 1.0 to 10.0 ug/L
 - 4.3.2. Soil – 33 to 330 ug/kg

5.0 HOLDING TIME AND PRESERVATION,**5.1. Holding Time**

- 5.1.1. Water samples must be extracted within 7 days from the time of sample collection.
- 5.1.2. Soil and sediment samples must be extracted within 14 days from the time of sample collection.
- 5.1.3. Analysis should be within 40 days after extraction completion date.

5.2. Preservation

- 5.2.1. Samples and extract should be kept at 4°C ± 2°C.

6.0 ASSOCIATED SOPs

- | | | |
|-------|-----------|--|
| 6.1. | EMAX-DM01 | Data Flow and Review |
| 6.2. | EMAX-QC02 | Analytical Standard Preparation |
| 6.3. | EMAX-SM04 | Analytical and QC Sample Labeling |
| 6.4. | EMAX-QA08 | Corrective Action |
| 6.5. | EMAX-3510 | Separatory Funnel Liquid/Liquid Extraction |
| 6.6. | EMAX-3520 | Extraction of Organic Compounds by Continuous Liquid/Liquid Extraction |
| 6.7. | EMAX-3550 | Extraction of Organic Compounds from Solid Samples by Pulse Sonication |
| 6.8. | EMAX-3540 | Soxhlet Extraction |
| 6.9. | EMAX-3620 | Florisil Cleanup |
| 6.10. | EMAX-3640 | GPC Cleanup |
| 6.11. | EMAX-3660 | Sulfur Cleanup |
| 6.12. | EMAX-3665 | Sulfuric Acid/Permanganate Cleanup |

7.0 SAFETY

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- 7.1. Read all MSDS of chemicals listed in this SOP.
- 7.2. All reagents, standards, and samples shall be treated as potential hazards. ECD contains minute quantity of Radioactive Ni (63), a wipe test performed by experienced personnel or manufacturer should be conducted semiannually or sooner if potential problem is suspected. Observe the standard laboratory safety procedures. Protective gear, i.e., lab coat, safety glasses, gloves, shall be worn at all times when performing this procedure.
- 7.3. All wastes generated during analytical process shall be placed in the waste containers. These wastes shall be endorsed to the waste disposal section for proper disposal.
- 7.4. Water samples shall be neutralized to pH 7 (± 2) prior to disposal.
- 7.5. If for any reason, solvent and/or other reagents get in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES**8.1. Instruments and Supplies**

- | | | |
|--------------------|---|--|
| Gas Chromatography | : | HP 5890 Series II |
| Detector | : | Dual Electron Capture Detectors |
| Column | : | RTX CLPESTI (30 m x 0.32 mm x 0.5 μ m) RTX CLPESTII (30 m x 0.32 mm x 0.25 μ m) (or equivalents) |
| Data System | : | EZ Chrom |
| Auto Sampler | : | HP Model 7673B or equivalent |
| Gas | : | Ultra-high purity hydrogen Ultra-high purity nitrogen |
| Microsyringes | : | 10, 25, 100 and 500 μ L with a 0.006 mm ID needle |
| Volumetric Flasks | : | 0,50, and 100 ml with ground glass stopper |
| Automatic Pipette | : | Pipetman, 1000 and 200 μ L |
| Transfer Pipette | : | Pasteur |

8.2. Chemicals and Reagents

- | | | |
|---------|---|---|
| Solvent | : | Pesticide-free grade hexane, methylene chloride |
|---------|---|---|

9.0 STANDARDS**9.1. Preparation**

- 9.1.1. Prepare analytical standards according to EMAX-QC02.

9.2. Stock Standard

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- 9.2.1. Calibration stock standards are purchased as certified solutions at 1000 mg/L. All stock standards should be transferred to an inert vial and stored with minimum headspace after opening.
- 9.2.2. Additional set of stock standards should be purchased from a different source to verify the concentration of the first set of standard. The verification should take place whenever a new lot of stock solution is used. It is prepared using the same procedure as that of the working standard.
- 9.2.3. Surrogate and matrix standards are purchased as certified solutions from various suppliers.
- 9.2.4. Store all standards at 4°C ($\pm 2^{\circ}\text{C}$).

9.3. Intermediate Standard

- 9.3.1. Prepare intermediate standards at 2000 $\mu\text{g/L}$ as suggested in Table 1.

9.4. Initial Calibration Standard (ICAL)

- 9.4.1. Prepare five calibration standards according to Table 1A – ICAL Standard Preparation.

9.5. Initial Calibration Verification (ICV)

- 9.5.1. Prepare ICV using an intermediate standard prepared from a second source stock standard similarly as described in Table 2 – Calibration Check Standards

9.6. Daily Calibration Check Standard (DCC)

- 9.6.1. Prepare DCC using the an intermediate standard prepared form the same source as the ICAL standard as described in Table 2 – Calibration Check Standard.

9.7. Surrogate Standard

- 9.7.1. Prepare surrogate standard as described in Table 3 – Surrogate Standard Preparation

9.8. LCS/MS Spike Standard

- 9.8.1. Prepare LCS/MS spike standard as described in Table 4 – Spike Standards Preparation

10.0 PROCEDURES**10.1. Sample Preparation**

- 10.1.1. Aqueous samples shall be prepared as described in EMAX-3520.
- 10.1.2. Solid samples shall be prepared as described in EMAX-3550 and clean up with concentrated sulfuric acid as described in EMAX-CLP-PEST.

10.2. Instrument Parameters

- 10.2.1. Method 8082 requires an analytical system complete with a temperature programmable gas chromatograph equipped with an autosampler suitable for on column injection of 2 to 5 μl .
- 10.2.2. **Gas Pressure**
 - Nitrogen Pressure : 40 psi
 - Hydrogen Pressure : 50 psi
- 10.2.3. **Temperature Program**
 - Initial Temp : 120°C, hold for 2 minutes

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Rate 1 : 20°C/min
 Temp 1 : 220°C, hold for 2 minutes
 Rate 2 : 2°C/min
 Temp 2 : 230°C, hold for 0 minutes
 Rate 3 : 25°C/min
 Final Temp : 300°C, hold for 5 minutes
 Injector : 280°C
 Detector : 300°C
 Injection Volume : 2µL

10.3. Calibration**10.3.1. Initial Calibration (ICAL)**

- 10.3.1.1. A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclor 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibration for each of the seven Aroclors.
- 10.3.1.2. The five levels of calibration standards of Aroclors 1016 and 1260 are analyzed by direct injection.
- 10.3.1.3. Three to five characteristic peaks from each Aroclor are chosen for quantitation. Peaks should be at least 25% of the height of the largest Aroclor peak. Tabulate each peak area response against concentration of injected standard. Calculate each calibration factor according to Equation 10.6.1.1. Five sets of calibration factors will be generated for each Aroclor. Calculate the average mean calibration factor (Equation 10.6.1.2), standard deviation (Equation 10.6.1.3) and the relative standard deviation (Equation 10.6.1.4) for each Aroclor peak. The average RSD of five calibration factors for each Aroclor should be equal to or less than 20 before linearity to be assumed and the average calibration factor to be used in quantitation.
- 10.3.1.4. Prepare a mid-level calibration standard of Aroclors (1260 + 1016) from a second source. Analyze it after the initial calibration to verify the standard. The %D should be equal to or less than 15%.
- 10.3.1.5. The standards of the other five Aroclors are used to determine a single-point calibration factor for each Aroclor and pattern recognition. It can be analyzed before or after the five 1016/1260 standards.
- 10.3.1.6. In situations where a particular Aroclor is of interest for a specific project, a five point calibration curve of that Aroclor may be employed instead of 1016/1260 mixture.

10.3.2. Daily Calibration Check (DCC)

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- 10.3.2.1. For daily analysis, a mid-level Aroclors (1016 + 1260) standard should be analyzed. A same calibration standard must also be injected at interval of not less than 20 samples interval and at the end of the analysis sequence.
- 10.3.2.2. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition.
- 10.3.2.3. The calibration factor calculated from each DCC must not exceed a difference of more than 15% when compared to the mean CF from the initial calibration curve (Equation 10.6.2.1).
- 10.3.2.4. Continuing calibration for sites that specific PCB is expected to be found, 500 µg/L of that particular PCB shall be analyzed.

10.4. Analysis**10.4.1. Sample Analysis**

- 10.4.1.1. Introduce sample into the gas chromatograph using direct injection technique (1 to 5 µl) after all quality control criteria have been met. Positive identification is made when a target peak falls within the retention time window on both columns established by the standard reference compound.
- 10.4.1.2. Identification of a multicomponent analyte in the sample is based on pattern recognition in conjunction with the elution of three or five sample peaks on both GC columns.
- 10.4.1.3. If the responses exceed the linear range of the system, dilute the sample and reanalyze.

10.4.2. Analytical Sequence

- 10.4.2.1. Before analysis, approximately 0.5 ml of extract is transferred to a 2 ml amber auto sampler vial (or equivalent) with a Pasteur pipette, sample vial is capped with a teflon septum and sealed with an aluminum rim. Standards and QC samples are sealed the same way.
- 10.4.2.2. Following the data acquisition software's instruction, prepare the analytical sequence as follows:
 - Instrument Blank
 - Five Calibration Standards of Aroclors 1016/1260
 - Calibration Standard of Aroclor 1254*
 - Calibration Standard of Aroclor 1248*
 - Calibration Standard of Aroclor 1242*
 - Calibration Standard of Aroclor 1232*
 - Calibration Standard of Aroclor 1221*
 - Initial Calibration Verification of Aroclor 1016/1260 by a Second Source
 - Method Blank

* Calibrate if the pattern is found in a sample

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- Samples (up to 20 samples)
- Continuing Calibration Standard of Aroclors 1016/1260 or other calibrated Aroclors

10.4.3. Sample Result Evaluation

10.4.3.1. Check QC parameters as soon as the data is available.

- ✓ Check surrogate recoveries against Appendix 1.
- ✓ Check concentration of target analytes. If the response exceeds the calibration range, dilute and re-analyze the sample until the response falls within the calibration range.
- ✓ If any of the above checkpoints indicate a problem, re-analysis is required. If re-analysis results are the same as the initial result, consult the Supervisor for further action. If results indicate extraction problem, fill-up an NCR and order re-extraction for the affected sample(s).

10.4.3.2. For positive identification, check that the PCB pattern characteristic peaks are detected in both columns within the established retention time window.

10.4.3.3. If one column meets the retention time criteria and a retention time shift is suspected on the other column, use the following guideline in reporting the data:

- ✓ Check that the expanded window does not exceed the RTW of the column in control or the established RTW or the CLP RTW (refer to table 7) which ever is greater.
- ✓ If the above condition is met, report the data and include a description of the observation in the case narrative.

10.4.4. Retention Time Windows

10.4.4.1. Establishing RTW

- 10.4.4.1.1. Collect at least three Daily Calibration Standards analyzed over a period of 72 hours.
- 10.4.4.1.2. Calculate the Standard Deviation (SD) of absolute retention time obtained for each of the major peaks used for calibration.
- 10.4.4.1.3. Determine the width of RTW by $\pm 3X$ SD.

10.4.4.2. Evaluating RTW

- 10.4.4.2.1. If the SD is equal to 0.00, default to the previous study until historical data is obtained or use the CLP¹ retention time window for Aroclors (± 0.07 minutes) which ever is narrower, until RTW is obtained for the instrument.
- 10.4.4.2.2. For new instruments, in the interim use the CLP retention time window for Aroclors (± 0.07 min) until RTW is obtained for the new instrument parameters condition.

10.4.4.3. Application of RTW

- 10.4.4.3.1. Establish the center of absolute retention time for each of the characteristic peak to include the surrogate(s) from the daily

¹ CLP-OLM4.2 Table 1 D-79/PEST

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calibration check at the beginning of the analytical shift then apply the established RTW.

- 10.4.4.3.2. Whenever the observe retention time is outside the established RTW, the analyst is advised to determine the cause and perform necessary corrective action before continuing analyses.

10.4.4.4. Updating RTW

- 10.4.4.4.1. Re-establish the RTW as described in Section 10.4.4.1 when any of the following conditions occur:

- Yearly RTW update
- Significant shifting is observed (e.g. succeeding calibration checks or LCS are out of RTW)
- Major instrument maintenance (e.g. replacements of detector or column; temperature program change, etc.)

10.4.5. **Manual Integration**

- 10.4.5.1. Refer to EMAX-DM01 for details of manual integration.

10.4.6. **Dealing with Carryover**

- ✓ Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- ✓ If there was no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
- ✓ If there was a target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.5. **Data Reduction**

- 10.5.1. Identification of a multicomponent analyte in the sample is based on pattern recognition in conjunction with the elution of three to five peaks on both GC columns.
- 10.5.2. The agreement between the quantitative results should be evaluated after the identification is made. Calculate the relative percent difference (RPD) between the two results according to Equation 10.6.1.4.
- 10.5.2.1. If the RPD is less than 40% and the pattern peaks do not indicate any anomalies, report the higher result.
- 10.5.2.2. If the RPD is less than 40% and the pattern peaks indicate an anomaly, report the result from the better patter peaks.
- 10.5.2.3. If the RPD is greater than 40%, use professional judgement. If no anomaly is found, report the higher result.

10.6. **Calculations**

- 10.6.1. Initial Calibration
- 10.6.1.1. Calculate for Calibration Factor (CF).

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$$CF = \frac{R_a}{C_a} \quad \text{Eq.-10.6.1.1}$$

where:

- R_a - response for analyte measured in peak area
 C_a - Concentration of analyte ($\mu\text{g/L}$) $\times 1/N$
 N - number of identification peak in each Aroclor

10.6.1.2. Calculate for Average Calibration Factor (ACF)

$$ACF = \frac{\sum CF_a}{n} \quad \text{Eq.-10.6.1.2}$$

where:

- ACF - average calibration factor
 $\sum CF_a$ - sum of calibration factors
 n - number of calibration points

10.6.1.3. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N-1}} \quad \text{Eq.-10.6.1.3}$$

where:

- SD - standard deviation
 x_i - result at i^{th} measurement
 \bar{x} - mean
 N - number of measurements

10.6.1.4. Calculate for % relative standard deviation (%RSD).

$$\% RSD = \frac{SD}{ACF} * 100\% \quad \text{Eq.-10.6.1.4}$$

where:

- RSD - relative standard deviation
 SD - standard deviation
 ACF - average calibration factor

10.6.2. Calibration Check/Continuing Calibration

10.6.2.1. Calculate Percent Difference.

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$$\% D = \frac{ACF - CF}{ACF} * 100\% \quad \text{Eq.-10.6.2.1}$$

where:

ACF - average calibration factor*CF* - calibration factor at calibration check standard

10.6.3. Sample Results

3 to 5 major peaks are identified as quantitative peaks for each Aroclor. Establish the ACF of each peak as outlined in Section 10.6.1.1 Calculate C of each peak in Aroclor according to Equations 10.6.3.1 or 10.6.3.2. The concentration of Aroclor equals to the sum of the concentrations of these major peaks.

10.6.4. Sample Results

10.6.4.1. Water Samples

$$C_n = \left(\frac{R_a}{AFC} \right) \left(\frac{V_e}{S_a} \right) DF \quad \text{Eq-10.6.4.1}$$

where:

C_n - Concentration of characteristic peak n measured in µg/L*R_a* - Total response of analyte in peak area*AFC* - Average response factor measure in ICAL*V_e* - Volume of extract in ml*S_a* - Sample amount in ml*DF* - Dilution factor of sample extract

10.6.4.2. Soil Samples

$$C = \left(\frac{R_a}{AFC} \right) \left(\frac{V_e}{S_a (\% \text{Solid})} \right) DF \quad \text{Eq.10.6.4.2}$$

where

C - Concentration of analyte to be measured (µg/kg)*R_a* - Total response of analyte in peak area*AFC* - Average response factor*V_e* - Volume of extract in ml*S_a* - Sample Amount in g*% Solid* - $\frac{100 - \% \text{moisture}}{100}$ *D* - Dilution factor of the sample extract

10.6.4.3. Final Aroclor Concentration

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$$C = C_1 + C_2 + \dots C_n \quad \text{Eq.-10.6.4.3}$$

where:

- C - Concentration of Aroclor in sample ($\mu\text{g/L}$ or $\mu\text{g/kg}$)
 C_1 - Concentration of characteristic peak 1
 C_2 - Concentration of characteristic peak 2
 n - Characteristic peak number in each Aroclor

10.6.5. Accuracy and Precision

10.6.5.1. Percent Recovery

$$\%R = \frac{C_f - C}{C_s} * 100 \quad \text{Eq.-10.6.4.1}$$

where:

- $\%R$ - percent recovery
 C_f - concentration found
 C_s - concentration of spike

10.6.5.2. Relative Percent Difference

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq.-10.6.4.2}$$

where:

- $\%RPD$ - Relative Percent Difference
 $C1$ - Measured concentration of the first sample aliquot
 $C2$ - Measured concentration of the second sample aliquot

10.6.6. Method Detection Limit

$$\text{MDL} = 3.14 * \text{SD} \quad \text{Eq.-10.6.5}$$

where:

- MDL - method detection limit
SD - standard deviation (see Eq-10.6.1.3) from 7 measurements.

10.7. Report Generation

- 10.7.1. Generate the method.txt file using WBDX².exe
10.7.2. Generate Lab Chronicle using Labchron.exe
10.7.3. Generate sample results using F1VX¹.exe

² X – version number

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- 10.7.4. Generate the QC Summary file using QCVX¹.exe
- 10.7.5. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration(s) and re-analyses.
- Sample Results
 - LCS Summary
 - MS/MSD Summary
 - DCC Summary
 - ICAL Summary
 - ICV Summary
 - Copy of Analysis Log
 - Copy of Preparation Log

10.8. Data Review

- 10.8.1. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
- ✓ Check the chromatogram of all positively identified Aroclor patterns to determine the final results according to Section 10.5.2.
 - ✓ Check surrogate recoveries against project specific criteria (PSR). In the absence of PSR, default to in-house QC limits.
 - ✓ Check concentration of target analytes if calibration range is exceeded.
 - ✓ If any of the above checkpoints indicate a problem, re-analysis is required.
- 10.8.2. Generate the case narrative to include discussion of the following as found in the review process:
- Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples extracted and analyzed within the holding time. For non-compliance, state the number of days that the sample(s) was out of holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Method Blank – That MB was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s) affected and the associated sample results were flagged with “B”.
 - Lab Control Samples (if applicable) – That LCS was prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”.

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Further more, if corrective action is not possible (e.g. no more samples to re-extract) state that results were qualified.

- Matrix Spike Samples – That MS/MSD (if applicable) were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Sample Analysis – That samples were analyzed in conformance to the method and project requirements.
- Other Anomalies (if any) – Discussed on a case by case basis concurred by the Supervisor or the Lab Director.

10.8.3. Submit the analysis package for secondary review.

10.9. Preventive Maintenance

10.9.1. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality.

11.0 QUALITY CONTROL**11.1. Preparative Batch**

11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD and a maximum of 20 field samples of similar matrix.

11.1.2. In the absence of MS/MSD, LCS/LCD is prepared.

11.2. Analytical Batch QC

11.2.1. Instrument Performance Evaluation Check must be analyzed daily. Acceptance criteria and corrective action are discussed in Section 10.3.1.4 and Appendix 1.

11.2.2. A continuing calibration shall be performed before any other analysis is done. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.5 and Appendix 1.

11.3. Method QC

11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.

11.3.2. A valid MDL must exist prior to sample analysis.

11.3.3. A valid ICAL must exist prior to sample analysis.

11.3.4. Instrument performance must be checked prior to sample analysis.

11.3.5. Check Appendix 1 for acceptance criteria.

11.3.6. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.

11.3.7. Surrogate standard shall be added to all samples, including method blank LCS/LCD and MS/MSD. Check PSR for QC Control Limits.

STANDARD OPERATING PROCEDURES

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- 11.3.8. Perform QC check prior to utilizing the surrogate and LCS/MS spike standards by analyzing the prepared standard at the spiking level. Results should be within $\pm 20\%$ of the expected value.

12.0 CORRECTIVE ACTION

- 12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures in Appendix 1. Document out-of-control event and corrective action in the analytical logbook. If the problem persists, consult the supervisor.
- 12.2. If Initial calibration is non-compliant, consider the following suggestions to correct the problem:
- 12.2.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.
 - 12.2.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and reanalyze.
 - 12.2.3. If the highest ICAL point appears to be saturated, drop the highest point.
 - 12.2.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.

Note: The lowest calibration point identifies the reporting limit (RL). Therefore, check that the RL is in conformance to the current projects where the ICAL will be used.
 - 12.2.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:
 - 12.2.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.
 - 12.2.5.2. Check the gas flow
 - 12.2.5.3. Prepare a fresh standard and repeat calibration
 - 12.2.6. If the problem persists, inform the supervisor.
- 12.3. If the ICV is non-compliant, consider the following suggestions to correct the problem:
- 12.3.1. Re-analyze ICV (to rule out poor injection)
 - 12.3.2. If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error
 - 12.3.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration
 - 12.3.4. If the problem persists, inform the supervisor
- 12.4. If the instrument blank is non-compliant, consider the following suggestions to correct problem:
- 12.4.1. Rule out instrument contamination by performing the instrument daily maintenance, such as changing septum, cleaning liner, cleaning or using new auto sampler syringe.
 - 12.4.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.
 - 12.4.3. Rule out preparation contamination by preparing a new instrument blank
 - 12.4.4. If the problem persists, inform the supervisor.
- 12.5. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:

STANDARD OPERATING PROCEDURES

POLYCHLORINATED BIPHENYLS (PCB) BY GAS CHROMATOGRAPHYSOP No.: EMAX-8082 Revision No. 2 Date: 03-Sep-07

- 12.5.1. Change the liner
- 12.5.2. Clean injection port
- 12.5.3. Prepare new standard
- 12.5.4. Cut or replace column
- 12.5.5. Clean the detector
- 12.5.6. Rule out leaks by checking all connections
- 12.5.7. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL
- 12.6. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
 - 12.6.1. Rule out instrument contamination by checking instrument blank
 - 12.6.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01
 - 12.6.3. Rule out glassware contamination used for extraction as described in EMAX-QC07
 - 12.6.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents
 - 12.6.5. If the problem persists, inform the supervisor
- 12.7. If LCS is non-compliant, perform the following suggestions to correct the problem:
 - 12.7.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level
 - 12.7.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and re-analyze the LCS and the associated samples.
 - 12.7.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.
- 12.8. Execute a Non-Conformance Report (NCR) when the following circumstances occur:
 - 12.8.1. If corrective action needs the function of other department; e.g., if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.
 - 12.8.2. If corrective action needs the assistance of the project manager; e.g. If the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.
- 12.9. For other problems encountered, inform the supervisor immediately for further instructions.

13.0 POLLUTION PREVENTION

- 13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.
- 13.2. All unused expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 WASTE MANAGEMENT

STANDARD OPERATING PROCEDURES

POLYCHLORINATED BIPHENYLS (PCB) BY GAS CHROMATOGRAPHYSOP No.: EMAX-8082 Revision No. 2 Date: 03-Sep-07

- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03

15.0 SUPPLEMENTARY NOTES**15.1. Definition of Terms**

- 15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents. Preparation batch is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. Analytical batch is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.3. Characteristic Peaks – are major identifying and quantifying peaks for each type of Aroclor.
- 15.1.4. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.5. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.6. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.7. Matrix – is a component or form of a sample.
- 15.1.8. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.9. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.
- 15.1.10. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.11. Re-analysis – is a repeated analysis from the same extract/digestate or sample, identified with the Lab Sample ID suffixed with "W".
- 15.1.12. Re-extract/digest – is a repeated sample preparation process identified with the Lab Sample ID suffixed with "R".
- 15.1.13. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.14. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

STANDARD OPERATING PROCEDURES

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15.1.15. Sample Duplicate – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.

15.2. Application of EMAX QC Procedures

15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing PCB analysis by GC. In instances where there is a project or program QAPP, the requirements given in the project shall take precedence over this SOP.

15.3. Air Force Center for Environmental Excellence (AFCEE) projects

15.3.1. When samples from AFCEE sponsored projects are analyzed for pesticides by GC, shall follow project specific requirements as specified by the QAPP. In the absence of a QAPP, the calibration, QC, corrective action, and data flagging requirements the AFCEE QAPP latest version shall be applied.

15.4. U.S. Army Corps of Engineers (USACE) Projects

15.4.1. When samples from USACE sponsored projects are analyzed for pesticides by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, DoD QSM latest version shall be applied.

15.5. Naval Facilities Engineering Service Center (NFESC) Projects

15.5.1. When samples from NFESC sponsored projects are analyzed for pesticides by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, DoD QSM latest version shall be applied.

15.6. Department of Energy (DOE) Projects

15.6.1. When samples from DOE sponsored projects are analyzed for pesticides by GC, the calibration, QC, corrective action, and data flagging requirements shall follow the specifics outlined in the project QAPP. In the absence of a project QAPP or directive from the client project manager, DOE QSAS shall be applied

16.0 REFERENCES

- 16.1. Method 8082, Test Methods for Evaluating Solid Wastes, USEPA SW846, 3rd edition
 16.2. Laboratory QA/QC Manual, as updated.

17.0 FIGURES, TABLES AND APPENDICES**17.1. Figures**

- 17.1.1. Figure 1 Peak Evaluation Technique
 17.1.2. Figure 2 Typical PCB Patterns
 17.1.3. Figure 3 Typical 1016/1260 Chromatogram

17.2. Tables

- 17.2.1. Table 1 Intermediate Standard
 17.2.2. Table 1A Initial Calibration Standard Preparation
 17.2.3. Table 2 Check Standard Preparation

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- 17.2.4. Table 3 LCS/MS Spike Standard Preparation
- 17.2.5. Table 5 Compound List
- 17.3. **Appendices**
 - 17.3.1. Appendix 1 Summary of Quality Control Procedures
 - 17.3.2. Appendix 2 Demonstration of Capability
- 17.4. **Forms**
 - 17.4.1. 8082FA Analysis Run Log
 - 17.4.2. 8082FM Instrument Maintenance Log

Figure 1 - Peak Evaluation Technique

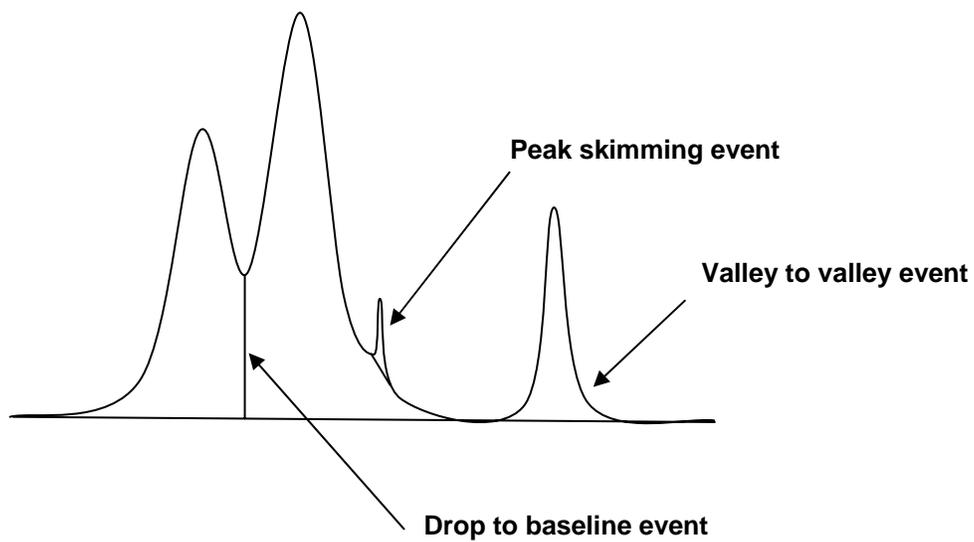
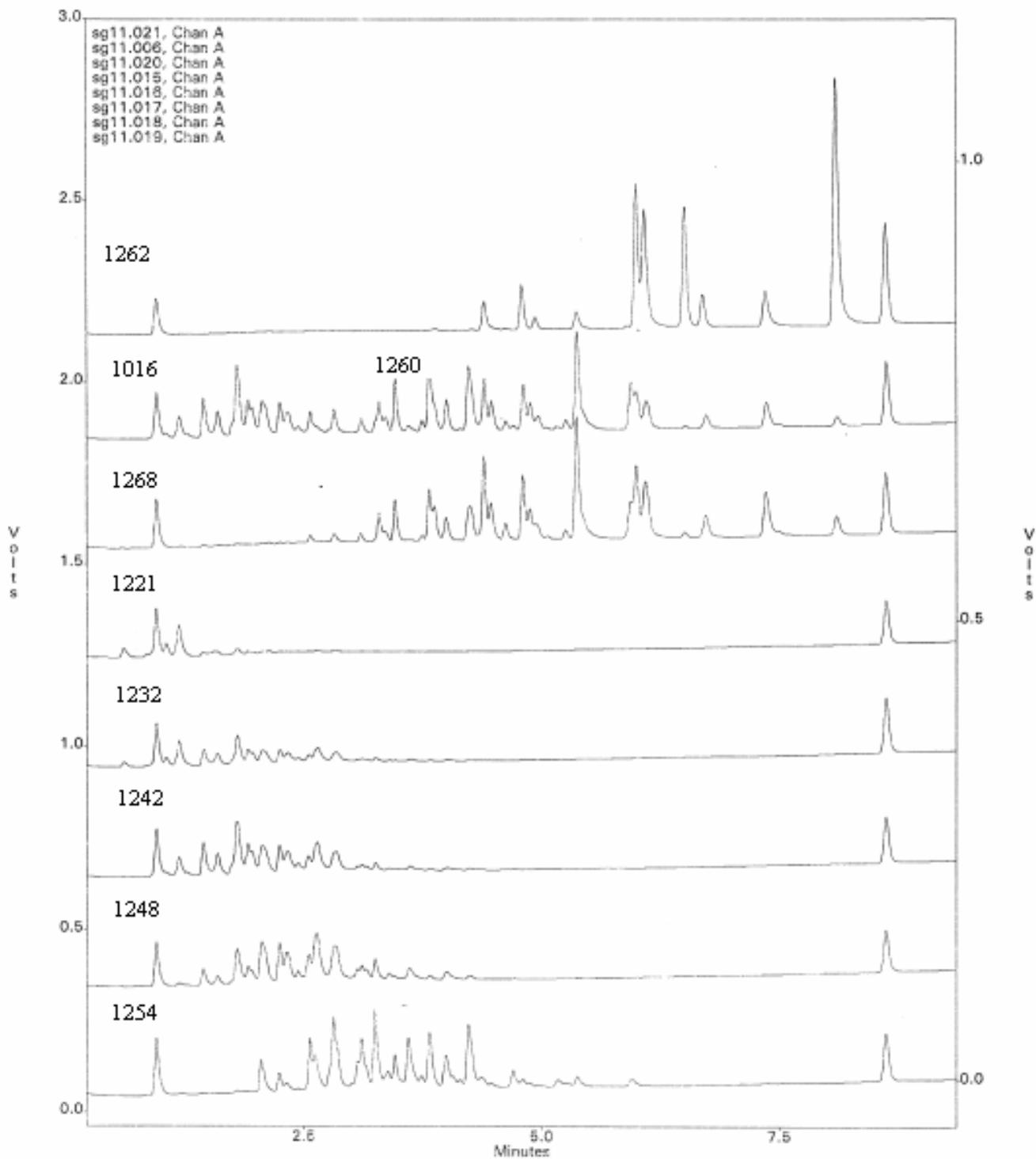


Figure 2

Typical PCB Patterns
Overlaid Traces



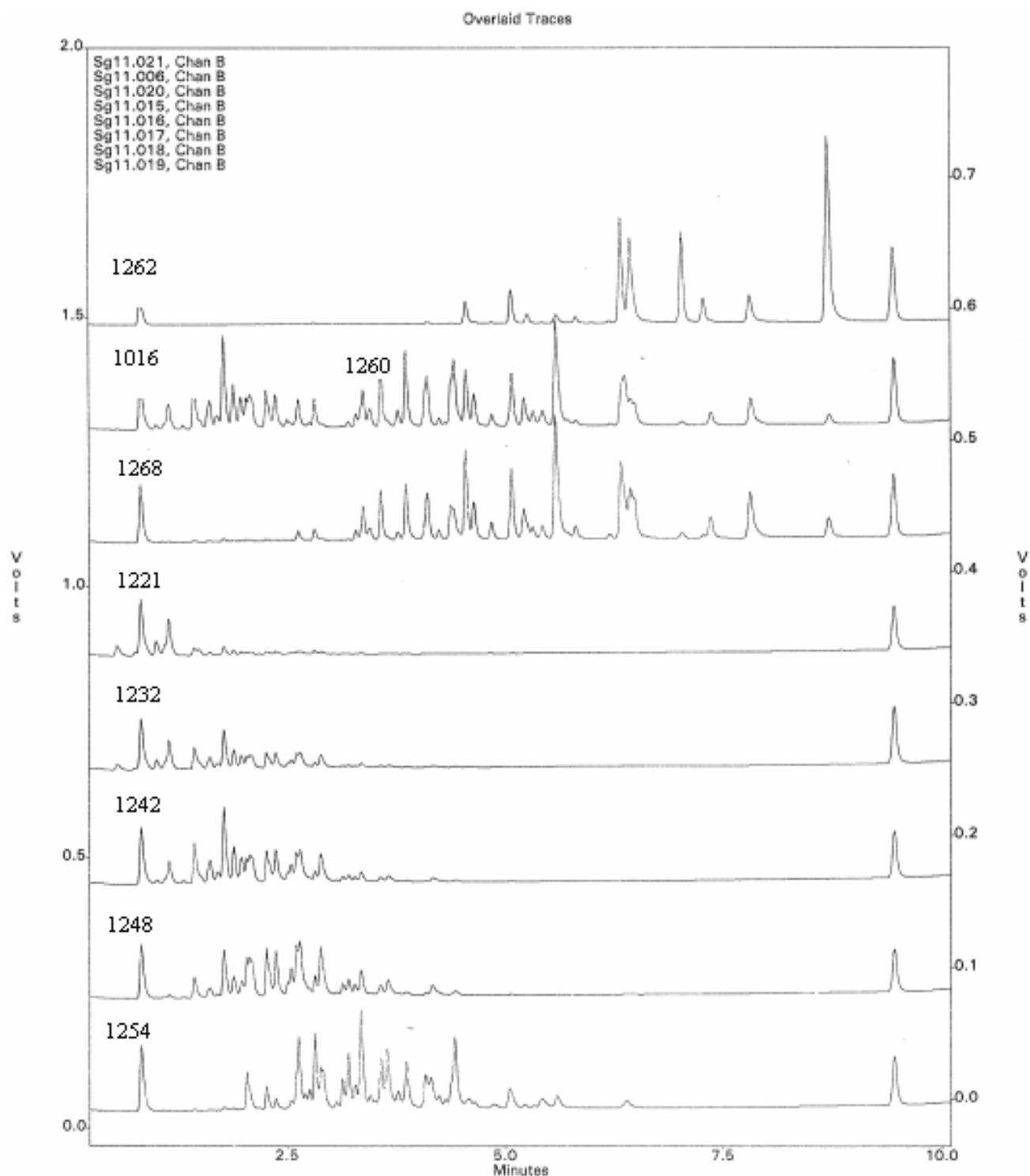


TABLE 1

Intermediate Standard Preparation

| Standard Name | Stock Standard Conc. (ppm) | Preparation | | | Final Conc. (µg/L) |
|------------------|----------------------------|--------------|---------|-----------------|--------------------|
| | | Aliquot (ml) | Solvent | Final Vol. (ml) | |
| PCB 1016 | 1000 | 0.2 | Hexane | 100 | 2000 |
| PCB 1260 | 1000 | 0.2 | | | 2000 |
| PCB 1221 | 1000 | 0.2 | Hexane | 100 | 2000 |
| PCB 1232 | 1000 | 0.2 | Hexane | 100 | 2000 |
| PCB 1242 | 1000 | 0.2 | Hexane | 100 | 2000 |
| PCB 1248 | 1000 | 0.2 | Hexane | 100 | 2000 |
| PCB 1254 | 1000 | 0.2 | Hexane | 100 | 2000 |
| Surrogate | | | | | |
| TCX | 200 | 0.05 | Hexane | 100 | 100 |
| DCB | 200 | 0.05 | Hexane | 100 | 100 |

TABLE 1A

Initial Calibration Standard Preparation

| Standard # | Standard Name | Intermediate Std. (µg/L) | Preparation | | | Final Conc. (µg/L) |
|------------|---------------|--------------------------|--------------|---------|-----------------|--------------------|
| | | | Aliquot (µl) | Solvent | Final Vol. (µl) | |
| 1 | PCB 1016/1260 | 2000 | 40 | Hexane | 800 | 100 |
| | Surrogate | 100 | | | | 5 |
| 2 | PCB 1016/1260 | 2000 | 100 | Hexane | 800 | 250 |
| | Surrogate | 100 | | | | 12.5 |
| 3 | PCB 1016/1260 | 2000 | 200 | Hexane | 800 | 500 |
| | Surrogate | 100 | | | | 25 |
| 4 | PCB 1016/1260 | 2000 | 300 | Hexane | 800 | 750 |
| | Surrogate | 100 | | | | 37.5 |
| 5 | PCB 1016/1260 | 2000 | 400 | Hexane | 800 | 1000 |
| | Surrogate | 100 | | | | 50 |

TABLE 2

Check Standard Preparation

| Standard Name | Stock Standard Conc. (mg/L) | Preparation | | | Final Conc. (µg/L) |
|------------------|-----------------------------|--------------|---------|-----------------|--------------------|
| | | Aliquot (ml) | Solvent | Final Vol. (ml) | |
| PCB 1016 | 1000 | 0.05 | Hexane | 100 | 500 |
| PCB 1260 | 1000 | 0.05 | | | |
| PCB 1221 | 1000 | 0.1 | Hexane | 100 | 1000 |
| PCB 1232 | 1000 | 0.05 | Hexane | 100 | 500 |
| PCB 1242 | 1000 | 0.05 | Hexane | 100 | 500 |
| PCB 1248 | 1000 | 0.05 | Hexane | 100 | 500 |
| PCB 1254 | 1000 | 0.05 | Hexane | 100 | 500 |
| Surrogate | | | | | |
| TCX | 200 | 0.0125 | Hexane | 100 | 25 |
| DCB | 200 | 0.0125 | Hexane | 100 | 25 |

TABLE 3

Spike Standard Preparation

| Standard Name | Stock Standard Conc. (mg/L) | Preparation | | | Final Conc. (µg/L) |
|------------------|-----------------------------|--------------|---------|-----------------|--------------------|
| | | Aliquot (ml) | Solvent | Final Vol. (ml) | |
| PCB 1016 | 1000 | 1.0 | Hexane | 100 | 10,000 |
| PCB 1260 | 1000 | 1.0 | Hexane | | |
| Surrogate | | | | | |
| TCX | 200 | 0.05 | Hexane | 100 | 100 |
| DCB | 200 | 0.05 | Hexane | 100 | 100 |

TABLE 5

Compound List

| Analyte | | Surrogate |
|----------|----------|--|
| PCB-1016 | PCB-1248 | Decachlorobiphenyl Tetrachloro-m-xylene |
| PCB-1221 | PCB-1254 | |
| PCB-1232 | PCB-1260 | |
| PCB-1242 | | |

SUMMARY OF IN-HOUSE QUALITY CONTROL LIMITS

| QC PROCEDURE | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1ST Rvw | 2ND Rvw |
|--|--|---|--|--|-------------------------------|-------------------------------|
| 5 Point Initial Calibration for PCB 1016 + 1260 | Initially, as needed | Mean RSD for each PCB ≤20% | Correct then problem then repeat initial calibration | | | |
| Initial calibration verification for PCB 1016/1260 mix (Second Source) | Once per 5-point initial calibration | Mix within ±15% of expected value | Correct then problem then repeat initial calibration | | | |
| Continuing calibration verification for PCB 1016/1260 mix | After every 12 hours and at the end of the analytical sequence | All analytes within ± 15% of expected value or mean RSD ≤ 15% | Correct the problem then reanalyze all samples since last successful continuing calibration | | | |
| Method Blank | One per preparation batch (≤ 20 samples per matrix) | No analytes detected ≥ RL | Reprep and reanalyze method blank and all samples processed with the contaminated blank | Apply B to specific analyte(s) on all associated samples | | |
| LCS (PCB 1016/1260) | One LCS per analytical batch (≤20 samples per matrix) | Within EMAX In-House QC Limits | Correct the problem then reprep and reanalyze the LCS and all samples in the affected AFCEE analytical batch | | | |
| Surrogate Spike | Every sample, spiked sample, standard, and method blank | Within EMAX In-House QC Limits | Re-extract and re-analyze the sample when both surrogates are out of control. | | | |
| Matrix Spike/ Matrix Spike Duplicate (PCB 1016/1260 mix) | One MS/MSD per every 20 project samples per matrix | Within EMAX In-House QC Limits | none | | | |
| Results reported between MDL and RL | None | None | none | Apply J to all values between MDL and RL | | |
| Comments: RL = Reporting Limit | | | | Reviewed By: | | |
| | | | | Date: | | |

SUMMARY OF METHOD DETECTION LIMIT STUDY

Final Report - Water Pollution Proficiency Testing

Study: WP0107

Opening Date: January 8, 2007 - Closing Date: February 22, 2007

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-618-8889 ext. 205

EPA Lab ID: CA00291

PCBs in Water - Ampule 2 (PT-PCBW-WP(2)) Lot #: 8058-33

| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
|------------|--------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|------------|
| 8880 | Aroclor 1016 | 10103603 | EPA808 | µg/L | 0.00 | 0 | | | Acceptable |
| 8880 | Aroclor 1016 | 10179007 | SW3520C/SW8082 | µg/L | 0.00 | 0 | | | Acceptable |
| 8885 | Aroclor 1221 | 10103603 | EPA808 | µg/L | 0.00 | 0 | | | Acceptable |
| 8885 | Aroclor 1221 | 10179007 | SW3520C/SW8082 | µg/L | 0.00 | 0 | | | Acceptable |
| 8890 | Aroclor 1232 | 10103603 | EPA808 | µg/L | 0.00 | 0 | | | Acceptable |
| 8890 | Aroclor 1232 | 10179007 | SW3520C/SW8082 | µg/L | 0.00 | 0 | | | Acceptable |
| 8895 | Aroclor 1242 | 10103603 | EPA808 | µg/L | 10.1 | 8.38 | 4.64 - 12.4 | 2.72 - 14.3 | Acceptable |
| 8895 | Aroclor 1242 | 10179007 | SW3520C/SW8082 | µg/L | 10.1 | 8.38 | 4.64 - 12.4 | 2.72 - 14.3 | Acceptable |
| 8900 | Aroclor 1248 | 10103603 | EPA808 | µg/L | 0.00 | 0 | | | Acceptable |
| 8900 | Aroclor 1248 | 10179007 | SW3520C/SW8082 | µg/L | 0.00 | 0 | | | Acceptable |
| 8905 | Aroclor 1254 | 10103603 | EPA808 | µg/L | 0.00 | 0 | | | Acceptable |
| 8905 | Aroclor 1254 | 10179007 | SW3520C/SW8082 | µg/L | 0.00 | 0 | | | Acceptable |
| 8910 | Aroclor 1260 | 10103603 | EPA808 | µg/L | 0.00 | 0 | | | Acceptable |
| 8910 | Aroclor 1260 | 10179007 | SW3520C/SW8082 | µg/L | 0.00 | 0 | | | Acceptable |

PCBs in Oil - Ampule 1 (PT-PCBO-WP(1)) Lot #: 8058-34

| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Warning Limits | Acceptance Limits | Evaluation |
|------------|-------------------|-------------|--------------------|-------|----------------|--------|----------------|-------------------|------------|
| 8912 | Aroclor 1016/1242 | 10103603 | EPA808 | mg/kg | 0.00 | 0 | | | Acceptable |
| 8912 | Aroclor 1016/1242 | 10179007 | SW3580/SW8082 | mg/kg | 0.00 | 0 | | | Acceptable |
| 8905 | Aroclor 1254 | 10103603 | EPA808 | mg/kg | 0.00 | 0 | | | Acceptable |
| 8905 | Aroclor 1254 | 10179007 | SW3580/SW8082 | mg/kg | 0.00 | 0 | | | Acceptable |
| 8910 | Aroclor 1260 | 10103603 | EPA808 | mg/kg | 29.0 | 25.7 | 11.3 - 35.1 | 5.35 - 41.1 | Acceptable |
| 8910 | Aroclor 1260 | 10179007 | SW3580/SW8082 | mg/kg | 29.0 | 25.7 | 11.3 - 35.1 | 5.35 - 41.1 | Acceptable |

SUMMARY OF METHOD DETECTION LIMIT STUDY

Final Report - Soil / Hazardous Waste PT

Study: HW0706

Opening Date: July 24, 2006 - Closing Date: September 7, 2006

Laboratory: EMAX Laboratories
 1825 205th Street
 Torrance, CA 90501

Contact: Ms. Kenette Pimentel
 310-618-8889 ext. 205

EPA Lab ID: CA00291

| PCBs (PT-PCB-SOIL) | | | | | | | | Lot #: 7025-17 |
|---------------------------|--------------|-------------|--------------------|-------|----------------|--------|-------------------|-----------------------|
| NELAC Code | Analyte | Method Code | Method Description | Units | Assigned Value | Result | Acceptance Limits | Evaluation |
| 8880 | Aroclor 1016 | 10179007 | SW3550B/SW8082 | mg/kg | <0.5 | 0 | | Acceptable |
| 8885 | Aroclor 1221 | 10179007 | SW3550B/SW8082 | mg/kg | <0.5 | 0 | | Acceptable |
| 8890 | Aroclor 1232 | 10179007 | SW3550B/SW8082 | mg/kg | <0.5 | 0 | | Acceptable |
| 8895 | Aroclor 1242 | 10179007 | SW3550B/SW8082 | mg/kg | <0.5 | 0 | | Acceptable |
| 8900 | Aroclor 1248 | 10179007 | SW3550B/SW8082 | mg/kg | 5.73 | 6.82 | 1.96 - 9.50 | Acceptable |
| 8905 | Aroclor 1254 | 10179007 | SW3550B/SW8082 | mg/kg | <0.5 | 0 | | Acceptable |
| 8910 | Aroclor 1260 | 10179007 | SW3550B/SW8082 | mg/kg | <0.5 | 0 | | Acceptable |

STANDARD OPERATING PROCEDURES

SEMIVOLATILE ORGANICS BY GC/MS
Method 8270SIM

SOP No.: EMAX-8270SIM Revision No. 1 Effective Date: 15-Nov-06

Prepared By: W. Tu Nisamanepong *W. Tu Nisamanepong* Date: 11-15-06

Approved By: Kenette Pimentel *Kenette Pimentel* Date: 11-15-06
QA Manager

Approved By: Kam Pang *Kam Pang* Date: 11/15/06
Laboratory Director

Control Number: 8270SIM-01-

1.0 SCOPE AND APPLICATION

- 1.1. Method 8270SIM is used to determine the trace levels of semi-volatile organic compounds in extracts prepared from many types of solid waste matrices, soils and water samples. Semi-volatile compounds that can be determined by this method are listed in Table 8. Additional compounds can be added after validation. It is an adaptation of method SW846 8270C.

2.0 SUMMARY OF METHOD

- 2.1. Samples are extracted with methylene chloride. Extracts are concentrated and appropriate cleanup procedure is applied if necessary.
- 2.2. Internal standards are added to an aliquot of the final product and are quantitatively and qualitatively analyzed by gas chromatography equipped with mass spectrometry (GCMS).
- 2.3. **Interference**
- 2.3.1. Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interference. Source of interference shall be determined in the preparation and/or cleanup of the samples and corrective action shall be undertaken to eliminate the problem.
- 2.3.2. Contamination by carry over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carry over, the sample syringe must be rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

3.0 QUANTITATION LIMITS**3.1. Method Detection Limits**

- 3.1.1. Prepare a minimum of seven samples for each matrix. Add MDL spike solution (preferably at the concentration of the lowest calibration point) to each of the samples and analyze the extracts as described in Section 10.4.
- 3.1.2. Refer to EMAX-QA04 for MDL evaluation and verification.

3.2. Reporting Limits

- 3.2.1. The quantitation limits of this method as written in this SOP are as follows:
- 3.2.1.1. Water = 1 µg/L to 2 µg/L
- 3.2.1.2. Soil = 5 µg/Kg to 10 µg/Kg

STANDARD OPERATING PROCEDURES

SEMIVOLATILE ORGANICS BY GC/MS
Method 8270SIMSOP No.: EMAX-8270SIMRevision No. 1Effective Date: 15-Nov-06

4.0 DYNAMIC RANGE

- 4.1. The highest quantifiable concentration requiring no dilution is equal to the highest calibration point. All samples analyzed above this concentration are considered "over-range" and shall require dilution for proper quantitation.
- 4.2. Likewise, the lowest quantifiable concentration of diluted samples is equal to the lowest calibration point. All diluted samples analyzed below this concentration are considered "under-range". A lower dilution factor is required for proper quantitation.
- 4.3. The linear dynamic range for this method are as follows:

Water = 1 to 20 µg/L

Soil = 5 to 666 µg/Kg

5.0 SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIME**5.1. Sample Preservation**

- 5.1.1. Store water and soil samples at 4°C (±2°C) away from light.
- 5.1.2. Store all extracts under 4°C (±2°C).

5.2. Holding Time

- 5.2.1. Extract water samples within 7 days from sampling date.
- 5.2.2. Extract soil samples within 14 days from sampling date.
- 5.2.3. Analyze all extracts within 40 days from extraction completion date.

6.0 ASSOCIATED SOPs

- 6.1. EMAX-QA04 Method Detection Limit Study
- 6.2. EMAX-QA08 Corrective Action
- 6.3. EMAX-QC02 Analytical Standard Preparation
- 6.4. EMAX-SM03 Waste Management
- 6.5. EMAX-SM04 Analytical and QC Sample Labeling
- 6.6. EMAX-3640 Clean Up, GPC
- 6.7. EMAX-3520 Extraction of Organic Compounds by Continuous Liquid/Liquid Extraction
- 6.8. EMAX-3510 Extraction of Organic Compounds by Separatory Funnel
- 6.9. EMAX-3550 Extraction of Organic Compounds from Solid Samples by Pulse Sonication
- 6.10. EMAX-3540 Soxhlet Extraction
- 6.11. EMAX-3580 Waste Dilution

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7.0 SAFETY

- 7.1. Read all MSDS for all chemicals listed in this SOP.
- 7.2. All reagents, standards, and samples shall be treated as potential hazards. Observe the standard laboratory safety procedures. Wear protective gear, i.e., lab coat, safety glasses, and gloves at all times when performing this procedure. Perform all sample and standard handling in the fume hood.
- 7.3. Place all wastes generated during analytical process in the wastes containers. Endorse these wastes to the waste disposal section for proper disposal.
- 7.4. If for any reason, solvent and/or other reagents gets in contact with your skin or any other part of your body, rinse the affected body part thoroughly with tap water. If irritations persist, inform your supervisor immediately so that proper action can be taken.

8.0 INSTRUMENTS, CHEMICALS, AND SUPPLIES**8.1. Instruments and Supplies**

| | |
|------------------------|---|
| Gas Chromatography | : HP 5890 Series II with electronic flow controller and split/splitless injection, Shimadzu GC-17A, or equivalent. |
| Mass Spectrometer | : HP5970 MSD or Shimadzu GCMS-GP5000 capable of scanning from 40 to 500 amu every 1 second using 70 volts electron energy in the electron impact ionization mode or equivalent. |
| GC/MS Interface | : The interface is capillary-direct into the mass spectrometer source or equivalent. |
| Chromatographic Column | : ZB-5MS (20m x 18mm x 18µm) |
| Data System | : MS ChemStation with Enviroquant software or equivalent. |
| GC Autosampler | : HP7673 or Shimadzu AOC-20i capable of direct injection of 1 µl and 100 µl of extract. |
| Gases | : Ultra high purity helium |
| Syringes | : 10 µl, 25 µl and 100 µl syringes; Hamilton 202N or equivalent. |

8.2. Chemicals and Reagents

- 8.2.1. Methylene chloride pesticides grade, high purity methanol and acid-washed Na₂SO₄.

9.0 STANDARDS**9.1. Standard Preparation**

- 9.1.1. Follow procedures for all standard preparations and labeling as described in EMAX-QC02 and EMAX SM04, respectively.
- 9.1.2. Other concentration levels may be prepared to meet the data quality objective of a project.

9.2. Stock Standard

- 9.2.1. Stock standards are purchased as certified solutions from Accustandard or other reputable vendor (refer to Table 1 for the listing of all certified solutions).

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9.2.2. Transfer the stock standard solutions into 2 ml amber vial with teflon lined screw caps and store at -10°C to -20°C or less and protect from the light.

9.3. Intermediate Standard

9.3.1. Prepare a 10-ml 200 $\mu\text{g/mL}$ intermediate standard (refer to Table 1 for details). Transfer the solution in a properly labeled 10-ml amber vial and store at -10°C to -20°C .

9.4. Internal Standard

9.4.1. The internal standard shall include 1,4-Dichlorobenzene-d₄, Naphthalene-d₈, Acenaphthalene-d₁₀, Phenanthrene-d₁₀, Chrysene-d₁₂ and Perylene-d₁₂ in methylene chloride solution.

9.4.2. Purchase internal standard solutions as certified solution from AccuStandard or other reputable vendor at 4,000 $\mu\text{g/mL}$.

9.4.3. Prepare a 10 ml 2000 $\mu\text{g/mL}$ working standard from 4000 $\mu\text{g/mL}$ stock internal standard.

9.4.4. Each 300 μl of sample extract undergoing analysis will be spike with 1.5 μl of the internal standard solution, resulting in a concentration of 10 $\text{g}/\mu\text{l}$ of the internal standard.

9.5. GC/MS Tuning

9.5.1. The tuning standard shall include decafluorotriphenylphosphine (DFTPP), 4,4-DDT, Pentachlorophenol, and Benzidine.

9.5.2. Purchase tuning standard solution as certified standard at 1000 $\mu\text{g/mL}$.

9.5.3. Prepare a 10 ml 50,000 $\mu\text{g/L}$ working standard tuning solution. Transfer the solution in a properly labeled 10-ml amber vial and store in -10°C to -20°C .

9.6. Surrogate Standard

9.6.1. Purchase basic neutral surrogate mixture, a stock standard as a certified standard at 5,000 $\mu\text{g/mL}$ and acid surrogate mixture stock standard as certified standard at 10,000 $\mu\text{g/mL}$.

9.6.2. Prepare a 1000-ml surrogate spiking solution by adding 10 ml of the base/neutral surrogate and 15-ml of the surrogate acid mixture in 1000 ml volumetric flask. Dilute to mark with Methanol and transfer to a properly labeled amber bottle. Store the surrogate solution at 4°C ($+ 2^{\circ}\text{C}$).

9.6.3. The resulting surrogate spiking solution will contain 50 $\mu\text{g/mL}$ of each base neutral surrogate and 150 $\mu\text{g/mL}$ of acid surrogate.

9.6.4. Spike each sample undergoing preparation with 1 ml of surrogate spiking solution resulting in a concentration of 50 $\mu\text{g/mL}$ for base neutral surrogates and 150 $\mu\text{g/mL}$ for acid surrogate.

9.7. Calibration Standard

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9.7.1. Prepare working standard solutions for initial calibration and daily calibration (refer to Table 2 for details). Transfer the solutions in 1-ml amber vial and store them at 4°C ($\pm 2^\circ\text{C}$)

9.8. ICAL Verification Standard (Second Source Verification) (ICV)

9.8.1. Purchase a certified ICV standard from a different vendor. The ICV standard contains the same list of compounds as the stock standard (refer to Table 1-B for the standard mix and the corresponding vendors).

9.8.2. Prepare a 10 ml of 200- $\mu\text{g}/\text{ml}$ check standard solution. Transfer the solution in a properly labeled 10-ml amber vial and store 4°C ($\pm 2^\circ\text{C}$).

9.9. MS/MSD/LCS/LCD Spike Standards

9.9.1. Purchase spiking standards as certified solutions.

9.9.2. Prepare 25 ml of 200 mg/L full spiking solution by adding 2.5 ml of each of Base Neutral Mix 1, B/N Mix 2, Toxic Substance Mix 1, Toxic Substance Mix 2, Phenol Mix, PAH Mix, Benzidine, Carbazole & Pyridin at 2,000 mg/L in 25 ml volumetric flask. Dilute to mark with methylene chloride and methanol (1:1 ratio).

9.9.3. Prepare short spike solution at 200 mg/L. Dilute 6 ml of 5000 mg/L Base Neutral matrix spike mix and 4 ml of 7500 mg/L Acid Spike Mix with methanol to a final volume of 150 ml..

9.9.4. Transfer the spiking solutions into a properly labeled amber bottle with Teflon lined septa cap. Store the spiking solutions at 4°C ($\pm 2^\circ\text{C}$).

9.9.5. Spike MS/MSD/LCS/LCD samples with 0.4 ml of full spiking solution prior to sample extraction.

9.9.6. Spike volume may be adjusted to normalize with the final extract volume and yield the same concentration.

10.0 PROCEDURES**10.1. Sample Preparation**

10.1.1. For aqueous samples, refer to EMAX-3510 or EMAX-3520.

10.1.2. For solid samples, refer to EMAX-3550 or EMAX-3540.

10.1.3. After extraction, examine the color and consistency of the supernatant. If the supernatant appeared to be opaque and/or viscous, the extracts should be cleaned up by GPC. Refer to EMAX-3640.

10.2. Instrument Parameters

10.2.1. Set the instrument parameters as suggested in Table 3. Fine tune the instrument to obtain optimum instrument condition.

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10.2.2. Print and display current condition on the instrument for easy access when performing daily instrument routine check.

10.2.3. In the event that instruments parameters necessitate a change, replace the instrument parameter printout with the new parameter setup and archive the previous instrument parameters in the instrument maintenance log.

10.2.4. Set injection volume to 1 μ l to 2 μ l.

10.3. Calibration

10.3.1. Set GC/MS operating condition as described in Section 10.2

10.3.2. Perform tune check

10.3.2.1. Analyze a solution containing 50- μ g/ml of tuning standard working solution.

10.3.2.2. Evaluate the tune check by either a single scan or the average of 3 scans (before, at, and after the APEX) Apply a background subtraction using a single scan no more than 20 scans prior to the elution of DFTPP. See Table-4 for acceptance criteria or follow the manufacturer's recommendation for tuning.

10.3.3. Initial Calibration (ICAL)

10.3.3.1. Perform ICAL when one of the conditions occurs.

- Instrument is new
- Instrument undergoes a major repair
- DCC failed to meet the acceptance criteria

10.3.3.2. Analyze a 5 to 9-point initial calibration curve as suggested in Figure 4 after a valid tune check.

10.3.3.3. Evaluate System Performance Check Compounds (SPCC) and calibration Check Compounds (CCC) in accordance to Appendix 1.

10.3.3.4. Check for completeness of target compound list. If there is/are missing compound(s), perform the following:

- Check the established retention time window
- Check the relative intensity of major ions
- Adjust accordingly if necessary.

10.3.3.5. Establish the relative retention time of each analyte with respect to the nearest internal standard.

10.3.3.6. Establish a summary of Relative Response Factors for each analyte at each concentration. Calculate the Average Relative Response Factor (RRF_m), the Standard Deviation (SD), and the Relative Standard Deviation (RSD) according to Eq-10.6.1.1, Eq-10.6.1.2 and Eq-10.6.1.3 and Eq-10.6.1.4 respectively.

10.3.3.7. Evaluate the ICAL for appropriate quantitation method.

- Use RRF_m - if the RSD of individual analyte \leq 15%.

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- Use RRFm - if the mean RSD of all target analyte is $\leq 15\%$ provided no individual RSD is above 30%.
- Use first order linear regression if $R > 0.995$
- Use second order regression if $COD > 0.99$ (based on six calibration points)
- Higher order regression is acceptable based on a minimum of seven calibration points.

10.3.3.8. Submit summary of ICAL, raw data and manual integration (if any) for secondary review.

10.3.4. Initial Calibration Verification (ICV)

10.3.4.1. Verify the concentration of the ICAL by analyzing the ICV from a second source (See Table 2 for standard preparation).

10.3.4.2. Check for completeness of analytes as described in 10.3.3.4.

10.3.4.3. Compare the retention times of the internal standards to the ICAL mid-point. Changes greater than 30 seconds indicates instrument malfunction. Corrective action is required prior to further analysis.

10.3.4.4. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

10.3.4.5. Check the project specific requirement (PSR) for ICV. Otherwise, refer to Appendix 1 for acceptance criteria or corrective action. Refer to Section 12 for possible measures for corrective action.

10.3.5. Daily Continuing Calibration (DCC)

10.3.5.1. Analyze DCC (See Table 2 for standard preparation) to check the validity of the ICAL.

10.3.5.2. Check SPCCs for minimum response factor (min. 0.05).

10.3.5.3. Check the CCCs for percentage difference (%Drift < 20%). If the CCCs are not included in the target analyte list, all analytes must meet the % drift criterion.

10.3.5.4. Check for completeness of analytes as described in 10.3.3.4.

10.3.5.5. Compare the retention times of the internal standards to the ICAL mid-point. Changes greater than 30 seconds indicates instrument malfunction. Corrective action is required prior to further analysis.

10.3.5.6. Compare the area of the Internal Standards (IS) acquired against the midpoint of the initial calibration point. The extracted ion current profile (EICP) must be within a factor of two (-50% to +100%).

10.3.5.7. Check the PSR for DCC. Otherwise, refer to Appendix 1 for acceptance criteria or corrective action. Refer to Section 12 for possible measures for corrective action.

10.4. **Analysis**

10.4.1. **Extract Preparation**

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- 10.4.1.1. Allow the extracts to equilibrate with the room temperature.
- 10.4.1.2. Measure 300 µl of extract, transfer into an autosampler vial.
- 10.4.1.3. Add 1.5 µl of 2000 ng/µl internal standard solution (refer to Section 9.4.3) .

10.4.2. Analytical Sequence

- 10.4.2.1. Analyze instrument blank.
- 10.4.2.2. Analyze DFTPP and evaluate tuning.
- 10.4.2.3. Analyze DCC and check ICAL validity.
- 10.4.2.4. Analyze Method Blank
- 10.4.2.5. Analyze Lab Control Sample and Lab Control Sample Duplicate (optional).
- 10.4.2.6. Analyze matrix spikes (MS/MSD) as per project requirement.
- 10.4.2.7. Analyze samples to a maximum number of 12 hours from the time of DFTPP injection.

10.4.3. Sample Result Evaluation

- 10.4.3.1. Check QC criteria
 - Check surrogate recoveries against PSR. In the absence of PSR, default to EMAX QC limits.
 - Check concentration of target analytes if calibration range is exceeded.
 - If any of the above checkpoints indicate a problem, re-analysis is required.
- 10.4.3.2. Qualitative Identification
 - The intensities of the characteristic ions must maximize in the same scan or within one scan of each other.
 - The relative retention time (RRT) of the sample component is within 0.06 RRT units of the RRT of the standard component.
 - The relative intensity of the characteristic ions agrees within 30% of the relative intensity of these ions in the reference spectrum.
 - Check the chromatogram for possible misidentified analytes. Investigate visible peaks in the chromatogram that were not identified in the data output. Manually integrate the peak if necessary. The analyst prior to submission shall initial all manual integration undertaken. All original copies will be submitted as file copies.
 - For samples containing components not associated with the calibration standards, perform a library search for purposes of tentative identification¹ (TIC).
 - Execute LSC (Chemstation program) to initiate the library search using NIST/EPA/USDC mass spectral library.

¹ Library search is performed only when indicated in the PSR.

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- Visually inspect each extracted mass ion chromatograph to determine the identification of the unknown before final reporting.

10.4.3.3. Quantitation

- Apply the appropriate quantitation method (Section 10.6.3) to calculate the concentration of any positively identified target analyte. Use the dilution factor for diluted samples to calculate for the final concentration of the sample.

10.4.3.4. Manual Integration

10.4.3.4.1. Refer to EMAX-DM01 for details of manual integration.

10.4.3.5. Dealing with Carryover

- Check the sample analyzed after a sample having target analyte concentrations exceeding the calibration range.
- If there is no target analyte detected as found in the sample that exceeded the calibration range, proceed with data reduction.
- If there is any target analyte detected as found in the sample that exceeded the calibration range, re-analyze the sample to rule-out carry over. If carry over is confirmed, proceed with data reduction and report the data from re-analysis.

10.5. Data Reduction

10.5.1.1. Make a copy of the analytical run log and highlight the data to be reported.

10.5.1.2. Collate the reportable raw data separating the QC results from the sample results.

10.5.1.3. Keep all other data generated with the analytical folder marked with "For record only".

10.5.1.4. Proceed to report generation.

10.6. Calculations

10.6.1. Initial Calibration

10.6.1.1. Calculate for Relative Response Factor (RRF)

$$RRF = \frac{A_x C_{is}}{A_{is} C_x} \quad \text{Eq.-10.6.1.1}$$

where:

- A – Area of characteristic ion for the compound being measured
- A_{is} – Area of characteristic ion for the specific internal standard
- C_x – Concentration of the compound being measured
- C_{is} – Concentration of the specific internal standard

10.6.1.2. Calculate for Average Relative Response Factor (RRF_m).

$$RRF_m = \frac{\sum RRF}{n} \quad \text{Eq-10.6.1.2}$$

where:

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RRF_m – average response factor
 $\sum RRF$ – summation of response factors
 n – number of measurements

10.6.1.3. Calculate for Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1}} \quad \text{Eq.-10.6.1.3}$$

where:

SD – standard deviation
 x_i – result at i^{th} measurement
 \bar{x} – mean
 n – number of measurements

10.6.1.4. Calculate for % relative standard deviation (%RSD).

$$\%RSD = \frac{SD}{RRF_m} * 100\% \quad \text{Eq-10.6.1.4}$$

where:

SD – standard deviation
 RRF_m – average response factor

10.6.2. Calibration Check/Continuing Calibration

10.6.2.1. Calculate Percent Difference (%D) when RRF_m is used for quantitation

$$\%D = \frac{[RRF_c - RRF_m]}{RRF_m} * 100\% \quad \text{Eq-10.6.2.1}$$

where:

RRF_c – response factor from continuing calibration standard
 RRF_m – average response factor

10.6.2.2. Calculate Percent Deviation (%D_t) when the first order linear regression or the second order regression is used for quantitation.

$$\%D_t = \frac{\text{abs}(T_t - T_f)}{T_t} * 100\% \quad \text{Eq-10.6.2.2}$$

where:

abs – absolute value
 T_t – true value of standard in $\mu\text{g/L}$
 T_f – found value of standard in $\mu\text{g/L}$

10.6.3. Calculation of Sample Concentration (Water and Soil/Sediment Samples). When a compound is identified, the quantitation of that compound shall be based on the integrated abundance from the EICP of the primary characteristic ion.

10.6.3.1. Water Samples

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$$\text{Concentration (ug/L)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(V_t)} \quad \text{Eq-10.6.3.1}$$

where:

- A_x – area of characteristic ion for the compound to be measured
 I_s – amount of internal standard added
 V_e – extract final volume from sample extraction, usually 1-ml
 DF – dilution factor = $\frac{\text{aliquot}(\text{mL}) + \text{solvent}(\text{mL})}{\text{aliquot}(\text{mL})}$
 A_{is} – area of characteristic ion for the internal standard
 RRF_m – average response factor
 V_i – volume of extract injected in μL , usually 1- μL
 V_t – volume of water extracted in ml, usually 1000-ml

10.6.3.2. Soil/Sediment Samples (Dry weight basis)

$$\text{Concentration (ug/Kg)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} \quad \text{Eq-10.6.3.2}$$

where:

- A_x – area of characteristic ion for the compound to be measured
 I_s – amount of internal standard injected in ng
 V_e – volume of extract in ml, usually 1-ml²
 DF – dilution factor = $\frac{\text{aliquot}(\text{mL}) + \text{solvent}(\text{mL})}{\text{aliquot}(\text{mL})}$
 A_{is} – area of characteristic ion for the internal standard
 RRF_m – average response factor
 V_i – volume of extract injected in μL , usually 1- μL
 W_s – wet soil weight in kg
 DW – % solid = $\frac{100 - \% \text{moisture}}{100}$

10.6.3.3. Extract is cleaned up by GPC

$$\text{Concentration (ug/Kg)} = \frac{(A_x)(I_s)(V_e)(DF)}{(A_{is})(RRF_m)(V_i)(W_s)(DW)} * \frac{V_{bg}}{V_{ig}} \quad \text{Eq-10.6.3.3}$$

where:

- A_x – area of characteristic ion for the compound to be measured
 I_s – amount of internal standard injected in ng

² For extracts subjected to GPC $V_i=0.5\text{-ml}$

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| | |
|----------|---|
| V_e | – volume of extract in ml |
| DF | – dilution factor = $\frac{\text{aliquot}(\text{mL}) + \text{solvent}(\text{mL})}{\text{aliquot}(\text{mL})}$ |
| A_{is} | – area of characteristic ion for the internal standard |
| RRF_m | – average response factor |
| V_i | – volume of extract injected in μL , usually 1- μL |
| W_s | – wet soil weight in kg |
| DW | – % solid = $\frac{100 - \% \text{moisture}}{100}$ |
| V_{bg} | – total volume of extract before GPC clean-up in mL |
| V_{ig} | – injected volume of extract to GPC in mL |

10.6.4. Alternatively, the regression line (area ratio of A_x/A_{is} versus concentration using first degree or higher regression) fitted to the initial calibration may be used for determination of the sample concentration when RSD of the analyte is greater than 15.

10.6.5. Concentration of TIC is estimated by the same method as target compounds with the following assumptions:

10.6.5.1. The area A_x and A_{is} are derived from total ion chromatogram. A_{is} refers to the closest internal standard (IS) free of interference.

10.6.5.2. RRF of the TIC is 1.

10.6.6. Accuracy and Precision

10.6.6.1. Percent Recovery

$$\% \text{ Recovery} = \frac{C_f - C}{C_s} * 100 \quad \text{Eq-10.6.6.1}$$

where:

| | |
|-------|---------------------------|
| C_f | – concentration found |
| C | – concentration of sample |
| C_s | – concentration of spike |

10.6.6.2. Relative Percent Difference (%RPD)

$$\%RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100 \quad \text{Eq-10.6.6.2}$$

where:

| | |
|-------|---|
| RPD | – Relative Percent Difference |
| C_1 | – Measured concentration of the first sample aliquot |
| C_2 | – Measured concentration of the second sample aliquot |

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10.7. Report Generation, Data Reduction & Review

- 10.7.1. Generate the method.txt file using WDB1C.exe
- 10.7.2. Generate Lab Chronicle using Labchron1.exe
- 10.7.3. Generate the sample results using F1V3C.exe
- 10.7.4. Generate the QC summary using QCV3C.exe
- 10.7.5. Arrange the analysis package in sequence as detailed below using section separators. Attach all raw data to every form generated, to include manual integration and re-analyses.
 - Sample Results
 - LCS Summary
 - MS/MSD Summary
 - DCC Summary
 - ICV Summary
 - ICAL Summary
- 10.7.6. Perform a 100% data review in accordance to EMAX-DM01 and the PSR.
- 10.7.7. Generate the case narrative to include discussion of the following as found in the review process:
 - Number of samples analyzed
 - Analytical method(s) applied
 - Holding Time – That samples extracted and analyzed within the holding time. For non-compliance, state the number of days that the sample(s) was out of holding time.
 - Initial Calibration – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Calibration Verifications – That all target analytes met calibration requirements. For non-compliance, state the analyte(s) that is non-compliant and the data qualified.
 - Method Blank – That MB was extracted with the samples and analyzed at a frequency specified by the project and that results are compliant to project requirement. For non-compliance, state the analyte(s) affected and the associated sample results were flagged with “B”.
 - Surrogate – That surrogate was added to MB, LCS, MS/MSD (if applicable) and every sample prior to analysis, and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. sample result form or QC Summary form, and that non-compliant results were indicated by an asterisk “*”.
 - Lab Control Samples (if applicable) – That LCS was prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the associated forms, e.g. Sample result form or QC Summary form, and state that non-compliant results were indicated by an asterisk “*”. Further more, if corrective action is not possible (e.g. no more samples to re-extract) state that results were qualified.

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- Matrix Spike Samples – That MS/MSD (if applicable) were prepared with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limits. For non-compliance, reference the MS Summary form, and that non-compliant results were indicated by an asterisk “*”.
- Sample Analysis – that samples were analyzed in conformance to the method and project requirements.
- Other Anomalies (if any) – discussed on a case by case basis concurred by the Supervisor or the Lab Director.

10.7.8. Submit the analysis package for secondary review.

10.8. Preventive Maintenance

10.8.1. Refer to form 8270SIMFM for daily routine maintenance check points.

10.8.2. Record instrument maintenance performed in the instrument maintenance log. Initial the column corresponding to the date when the instrument was back to control.

10.8.3. Instruments should receive routine preventive maintenance and recorded in instrument-specific maintenance logs. Routine maintenance ensures that all equipment is operating under optimum conditions, thus reducing the possibility of instrument malfunction and consequently affecting data quality.

11.0 QUALITY CONTROL**11.1. Preparative Batch**

11.1.1. A preparative batch shall consist of a method blank, LCS, MS/MSD (when required by the project) and a maximum of 20 field samples of similar matrix.

11.1.2. In the absence of MS/MSD, prepare LCS/LCD to check for precision.

11.2. Analytical Batch QC

11.2.1. The GC/MS tuning standard must be analyzed at the beginning of every 12-hour shift. GC/MS tuning criteria is listed in Table 4 and Section 10.3.2.

11.2.2. A continuing calibration shall be performed before any other analysis is done, and after analysis of tuning standard. The continuing calibration procedure and the acceptance criteria are discussed in Section 10.3.6.

11.3. Method QC

11.3.1. Analyst demonstration of proficiency is a must prior to performing this analysis.

11.3.2. A valid MDL must exist prior to sample analysis.

11.3.3. A valid ICAL must exist prior to sample analysis.

11.3.4. Instrument performance must be checked prior to sample analysis.

11.3.5. Check Appendix 1 for acceptance criteria.

11.3.6. Prepare and analyze QC samples, to include, method blank, LCS (LCD), and MS/MSD. QC Control Limits shall follow the Project Specific Requirement (PSR) in each analytical folder.

11.3.7. Surrogate standard shall be added to all samples, including method blank LCS/LCD and

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Method 8270SIMSOP No.: EMAX-8270SIMRevision No. 1Effective Date: 15-Nov-06

MS/MSD. Check PSR for QC Control Limits.

12.0 CORRECTIVE ACTION

- 12.1. Corrective actions associated with this analytical procedure are described in the Summary of Quality Control Procedures in Appendix 1. Document out-of-control event and corrective action in the analytical logbook. If the problem persists, consult the supervisor.
- 12.2. If the tune is non-compliant, consider the following suggestions to correct the problem:
 - 12.2.1. Check the instrument settings and make sure that the instrument parameters are properly set up
 - 12.2.2. Check gas flow
 - 12.2.3. Perform auto tune or visual optimization
 - 12.2.4. If the problem persists, inform the supervisor
- 12.3. If initial calibration is non-compliant, consider the following suggestions to correct the problem:
 - 12.3.1. If %RSD is out of acceptance criteria, review result and identify presence of an outlier.
 - 12.3.2. If one of the standard returns a bias-low or bias-high on all of the analytes, then that point is considered an outlier, prepare a standard at that ICAL point and re-analyze.
 - 12.3.3. If the highest ICAL point appears to be saturated, drop the highest point.
 - 12.3.4. If the lowest point returns a bias-low response or the peaks are not distinct and sharp, consider the point not usable.
Note: The lowest calibration point identifies the reporting limit (RL). Therefore, check that the RL is in conformance to the current projects where the ICAL will be used.
 - 12.3.5. If instrumentation problem is suspected, consider the following suggestions to correct the problem:
 - 12.3.5.1. Check the connections and make sure they are air-tight and perform maintenance as needed.
 - 12.3.5.2. Check the gas flow
 - 12.3.5.3. Re-tune the MS
 - 12.3.5.4. Prepare a fresh standard and repeat calibration
 - 12.3.5.5. Clean the MS source and repeat the calibration
 - 12.3.6. If the problem persists, inform the supervisor.
- 12.4. If the ICV is non-compliant, consider the following suggestions to correct the problem:
 - 12.4.1. Re-analyze ICV (to rule out poor injection)
 - 12.4.2. If ICV is still out of acceptance criteria, prepare a fresh standard and re-analyze to rule out any preparation error
 - 12.4.3. If ICV is still out of acceptance criteria, prepare a fresh ICAL standard and repeat calibration
 - 12.4.4. If the problem persists, inform the supervisor

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- 12.5. If the instrument blank is non-compliant, consider the following suggestions to correct the problem:
- 12.5.1. Rule out instrument contamination by performing the daily instrument maintenance, such as changing septum, cleaning liner, cleaning or using new autosampler syringe.
 - 12.5.2. Rule out reagent contamination by testing solvent used for analysis and working internal standard.
 - 12.5.3. Rule out preparation contamination by preparing a new instrument blank
 - 12.5.4. If the problem persists, inform the supervisor.
- 12.6. If Continuing Calibration is non-compliant, consider the following suggestions to correct the problem:
- 12.6.1. Change the liner
 - 12.6.2. Clean injection port
 - 12.6.3. Prepare new standard
 - 12.6.4. Cut or replace column
 - 12.6.5. Rule out leaks by checking all connections
 - 12.6.6. If continuing calibration is still non-compliant, prepare a new standard and repeat the ICAL
- 12.7. If Method Blank is non-compliant, consider the following suggestions to correct the problem:
- 12.7.1. Rule out instrument contamination by checking instrument blank
 - 12.7.2. Rule out reagent contamination by testing each reagent used for extraction as described in EMAX-QC01
 - 12.7.3. Rule out glassware contamination used for extraction as described in EMAX-QC07
 - 12.7.4. Re-extract MB and the associated samples with reagents free of contamination or with newly opened reagents
 - 12.7.5. If the problem persists, inform the supervisor
- 12.8. If LCS is non-compliant, perform the following suggestions to correct the problem:
- 12.8.1. If result is bias-high or bias-low, check the LCS Standard by analyzing at the spike level
 - 12.8.2. If LCS check is within 80-120% of expected value, check calibration of the micropipette or syringe used for spiking. Re-extract and reanalyze the LCS and the associated samples.
 - 12.8.3. If LCS check is not within 80-120% of expected value, prepare a fresh LCS Standard, re-extract and re-analyze LCS and the associated samples.
- 12.9. Execute a Non-Conformance Report (NCR) when the following circumstances occur:
- 12.9.1. If corrective action needs the function of other department; e. g. if the sample needs to be re-extracted, refer to EMAX-QA08 for details of completing an NCR.
 - 12.9.2. If corrective action needs the assistance of the project manager; e. g. if the sample is non-compliant to the technical holding time requirement, insufficient amount of sample, or other non-conforming issues.
- 12.10. For other problems encountered, inform the supervisor immediately for further instructions.

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13.0 POLLUTION PREVENTION

- 13.1. All unused samples shall be endorsed to the Waste Disposal Unit (WDU) for proper disposal. No samples shall be dumped on the laboratory sink.
- 13.2. All unused, expired analytical standards shall be separated and properly identified prior to endorsing them to WDU for proper disposal.

14.0 WASTE MANAGEMENT

- 14.1. All unused samples, expired analytical standards and other waste generated during the analytical process, endorsed to WDU shall be disposed in accordance to EMAX-SM03.

15.0 SUPPLEMENTARY NOTES**15.1. Definition of Terms**

- 15.1.1. Batch – is a group of samples that are prepared and/or analyzed at the same time using the same lot of reagents. **Preparation batch** is composed of one to 20 samples of the same matrix, a method blank, a lab control sample and matrix spike/matrix spike duplicate. **Analytical batch** is composed of prepared samples (extracts, digestates, or concentrates), which are analyzed together as a group using an instrument in conformance to the analytical requirement. An analytical batch can include samples originating from various matrices, preparation batches, and can exceed 20 samples.
- 15.1.2. Calibration – is a determinant measured from a standard to obtain the correct value of an instrument output.
- 15.1.3. Duplicate Sample – is a replicate of a sub-sample taken from one sample, prepared and analyzed within the same preparation batch.
- 15.1.4. Instrument Method – is a file generated to contain the instrument calibration and instrument parameter settings for a particular analysis.
- 15.1.5. Instrument Blank – is a target-analyte-free solvent subjected to the entire analytical process to establish zero baseline or background value.
- 15.1.6. Lab Control Sample (LCS) – is a target-analyte-free sample spiked with a verified known amount of target analyte(s) or a reference material with a certified known value subjected to the entire sample preparation and/or analytical process. LCS is analyzed to monitor the accuracy of the analytical system.
- 15.1.7. Lab Control Sample Duplicate (LSD) – is a replicate of LCS analyzed to monitor precision when MS/MSD samples are not analyzed.
- 15.1.8. Matrix – is a component or form of a sample.
- 15.1.9. Matrix Spike (MS) – is a sample spiked with a verified known amount of target analyte(s) subjected to the entire sample preparation and/or analytical process. MS is analyzed to monitor matrix effect on a method's recovery efficiency.
- 15.1.10. Matrix Spike Duplicate (MSD) – is a replicate of MS analyzed to monitor precision or recovery.

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- 15.1.11. Method Blank – is a target-analyte-free sample subjected to the entire sample preparation and/or analytical to monitor contamination.
- 15.1.12. Sample – is a specimen received in the laboratory bearing a sample label traceable to the accompanying COC. Samples collected in different containers having the same field sample ID are considered the same and therefore labeled with the same lab sample ID unless otherwise specified by the project.
- 15.1.13. Sub-sample – is an aliquot taken from a sample for analysis. Each sub-sample is uniquely identified by the sample preparation ID.

15.2. Application of QC Procedures

- 15.2.1. The procedures and QC criteria summarized in this SOP shall be applied to all projects when performing semi-volatile analysis by GC/MS. In instances where there is a project or program specific quality control, the requirements given in the project shall take precedence over this SOP.

15.3. Air Force Center for Environmental Excellence (AFCEE) Projects

- 15.3.1. When samples from AFCEE sponsored projects are analyzed for semi-volatiles by GC/MS, the Quality Assurance Project Plan shall be applied otherwise default to the current version of AFCEE QAPP.

15.4. U.S. Army Corps of Engineers (USACE) Projects

- 15.4.1. In the absence of project QAPP, the default QAPP is the Shell Document, latest version.

15.5. Naval Facilities Engineering Service Center (NFESC) Projects

- 15.5.1. In the absence of project QAPP, the default QAPP is the NFESC Interim Guidance Document, latest version.

15.6. Department of Energy Basic Ordering Agreement (DOE-BOA) Projects

- 15.3.1. For samples from DOE-BOA sponsored projects follow BOA Guidance Document, latest version in the absence of project QAPP.

16.0 REFERENCES

- 16.1. “Test Methods for Evaluation of Solid Wastes”, EPA SW846, as updated.
- 16.2. Laboratory QA/QC Manual, as updated.

17.0 FIGURES, TABLES AND APPENDICES**17.1. Figures**

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- 17.1.2. Figure 2 Typical Chromatogram
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Figure 1 - Peak Evaluation Technique

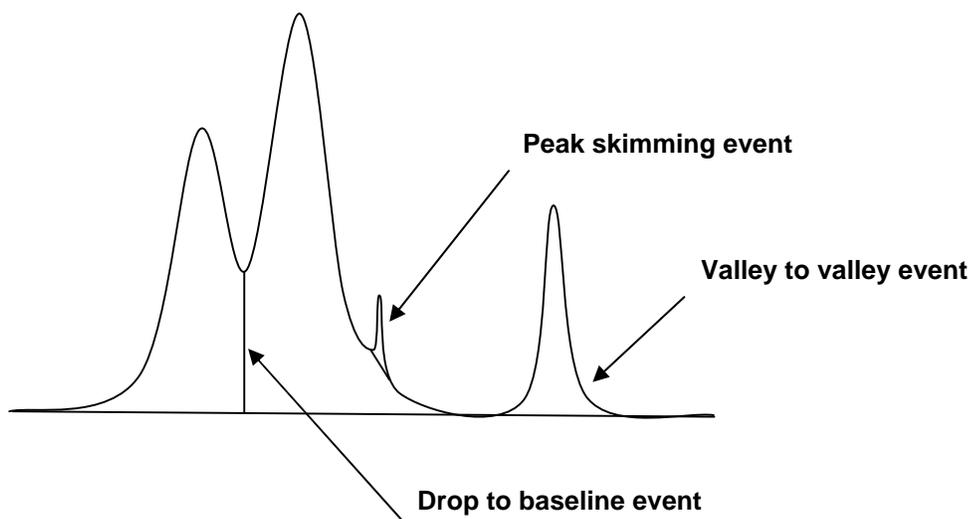
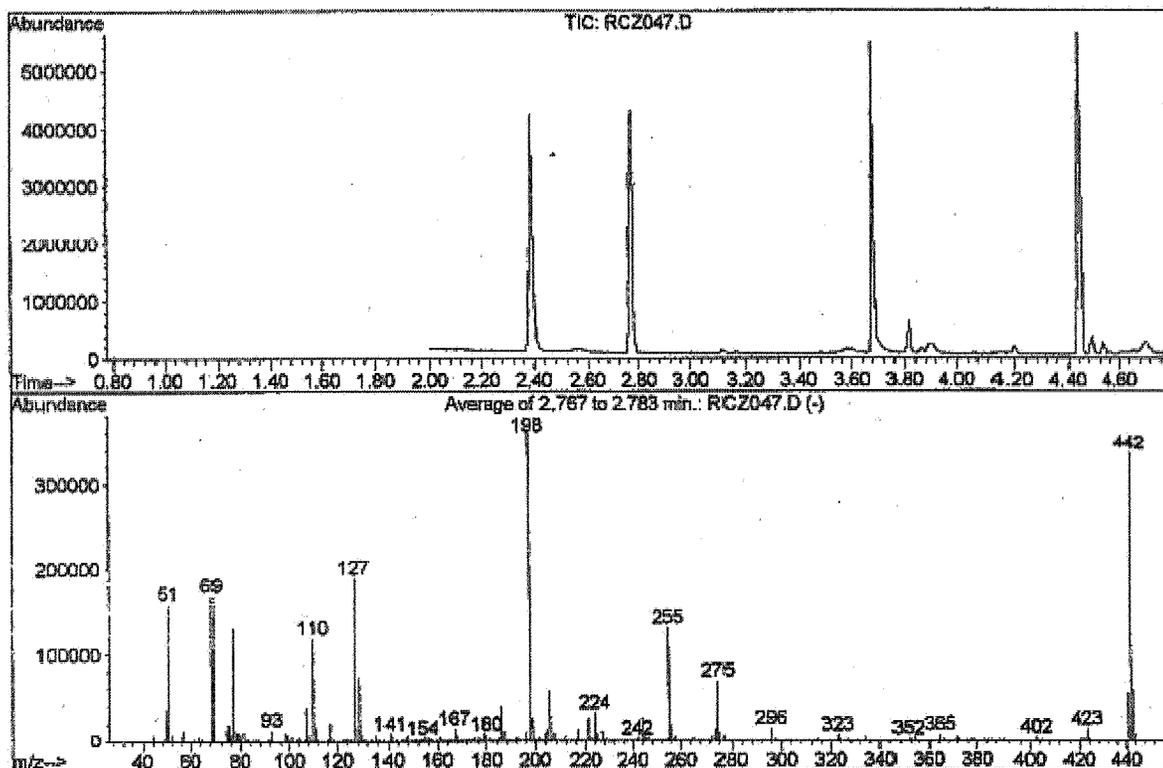


FIGURE 3 - TYPICAL DFTPP CHROMATOGRAM

DFTPP

Data File : D:\CHEMDATA\06C02\RCZ047.D
 Acq On : 2 MAR 2006 19:31
 Sample : DFT48C0201
 Misc :
 MS Integration Params: rteint.p
 Method : C:\HPCHEM\1\METHODS\DFTPPSIM.M (RTE Integrator)
 Title : DFTPP TUNE 8270C SHIMADZU GCMS-QP5000

Vial: 2
 Operator: KV
 Inst : TO48
 Multiplr: 1.00



AutoFind: Scans 93, 94, 95; Background Corrected with Scan 89

| Target Mass | Rel. to Mass | Lower Limit% | Upper Limit% | Rel. Abn% | Raw Abn | Result Pass/Fail |
|-------------|--------------|--------------|--------------|-----------|---------|------------------|
| 51 | 198 | 30 | 60 | 43.8 | 159512 | PASS |
| 68 | 69 | 0.00 | 2 | 0.3 | 548 | PASS |
| 69 | 198 | 0.00 | 100 | 46.4 | 168752 | PASS |
| 70 | 69 | 0.00 | 2 | 0.5 | 795 | PASS |
| 127 | 198 | 40 | 60 | 51.9 | 188901 | PASS |
| 197 | 198 | 0.00 | 1 | 0.8 | 2825 | PASS |
| 198 | 198 | 100 | 100 | 100.0 | 363768 | PASS |
| 199 | 198 | 5 | 9 | 7.2 | 26133 | PASS |
| 275 | 198 | 10 | 30 | 18.8 | 68232 | PASS |
| 365 | 198 | 1 | 100 | 1.6 | 5897 | PASS |
| 441 | 443 | 0.01 | 100 | 93.2 | 54259 | PASS |
| 442 | 198 | 40 | 100 | 92.3 | 335890 | PASS |
| 443 | 442 | 17 | 23 | 17.3 | 58248 | PASS |

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FIGURE 4 – TYPICAL ICAL SUMMARY

INITIAL CALIBRATION - RELATIVE RESPONSE FACTOR

Instrument ID :TO48
Beginning DateTime :03/02/06 19:43
Spike Units :PPM
IC File :RCZ053

Column Spec :ZB-5MS ID :0.18MM
Ending DateTime :03/02/06 22:35
HPCChem Method :SV48C02

| IDX | Parameters | .15 | .5 | 1 | 2 | 5 | 10 | 20 | 40 | 80 | 100 | Av_RRF | %_RSD | Av_Rt_M |
|-----|----------------------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|--------|-------|---------|
| | | 19:43 RCZ048 | 20:02 RCZ049 | 20:21 RCZ050 | 20:40 RCZ051 | 20:59 RCZ052 | 21:18 RCZ053 | 21:37 RCZ054 | 21:57 RCZ055 | 22:16 RCZ056 | 22:35 RCZ057 | | | |
| 1 | 1,4-Dichlorobenzene-d4 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 2.7525 |
| 2 | N-Nitrosodimethylamine | 1.600 | 1.904 | 1.753 | 1.843 | 1.787 | 1.722 | 1.731 | ----- | ----- | ----- | 1.763 | 5.50 | 1.2917 |
| 3 | Phenol-d5 | 1.524 | 1.463 | 1.521 | 1.480 | 1.611 | 1.593 | 1.578 | 1.314 | 1.419 | 1.509 | 1.501 | 5.92 | 2.4742 |
| 4 | Phenol | 1.706 | 1.621 | 1.671 | 1.637 | 1.793 | 1.854 | 1.653 | ----- | ----- | ----- | 1.705 | 5.10 | 2.4881 |
| 5 | Bis(2-chloroethyl)ether | 1.333 | 1.431 | 1.310 | 1.382 | 1.352 | 1.362 | 1.473 | ----- | ----- | ----- | 1.378 | 4.14 | 2.5679 |
| 6 | 2-Chlorophenol | 1.589 | 1.608 | 1.664 | 1.577 | 1.727 | 1.766 | 1.767 | ----- | ----- | ----- | 1.671 | 4.94 | 2.5976 |
| 7 | N-Nitroso-di-n-propylamine | 0.661 | 0.632 | 0.600 | 0.642 | 0.667 | 0.686 | 0.693 | ----- | ----- | ----- | 0.655 | 4.97 | 3.0691 |
| 8 | 2,4-Dimethylphenol | 1.103 | 1.139 | 1.059 | 1.033 | 1.075 | 1.004 | 1.019 | ----- | ----- | ----- | 1.062 | 4.54 | 3.5071 |
| 9 | 2,4-Dichlorophenol | 1.186 | 1.018 | 1.070 | 1.033 | 1.103 | 1.138 | 1.155 | ----- | ----- | ----- | 1.100 | 5.75 | 3.6643 |
| 10 | Naphthalene | 3.878 | 3.713 | 3.770 | 3.736 | 3.797 | 3.717 | 3.752 | ----- | ----- | ----- | 3.766 | 1.53 | 3.8095 |
| 11 | 4-Chloro-3-methylphenol | 1.196 | 1.028 | 1.050 | 0.998 | 1.102 | 1.074 | 1.028 | ----- | ----- | ----- | 1.068 | 6.15 | 4.3071 |
| 12 | 2-Methylnaphthalene | 2.213 | 2.131 | 2.085 | 1.991 | 2.160 | 2.151 | 2.189 | ----- | ----- | ----- | 2.131 | 3.48 | 4.4321 |
| 13 | 1-Methylnaphthalene | 1.914 | 1.833 | 1.882 | 1.813 | 1.925 | 1.782 | 1.753 | ----- | ----- | ----- | 1.843 | 3.58 | 4.5155 |
| 14 | 2,4,6-Trichlorophenol | 0.640 | 0.551 | 0.613 | 0.564 | 0.664 | 0.611 | 0.595 | ----- | ----- | ----- | 0.605 | 6.57 | 4.6821 |
| 15 | 2,4,5-Trichlorophenol | 0.681 | 0.548 | 0.582 | 0.529 | 0.630 | 0.611 | 0.613 | ----- | ----- | ----- | 0.599 | 8.56 | 4.7071 |
| 16 | Acenaphthylene | 3.599 | 2.950 | 2.975 | 2.807 | 3.062 | 2.984 | 2.914 | ----- | ----- | ----- | 3.041 | 8.47 | 5.2405 |
| 17 | Acenaphthene | 2.435 | 1.795 | 1.735 | 1.731 | 1.760 | 1.711 | 1.771 | ----- | ----- | ----- | 1.848 | 14.08 | 5.4047 |
| 18 | Fluorene | 2.469 | 1.907 | 1.987 | 1.919 | 2.069 | 1.975 | 1.913 | ----- | ----- | ----- | 2.034 | 9.84 | 5.8821 |
| 19 | Azobenzene | 2.558 | 1.982 | 2.108 | 2.083 | 2.233 | 2.222 | 2.112 | ----- | ----- | ----- | 2.185 | 8.47 | 6.0500 |
| 20 | Phenanthrene-d10 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 6.7434 |
| 21 | Hexachlorobenzene | 0.467 | 0.338 | 0.350 | 0.350 | 0.356 | 0.354 | 0.332 | ----- | ----- | ----- | 0.364 | 12.70 | 6.3821 |
| 22 | Pentachlorophenol | ----- | 0.650 | 0.727 | 0.698 | 0.752 | 0.758 | 0.759 | ----- | ----- | ----- | 0.724 | 5.94 | 6.5722 |
| 23 | Phenanthrene | 2.089 | 1.543 | 1.526 | 1.547 | 1.563 | 1.546 | 1.582 | ----- | ----- | ----- | 1.628 | 12.53 | 6.7691 |
| 24 | Anthracene | 2.019 | 1.507 | 1.556 | 1.560 | 1.563 | 1.603 | 1.592 | ----- | ----- | ----- | 1.628 | 10.73 | 6.8167 |
| 25 | Fluoranthene | 1.678 | 1.240 | 1.266 | 1.304 | 1.357 | 1.346 | 1.431 | ----- | ----- | ----- | 1.375 | 10.76 | 7.8750 |
| 26 | Pyrene | 1.839 | 1.334 | 1.369 | 1.342 | 1.438 | 1.423 | 1.399 | ----- | ----- | ----- | 1.449 | 12.17 | 8.0821 |
| 27 | Terphenyl-d14 | 0.684 | 0.491 | 0.474 | 0.450 | 0.499 | 0.510 | 0.504 | 0.537 | 0.572 | 0.522 | 0.524 | 12.43 | 8.2767 |
| 28 | Perylene-d12 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 0 | 10.4600 |
| 29 | Benzo(a)anthracene | 2.555 | 2.048 | 1.915 | 1.975 | 1.976 | 1.959 | 2.087 | ----- | ----- | ----- | 2.074 | 10.60 | 9.2179 |
| 30 | Chrysene | 2.533 | 2.191 | 2.083 | 2.146 | 2.125 | 2.088 | 2.034 | ----- | ----- | ----- | 2.171 | 7.71 | 9.2500 |
| 31 | bis(2-Ethylhexyl)phthalate | 2.558 | 2.102 | 2.213 | 2.327 | 2.526 | 2.498 | 2.555 | ----- | ----- | ----- | 2.397 | 7.68 | 9.3536 |
| 32 | Benzo(b)fluoranthene | 1.894 | 1.569 | 1.553 | 1.749 | 1.799 | 1.889 | 2.055 | ----- | ----- | ----- | 1.787 | 10.15 | 10.1571 |
| 33 | Benzo(k)fluoranthene | 2.453 | 1.987 | 2.075 | 2.058 | 2.082 | 2.101 | 1.744 | ----- | ----- | ----- | 2.072 | 10.07 | 10.1809 |
| 34 | Benzo(a)pyrene | 1.929 | 1.529 | 1.619 | 1.880 | 1.896 | 1.926 | 1.982 | ----- | ----- | ----- | 1.823 | 9.60 | 10.4167 |
| 35 | Indeno(1,2,3-cd)pyrene | 2.456 | 1.762 | 1.986 | 2.182 | 2.175 | 2.276 | 2.383 | ----- | ----- | ----- | 2.174 | 10.93 | 11.2691 |
| 36 | Dibenzo(a,h)anthracene | 1.771 | 1.305 | 1.440 | 1.605 | 1.626 | 1.697 | 1.784 | ----- | ----- | ----- | 1.604 | 10.98 | 11.2917 |
| 37 | Benzo(g,h,i)perylene | 2.431 | 1.683 | 1.932 | 2.001 | 2.000 | 2.027 | 2.047 | ----- | ----- | ----- | 2.017 | 10.94 | 11.4583 |

Ave_%RSD : 8.2 Max_%RSD : 14.1

FIGURE 5 – TYPICAL ICV SUMMARY

CONTINUE_CALIBRATION - CALIBRATION VERIFICATION

Instrument ID :TO48
IC_Beginning DateTime :03/02/06 19:43
Spike Amount :10 PPM
CC/CV File :REZ050
IC File :RCZ053

Column Spec :ZB-5MS ID :0.18MM
IC_Ending DateTime :03/02/06 22:35
HPChem Method :SV48C02
Date_Time :05/05/06 15:38

| IDX | Parameters | CC_Con | CC%_D | CC_Resp | CCRRF | AvRRF | CC_Rtm | AvRtm | %_RSD | Co_X0 | Co_X1 | Co_X2 | Co_Cor |
|-----|----------------------------|--------|-------|---------|-------|-------|--------|--------|-------|-------|-------|-------|--------|
| 1 | 1,4-Dichlorobenzene-d4 | 10.000 | 0 | 196265 | 1 | 1 | 2.758 | 2.753 | 0 | | | | |
| 2 | N-Nitrosodimethylamine | 10.619 | 6.2 | 367373 | 1.872 | 1.763 | 1.325 | 1.292 | 5.50 | | | | |
| 3 | Phenol-d5 | 11.197 | 12.0 | 329908 | 1.681 | 1.501 | 2.492 | 2.474 | 5.92 | | | | |
| 4 | Phenol | 11.836 | 18.4 | 396096 | 2.018 | 1.705 | 2.508 | 2.488 | 5.10 | | | | |
| 5 | Bis(2-chloroethyl)ether | 10.017 | 0.2 | 270855 | 1.380 | 1.378 | 2.575 | 2.568 | 4.14 | | | | |
| 6 | 2-Chlorophenol | 9.695 | -3.1 | 317998 | 1.620 | 1.671 | 2.608 | 2.598 | 4.94 | | | | |
| 7 | N-Nitroso-di-n-propylamine | 9.448 | -5.5 | 121402 | 0.619 | 0.655 | 3.075 | 3.069 | 4.97 | | | | |
| 8 | 2,4-Dimethylphenol | 9.853 | -1.5 | 205340 | 1.046 | 1.062 | 3.517 | 3.507 | 4.54 | | | | |
| 9 | 2,4-Dichlorophenol | 10.076 | 0.8 | 217605 | 1.109 | 1.100 | 3.675 | 3.664 | 5.75 | | | | |
| 10 | Naphthalene | 9.489 | -5.1 | 701435 | 3.574 | 3.766 | 3.817 | 3.809 | 1.53 | | | | |
| 11 | 4-Chloro-3-methylphenol | 10.013 | 0.1 | 209933 | 1.070 | 1.068 | 4.325 | 4.307 | 6.15 | | | | |
| 12 | 2-Methylnaphthalene | 9.945 | -0.6 | 416031 | 2.120 | 2.131 | 4.433 | 4.432 | 3.48 | | | | |
| 13 | 1-Methylnaphthalene | 10.072 | 0.7 | 364378 | 1.857 | 1.843 | 4.517 | 4.516 | 3.58 | | | | |
| 14 | 2,4,6-Trichlorophenol | 7.660 | -23.4 | 91005 | 0.464 | 0.605 | 4.692 | 4.682 | 6.57 | | | | |
| 15 | 2,4,5-Trichlorophenol | 9.244 | -7.6 | 108689 | 0.554 | 0.599 | 4.725 | 4.707 | 8.56 | | | | |
| 16 | Acenaphthylene | 9.965 | -0.3 | 594820 | 3.031 | 3.041 | 5.242 | 5.240 | 8.47 | | | | |
| 17 | Acenaphthene | 9.477 | -5.2 | 343801 | 1.752 | 1.848 | 5.408 | 5.405 | 14.08 | | | | |
| 18 | Fluorene | 9.688 | -3.1 | 386769 | 1.971 | 2.034 | 5.892 | 5.882 | 9.84 | | | | |
| 19 | Azobenzene | 9.182 | -8.2 | 393838 | 2.007 | 2.185 | 6.050 | 6.050 | 8.47 | | | | |
| 20 | Phenanthrene-d10 | 10.000 | 0 | 310403 | 1 | 1 | 6.750 | 6.743 | 0 | | | | |
| 21 | Hexachlorobenzene | 10.074 | 0.7 | 113742 | 0.366 | 0.364 | 6.392 | 6.382 | 12.70 | | | | |
| 22 | Pentachlorophenol | 8.800 | -12.0 | 197795 | 0.637 | 0.724 | 6.592 | 6.572 | 5.94 | | | | |
| 23 | Phenanthrene | 9.230 | -7.7 | 466468 | 1.503 | 1.628 | 6.783 | 6.769 | 12.53 | | | | |
| 24 | Anthracene | 9.813 | -1.9 | 496035 | 1.598 | 1.628 | 6.825 | 6.817 | 10.73 | | | | |
| 25 | Fluoranthene | 9.663 | -3.4 | 412293 | 1.328 | 1.375 | 7.892 | 7.875 | 10.76 | | | | |
| 26 | Pyrene | 9.090 | -9.1 | 408897 | 1.317 | 1.449 | 8.100 | 8.082 | 12.17 | | | | |
| 27 | Terphenyl-d14 | 10.589 | 5.9 | 172331 | 0.555 | 0.524 | 8.283 | 8.277 | 12.43 | | | | |
| 28 | Perylene-d12 | 10.000 | 0 | 159040 | 1 | 1 | 10.492 | 10.460 | 0 | | | | |
| 29 | Benzo(a)anthracene | 8.585 | -14.1 | 283132 | 1.780 | 2.074 | 9.233 | 9.218 | 10.60 | | | | |
| 30 | Chrysene | 9.150 | -8.5 | 315969 | 1.987 | 2.171 | 9.267 | 9.250 | 7.71 | | | | |
| 31 | bis(2-Ethylhexyl)phthalate | 9.847 | -1.5 | 375410 | 2.360 | 2.397 | 9.350 | 9.354 | 7.68 | | | | |
| 32 | Benzo(b)fluoranthene | 9.727 | -2.7 | 276414 | 1.738 | 1.787 | 10.183 | 10.157 | 10.15 | | | | |
| 33 | Benzo(k)fluoranthene | 9.314 | -6.9 | 306859 | 1.929 | 2.072 | 10.208 | 10.181 | 10.07 | | | | |
| 34 | Benzo(a)pyrene | 9.831 | -1.7 | 285078 | 1.792 | 1.823 | 10.450 | 10.417 | 9.60 | | | | |
| 35 | Indeno(1,2,3-cd)pyrene | 9.626 | -3.7 | 332836 | 2.093 | 2.174 | 11.308 | 11.269 | 10.93 | | | | |
| 36 | Dibenzo(a,h)anthracene | 9.730 | -2.7 | 248241 | 1.561 | 1.604 | 11.325 | 11.292 | 10.98 | | | | |
| 37 | Benzo(g,h,i)perylene | 8.938 | -10.6 | 286789 | 1.803 | 2.017 | 11.500 | 11.458 | 10.94 | | | | |

FIGURE 6 – TYPICAL SAMPLE RESULT SUMMARY

SW 3520C/8270C SIM
 SEMI VOLATILE ORGANICS BY GC/MS

```

=====
Client       : XYZ, INC                      Date Collected: 03/17/06
Project      : CLEAN LAND PROJECT           Date Received: 03/17/06
Batch No.    : 06C177                       Date Extracted: 03/23/06 13:00
Sample ID    : 0009-005                     Date Analyzed: 03/28/06 19:36
Lab Samp ID  : C177-05                      Dilution Factor: .94
Lab File ID  : RCZ431                       Matrix          : WATER
Ext Btch ID  : SVC028W                      % Moisture     : NA
Calib. Ref.  : RCZ053                       Instrument ID   : T-048
=====
  
```

| PARAMETERS | RESULTS (ug/L) | RL (ug/L) | MDL (ug/L) |
|------------------------|-------------------|--------------|---------------|
| ACENAPHTHENE | ND | .94 | .19 |
| ACENAPHTHYLENE | ND | .94 | .19 |
| ANTHRACENE | ND | 1.9 | .19 |
| BENZO(A)ANTHRACENE | ND | 1.9 | .19 |
| BENZO(A)PYRENE | ND | .94 | .19 |
| BENZO(B)FLUORANTHENE | ND | .94 | .19 |
| BENZO(K)FLUORANTHENE | ND | 1.9 | .19 |
| BENZO(G,H,I)PERYLENE | ND | .94 | .19 |
| CHRYSENE | ND | 1.9 | .19 |
| DIBENZO(A,H)ANTHRACENE | ND | .94 | .19 |
| FLUORANTHENE | ND | 1.9 | .19 |
| FLUORENE | ND | 1.9 | .19 |
| INDENO(1,2,3-CD)PYRENE | ND | .94 | .19 |
| NAPHTHALENE | ND | .94 | .19 |
| PHENANTHRENE | ND | .94 | .19 |
| PYRENE | ND | 1.9 | .19 |

| SURROGATE PARAMETERS | % RECOVERY | QC LIMIT |
|----------------------|------------|----------|
| TERPHENYL-D14 | 76 | 50-130 |

RL: Reporting Limit

FIGURE 7-TYPICAL LCS REPORT SUMMARY

EMAX QUALITY CONTROL DATA
LCS/LCD ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN LAND PROJECT
BATCH NO.: 06C177
METHOD: SW 3520C/8270C SIM

MATRIX: WATER % MOISTURE: NA
DILUTION FACTOR: 1 1 1
SAMPLE ID: MBLK1W
LAB SAMP ID: SVC028WB SVC028WL SVC028WC
LAB FILE ID: RCZ423 RCZ424 RCZ425
DATE EXTRACTED: 03/23/0613:00 03/23/0613:00 03/23/0613:00 DATE COLLECTED: NA
DATE ANALYZED: 03/28/0617:04 03/28/0617:23 03/28/0617:42 DATE RECEIVED: 03/23/06
PREP. BATCH: SVC028W SVC028W SVC028W
CALIB. REF: RCZ053 RCZ053 RCZ053

ACCESSION:

| PARAMETER | BLNK RSLT (ug/L) | SPIKE AMT (ug/L) | BS RSLT (ug/L) | BS % REC | SPIKE AMT (ug/L) | BSD RSLT (ug/L) | BSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|------------------------|---------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|--------------|-------------------|------------------|
| Acenaphthene | ND | 10 | 6.35 | 64 | 10 | 5.71 | 57 | 11 | 40-130 | 30 |
| Acenaphthylene | ND | 10 | 6.75 | 68 | 10 | 6.06 | 61 | 11 | 40-130 | 30 |
| Anthracene | ND | 10 | 7.1 | 71 | 10 | 6.76 | 68 | 5 | 50-130 | 30 |
| Benzo(a)anthracene | ND | 10 | 8.11 | 81 | 10 | 8.26 | 83 | 2 | 50-130 | 30 |
| Benzo(a)pyrene | ND | 10 | 8.39 | 84 | 10 | 8.49 | 85 | 1 | 50-130 | 30 |
| Benzo(b)fluoranthene | ND | 10 | 10.1 | 101 | 10 | 9.52 | 95 | 6 | 50-130 | 30 |
| Benzo(k)fluoranthene | ND | 10 | 6.44 | 64 | 10 | 6.9 | 69 | 7 | 30-150 | 30 |
| Benzo(g,h,i)perylene | ND | 10 | 8.11 | 81 | 10 | 8.05 | 81 | 1 | 50-130 | 30 |
| Chrysene | ND | 10 | 7.69 | 77 | 10 | 7.92 | 79 | 3 | 50-130 | 30 |
| Dibenzo(a,h)anthracene | ND | 10 | 8.92 | 89 | 10 | 8.81 | 88 | 1 | 40-140 | 30 |
| Fluoranthene | ND | 10 | 7.3 | 73 | 10 | 7.09 | 71 | 3 | 50-130 | 30 |
| Fluorene | ND | 10 | 6.77 | 68 | 10 | 6.02 | 60 | 12 | 40-130 | 30 |
| Indeno(1,2,3-cd)pyrene | ND | 10 | 8.78 | 88 | 10 | 8.76 | 88 | 0 | 30-140 | 30 |
| Naphthalene | ND | 10 | 6.32 | 63 | 10 | 5.67 | 57 | 11 | 30-130 | 30 |
| Phenanthrene | ND | 10 | 6.65 | 66 | 10 | 6.45 | 65 | 3 | 40-130 | 30 |
| Pyrene | ND | 10 | 7.25 | 72 | 10 | 7.17 | 72 | 1 | 40-130 | 30 |

| SURROGATE PARAMETER | SPIKE AMT (ug/L) | BS RSLT (ug/L) | BS % REC | SPIKE AMT (ug/L) | BSD RSLT (ug/L) | BSD % REC | QC LIMIT (%) |
|---------------------|-----------------------------|-------------------|-------------|---------------------|--------------------|--------------|-------------------|
| Terphenyl-d14 | 10 8.25 83 10 8.1 81 50-130 | | | | | | |

FIGURE 8 – TYPICAL MATRIX SPIKE SUMMARY

EMAX QUALITY CONTROL DATA
MS/MSD ANALYSIS

CLIENT: XYZ, INC
PROJECT: CLEAN LAND PROJECT
BATCH NO.: 06C177
METHOD: SW 3520C/8270C SIM

```

=====
MATRIX: WATER                               % MOISTURE: NA
DILUTION FACTOR: .94                        .96                .96
SAMPLE ID: 0009-005
LAB SAMP ID: C177-05                        C177-05M            C177-05S
LAB FILE ID: RCZ431                         RCZ426              RCZ427
DATE EXTRACTED: 03/23/0613:00              03/23/0613:00      03/23/0613:00      DATE COLLECTED: 03/17/06
DATE ANALYZED: 03/28/0619:36               03/28/0618:00      03/28/0618:20      DATE RECEIVED: 03/17/06
PREP. BATCH: SVC028W                       SVC028W             SVC028W
CALIB. REF: RCZ053                          RCZ053              RCZ053
=====

```

ACCESSION:

| PARAMETER | SMPL RSLT (ug/L) | SPIKE AMT (ug/L) | MS RSLT (ug/L) | MS % REC | SPIKE AMT (ug/L) | MSD RSLT (ug/L) | MSD % REC | RPD (%) | QC LIMIT (%) | MAX RPD (%) |
|------------------------|---------------------|---------------------|-------------------|-------------|---------------------|--------------------|--------------|--------------|-------------------|------------------|
| Acenaphthene | ND | 9.6 | 5.22 | 54 | 9.6 | 5.78 | 60 | 10 | 40-130 | 30 |
| Acenaphthylene | ND | 9.6 | 5.5 | 57 | 9.6 | 6.14 | 64 | 11 | 40-130 | 30 |
| Anthracene | ND | 9.6 | 6.56 | 68 | 9.6 | 6.74 | 70 | 3 | 50-130 | 30 |
| Benzo(a)anthracene | ND | 9.6 | 7.59 | 79 | 9.6 | 8.1 | 84 | 6 | 50-130 | 30 |
| Benzo(a)pyrene | ND | 9.6 | 7.88 | 82 | 9.6 | 8.34 | 87 | 6 | 50-130 | 30 |
| Benzo(b)fluoranthene | ND | 9.6 | 9.71 | 101 | 9.6 | 9.78 | 102 | 1 | 50-130 | 30 |
| Benzo(k)fluoranthene | ND | 9.6 | 5.58 | 58 | 9.6 | 6.25 | 65 | 11 | 30-150 | 30 |
| Benzo(g,h,i)perylene | ND | 9.6 | 7.3 | 76 | 9.6 | 7.91 | 82 | 8 | 50-130 | 30 |
| Chrysene | ND | 9.6 | 7.29 | 76 | 9.6 | 7.74 | 81 | 6 | 50-130 | 30 |
| Dibenzo(a,h)anthracene | ND | 9.6 | 7.96 | 83 | 9.6 | 8.52 | 89 | 7 | 40-140 | 30 |
| Fluoranthene | ND | 9.6 | 6.61 | 69 | 9.6 | 6.89 | 72 | 4 | 50-130 | 30 |
| Fluorene | ND | 9.6 | 5.8 | 60 | 9.6 | 6.4 | 67 | 10 | 40-130 | 30 |
| Indeno(1,2,3-cd)pyrene | ND | 9.6 | 7.97 | 83 | 9.6 | 8.6 | 90 | 8 | 30-140 | 30 |
| Naphthalene | ND | 9.6 | 5.23 | 54 | 9.6 | 5.78 | 60 | 10 | 30-130 | 30 |
| Phenanthrene | ND | 9.6 | 6.26 | 65 | 9.6 | 6.54 | 68 | 4 | 40-130 | 30 |
| Pyrene | ND | 9.6 | 6.47 | 67 | 9.6 | 6.92 | 72 | 7 | 40-130 | 30 |

```

=====
SURROGATE PARAMETER      SPIKE AMT      MS RSLT      MS      SPIKE AMT      MSD RSLT      MSD      QC LIMIT
                          (ug/L)        (ug/L)        % REC    (ug/L)        (ug/L)        % REC    ( % )
-----
Terphenyl-d14            9.6            6.64         69       9.6            7.57         79       50-130
=====

```

CASE NARRATIVE

CLIENT: XYZ, INC.
PROJECT: CLEAN LAND PROJECT
SDG: 06C177

METHOD SW 3520C/8270C SIM SEMI VOLATILE ORGANICS BY GC/MS

Seven (7) water samples were received on 03/17/06 for Semi Volatile Organic analysis by Method 3520C/8270C SIM in accordance with USEPA SW846, 3rd ed.

1. Holding Time

Samples and extracts were analyzed within holding time.

2. Tuning and Calibration

Tuning and calibration were carried out at 12-hour interval. All QC requirements were met.

3. Method Blank

Method blank was extracted and analyzed at a frequency specified by the project and that results are compliant to project requirement.

4. Surrogate

Surrogate was added to MB, LCS, MS/MSD and every sample prior to analysis, and that recoveries met the project QC limits.

5. Lab Control Sample/Lab Control Sample Duplicate

LCS/LCD were analyzed at a frequency specified by the project and that recoveries met the project QC limit.

6. Matrix Spike/Matrix Spike Duplicate

MS/MSD were extracted with the samples and analyzed at a frequency specified by the project and that recoveries met the project QC limit.

7. Sample Analysis

Samples were analyzed in conformance to the method and project requirements.

TABLE 1
INTERMEDIATE STANDARD PREPARATION
METHOD: Semivolatiles by GCMS (8270SIM)

A. Primary Source: Accua

| Compound Name | Stock/Internal Soln. Conc. mg/ml | Source | Preparation | | | Final Conc. (mg/ml) |
|--------------------------------------|----------------------------------|---------------|--------------|-------------------|-----------------|---------------------|
| | | | Aliquot (mL) | Dil. Solution | Final Vol. (ml) | |
| Base/Neutral Composite Mix | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Toxic Substance Mix #1 | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Phenol Mix | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Composite Mix 3 | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Benzidine and 3,3' dichlorobenzidine | 2000 | Accu Standard | 1000 | MeCl ₂ | 10 | 200 |
| Acid Surrogate Mix | 7500 | Restek | 267 | MeCl ₂ | 10 | 200 |
| Base/Neutral Surrogate Mix | 5000 | Restek | 400 | MeCl ₂ | 10 | 200 |

B. Secondary Source: Supelco,Ultra

| Compound Name | Stock Soln. Conc. mg/ml | Source | Preparation | | | Final Conc. (mg/ml) |
|-----------------------|-------------------------|------------------|--------------|-------------------|-----------------|---------------------|
| | | | Aliquot (ml) | Solvent | Final Volume,mL | |
| Base/Neutral Mix 1 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Base/Neutral Mix 2 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Toxic Substance Mix 1 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Toxic Substance Mix 2 | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Phenol Mix | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| PAH Mixture | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Benzidine | 2000 | Ultra Scientific | 1 | MeCl ₂ | 10 | 200 |
| Carbazole | 2000 | Supelco | 1 | MeCl ₂ | 10 | 200 |
| Pyridine | 2000 | Supelco | 1 | MeCl ₂ | 10 | 200 |

TABLE 2
WORKING STANDARD CALIBRATION
METHOD: Semivolatiles by GC/MS (8270SIM)

| Standard Name | Intermediate Standard, 200 mg/ml (primary source)See Table 1 | Internal Standard, 2000 mg/ml | Amount of MeCl ₂ Needed | Final Volume | Final Concentration |
|------------------------------|--|-------------------------------|------------------------------------|--------------|--|
| Standard 1 0.15 µg/ml Std | 0.75 µl | 5 µl | 994.25 µl | 1000 µl | 0.15 µg/ml of Cal. Std + 10. µg/ml of Int. std |
| Standard 2 0.5 µg/ml Std | 1.25 µl | 2.5 µl | 496.25 µl | 500 µl | 0.5 µg/ml of Cal. Std + 10. µg/ml of Int. std |
| Standard 3 1 µg/ml Std | 2.5 µl | 2.5 µl | 495 µl | 500 µl | 1.0 µg/ml of Cal. Std + 10. µg/ml of Int. std |
| Standard 4 2 µg/ml Std | 5 µl | 2.5 µl | 492.5 µl | 500 µl | 2.0 µg/ml of Cal. Std + 10. µg/ml of Int. std |
| Standard 5 5 µg/ml Std | 12.5 µl | 2.5 µl | 485 µl | 500 µl | 5.0 µg/ml of Cal. Std + 10. µg/ml of Int. std |
| Standard 6 10 µg/ml Std * | 25 µl | 2.5 µl | 472.5 µl | 500 µl | 10.0 µg/ml of Cal. Std + 10. µg/ml of Int. std |
| Standard 7 20 µg/ml Std | 50 µl | 2.5 µl | 447.5 µl | 500 µl | 20.0 µg/ml of Cal. Std + 10. µg/ml of Int. std |

*Used as DCC Standard

WORKING SECONDARY SOURCE STANDARD
METHOD: Semivolatiles by GC/MS (8270SIM)

| Standard Name | Intermediate Standard | Internal Standard | Amount of MeCl ₂ Needed | Final Volume | Final Conc. |
|--|-----------------------|-------------------|------------------------------------|--------------|--|
| Secondary Source Std. Mix(see Table 1-B) | 25.0 µl | 2.5 µl | 472.5 µl | 500 µl | 10 µg/ml of Cal. Ver.Std.+ 10 µg/ml of Internal Std |

TABLE 3

**INSTRUMENT PARAMETERS
 METHOD (8270SIM)**

| | | |
|----------------------------|-------------------------------------|-------------------------------------|
| Instrument No: | 041; 042 | 048, 052 |
| Carrier Gas | Helium at 60 psi at outlet | Helium at 90 psi at outlet |
| Column head pressure | 10-15 psi at 30°C | 80-90 psi at 45°C |
| Injection port temperature | 260°C | 260°C |
| Interface | Direct column interference at 300°C | Direct column interference at 300°C |
| Valve time | Splitless 2.0 minute | Split 2.0 minute |

Oven Temperature Program

| | | |
|---------------------|--------------------------------------|--------------------------------------|
| Instrument No: | 041; 042 | 048, 052 |
| Initial Temperature | 30°C;/min. to 100°C | 45°C; hold for 0.5 minutes |
| Rate | 17°C/min to 320°C; hold for 4.39 min | 22°C/min to 340°C; hold for 0.09 min |
| Run Time | 20 minutes | 14 minutes |

Scan Parameters

| | | |
|-------------------------|--------------------|--------------------|
| Instrument No: | 041; 042 | 048, 052 |
| Scan start time | After Solvent Peak | After Solvent Peak |
| Mass range | 40 to 500 AMU | 40 to 450 AMU |
| Multiplier voltage | 1000-3000 | 0.7-3 |
| Number of sampling rate | 2 | 2 |
| Threshold | 200-1500 | 500-1500 |
| Tuning File | DFTPP | DFTPP |

TABLE 4
DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

| Mass | Ion Abundance Criteria |
|-------------|---|
| 51 | 30.0 to 60.0% of mass 198 |
| 68 | Less than 2.0% of mass 69 |
| 69 | Present |
| 70 | Less than 2.0% of mass 69 |
| 127 | 40.0 to 60.0% of mass 198 |
| 197 | Less than 1.0% of mass 198 |
| 198 | Base peak, 100% relative abundance (See Note) |
| 199 | 5.0 to 9.0% of mass 198 |
| 275 | 10.0 to 30.0% of mass 198 |
| 365 | Greater than 1.0% of mass 198 |
| 441 | Present but less than mass 443 |
| 442 | Greater than 40% of mass 198 |
| 443 | 17.0 to 23.0% of mass 442 |

NOTE: All ion abundance MUST be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 110 percent that of m/z 198.

TABLE 5
CALIBRATION CHECK COMPOUNDS (CCC)

| Base/Neutral Fraction |
|------------------------------|
| Acenaphthene |
| Fluoranthene |
| Benzo(a)pyrene |

TABLE 6 - ANALYTE LIST AND QUANTITATION IONS

| Compound | TS/IS | Primary Quant-Ion | Secondary Quant-Ion |
|----------------------------|-------------------|-------------------|---------------------|
| Acenaphthene | Target | 154 | 152, 153 |
| Acenaphthylene | Target | 152 | 151 |
| Anthracene | Target | 178 | 176 |
| Azobenzene | Target | 77 | 105 |
| benzo(a)anthracene | Target | 228 | 229, 226 |
| benzo(a)pyrene | Target | 252 | 253 |
| benzo(b)fluoranthene | Target | 252 | 253 |
| benzo(g,h,i)perylene | Target | 276 | 138 |
| benzo(k)fluoranthene | Target | 252 | 253, 125 |
| bis(2-chloroethyl)ether | Target | 93 | 63 |
| Chrysene | Target | 228 | 226 |
| Dibenzo(a,h)anthracene | Target | 278 | 139 |
| Fluoranthene | Target | 202 | 101 |
| Fluorene | Target | 166 | 165 |
| Hexachlorobenzene | Target | 142 | 284 |
| Indeno(1,2,3-cd)pyrene | Target | 276 | 138 |
| 1-Methylnaphthalene | Target | 142 | 141 |
| 2-Methylnaphthalene | Target | 142 | 141 |
| 2-Chlorophenol | Target | 128 | 64, 130 |
| Naphthalene | Target | 128 | 129 |
| n-Nitroso-di-n-propylamine | Target | 70 | 42, 101 |
| Phenanthrene | Target | 178 | 179, 176 |
| Pyrene | Target | 202 | 200 |
| N-Nitrodimethylamine | Target | 74 | 42 |
| Phenol | Target | 94 | 66 |
| 2,4-Dimethylphenol | Target | 122 | 107 |
| 2,4-Dichlorophenol | Target | 162 | 164 |
| 4-Chloro-3-methylphenol | Target | 107 | 142 |
| 2,4,6-Trichlorophenol | Target | 196 | 198, 132 |
| 2,4,5-Trichlorophenol | Target | 196 | 198, 97 |
| Pentachlorophenol | Target | 266 | 264, 268 |
| Bis(2-Ethylhexyl)phthalate | Target | 149 | 167, 52 |
| Phenol-d5 | Surrogate | 99 | 71 |
| Terphenyl-d14 | Surrogate | 244 | 122 |
| 1,4-Dichlorobenzene-d4 | Internal Standard | 152 | 150 |
| Phenanthrene-d10 | Internal Standard | 188 | 94 |
| Perylene-d12 | Internal Standard | 264 | 260 |

TABLE 7
INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

| 1,4-Dichlorobenzene-d₄ | Phenanthrene-d₁₀ | Perylene-d₁₂ |
|--|------------------------------------|--------------------------------|
| 1-Methylnaphthalene | Anthracene | benzo(a)anthracene |
| 2-Methylnaphthalene | Fluoranthene | benzo(a)pyrene |
| Acenaphthene | Hexachlorobenzene | benzo(b)fluoranthene |
| Acenaphthylene | Phenanthrene | benzo(g,h,i)perylene |
| Azobenzene | Pyrene | benzo(k)fluoranthene |
| bis(2-chloroethyl)ether | Terphenyl-d14 | Chrysene |
| Fluorene | Pentachlorophenol | Dibenzo(a,h)anthracene |
| Naphthalene | | Indeno(1,2,3-cd)pyrene |
| n-Nitroso-di-n-propylamine | | Bis(2-Ethylhexyl)phthalate |
| n-Nitrosodimethylamine | | |
| Phenol | | |
| Phenol – d5 | | |
| 2-Chlorophenol | | |
| 2,4-Dimethylphenol | | |
| 2,4-Dichlorophenol | | |
| 4-Chloro-3-methylphenol | | |
| 2,4,6-Trichlorophenol | | |
| 2,4,5-Trichlorophenol | | |

TABLE 8 - TARGET COMPOUND STANDARD LIST & RL

| PARAMETER | WATER(µg/L) | | SOIL(µg/Kg) | |
|----------------------------|-------------|----|-------------|----|
| | MDL | RL | MDL | RL |
| 2-Methylnaphthalene | 0.2 | 1 | 10 | 20 |
| Acenaphthene | 0.2 | 1 | 10 | 20 |
| Acenaphthylene | 0.2 | 1 | 10 | 20 |
| Anthracene | 0.2 | 1 | 10 | 20 |
| Benzo(a)anthracene | 0.2 | 1 | 10 | 20 |
| Benzo(a)pyrene | 0.2 | 1 | 10 | 20 |
| Benzo(b)fluoranthene | 0.2 | 1 | 10 | 20 |
| Benzo(g,h,i)perylene | 0.2 | 1 | 10 | 20 |
| Benzo(k)fluoranthene | 0.2 | 1 | 10 | 20 |
| Bis(2-chloroethyl)ether | 0.2 | 1 | 10 | 20 |
| Bis(2-ethylhexyl)phthalate | 1 | 2 | 10 | 20 |
| Chrysene | 0.2 | 1 | 10 | 20 |
| Dibenzo(a,h)anthracene | 0.2 | 1 | 10 | 20 |
| Fluoranthene | 0.2 | 1 | 10 | 20 |
| Fluorene | 0.2 | 1 | 10 | 20 |
| Hexachlorobenzene | 0.2 | 1 | 10 | 20 |
| Indeno(1,2,3-cd)pyrene | 0.2 | 1 | 10 | 20 |
| Naphthalene | 0.2 | 1 | 10 | 20 |
| n-Nitrosodimethylamine | 0.3 | 1 | 15 | 30 |
| n-Nitroso-di-n-propylamine | 0.2 | 1 | 10 | 20 |
| Pentachlorophenol | 0.2 | 1 | 10 | 20 |
| Phenanthrene | 0.2 | 1 | 10 | 20 |
| Phenol | 0.3 | 1 | 10 | 20 |
| Pyrene | 0.2 | 1 | 10 | 20 |

SUMMARY OF IN-HOUSE QUALITY CONTROL PROCEDURES

| QC PROCEDURE | FREQUENCY | ACCEPTANCE CRITERIA | CORRECTIVE ACTION | FLAGGING CRITERIA | 1st Rvw | 2nd Rvw |
|---|---|--|--|--|---------|---------|
| Check of mass spectral ion intensities using DF TPP | Prior to initial calibration and calibration verification | Refer to criteria listed in the method description (Table 4) | Retune instrument and verify | | | |
| Five-point initial calibration for all analytes | Initially; as needed | SPCCs average RF ± 0.050 and %RSD for RFs for CCCs $\leq 30\%$ and one option below 1). linear- mean RSD for all analytes $\leq 15\%$ 2). linear – least squares regression $r \geq 0.995$, when RSD $> 15\%$ 3). non-linear – COD > 0.990 (6 points shall be used for second order, 7 points shall be used for third) | Correct the problem then repeat initial calibration | | | |
| Second-source calibration verification | After initial calibration | All analytes within $\pm 25\%$ of expected value [* within $\pm 35\%$ of expected value] | Correct the problem then repeat initial calibration | | | |
| Retention time window calculated for each analyte | Each sample | Relative retention time (RRT) of the analyte within ± 0.06 RRT units of the RRT | Correct the problem then re-analyze all samples analyzed since the last retention time check | | | |
| Calibration verification | Daily, before sample analysis and every 12 hours of analysis time | RRF ≥ 0.050 for all analytes; and CCCs $\leq 20\%$ difference (when using RFs) or drift (when using least squares regression or non-linear calibration) | Correct the problem then repeat initial calibration | | | |
| Internal Standard | Every sample, spiked sample, standard, and method blank | Retention time ± 30 seconds from retention time of the mid-point std. In the ICAL. EICP area within -50% to $+100\%$ of ICAL mid-point std. | Inspect mass spectrometer and GC for malfunctions; mandatory re-analysis of samples analyzed while system was malfunctioning | | | |
| Method blank | One per preparation batch (≤ 20 samples per matrix) | No analytes detected \geq RL | Re-prep and re-analyze method blank and all samples processed with the contaminated blank | Apply B to specific analyte(s) on all associated | | |
| LCS | One LCS per preparation (≤ 20 samples per matrix) | Within EMAX In-House QC Limits | Re-prep and re-analyze the LCS and all associated samples | | | |
| MS/MSD | One MS/MSD per every 20 project samples per matrix | Within EMAX In-House QC Limits | None | | | |
| Surrogate spike | Every sample, spiked sample, standard, and method blank | Surrogate spike should be within EMAX In-house QC Limits. | Correct the problem then re-extract and analyze sample. | | | |
| Results reported between MDL and RL | None | None | None | Apply J to all values between MDL and RL | | |
| Comments: RL = Reporting Limit | | | | Reviewed by: | | |
| | | | | Date: | | |

DEMONSTRATION OF CAPABILITY

Applicable SOP : EMAX-8270SIM

Extracted date: 4/13/2006

Conc Unit: µg/L

04/17/06

Analyzed date: 04/24/06

Sample Amount(ml): 1000

Extracted by: Juanita Muertigue

Sample Extracted(ml): 1

Analyzed by: Souzan Greas

| PARAMETER | SVD011WL | SVD011WC | SVD023WL | SVD023WC | TV | Ave. Conc. | Ave. %Rec | SD | QC Criteria | COMMENTS |
|----------------------------|----------|----------|----------|----------|----|------------|-----------|----|-------------|----------|
| | RDZ096 | RDZ097 | REZ122 | REZ123 | | | | | | |
| Acenaphthene | 45.90 | 47.50 | 53.21 | 50.87 | 80 | 49.4 | 62 | 3 | 30 - 130 | Passed |
| Acenaphthylene | 48.70 | 51.55 | 54.80 | 54.91 | 80 | 52.5 | 66 | 3 | 30 - 130 | Passed |
| Anthracene | 52.97 | 54.75 | 60.79 | 59.38 | 80 | 57.0 | 71 | 4 | 30 - 130 | Passed |
| Benzo(a)anthracene | 62.00 | 67.61 | 79.12 | 75.29 | 80 | 71.0 | 89 | 8 | 30 - 130 | Passed |
| benzo(a)pyrene | 61.90 | 66.56 | 80.53 | 76.44 | 80 | 71.4 | 89 | 9 | 40 - 130 | Passed |
| Benzo(b)fluoranthene | 74.32 | 65.51 | 82.05 | 81.68 | 80 | 75.9 | 95 | 8 | 30 - 130 | Passed |
| Benzo(g,h,i)perylene | 59.98 | 65.44 | 72.84 | 69.34 | 80 | 66.9 | 84 | 6 | 30 - 130 | Passed |
| Benzo(k)fluoranthene | 51.27 | 65.93 | 82.00 | 73.04 | 80 | 68.1 | 85 | 13 | 30 - 130 | Passed |
| bis(2-chloroethyl)ether | 43.82 | 45.79 | 53.94 | 43.63 | 80 | 46.8 | 58 | 5 | 40 - 140 | Passed |
| bis(2-Ethylhexyl)phthalate | 60.54 | 65.56 | 90.98 | 84.98 | 80 | 75.5 | 94 | 15 | 50 - 130 | Passed |
| Chrysene | 61.91 | 64.51 | 78.64 | 75.56 | 80 | 70.2 | 88 | 8 | 50 - 130 | Passed |
| Dibenzo(a,h)anthracene | 65.79 | 70.44 | 80.41 | 74.60 | 80 | 72.8 | 91 | 6 | 30 - 130 | Passed |
| 2,4-Dichlorophenol | 48.87 | 50.06 | 63.54 | 59.01 | 80 | 55.4 | 69 | 7 | 30 - 130 | Passed |
| 2,4-Dimethylphenol | 35.86 | 36.20 | 46.26 | 41.09 | 80 | 39.9 | 50 | 5 | 40 - 130 | Passed |
| Fluoranthene | 53.74 | 56.52 | 67.36 | 65.13 | 80 | 60.7 | 76 | 7 | 40 - 130 | Passed |
| Fluorene | 50.90 | 54.33 | 59.66 | 56.29 | 80 | 55.3 | 69 | 4 | 40 - 130 | Passed |
| Hexachlorobenzene | 50.59 | 54.63 | 64.03 | 60.67 | 80 | 57.5 | 72 | 6 | 40 - 130 | Passed |
| Indeno(1,2,3-cd)pyrene | 63.42 | 68.05 | 79.35 | 75.39 | 80 | 71.6 | 89 | 7 | 30 - 130 | Passed |
| 2-Methylnaphthalene | 42.59 | 42.33 | 47.95 | 43.24 | 80 | 44.0 | 55 | 3 | 40 - 130 | Passed |
| Naphthalene | 41.30 | 42.00 | 47.64 | 45.21 | 80 | 44.0 | 55 | 3 | 30 - 160 | Passed |
| n-Nitrosodimethylamine | 49.98 | 37.65 | 56.16 | 36.81 | 80 | 45.2 | 56 | 9 | 40 - 130 | Passed |
| n-Nitroso-di-n-propylamine | 52.07 | 53.35 | 62.01 | 59.96 | 80 | 56.8 | 71 | 5 | 40 - 130 | Passed |
| Pentachlorophenol | 50.97 | 54.14 | 69.34 | 67.40 | 80 | 60.5 | 76 | 9 | 40 - 130 | Passed |
| Phenanthrene | 48.24 | 51.97 | 56.33 | 55.60 | 80 | 53.0 | 66 | 4 | 40 - 130 | Passed |
| Phenol | 47.03 | 44.54 | 52.73 | 49.79 | 80 | 48.5 | 61 | 4 | 40 - 130 | Passed |
| Pyrene | 52.13 | 53.74 | 64.95 | 63.05 | 80 | 58.5 | 73 | 6 | 30 - 140 | Passed |

8270SIMF1

ANALYSIS RUN LOG FOR SEMIVOLATILES

SOP EMAX-8270 Rev. No. 2 EMAX-8270SIM Rev. No. 0 EMAX-CLPSVOA Book # A42-007

Method File: Tune File: Start Date/Time: End Date/Time:



| Preparative Batch | Data File Name | Run ID | DF | Matrix | | Notes | Instrument No: 42 | | |
|-------------------|----------------|--------|----|--------|---|-------|--|----|--------------|
| | | | | S | W | | INITIAL CALIBRATION REFERENCE | | |
| | | | | | | | Date | | |
| | | | | | | | ICAL ID | | |
| | | | | | | | Standards | | |
| | | | | | | | Name | ID | Conc. (mg/L) |
| | | | | | | | DFTPP | | |
| | | | | | | | DCC | | |
| | | | | | | | | | |
| | | | | | | | Solvent | ID | |
| | | | | | | | CH ₂ Cl ₂ | | |
| | | | | | | | Data File | | |
| | | | | | | | Comments: | | |
| | | | | | | | | | |
| | | | | | | | Analyzed By: | | |
| | | | | | | | This page is checked during data review. | | |

ANALYTICAL BATCH

8270SIMF2

EXTRACTION LOG FOR SEMIVOLATILES

| SOP | | | | | | | | | | | | |
|---|-------------|---------------|------------------|----------------------|----|---------------------|--------------------------|-------|----------------------|---|---------------|-------------------|
| ☞ EMAX 3540 Rev. No.: 0 ☞ EMAX3510 Rev. No.: 0 ☞ EMAX3550 Rev. No.: 1 ☞ EMAX-3520 Rev. No.: 1 ☞ EMAX-CLP-SVOA ☞ | | | | | | | | | | | | |
| Matrix: | | Start Date: | | Time: | | Ending Date: | | Time: | | Book # ESV-017 | | |
| PREPARATION BATCH: * | Sample Prep | Lab Sample ID | Sonicator Number | Sample Amount (g/ml) | pH | Extract Volume (ml) | Clean-up [G] [F] [A] [S] | Notes | | Standards | ID | Amount Added (ml) |
| | ID | ID | Number | (g/ml) | | (ml) | [A] [S] | | | Surrogate | | |
| | 01 | | | | | | | | | LC/MS | | |
| | 02 | | | | | | | | | | | |
| | 03 | | | | | | | | | Reagent | Lot# / ID | |
| | 04 | | | | | | | | | CH ₂ Cl ₂ | | |
| | 05 | | | | | | | | | Na ₂ SO ₄ | | |
| | 06 | | | | | | | | | H ₂ SO ₄ | | |
| | 07 | | | | | | | | | NaOH | | |
| | 08 | | | | | | | | | Silica Sand | | |
| | 09 | | | | | | | | | TUNING | | |
| | 10 | | | | | | | | | Sonicator # | Reading | |
| | 11 | | | | | | | | | | | |
| | 12 | | | | | | | | | | | |
| | 13 | | | | | | | | | | | |
| | 14 | | | | | | | | | | | |
| | 15 | | | | | | | | | Concentrator Water Bath Temp, T ₁ (°C) | | |
| | 16 | | | | | | | | | 1 | | |
| | 17 | | | | | | | | | 2 | | |
| | 18 | | | | | | | | | 3 | | |
| | 19 | | | | | | | | | 4 | | |
| | 20 | | | | | | | | | 5 | | |
| | 21 | | | | | | | | | Comments: Test Thermometer = T ₁ | | |
| | 22 | | | | | | | | | Prepared By: | Witnessed By: | |
| | 23 | | | | | | | | | Standard Added By: | | |
| | 24 | | | | | | | | | Checked By: | | |
| 25 | | | | | | | | | Extract Received by: | | Location: | |
| 26 | | | | | | | | | Disposed by: | | Disposed on: | |

Corvallis ASL Standard Operating Procedure

**STANDARD OPERATING PROCEDURE
DETERMINATION OF TOTAL PETROLEUM HYDROCARBONS AS
GASOLINE BY AK-101, EPA SW8021B, SW5030 AND SW5035 FOR USE
AT THE CH2M HILL, INC. KALAKAKET CREEK FIELD LAB**

APPROVED:

Singer Collins

6/6/07

QA Officer

Date

Mark Goehry

6/6/07

Laboratory Director

Date

Documentation of reading this SOP will be kept in the ASL QAQC training database. Each analyst is responsible for entering their own training dates. By entering their name and date of reading the SOP each analyst is agreeing to the following statement:

I have read and understood the following Standard Operating Procedure (SOP) and agree to follow the SOP as written. Any exceptions to the SOP will be recorded in the appropriate logbook or benchsheet and changes will be noted in the case narrative of the report to the client.

STANDARD OPERATING PROCEDURE
DETERMINATION OF TOTAL PETROLEUM HYDROCARBONS AS GASOLINE BY
AK-101, EPA SW8021B, SW5030 AND SW5035 FOR USE
AT THE CH2M HILL, INC. KALAKAKET CREEK FIELD LAB

1.0 SCOPE AND APPLICATION

- 1.1 This standard operating procedure is specific to the CH2M HILL on-site laboratory in Kalakaket Creek, Alaska.
- 1.2 This method is designed to measure the concentration of Gasoline Range Organics (GRO) in water and soil. This corresponds to an alkane range from the peak start of n-hexane (C6) to the peak start of n-decane (C10), and to a boiling point range between approximately 60°C and 170°C.
- 1.3 With the optional photo ionization detector (PID), this method can be extended for specific determination of volatile aromatics (BTEX) as specified in EPA Method SW-846 8021B.
- 1.4 Please be aware that any reference to 8021B is in regard to apparatus and not sample preparation.
- 1.5 This method is based upon the Alaska Department of Environmental Quality SOP AK101 and includes QC measures/information from EPA method SW8015, 5030, 5035, 8021, and AFCEE QAPP version 4.0.01.
- 1.6 As this SOP is written to satisfy multiple methods and agencies, the tightest criteria for reporting limits, QC acceptance criteria and other parameters will be employed in all cases unless otherwise specified.

2.0 OVERVIEW OF THE ANALYTICAL PROCESS

- 2.1 *Water samples are not expected to be analyzed at the Kalakaket Creek laboratory.* In the event the situation changes, the water samples are prepared by addition of surrogate spikes and/or matrix spikes when applicable. A 5.0 mL aliquot of sample is introduced into a sparge cell where it is purged for 11.0 minutes at ambient temperature. The purged gas flows through a hydrocarbon specific trap, where the species of interest are adsorbed onto a stationary phase also held at ambient temperature. Once the purge cycle is complete, the trap is heated rapidly to release the hydrocarbon analytes into the helium flow, where they are swept into the GC column. The analytes are separated chromatographically in the column by means of oven temperature programming and are detected by a tandem FID/PID detector and quantitated against a calibration curve.
- 2.2 Soil samples follow the same general procedure with the addition of an initial methanol extraction of the soil sample. A portion of the methanol extract is added to a 44mL VOA vial of water and is then purged the same way as a water sample. Soil samples are preserved with surrogated methanol at the time of sampling in pre-weighed VOA vials.
- 2.3 This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline. Other non-petroleum compounds with similar characteristics and boiling points may also be detected with this method.
- 2.4 The gas chromatograph is temperature programmed to facilitate separation of organic compounds. A PID/FID in series provides detection. Quantitation must be performed by comparing the total chromatographic area between and including C6 (n-hexane) and C9 (n-nonane), to the peak start time of C10 (n-decane), including resolved and unresolved components, based on FID response compared to a blended commercial gasoline standard and using forced baseline/baseline integration.
- 2.5 Special field sampling techniques are required to minimize the loss of volatile organic compounds from soil. Conventional sampling and sample handling techniques are not acceptable.

- 2.6 Benzene, toluene, ethylbenzene and total xylene isomers (BTEX) shall be determined simultaneously by means of a tandem PID detector system following the requirements of EPA SW-846 Method 8021B.

3.0 TARGET ANALYTES, REPORTING LIMITS AND DETECTION LIMITS

- 3.1 Target analytes for this method are gasoline range organics eluting between C6 and the beginning of C10 on the analytical column(s) described herein.
- 3.2 Method detection limits must be verified by performance of method detection limit studies on an annual basis in accordance with the procedure stated in 40 CFR 136, App. B. This process requires the analysis of a minimum of seven replicates of a known concentration of the compounds in water or soil matrix. The concentration of the replicate analyses should be one to five times the estimated detection limits. The method detection limit is calculated by multiplying the standard deviation of the replicate results by the appropriate student's T factor at the 99% confidence level. The goal for the spiking procedure is to produce a calculated method detection limit that is not less than 1/10 the spike level for each analyte.
- 3.3 The method detection limits must be equal to or less than the reporting limit and equal to or less than ½ of the AFCEE reporting limit.
- 3.4 For AK101 methods the PQL must be >5 times the calculated MDL.
- 3.5 The reporting limits required for each method are as follows:

| | AK101 | AFCEE | Kalakaket Lab Limits |
|--------------------------|------------|----------|----------------------|
| Water – gasoline | 100 ug/L | 100ug/L | n/a |
| Benzene | 1.0 ug/L | n/a | n/a |
| Toluene | 1.0 ug/L | n/a | n/a |
| Ethyl benzene | 1.0 ug/L | n/a | n/a |
| Total xylenes | 3.0 ug/L | n/a | n/a |
| Soil/Sediment - gasoline | 20 mg/kg | 10 mg/kg | 10 mg/kg |
| Benzene | 0.02 mg/kg | n/a | 0.02 mg/kg |
| Toluene | 0.05 mg/kg | n/a | 0.05 mg/kg |
| Ethyl benzene | 0.05 mg/kg | n/a | 0.05 mg/kg |
| Total xylenes | 0.05 mg/kg | n/a | 0.05 mg/kg |

- 3.6 Retention time standard used are as follows:

| |
|---|
| AK101 |
| Hexane to decane (beginning of C6 - beginning of C10) |

4.0 INTERFERENCES

Significant interferences may be encountered due to the presence of non-petroleum products such as polar organic analytes, biogenic interferences, using an incorrect solvent, or carryover from highly concentrated samples, etc. Analysts should minimize interferences such as carryover between samples by adequate syringe rinses. Daily changing of ultra pure water will reduce false positive blank and blank spike detections. Care shall be taken to avoid cross contamination of the surrogate spike solution with the calibration check solution or by highly concentrated samples. The retention time standard has exhibited carryover between samples in some cases, and should be spiked at the lowest possible concentration to minimize this problem.

5.0 SAFETY, WASTE MINIMIZATION AND POLLUTION PREVENTION

- 5.1 Samples contaminated with TPH constituents may be hazardous. Samples may include flammables, explosives, and potentially carcinogenic compounds. All samples are assumed to be hazardous and should be handled as such. All stock and working calibration standards, as well as all samples, shall be handled with the utmost care using good laboratory techniques in order to avoid harmful exposure. Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager. Personnel are encouraged to review the Chemical Hygiene Plan for general safety policies and Material Safety Data Sheets for reagents used in the laboratory.
- 5.2 Standards and samples shall be prepared in a fume hood. Sample preparation should be performed in a fume hood with adequate skin, eye, and hearing protection provided for and used by the analysts. Any situation creating odor levels should be immediately corrected.
- 5.3 Sample extracts and standards prepared in flammable solvents shall be stored in an explosion-proof refrigerator or a cooler.
- 5.4 Safety equipment including fire extinguisher, first aid kit, eye wash, and chemical spill cleanup kit shall be available for use at all times.
- 5.5 Laboratory wastes shall be separated and properly disposed complying with all federal, state, and local regulations. The wastes include collected solvent rinses; expired sample extracts and disposable labware (or other item as applicable) used in the preparation of the samples. These wastes shall be handled according to CVO SOP HAZ01, Waste Disposal.
- 5.6 Analysts are encouraged to reduce the amount of solvent or disposable labware waste whenever possible. More information on this topic can be found in "Less is Better: Laboratory Chemical Management Waste Reduction" located on the American Chemical Society website at http://membership.acs.org/c/ccs/pub_9.htm.

6.0 SAMPLE COLLECTION, STORAGE, HOLDING TIMES AND PRESERVATION

- 6.1 Aqueous Samples – *water samples are not expected to be analyzed at the Kalakaket field lab.*
- 6.1.1 Aqueous samples should be collected without agitation and without headspace in contaminant-free, amber glass 40-mL VOA vials with Teflon-lined septa. A sufficient number of samples should be collected to provide for quality control criteria and for back-up in the event of breakage. If amber glass vials are not available, clear glass may be substituted if the samples are protected from light. The Teflon layer must contact the sample (zero headspace). Sample vials should contain 200 μ L of 50% hydrochloric acid (HCl) as a preservative. Refrigerated samples ($4 \pm 2^\circ\text{C}$) must be analyzed within 14 days of collection.
- 6.1.2 A trip blank (contaminant-free amber glass 40-mL vial with Teflon-lined septum, filled to zero headspace with purged, organic free water preserved with the same acid as the samples, if possible) must accompany each shipping container and should be stored and analyzed with the field samples. Trip blank analysis is not required if all samples in a shipping container are less than the project specific cleanup level.
- 6.2 Soil/Sediment Samples - *The samples in this onsite laboratory are expected to be hand delivered daily to the laboratory with no external shipping necessary.*
- 6.2.1 Solid samples must be collected with minimum disturbance into 4-oz large mouth amber jars with Teflon lined lids. 25-mL aliquots of surrogated methanol (includes 1.2 mL of a surrogate solution at 50 μ g/mL)(see 8.6.5) or as close to a 1:1 ratio of surrogated methanol and soil as possible should be carefully added to the undisturbed soil until the sample is submerged.

- 6.2.2 It is extremely important that the weight of the jar, the weight of the methanol/surrogate solution, and the weight of the sample collected be known. These must either be measured directly, or sufficient information documented so that these weights can be calculated.
- 6.2.3 The ratio of soil to methanol used to calculate the MDL and PQL offered in this method was 1:1 (w/w). However, absorbent, organic soils such as muskeg and tundra will require a higher methanol-to-sample ratio, while beach sand may tolerate a lower ratio. Once the onsite lab is set up, testing will be performed to determine the best methanol to soil/sediment ratio.
- 6.2.4 Soil for volatiles analysis can be collected using any coring device that minimizes soil disturbance. Any scraping, stirring, or similar activity will result in a loss of volatiles during sampling. A sufficient number of samples should be collected to provide for backup in case of breakage.
- 6.2.5 Although it is not necessary to refrigerate all methanol preserved samples at $4^{\circ} \pm 2^{\circ}\text{C}$ after collection and until analysis is complete, collected samples must be kept below 25°C .
- 6.2.6 A reagent methanol trip blank must be prepared in the same manner as the sample vials, and must contain surrogated methanol. One trip blank must be included with each shipping container and must be stored and analyzed with the field samples. Trip blank analysis is not required if all samples in a shipping container are less than the project specific cleanup level.
- 6.2.7 Field blanks may be added to the sampling protocol and are prepared in the field by addition of surrogated methanol to the prepared container, as required by the Assessment Firm or the Project Manager.
- 6.2.8 A sample of the same soil to be analyzed for GRO should be collected into a moisture-proof container for percent moisture determination. This sample should be processed as soon as possible upon arrival at the laboratory to assure that the resulting moisture determination is representative of the preserved sample as surveyed.
- 6.2.9 Trip blanks, field blanks, method blanks, etc. should be prepared from the same batch of solvent, reagents and vials as are used for sample preservation. Twenty-eight days is the maximum holding time for soil and sediment samples.

7.0 APPARATUS AND MATERIALS

- 7.1 VOA sample vials – 40 mL capacity with septum screw caps; precleaned as purchased from I-Chem or similar source.
- 7.2 Standard dilution vials - 2 mL screw top vials, etc.
- 7.3 Sample jars – 4 oz. pre cleaned jars with Teflon lined lid for soil sample collection.
- 7.4 Balance–Sartorius; top loading electronic with 3,600-gm capacity with 0.1 g sensitivity.
- 7.5 Glassware–Class A volumetric pipets and flasks; beakers, vials, Pasteur pipets, and miscellaneous glassware as necessary for preparation and handling of samples and standards. All glassware should be washed with Liquinox and warm water, rinsed three times with both hot tap water and DI water, and then rinsed with methanol to dry.
- 7.6 Labware–Necessary for preparation and handling of samples and standards.
- 7.7 Syringes–Hamilton glass type as required for injection of sample extracts and standards, preparation of dilutions, and spiking of samples.
- 7.8 Archon autosampler for sample transfer to purge and trap system.
- 7.9 Tekmar Velocity XPT purge and trap system with Purge Trap J (BTEX trap). System will be fitted with a 5mL sparge tube initially. If samples exhibit surfactant properties a larger 25mL sparge tube may be employed.
- 7.10 Gas chromatograph (GC) – Hewlett-Packard Model 6890 or 7890 with temperature programming, electronic integration, report annotation, automated sample injection and tandem PID/FID detection system from OI analytical.

- 7.11 RTX-502.2 (Restek Corp.) 60m x 0.53mm with a film thickness of 3.0 µm or an equivalent column capable of resolving ethylbenzene from m/p-xylenes. The J&W DB-VRX 0.45 mm x 75 m x 2.55 um film has also been found to separate these analytes at the ASL Corvallis laboratory.

8.0 STANDARDS, GASES AND REAGENTS

- 8.1 Burdick and Jackson purge and trap grade methanol or equivalent.
- 8.2 Organics free water purchased and sent to lab site.
- 8.3 Hydrogen gas, for FID and PID.
- 8.4 Helium gas, carrier gas and makeup gas.
- 8.5 Zero air gas for FID use.
- 8.6 Calibration/QC standards
- 8.6.1 Petroleum primary calibration standard - a mixture of regular, plus, and premium gasoline that is certified as non-oxygenate. Accustandard part number AK-101.0-GCS-PAK, 5.0 mg/mL (total gasoline) in methanol, 5 individual ampules (Accustandard phone # 1-800-442-5290). Make a dilution of this standard at 2000 ug/mL.
- 8.6.2 Second source calibration check standard – a mixture of regular, plus, and premium gasoline that is certified as non-oxygenate from a source other than used for the primary source calibration standard. Ultra scientific standard part #RGO-605, 2500 ug/mL in methanol. No dilution necessary.
- 8.6.3 BTEX standard – contains benzene, toluene, ethylbenzene and m,p,o-xylenes. Accustandard part # AK-101.0-GCS-BTEX, 5.0 mg/mL in methanol. Dilute to 20.0 ug/mL and 200 ug/mL in methanol.
- 8.6.4 BTEX second source standard – contains benzene, toluene, ethylbenzene and m,p,o-xylenes. Ultra scientific part number BTX-2000N, 2.0 mg/mL in methanol. Dilute to 200 ug/mL in methanol.
- 8.6.5 Surrogate spiking standard for field collection (surrogated methanol) – a,a,a-trifluorotoluene, Sigma-Aldrich part number T63703-100G, 100g of >99% pure material. The methanol used for field preservation of samples is spiked with this surrogate at a concentration of 4.0 ug/mL. Two steps are necessary here-A) create a stock spiking solution at 8000 ug/mL by diluting 672 uL of neat a,a,a-TFT into 100 mL P&T grade methanol and B) spike 0.50 mL of this 8000 ug/mL stock into 1.0 liter of methanol which will yield a final field a,a,a-TFT concentration of 4.0 ug/mL in the methanol (density of a,a,a-TFT is 1.19 g/mL at 20°C).
- 8.6.6 Surrogate spiking standard for lab addition – 4-bromofluorobenzene. Accustandard part # M-624-SS-03-10X, 2000 ug/mL in methanol.
- 8.6.7 Retention Time Standard–Prepare a composite standard containing hexane (C6) and n-decane (C10) at approximately 2000 ug/mL in methanol.
- 8.7 Store all standards in a freezer at -10 to -20°C. Standards may be stored for up to 6 months. In this fashion. Standards from outside suppliers are good until the listed expiration date from that vendor.

9.0 QA/QC

- 9.1 All calculations for this section may be found at the end of the document in Table 5 at the end of this document. All reporting limits and QC acceptance criteria listed herein will be superseded by project specific requirements when applicable.
- 9.2 Instrument calibration– There must be an initial calibration of the GC system as described below.
- 9.2.1 An initial calibration curve of 5 points of more covering the range of 100 to 4000 µg/L for the gasoline target constituents and 0.50 ug/L to 500 ug/L for PID analytes will be analyzed

- to determine instrument sensitivity and the linearity of response. Once the curve has been verified by second source standard(s), analysis may proceed.
- 9.2.2 The lowest calibration point in the curve must be at or below the reporting limit. Since this point drives the reporting limit, it must always be included in the calibration curve, and may not be discarded. If the low-point is an outlier, the calibration must be repeated. The only point that may be readily removed from a calibration curve is the highest point, if linearity of the detector is exceeded or column overloading occurs. If this is the case, clear documentation of this removal and reason for removal will be written and included with calibration curve raw data.
- 9.2.3 SW-846 methods allow the use of linear and non-linear models for calibration data. The option for non-linear calibration may be necessary to achieve low detection limits or to address specific instrument techniques. However, one shall not allow non-linear calibration to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.
- 9.2.4 The following steps shall be adhered to when deciding on a calibration fit (refer to method 8000B, pages 17-23). *It should be noted that AK101 requirements are less stringent than the ones listed, which satisfy AFCEE requirements.*
- 9.2.4.1 Use the simplest curve fit first, which is linear calibration using average calibration or average response factor (called 'Average RF/Amount' in HP Chemstation software). If the RSD for any analyte is > 20% RSD then utilize another curve type (and section 9.2.3 is adhered to), proceed to least squares regression curve fit. [Line equation: $Y = \text{average of (response/amount)} * x$]
- 9.2.4.2 Linear Least squares regression is the next possible choice in curve fit factor (called 'Linear' in HP Chemstation software). This fit is commonly known as a linear curve fit. The correlation coefficient (r) must meet or exceed 0.995 to utilize this curve type. If the curve does not meet this acceptance limit and section 9.2.3 is adhered to, then non-linear calibration models may be employed. *Please note that any fit beyond linear for an FID detector may indicate a system/detector problem.* [Line equation: $Y = mx + b$]
- 9.2.4.3 Non-linear calibration (called Quadratic or Cubic in HP Chemstation software requiring 6 and 7 points, respectively) models may be used in situations where the analyst knows the instrument response does not follow a linear model over a sufficiently wide working range, or when other approaches above do not meet acceptance criteria. The coefficient of determination r^2 value >0.990 must be met or exceeded to utilize this curve type [Line equation: $Y = Ax^2 + Bx + C$ for quadratic, 3rd order for cubic].
- 9.2.5 Surrogate standards are calibrated at multiple levels, just as target analytes. The surrogate standards are added to the calibration standards.
- 9.3 Initial calibration verification-The ICV is performed any time a new calibration curve is run to ensure the curve was not created from faulty standards. The ICV must be a second source standard and prepared to fall within the normal range of the calibration curve. The ICV must recover between 80-120% to begin sample analysis. If the ICV does not meet acceptance criteria, re-analyze one time to ensure proper injection. If it still fails, the standards used to make the curve may be bad (evaporated, contaminated, etc.). A new primary and second source standard should be ordered to determine the nature of the problem. No samples may be analyzed until a passing ICV is analyzed.
- 9.4 IDC- An initial demonstration of capability (IDC) study must be performed prior to use of the method by each analyst or after any significant changes to the method. An IDC study consists of four aliquots of reagent water spiked with target analytes and processed through the entire analytical method. For NELAC certification purposes the IDC study may be used to satisfy the yearly training requirement for an analyst or work cell.

- 9.4.1 Prepare and analyze four spiked blank samples at a concentration of approximately 100ug/L, or the midpoint of the calibration curve.
- 9.4.2 Calculate the mean concentration found (X) in ug/L and the standard deviation of the concentration in ug/L for each analyte.
- 9.4.3 For each analyte X should be within the acceptance criteria in Table 1. The RSD should be 30% or less for water, 50% or less for soils (see Table 1 for test specific information). If the results from all analytes meet these criteria then the system and analyst performance are acceptable. If any analyte fails to meet the criteria then investigate and correct the source of the problem and repeat the test.
- 9.5 MDL Study- A method detection limit study shall be performed on each matrix (soil/water) on a yearly basis as the minimum frequency. An MDL study should be performed whenever changes to the instrument affect response in a fashion to render the previous MDL study meaningless. For further information, refer to ASL SOP14.
- 9.6 Uncertainty of measurements-The uncertainty of measurements shall be calculated by following ASL SOP30. In brief, SOP30 defines the uncertainty of measurements for an analysis as being 3 times the standard deviation of at least 20 blank spike analyses. At the request of each client, the uncertainty of measurement shall be included with the analytical report. The major source of uncertainty for this method is sample preparation; specifically, the spiking of standards and samples.
- 9.7 Continuing calibration verification- A continuing calibration standard shall be analyzed before sample analysis, after every ten samples, and after all samples are analyzed. The calibration verification shall be a mid level standard, and will not be varied in concentration for AFCEE clients. If the concentrations vary by more than $\pm 20\%$ from the initial calibration then recalibration may need to be performed. In the event samples are analyzed and a cal check fails in the run and not enough sample is left to re-analyze, the data must be reported with qualifiers and the discrepancy noted on the case narrative. Notify the project manager immediately in this case.
- 9.8 Method blank and analytical blank - Two blank samples are required for AK101 methodology.
- 9.8.1 The analytical blank is a water sample containing only an equivalent aliquot of methanol as used in standard sample analysis (in most cases herein 2.2 mL methanol will suffice). This sample is reported only for use as a monitoring device. Run one per 20 samples.
- 9.8.2 Analyze method blank samples at a frequency of 1 in 20 samples analyzed or 1 per day, whichever is more frequent. If the laboratory blanks show contamination above $\frac{1}{2}$ the reporting limit, the cause of the contamination will be investigated and corrective action taken before sample analysis begins. A proper method blank for soils will contain a similar aliquot of methanol added to the VOA vial as for the field samples. No method blank subtraction from any sample or QC data is allowed.
- 9.9 Blank spike/Lab fortified blank (BS/LFB) - Analyze a blank spike sample at a frequency of 1 in 20 samples analyzed or 1 per day, whichever is more frequent. Spike the samples at the midpoint of the calibration curve. The BS shall be prepared with the primary calibration stock standard, not a second source standard. The acceptance criteria are listed in Table 1. If the BS fails, refer to section 9.14 for reporting.
- 9.10 Matrix spike- A matrix spike sample and duplicate, (MS/MSD) are prepared at a rate of 1 set per 20 field samples or 1 set per day, whichever is more frequent. MS samples should be chosen randomly from a client batch of samples unless they are pre-selected by the client. Analysts should rotate the client selected for matrix spikes so that recovery and precision data is collected from a wide variety of sample matrices. An MS sample pair should be processed with each analytical batch if there is sufficient sample. If sufficient sample is not provided then the BS/BSD sample results will be used to evaluate analytical batch recovery and precision. This should be noted in the case narrative. Acceptance criteria for the MS/MSD are noted in Table 1. Poor recoveries of analytes from a matrix spike sample may indicate matrix interference from the sample or instrument problems. Poor matrix spike recovery or other evidence of method matrix interference should be reported to the client whose sample was used to prepare the MS. If the MS fails, refer to section 9.14 for reporting.

Table 1-TPH Gas BS/MS Recovery/RPD limits

| Test | Matrix | % Rec. Limits | RPD Limits |
|------------------------|--------|---------------|------------|
| AFCEE (5035/8015) | Soil | 57-146% | ≤ 50% |
| AFCEE (5030/8015) | Water | 67-136% | ≤ 30% |
| AK 101 | Soil | 60-120% | ≤ 20% |
| AK 101 | Water | 60-120% | ≤ 20% |
| Most stringent limits* | Soil | 60-120% | ≤ 20% |
| Most stringent limits* | water | 67-120% | ≤ 20% |

*These are the requirements the onsite lab needs to meet to satisfy all applicable parties.

9.11 Duplicate - A duplicate sample is prepared at a rate of 1 set per 20 field samples. In all cases, a matrix spike/spike duplicate pair should be chosen if sample volume is sufficient. If sufficient sample is not provided then the BS/BSD sample results will be used to evaluate analytical batch precision. This should be noted in the case narrative. Poor duplicate precision may indicate problems with the sample composition and should be reported to the client whose sample was selected for the duplicate. If the duplicate fails to meet the criteria in Table 1, refer to section 9.14.

9.12 Surrogate - a,a,a-Trifluorotoluene and 4-bromofluorobenzene will be used as the field sampling surrogate and the laboratory fortified surrogate, respectively. Upon failure of a surrogate spike to meet recovery limits, re-analyze the sample one time to ensure proper instrument performance. If the field added surrogate spike does not meet acceptance criteria and the laboratory added spike *does* meet criteria, notify acting project manager for instructions. For a second failure of surrogate spikes refer to section 9.14. Reporting of data with high surrogate recoveries that exhibit no analyte hits above the MDL is acceptable with a note in the case narrative. In the event of limited sample to re-analyze, inform acting onsite project manager of the situation.

Table 2 - Surrogate recovery table

| Test | SS Compound | Matrix | % Rec. Limits |
|------------------------|--|--------------|---------------|
| AFCEE (lab spike) | 4-bromofluorobenzene | Soil | 60-140% |
| AFCEE (lab spike) | 4-bromofluorobenzene | Water | 75-125% |
| AFCEE (field spike) | a,a,a-trifluorotoluene | Soil | 50-150%** |
| AFCEE (lab spike) | a,a,a-trifluorotoluene | Water | 80-114% |
| AK 101 (lab spike) | 4-bromofluorobenzene | Soil / Water | 60-120% |
| AK 101 (field spike) | a,a,a-trifluorotoluene | Soil | 50-150% |
| AK 101 (field spike) | a,a,a-trifluorotoluene | Water | 50-150% |
| Most stringent limits* | 4-bromofluorobenzene (lab spiked surrogate) | Soil | 60-120% |
| Most stringent limits* | a,a,a-trifluorotoluene (field spiked surrogate) | Soil | 50-150%** |

*These are the requirements the onsite lab needs to meet to satisfy all applicable parties.

** AFCEE does not state field surrogate limits; as directed by AFCEE contact, CH2M HILL will use ADEQ limits here.

9.13 Retention Time Standard- At the beginning of each batch or every 24 hours, whichever comes first, a retention time standard appropriate to the method will be analyzed to ensure proper instrument function. This RTS will contain n-hexane (C6) and n-decane (C10). The integration range is from the beginning of the C6 peak to the beginning of the C10 peak. RTS sample concentrations will be approximately 500 ug/L. Retention times must be within 3 standard deviations of the mean retention time shift over a 72 hour period (1 injection every 24 hours; calculate standard deviation; 3sd is maximum allowable time shift).

- 9.14 If any one of the QC parameters on this list is out of compliance, then the cause will be determined and corrective action taken. Record the out-of-compliance events and remedy in the corrective action logbook for the onsite laboratory. The on-site project manager must be made aware of the situation immediately in order to contact the client if applicable. If necessary, rerun all samples analyzed while the system was out of compliance. If insufficient sample is left to re-extract and analyze, then report the data with qualifiers and make a statement of the occurrence on the case narrative. Re-analysis and/or re-sampling and re-analysis will be determined on a case by case basis via the laboratory project manager contacting the project manager for the client.

10.0 PROCEDURE

- 10.1 All volumes, spikes, and ratios herein are based on the following assumptions:
- 10.1.1 The GC is calibrated to 0.50 ug/L for the BTEX analytes (PID) and 50 ug/L for TPH-gasoline range organics (FID).
 - 10.1.2 All field samples are collected in a 1:1 ratio of sample/methanol with a nominal value of 25 mL surrogated methanol and 25 g of sample.
 - 10.1.3 2.2 mL of surrogated methanol from each soil sample will be added to a 44.0 mL VOA vial containing 41.8 mL of organic free water; 5.0 mL of this VOA vial will be transferred to a sparge tube and purged. The concentration of a,a,a-trifluorotoluene in the sparge tube will be 200 ug/L.
 - 10.1.4 Any change in these items will change the reporting limits of the method and shall be verified with the acting project manager.
- 10.2 Tared sampling jars
- 10.2.1 Jars sent to the field for sample collection must be pre-tared to the nearest 0.01g. It is recommended that stickers not be used on the sample vials due to the necessity of pre and post weighing each sample. Instead, writing with sharpie on the sample top and side will be used to identify samples. Note that sharpie pen will come off with methanol. Each individual sample container should be placed in a bag that identifies the sample with a proper ID tag on the bag. In no case should this tag be attached to the sample jar.
 - 10.2.2 Pre-weigh the sample jars and label as date weighed with an added number afterwards (i.e. 051107-01) on the top and side of the container. Keep this information in a spreadsheet on the TPH computer in the onsite laboratory. The files will be updated with the final weights when the sample jars are returned from the field. After weighing out a complete batch, save the excel sheet and print off a hard copy to keep in a 3 ring binder.
- 10.3 Percent solids
- 10.3.1 Percent solids must be performed on all soil and sediment samples for use in final calculations. Because of the potential for gasoline or related compounds at high concentrations all drying shall be performed near or in a functioning hood.
 - 10.3.2 Open a new solids bench sheet on the computer, print off for use.
 - 10.3.3 Label and pre-weigh an aluminum weigh-boat.
 - 10.3.4 Weigh out approximately 10 g of soil, record the weight of sample and weigh boat to the nearest 0.01 g on the bench sheet or the computer if available.
 - 10.3.5 Dry the sample overnight in a 105°C oven.
 - 10.3.6 Remove the sample from the oven and place in a dessicator to cool to room temperature. Weigh the dried sample and record this post weight. Divide the post weight by the initial weight and multiply by 100 to get the % solids of the sample. Record all data on an excel sheet and print daily, keeping in a binder for solids data.
- 10.4 Soil samples
- 10.4.1 Allow standards and samples to equilibrate to room temperature before analysis.
 - 10.4.2 Document all sample preparations on the volatiles extraction log sheets, including lot #'s and spike Id's for all samples analyzed.

- 10.4.3 Samples arriving from the field must be weighed prior to removing any sample for analysis. These shall be weighed to the nearest 0.01g and the weights recorded onto the proper log sheet/computer file for later use in calculating the concentration of analyte in each sample. Swirl the contents of the jar to break up any soil chunks and mark the meniscus of the methanol once the solids have settled.
- 10.4.4 Best results are achieved after samples sit in the methanol for 48 hours before analyzing. This 48 hour wait time is not expected to be acceptable at the onsite laboratory, as fast turn around time is the reason for the on-site lab. Both the client and regulating body(s) need to accept this modification or provide a suitable alternative. Noting in the case narrative may be acceptable, but all parties need to be aware of this modification.
- 10.4.5 For calculation purposes, based on total mass, the density of methanol is 0.791 g/mL at 25°C.
- 10.5 Spiking regiment – refer to Table 3 and Table 4 below regarding spiking of all samples.
- 10.6 Sample preparation
- 10.6.1 Client samples are prepared by spiking 2.2 mL of the surrogated methanol/sample extract into a VOA vial filled within 3 mL of the top. After adding the methanol, the VOA vial is quickly filled to a slightly convex meniscus with water and capped. The proper spikes from Table 4 are added depending on the sample type.
- 10.6.1.1 Sample dilutions are performed by adding lower volumes of methanol to the VOA vial and calculating the change in volume as the dilution factor. This is acceptable up to a 100x dilution (22 uL methanol added to a VOA); beyond this point secondary dilutions in volumetric flasks are necessary.

Table 3A - Summary of gasoline calibration spikes

| Standard | Spike Volume (uL) | Gas Std Conc., (ug/mL) | SS 1* Vol., (ul) | SS 2** Vol., (ul) | SS Std 1 & 2 Conc., ug/mL | Spl Conc./ SS conc., ug/L in 44mL VOA |
|-------------|-------------------|------------------------|------------------|-------------------|---------------------------|---------------------------------------|
| Level 1 gas | 2.2 | 2000 | 0.55 | 0.55 | 2000 / 2000 | 100 / 25 ug/L |
| Level 2 gas | 4.4 | 2000 | 1.1 | 1.1 | 2000 / 2000 | 200 / 50 ug/L |
| Level 3 gas | 8.8 | 2000 | 2.2 | 2.2 | 2000 / 2000 | 400 / 100 ug/L |
| Level 4 gas | 22 | 2000 | 4.4 | 4.4 | 2000 / 2000 | 1000 / 200 ug/L |
| Level 5 gas | 44 | 2000 | 8.8 | 8.8 | 2000 / 2000 | 2000 / 500 ug/L |
| Level 6 gas | 88 | 2000 | 22 | 22 | 2000 / 2000 | 4000 / 1000 ug/L |
| ICV-Gas | 22 | 2000 | --- | --- | --- | 1000 ug/L |

Table 3B - Summary of BTEX calibration spikes

| Standard | Spike Volume (uL) | BTEX Std Conc., (ug/mL) | SS 1* Vol., (ul) | SS 2** Vol., (ul) | SS Std 1 & 2 Conc., ug/mL | Spl. conc., ug/L in 44mL VOA |
|--------------|-------------------|-------------------------|------------------|-------------------|---------------------------|------------------------------|
| Level 1 BTEX | 1.1 | 20.0 | N/A | N/A | N/A | 0.5 ug/L |
| Level 2 BTEX | 4.4 | 20.0 | N/A | N/A | N/A | 2.0 ug/L |
| Level 3 BTEX | 8.8 | 20.0 | N/A | N/A | N/A | 4.0 ug/L |
| Level 4 BTEX | 2.2 | 200 | N/A | N/A | N/A | 10.0 ug/L |
| Level 5 BTEX | 4.4 | 200 | N/A | N/A | N/A | 20.0 ug/L |
| Level 6 BTEX | 22.0 | 200 | N/A | N/A | N/A | 100 ug/L |
| ICV-BTEX | 4.4 | 200 | N/A | N/A | N/A | 20.0 ug/L |

Table 4 - Summary of sample preparation spikes

| Standard | Spike Volume (uL) | Gas Std Conc., (ug/mL) | SS 1* Vol. (ul) – Field spiked SS | SS 2** Vol., (ul) | SS Std 1 & 2 Conc., ug/mL | Spl Conc./ SS conc. in 44mL VOA |
|-----------------|--|------------------------|-----------------------------------|-------------------|---------------------------|---------------------------------|
| CV | 22 | 2000 | 4.4 | 4.4 | 2000 / 2000 | 1000 / 200 ug/L |
| Method blank | --- | --- | 4.4 | 4.4 | 2000 / 2000 | NA / 200 ug/L |
| Water blank | Add 2.2 mL of methanol to 44 mL VOA filled with organics free water. | | | | | |
| Equipment blank | --- | --- | 4.4 | 4.4 | 2000 / 2000 | NA / 200 ug/L |
| Trip Blank | --- | --- | 4.4 | 4.4 | 2000 / 2000 | NA / 200 ug/L |
| Matrix Spike† | 22 | 2000 | Field spiked | 4.4 | 2000 / 2000 | 1000 / 200 ug/L |
| Blank Spike | 22 | 2000 | 4.4 | 4.4 | 2000 / 2000 | 1000 / 200 ug/L |
| Field Sample | --- | --- | Field spiked | 4.4 | 2000 / 2000 | NA / 200 ug/L |

* Surrogate 1 is a,a,a-trifluorotoluene and is used as the field preservative surrogate spike.

**Surrogate 2 is 4-bromofluorobenzene and is the laboratory spiked surrogate.

† The matrix spike, if requested, can either be field sampled as a separate sample or from a second portion of the methanol from the native sample. If a portion of the native sample is used, follow this regiment. If a second sample is taken, spike the methanol preservative in the jar (with the soil) accordingly and follow the “field sample” spiking.

- 10.6.2 All client samples, calibration samples, and QC samples are diluted into a 44 mL VOA vial with organics free water prior to analysis. Using an Archon auto sampling device, 5.0 mL of sample is transferred to a sparge tube and purged for 11 min at 40 mL/min purge flow (at ambient temperature).
- 10.6.3 Archon setup
- 10.6.3.1 Place samples in proper Archon spot.
- 10.6.3.2 Program the system for the total number of vials, the proper method to use, and start the run.
- 10.6.4 Purge and trap system-set system to purge 5 mL of water for 11 minutes at ambient temperature using a 40 mL/min flow rate. The desorb time is 4 minutes (minimum) and the bake time shall be 8 minutes (minimum) at the trap maximum, normally 260°C for BTEX traps.
- 10.6.5 Sample analysis batch - a proper analysis batch will contain the following, in this order:
- 10.6.5.1 ADEQ instrument blank (2.2 mL methanol in 44 mL water)
- 10.6.5.2 Retention time standard
- 10.6.5.3 Calibration verification
- 10.6.5.4 Laboratory fortified blank / blank spike sample and LFB/BS duplicate
- 10.6.5.5 Method blank
- 10.6.5.6 Up to 10 samples counting client specified matrix spike, if applicable
- 10.6.5.7 Calibration verification
- 10.6.5.8 10 samples
- 10.6.5.9 Calibration verification
- 10.6.6 Calibration batch - A proper calibration sequence will contain the following, in this order:
- 10.6.6.1 ADEQ instrument blank
- 10.6.6.2 Retention time standard
- 10.6.6.3 Method blank
- 10.6.6.4 All calibration levels for gasoline

- 10.6.6.5 ICV (second source verification) for gasoline
- 10.6.6.6 All calibration levels for BTEX
- 10.6.6.7 ICV (second source verification) for BTEX
- 10.8 GC Analysis
 - 10.8.1 GC analysis is performed using these operating parameters
 - Injector Temperature = 250°C
 - Detector Temperature = 250°C
 - Head pressure = 20psig at 40°C, constant flow
 - Hydrogen Flow = 25-35mL/min
 - Air Flow = 350mL/min
 - Make-up Gas Flow = 30mL/min
 - PID power-set to a low range (20-40% power) using the H mode.
 - GC Temperature Program = Initial temperature of 40°C and hold for 1.0 minutes; ramp the temperature 10°C/min to 240°C, hold 2 minutes. Total run time is 23 minutes. This may be run faster as long as AK101 separatory criteria are met.
 - 10.8.2 Purge and trap conditions:
 - Purge flow--40mL/minute.
 - Purge time--11minutes.
 - Desorb time--4minutes.
 - Bake time--8minutes.
 - Purge temperature – ambient
 - Trap temperature -- 25°C (ambient temperature will dictate this)
 - Desorb temperature -- 240°C.
 - Bake temperature -- 260°C.
 - Valve temperature -- 120°C
 - XFER temperature -- 120°C
 - 10.8.3 Capillary Column: J&W Scientific DB-VRX 75m x 0.45mm I.D. x 2.55um film thickness (part number 124-1574) or equivalent.

11.0 DATA REDUCTION

- 11.1 Qualitative Analysis: The identification of gasoline is based on pattern recognition of the chromatographic fingerprint of the sample with that from the gasoline standard. If compounds are detected within the quantitation range of gasoline, but the pattern does not match the chromatographic fingerprint of the gasoline standard, a value is reported based on the response factor for gasoline and a comment is made in the case narrative. Significant interferences may be encountered due to the presence of other petroleum products (or non-petroleum products) eluting within the retention time range of the volatile petroleum product being analyzed. Most associated calculations for this method may be found in Table 5 at the end of this document.
- 11.2 Sum the area of all the peaks eluting in the specified carbon ranges. This area is generated by projecting a horizontal baseline between the retention times of the initial and final carbon compounds. The surrogate(s) standard areas must be removed from the target area by tangent skimming the peaks from the TPH “hump”.
- 11.3 Sample and standards must be integrated in the same fashion. If the sample area exceeds the calibration standard area, then the sample is diluted and reanalyzed. If the sample area is below the reporting limit, then the sample is reported as a non-detect.
- 11.4 Samples are quantitated using the following equations:

Aqueous Samples:

$$C_s \text{ (mg/L)} = (A_x)(D) / [(RF)(V_s)]$$

Where:

C_s = Concentration of Gasoline Range Organics

RF = Response factor, as described in Section 9.8.4

A_x = Response for the Gasoline Range Organics in the sample, units in area

V_s = Volume of sample purged, in liters.

D = Dilution factor, if dilution was performed on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

Solid samples (methanol extraction):

$$C_s \text{ (mg/kg)} = (A_x)(V_t)(D) / [(RF)(W)(V_i)]$$

Where:

V_t = Volume of total extract (uL) (use 10000uL for standard 25mL extract volume).

V_i = Volume of extract actually purged (uL)

W = Weight of sample extracted, kg. The dry wet weight is used. A_x, RF, and D have the same definition as above.

12.0 DOCUMENTATION

- 12.1 All raw electronic data and any associated electronic bench sheets will be backed up to CD/DVD on a weekly basis.
- 12.2 Instrument printouts and all raw data are kept in the file cabinet in the instrument lab and filed by date of analysis. This data will be returned to the CH2M HILL Applied Sciences Laboratory to be kept for one year onsite, then archived a total of 7 years time from date of creation.
- 12.3 Extraction logs used herein are printed and kept in a dedicated laboratory binder.
- 12.4 All chromatograms are to be initialed and dated by the analyst. In the case of tangent skimmed peaks (all samples but the RTS), a reviewer needs to initial the integration as being acceptable. There is no "before" manual integration chromatogram printed off for TPH analysis, as all samples need to be tangent skimmed.
- 12.5 Calibration sample chromatograms need to be initialed and dated by the analyst, a peer reviewer, and the acting onsite QA Officer.

13.0 REFERENCES

- 13.1 *Test Methods for Evaluating Solid Waste*, 3rd ed. Update III, December 1996, Method 8015.
- 13.2 *Test Methods for Evaluating Solid Waste*, 3rd ed. Update III, December 1996, Method 8021B.
- 13.3 *Test Methods for Evaluating Solid Waste*, 3rd ed. Update III, December 1996, Method 5030.
- 13.4 *Test Methods for Evaluating Solid Waste*, 3rd ed. Update III, December 1996, Method 5035.
- 13.5 Leaking Underground Fuel Tank Field Manual: *Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure*, State of CA LUFT Task Force, May 1988.
- 13.6 Washington State Dept. of Ecology, *NWTPH-Gx: Volatile Petroleum Products Method for Soil and Water*. Analytical Methods for Petroleum Hydrocarbons, Publication No. ECY 97-602, June 1997.
- 13.7 AFCEE QAPP Version 3.0, 3.1, and 4.0.
- 13.8 Pilgrim, Mary Jane. *Method AK101-For the Determination of Gasoline Range Organics*. Version 04/08/02, State of Alaska Department of Environmental Conservation.

14.0 DEFINITIONS

- 14.1 Surrogate Standard (SS)—A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(S) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SS is to monitor method performance with each sample.
- 14.2 Laboratory Duplicates—Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of duplicates indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
- 14.3 Field Duplicates—Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedure. Analyses of Duplicates gives a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 14.4 Laboratory Replicates—The same sample run two times on any instrument.
- 14.5 Laboratory Reagent Blank or Method Blank (WB1, SB1, XB1)—An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 14.6 Trip Blank (TB)—An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the TB is to determine if method analytes or other interferences are present in the field environment.
- 14.7 Calibration Check Verification (CCV)—A solution of one or more compounds (analytes, surrogates, internal standard, or other test compounds) used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
- 14.8 Blank Spike/Laboratory fortified blank (BS1S)—An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 14.9 Blank Spike Duplicate/Laboratory fortified blank duplicate (BD1S)—An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BD is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 14.10 Matrix Spikes/Spike Duplicates (MS/MSD)—An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.
- 14.11 Stock Standard Solution (SSS)—A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.
- 14.12 Primary Standard Solution (PSS)—A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 14.13 Calibration Standard (CAL)—A solution prepared from the primary standard solution or stock standard solution and the internal standards and surrogate analytes. The Cal solutions are used to calibrate the instrument response with respect to analyte concentration.

- 14.14 Initial Calibration Verification (ICV)—A solution of method analytes of known concentrations which is used to fortify an aliquot of WB1 or sample matrix (MS). The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.
- 14.15 Procedural Standard Calibration—A calibration method where aqueous calibration standards are prepared and processed (e.g. purged, extracted, and /or derivatized) in exactly the same manner as a sample. All steps in the process from addition of sampling preservatives through instrumental analyses are included in the calibration. Using procedural standard calibration compensates for any inefficiencies in the processing procedure.

Table 5

| STATISTIC | SYMBOL | FORMULA | DEFINITION | USES |
|-----------------------------------|-----------|---|---|---|
| 1. Mean | \bar{X} | $\frac{\left(\sum_{i=1}^n X_i\right)}{n}$ | Measure of central tendency | Used to determine the central value of a set of measurements. |
| 2. Standard Deviation | S | $S = \left[\frac{\sum (X_i - \bar{X})^2}{n - 1} \right]^{1/2}$ | Measure of the relative scatter of data | Used in variation of measurements |
| 3. Relative Standard Deviation | RSD | $\left(S / \bar{X} \right) \times 100$ | Adjusts for the magnitude of observations when calculating (S) | Used to assess precision for replicate results |
| 4. Percent Difference | %D | $\left[\frac{X_1 - X_2}{X_1} \right] \times 100$ | Measure of the difference of two observations | Used to assess accuracy |
| 5. Relative Percent Difference | RPD | $\left[\frac{X_1 - X_2}{X_1 + X_2 / 2} \right] \times 100$ | Measure of variability that adjusts for the magnitude of observations | Used to assess total and analytical precision of duplicate measurements |
| 6. Percent Recovery (Blank spike) | %Rec | $\left[\frac{\text{Experimental Value}}{\text{True value}} \right] \times 100$ | Recovery of spiked compound in pure matrix | Used to assess accuracy |
| 7. Percent Recovery (Sample) | %Rec | $\left[\frac{\text{spiked sample result} - \text{unspiked sample result}}{\text{value of spike added}} \right] \times 100$ | Recovery of spiked compound in sample matrix | Used to assess matrix effects and total precision |

Figure 1- Volatile extraction log sheet

| CH2M HILL APPLIED SCIENCES VOLATILES SOIL EXTRACTION LOG METHOD 5030/5035 | | | | | | | | | | |
|--|-----------|----------------------|-------------------------|-----------------------|-------------|-------------|---------------|----------------|----------|--|
| Project Name: _____ | | | Lab Batch: _____ | | | | | | | |
| Date Extracted: _____ | | | Analytical Batch: _____ | | | | | | | |
| Analyst: _____ | | | | | | | | | | |
| LAB ID # | CLIENT ID | VIAL TARE WEIGHT (g) | SAMPLE+VIAL MASS (g) | FINAL SAMPLE MASS (g) | SS VOL (uL) | EXTR LEVEL* | MEOH VOL (mL) | WATER VOL (mL) | COMMENTS | |
| 1 | Blank | | | | | | | 5.0mL | | |
| 2 | LCS | | | | | | | 5.0mL | | |
| 3 | | | | | | | | 5.0mL | | |
| 4 | | | | | | | | 5.0mL | | |
| 5 | | | | | | | | 5.0mL | | |
| 6 | | | | | | | | 5.0mL | | |
| 7 | | | | | | | | 5.0mL | | |
| 8 | | | | | | | | 5.0mL | | |
| 9 | | | | | | | | 5.0mL | | |
| 10 | | | | | | | | 5.0mL | | |
| 11 | | | | | | | | 5.0mL | | |
| 12 | | | | | | | | 5.0mL | | |
| 13 | | | | | | | | 5.0mL | | |
| 14 | | | | | | | | 5.0mL | | |
| 15 | | | | | | | | 5.0mL | | |
| 16 | | | | | | | | 5.0mL | | |
| 17 | | | | | | | | 5.0mL | | |
| 18 | | | | | | | | 5.0mL | | |
| 19 | | | | | | | | 5.0mL | | |
| 20 | | | | | | | | 5.0mL | | |
| 21 | | | | | | | | 5.0mL | | |
| 22 | | | | | | | | 5.0mL | | |
| 23 | | | | | | | | 5.0mL | | |
| 24 | | | | | | | | 5.0mL | | |
| 25 | | | | | | | | 5.0mL | | |
| 26 | | | | | | | | 5.0mL | | |
| 27 | | | | | | | | 5.0mL | | |
| 28 | | | | | | | | 5.0mL | | |
| 29 | | | | | | | | 5.0mL | | |
| 30 | | | | | | | | 5.0mL | | |

* = Extraction Level High = Methanol extraction Low = direct heated sparge

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Appendix A

Taken from AFCEE Version 4.0.01, 2007.

7.2.1.10 QC Checks for GC Methods

Table 7.2.1.10-1 details the QC checks and associated minimum frequencies, acceptance criteria, corrective actions, and flagging criteria for the GC methods listed in Sections 7.2.1.1 through 7.2.1.9. The QC checks include initial and continuing calibration requirements, retention time verifications, and other checks for precision and accuracy.

Table 7.2.1.10-1. Summary of Calibration and QC Procedures for GC Methods SW8011, SW8015B, SW8021B, SW8070A, SW8081A, SW8082, SW8141A, SW8151A, and RSK-175

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action ^a | Flagging Criteria ^b |
|--|--|---|---|---|
| Breakdown check (Endrin and DDT, Method SW8081A only) | Daily prior to analysis of sample | Degradation $\leq 15\%$ for each analyte | Correct problem then repeat breakdown check. | Problem must be corrected. Samples shall not be run until degradation is $\leq 15\%$. |
| Initial multi-point calibration for all analytes (minimum five standards) (ICAL) | Initial calibration prior to sample analysis | One of the options below (except for Method 8082 which may only use Option 1 or 2): <i>Option 1:</i> linear – RSD for each analyte $\leq 20\%$ <i>Option 2:</i> linear – least squares regression $r \geq 0.995$ for each analyte. <i>Option 3:</i> non-linear – COD ≥ 0.99 (six points shall be used for second order, seven points shall be used for third order) not applicable for SW8082 | Correct problem then repeat initial calibration. | Problem must be corrected. Samples may not be analyzed until there is a valid ICAL. |
| Second-source calibration verification | Once after each ICAL | All analytes within $\pm 20\%$ of expected value | Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. | Problem must be corrected. Samples may not be analyzed until the calibration has been verified. |

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action ^a | Flagging Criteria ^b |
|---|--|--|--|--|
| Retention time window position established for each analyte and surrogate | Each ICAL and after the initial daily CCV | Position shall be set using the midpoint standard of the initial calibration curve. | N/A | N/A |
| Retention time window width established for each analyte and surrogate | At method set-up and after major maintenance (e.g., column change) | 3 times standard deviation for each analyte (each quantitation peak SW8082) retention time from 72-hour study GRO: calculate retention time based on 2-methylpentane and 1,2,4-trimethylbenzene (see 7.4.2 in Method SW8015A). DRO: calculate retention time based on C10 and C28 alkanes (see 7.4.3 in Method SW8015A). | N/A | N/A |
| Retention time window verification for each analyte and surrogate | Each calibration verification | Analyte within established window | Correct problem then reanalyze all samples analyzed since the last acceptable retention time check. | ICV: Flagging criteria are not appropriate for initial verification. CCV: Apply Q-flag to all results for the specific analyte(s) in the sample which are outside the established window. |
| Calibration verification: initial (ICV) and continuing (CCV) | ICV: Daily, before sample analysis, unless ICAL performed on same day CCV: After every 10 samples and at the end of the analysis sequence | All analytes within $\pm 20\%$ of expected value (% D) | ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. CCV: Correct problem then repeat CCV. Reanalyze all samples since last successful calibration verification. | ICV: Flagging criteria are not appropriate. CCV: Apply Q-flag to all results for the specific analyte(s) >20 %D for all samples associated with the calibration verification. |

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action ^a | Flagging Criteria ^b |
|--|---|---|---|--|
| Method blank | One per analytical batch | No analytes detected > ½ RL. For common lab contaminants no analytes detected > RL. | Assess data. Correct problem. If necessary, reprep and analyze method blank and all samples processed with the contaminated blank. | Apply B-flag to all associated positive results for the specific analyte(s) as appropriate. See guidance Section 8.2.1.4 |
| LCS for all analytes (For SW8082 PCB 1016/1260 mix) | One LCS per analytical batch | Acceptance criteria: Tables 7.2.1.1-2 through 7.1.1.9-2, respectively. See Section 4.4.1.2 for guidance on determining marginal exceedances. | Correct problem then reanalyze. If still out, reprep and re-analyze the LCS and all samples in the affected AFCEE batch. | If corrective action fails, apply Q-flag to the specific analyte(s) which are not marginal exceedances in all samples in the associated preparatory batch. |
| Surrogate spike | Every sample, spiked sample, standard, and method blank | Acceptance criteria: Tables 7.2.1.1-2 through 7.1.1.9-2, respectively. | Correct problem then re-extract and reanalyze the affected samples. If matrix effect is verified, discuss in case narrative. | For the samples: If the %R > UCL for any surrogate, apply J-flag to all positive results for associated analytes. If the %R < LCL for any surrogate, apply J-flag to all positive results for associated analytes and UJ -flag to all associated non-detects. If any surrogate recovery is <10%, apply Q-flag to all results for all associated analytes. |

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action ^a | Flagging Criteria ^b |
|--|--|--|--|---|
| MS/MSD | One MS/MSD per every 20 Air Force project samples per matrix | Acceptance criteria: Tables 7.2.1.1-2 through 7.1.1.9-2, respectively. | Assess data to determine whether there is a matrix effect or analytical error. Review LCS for failed target analytes. Potential matrix effects should be communicated to the prime contractor so an evaluation can be made with respect to the PQOs. | For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply M-flag if; (1)%R for MS or MSD > UCL (2)%R for MS or MSD < LCL or (3)MS/MSD RPD > CL |
| Second-column confirmation (not required for Method 8081A toxaphene and chlordane; Method 8015B DRO or GRO; RSK-175) | 100% for all positive results | Same as for initial or primary column analysis | Same as for initial or primary column analysis | Apply J-flag if RPD >40% from first column result. Apply Q-flag to all results for the specific analyte(s) in the sample not confirmed. |
| MDL study | At initial setup and subsequently once per 12-month period or quarterly MDL verification checks. | Detection limits established shall be $\leq \frac{1}{2}$ the RLs in Tables 7.2.1.1-1 through 7.2.1.9-1. See 40 CFR, Part 136 Appendix B. All analytes must be detected and identified by method-specified criteria for the for the verification check to be valid, or the verification check must produce a response that is at least 3X the instrument noise level and greater than the response in the blanks associated with the MDL study. | Run MDL verification check at higher level and set higher MDL or reconduct MDL study. | N/A |
| Results reported between MDL and RL | None | None | None | Apply F-flag to all results between MDL and RL. |

- a. All corrective actions associated with AFCEE project work shall be documented, and all records shall be maintained by the laboratory.
- b. Flagging criteria are applied when acceptance criteria were not met and corrective action was not successful or corrective action was not performed.

**Table 7.2.1.2-2. QC Acceptance Criteria for Method SW8015B
Volatile and Extractable Total Petroleum Hydrocarbons (TPH)**

| Analyte | Accuracy Water (% R) | Precision Water RPD (%) | Accuracy Soil (% R) | Precision Soil RPD (%) |
|-----------------------------------|----------------------|-------------------------|---------------------|------------------------|
| TPH–Gasoline Range Organics (GRO) | 67–136 | ≤ 30 | 57–146 | ≤ 50 |
| <i>Surrogates (choose 1):</i> | | | | |
| a,a,a-Trifluorotoluene | 80–114 | - | 60–140 | - |
| Chlorobenzene | 74–138 | - | 64–148 | - |
| 4-Bromofluorobenzene | 75–125 | - | 60–140 | - |
| TPH–Diesel Range Organics (DRO) | 61–143 | ≤ 30 | 51–153 | ≤ 50 |
| TPH–Jet Fuel | 61–143 | ≤ 30 | 51–153 | ≤ 50 |
| <i>Surrogates (choose 2):</i> | | | | |
| Octacosane | 26–152 | - | 25–162 | - |
| o-Terphenyl | 57–132 | - | 47–142 | - |
| Fluorobenzene | 75–125 | - | 65–135 | - |
| Tricontane | 40–140 | - | 30–150 | - |

**Table 7.2.1.3-2. QC Acceptance Criteria for Method SW8021B
Aromatic and Halogenated Volatile Organics**

| Analyte | Accuracy Water (% R) | Precision Water RPD (%) | Accuracy Soil (% R) | Precision Soil RPD (%) |
|---------------------------|----------------------|-------------------------|---------------------|------------------------|
| 1,1,1,2-Tetrachloroethane | 75–125 | ≤ 20 | 65–125 | ≤ 30 |
| 1,1,1-TCA | 69–134 | ≤ 20 | 59–134 | ≤ 30 |
| 1,1,2,2-Tetrachloroethane | 30–166 | ≤ 20 | 25–166 | ≤ 30 |
| 1,1,2-TCA | 61–130 | ≤ 20 | 51–130 | ≤ 30 |
| 1,1-DCA | 64–127 | ≤ 20 | 54–127 | ≤ 30 |
| 1,1-DCE | 53–147 | ≤ 20 | 43–147 | ≤ 30 |

| Analyte | Accuracy Water (% R) | Precision Water RPD (%) | Accuracy Soil (% R) | Precision Soil RPD (%) |
|-----------------------------|----------------------|-------------------------|---------------------|------------------------|
| 1,1-Dichloropropene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,2,3-Trichlorobenzene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,2,3-Trichloropropane | 75–125 | ≤ 20 | 65–125 | ≤ 30 |
| 1,2,4-Trichlorobenzene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,2,4-Trimethylbenzene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,2-Dibromo-3-chloropropane | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,2-Dibromoethane | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,2-DCA | 68–137 | ≤ 20 | 58–137 | ≤ 30 |
| 1,2-DCB | 61–134 | ≤ 20 | 51–134 | ≤ 30 |
| 1,2-Dichloropropane | 73–125 | ≤ 20 | 63–125 | ≤ 30 |
| 1,3,5-Trimethylbenzene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,3-DCB | 63–137 | ≤ 20 | 53–137 | ≤ 30 |
| 1,3-Dichloropropane | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 1,4-DCB | 66–135 | ≤ 20 | 56–135 | ≤ 30 |
| 2,2-Dichloropropane | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 2-Chlorotoluene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| 4-Chlorotoluene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| Benzene | 75–125 | ≤ 20 | 65–125 | ≤ 30 |
| Bromobenzene | 75–125 | ≤ 20 | 65–125 | ≤ 30 |
| Bromochloromethane | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| Bromodichloromethane | 61–135 | ≤ 20 | 51–135 | ≤ 30 |
| Bromoform | 58–129 | ≤ 20 | 48–129 | ≤ 30 |
| Bromomethane | 68–125 | ≤ 20 | 58–125 | ≤ 30 |
| Carbon Tetrachloride | 69–139 | ≤ 20 | 59–139 | ≤ 30 |
| Chlorobenzene | 75–129 | ≤ 20 | 65–129 | ≤ 30 |
| Chloroethane | 75–130 | ≤ 20 | 65–130 | ≤ 30 |
| Chloroform | 49–133 | ≤ 20 | 39–133 | ≤ 30 |
| Chloromethane | 59–154 | ≤ 20 | 49–154 | ≤ 30 |
| Cis-1,2-DCE | 75–120 | ≤ 20 | 65–125 | ≤ 30 |
| Cis-1,3-Dichloropropene | 75–130 | ≤ 20 | 65–130 | ≤ 30 |
| Dibromochloromethane | 75–131 | ≤ 20 | 65–131 | ≤ 30 |
| Dibromomethane | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| Dichlorodifluoromethane | 68–125 | ≤ 20 | 58–125 | ≤ 30 |
| EDB | 75–131 | ≤ 20 | 65–131 | ≤ 30 |
| Ethylbenzene | 71–129 | ≤ 20 | 61–129 | ≤ 30 |
| Hexachlorobutadiene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| Isopropylbenzene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| m,p -Xylene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| Methylene Chloride | 42–176 | ≤ 20 | 32–176 | ≤ 30 |
| n-Butylbenzene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| n-Propylbenzene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| Naphthalene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |
| o-Xylene | 65–135 | ≤ 20 | 55–145 | ≤ 30 |

| Analyte | Accuracy Water (% R) | Precision Water RPD (%) | Accuracy Soil (% R) | Precision Soil RPD (%) |
|---------------------------|----------------------|-------------------------|---------------------|------------------------|
| p-Isopropyltoluene | 65-135 | ≤ 20 | 55-145 | ≤ 30 |
| Sec-Butylbenzene | 65-135 | ≤ 20 | 55-145 | ≤ 30 |
| Styrene | 65-135 | ≤ 20 | 55-145 | ≤ 30 |
| TCE | 75-141 | ≤ 20 | 65-141 | ≤ 30 |
| Tert-Butylbenzene | 65-135 | ≤ 20 | 55-145 | ≤ 30 |
| Tetrachloroethene | 75-142 | ≤ 20 | 65-142 | ≤ 30 |
| Toluene | 70-125 | ≤ 20 | 60-125 | ≤ 30 |
| Trans-1,2-DCE | 75-130 | ≤ 20 | 68-130 | ≤ 30 |
| Trans-1,3-Dichloropropene | 42-156 | ≤ 20 | 32-156 | ≤ 30 |
| Trichlorofluoromethane | 75-130 | ≤ 20 | 69-130 | ≤ 30 |
| Vinyl Chloride | 47-142 | ≤ 20 | 37-142 | ≤ 30 |
| Xylenes, Total | 71-133 | ≤ 20 | 61-133 | ≤ 30 |
| <i>Surrogates:</i> | | | | |
| 1,4-Dichlorobutane | 35-135 | - | 35-135 | - |
| Bromochlorobenzene | 37-137 | - | 37-137 | - |

Corvallis ASL Standard Operating Procedure

**STANDARD OPERATING PROCEDURE
FOR THE DETERMINATION OF
DIESEL AND RESIDUAL RANGE ORGANICS
IN WATER, SOIL, AND SEDIMENT BY GAS CHROMATOGRAPHY
(GC) FOLLOWING AK 102 AND AK 103 FOR USE AT THE CH2M HILL,
INC. KALAKAKET CREEK FIELD LAB**

| | |
|---|----------|
| APPROVED:  | 06/07/07 |
| QA Officer | Date |
|  | 06/07/07 |
| Laboratory Director | Date |

Documentation of reading this SOP will be kept in the ASL QAQC training database. Each analyst is responsible for entering their own training dates. By entering their name and date of reading the SOP each analyst is agreeing to the following statement:

I have read and understood the following Standard Operating Procedure (SOP) and agree to follow the SOP as written. Any exceptions to the SOP will be recorded in the appropriate logbook or benchsheet and changes will be noted in the case narrative of the report to the client.

**STANDARD OPERATING PROCEDURE
FOR THE DETERMINATION OF
DIESEL AND RESIDUAL RANGE ORGANICS
IN WATER, SOIL, AND SEDIMENT BY GAS CHROMATOGRAPHY (GC) FOLLOWING AK
102 AND AK 103 FOR USE AT THE CH2M HILL, INC. KALAKAKET CREEK FIELD LAB**

1.0 SCOPE AND APPLICATION

- 1.1 This standard operating procedure (SOP) is designed for analysis of petroleum hydrocarbons in water, sediment, and soil by gas chromatography. This method applies to diesel and oil range petroleum hydrocarbons in water, sediment, and soil from C10 to C36.
- 1.2 This document provides specific procedures for analyzing samples by Alaska Dept. of Environmental Quality (ADEQ) method AK 102 and AK 103 and AFCEE Version 4.0.01. This SOP is designed for the analysis of diesel and residual range organics in soil, sediment, and water at the CH2M Hill, Inc. Kalakaket Creek onsite laboratory. All samples received in the laboratory will be analyzed by this SOP unless project-specific amendments are made in advance.
- 1.3 Samples are extracted following EPA method 3510C (liquids) or EPA 3550B (solids). Sample extracts are then analyzed by GC-FID and follow the methods listed above. Quantitation is performed against standards of diesel fuel #2 and a 1:1 mix of 30w and 40w motor oils.

2.0 OVERVIEW OF THE ANALYTICAL PROCESS

An aliquot of sample is extracted with methylene chloride via SW3510 (water) or SW3550 (soil) and concentrated to a volume of 2 mL. Depending on client request, samples may be passed through silica gel to remove non-petroleum interferences prior to analysis. The samples are then analyzed by a gas chromatograph equipped with a flame ionization detector (FID), automated injection tower, and HP Chemstation software for data collection. The hydrocarbon ranges used can be found in Table 1.

3.0 TARGET ANALYTES, REPORTING LIMITS AND DETECTION LIMITS

- 3.1 Target analytes for this method are diesel range and oil range organics (see Table 1). Reporting limits can be found in Table 1A.
- 3.2 The MDL value is not a static number and changes every time it is run. Current MDL's will be available upon request from the on-site lab. The reporting limits will be at least 5 times the calculated MDL.

Table 1 – Hydrocarbon Ranges

| Method | Diesel range organics | Residual range organics |
|--------------|--------------------------------------|--------------------------------|
| AK 102 (DRO) | Beginning of C10 to beginning of C25 | --- |
| AK 103 (ORO) | --- | Beginning of C25 to end of C36 |

Table 1A – Reporting Limits

| Method | RL, Soil (mg/kg) | RL, Water (ug/L) |
|---------------------|------------------|------------------|
| AK 102 (DRO) | 20.0 | 800 |
| AK 103 (ORO) | 100 | Not listed |
| AFCEE (DRO) | 10.0 | 1000 |
| AFCEE (ORO) | Not listed | Not listed |
| Lowest Limits (DRO) | 10.0 | 800 |
| Lowest Limits (ORO) | 100 | Not listed |

- 3.3 For details on the procedures and criteria used to establish MDLs, consult *SOP14* and *ASL Quality Assurance Program Manual*.

4.0 INTERFERENCES

- 4.1 Samples containing non-petroleum organics that elute in the same retention windows as the target constituents may cause a positive bias in the results.
- 4.2 Biogenic material such as bark, potting soils, biota, peat moss and other commonly found soil contaminants may be falsely identified as petroleum products. Sample extract cleanup via silica gel may help to minimize the bias caused by naturally occurring organic material / biogenic interferences.

5.0 SAFETY, WASTE MINIMIZATION, AND POLLUTION PREVENTION

- 5.1 Samples contaminated with TPH constituents should be considered hazardous. Samples may include flammables, explosives, and potentially carcinogenic compounds. All samples are assumed to be hazardous and should be handled as such.
- 5.2 All stock and working calibration standards, as well as all samples, shall be handled with the utmost care using good laboratory techniques in order to avoid harmful exposure.
- 5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement.
- 5.4 The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.
- 5.5 Standards and samples shall be prepared in a fume hood. Sample preparation should be performed in a fume hood with adequate skin, eye, and hearing protection provided for and used by the analysts. Any situation creating odor levels should be immediately corrected.
- 5.6 Sample extracts and standards prepared in flammable solvents shall be stored in an explosion-proof refrigerator or a cooler.
- 5.7 Safety equipment including fire extinguisher, first aid kit, eye wash, and chemical spill cleanup kit shall be available for use at all times.
- 5.8 Laboratory wastes shall be separated and properly disposed complying with all federal, state, and local regulations. The wastes include collected solvent rinses; expired sample extracts and disposable labware (or other item as applicable) used in the preparation of the samples. These wastes shall be handled according to CVO SOP HAZ01, Waste Disposal.
- 5.9 Analysts are encouraged to reduce the amount of solvent or disposable labware waste whenever possible. More information on this topic can be found in "Less is Better: Laboratory Chemical Management Waste Reduction" located on the American Chemical Society website at http://membership.acs.org/c/ccs/pub_9.htm.

6.0 SAMPLE COLLECTION, STORAGE, HOLDING TIMES AND PRESERVATION

- 6.1 Water samples: Water samples are not expected to be analyzed at the Kalakaket Creek field lab. In the event of a change, the following procedure shall be employed.
- 6.1.1 The samples shall be collected in clean glass bottles with Teflon lined lids. Care should be taken to minimize the headspace in each sample container. Water samples should be preserved with 1+1 HCl to a pH<2 to extend sample holding time unless the project requirements specify using non-preserved samples. If the pH of the samples cannot be adjusted by acidification due to the nature of the matrix, the holding time is 7 days from the time of collection. The samples should be shipped as soon as possible and must be kept cool, 4 °C, with ice during shipment.
- 6.1.2 Soil/sediment samples: The samples shall be collected in 4-oz wide mouth amber jars with Teflon lined lids. Care should be taken to minimize the headspace in each sample container. No preservative is necessary for soil samples. The samples should be shipped as soon as possible and must be kept cool, 4 °C, with ice during shipment.
- 6.1.3 The samples must be stored at 4°C at all times until extraction is performed.

- 6.1.4 The holding time for soils or preserved water samples is 14 days. For unpreserved water samples the holding time is 7 days.
- 6.1.5 Sample extract holding time is 40 days after the date of extraction if stored at a temperature of <-10°C.

7.0 APPARATUS AND MATERIALS

- 7.1 VOA sample vials—40-mL capacity with Teflon lined septum screw caps; precleaned as purchased from I-Chem or similar quality.
- 7.2 Balance—top loading electronic with at least 2000-g capacity and 0.01g sensitivity.
- 7.3 Glassware - Class A volumetric pipets and flasks; beakers, vials, Pasteur pipets, K-D flasks, Snyder columns, and other miscellaneous glassware as necessary for extraction, preparation and handling of samples and standards.
- 7.4 Labware—Necessary for preparation and handling of samples and standards.
- 7.5 Syringes—Hamilton glass type as required for injection of sample extracts and standards, preparation of dilutions, and spiking of samples.
- 7.6 Sonicator —Heat Systems Ultrasonic Sonicator with variable control up to 375-watt output and ½” microtip disrupter horn.
- 7.7 Water bath- For concentrating sample extracts. Must be capable of maintaining 60±5°C and deep enough to hold 250 mL Kuderna-Danish concentration assemblies with 25 mL concentrating tubes.
- 7.8 Gas chromatograph (GC)—Hewlett-Packard Model 6890 or similar with temperature programming, electronic integration, report annotation, automatic sampler and flame ionization detector.
- 7.9 GC column - fused silica capillary column, ZB-5 (Zebron), 30 m x 0.32 mm with a film thickness of 0.25 µm or an equivalent column. This column must be capable of separating C24 and C25 to allow proper retention time programming.
- 7.10 Separatory funnels-2 L

8.0 STANDARDS, GASES AND REAGENTS

- 8.1 Methylene chloride – Pesticide grade or equivalent so as to produce blank samples at or below the reporting/quantitation limit.
- 8.2 Silica Gel—Grade 27, 60-200 mesh, activated at a minimum of 130°C for 16 hours.
- 8.3 Sodium sulfate—Reagent grade, anhydrous powder form baked at 400°C for >4 hours. This shall be prepared at the Corvallis lab and shipped in sealed containers to the Kalakaket Creek laboratory.
- 8.4 Calibration, surrogate, and spiking standards
 - 8.4.1 DRO stock standard – commercial grade diesel #2, diluted to 20,000 mg/L in methylene chloride solvent. Accustandard part # FU-009-D-40x.
 - 8.4.2 DRO second source stock standard – commercial grade diesel #2 from a supplier independent of the primary calibration standard. Absolute Standards part # 51006. Dilute this standard 1:20 in methylene chloride for a standard at 1000 mg/L.
 - 8.4.3 RRO stock composite mix – a 1:1 w/w mix of 30W and 40W motor oils. Absolute Standards part #51178 (a straight weight/weight mix, not diluted in solvent). Add 2.00 g of this oil mix to a 100 mL volumetric flask partially filled with methylene chloride to make a standard at 20,000 mg/L.
 - 8.4.4 RRO second source stock standard – a 1:1 mix of 30W and 40W motor oil from a supplier independent of the primary calibration standard. This 1:1 w:w mix will be prepared at the Corvallis lab and sent to the Kalakaket Creek lab.
 - 8.4.4.1 At the on-site lab, add 2.0 g of this oil to a 100 mL volumetric flask partially filled with methylene chloride, record the weight, and fill the flask to the mark with methylene chloride. Transfer this to two VOA vials or similar, log in and label properly as a 20,000 mg/L stock.
 - 8.4.4.2 Dilute this stock volumetrically to 1000mg/L by adding 5 mL of the 20,000 mg/L stock to a 100 mL volumetric flask and diluting to the mark with methylene chloride. This standard is now used in section 8.4.8.

- 8.4.5 Surrogate spike mix - ortho-terphenyl (OTP) and n-octacosane at 2000 mg/L each in methylene chloride. Order neat materials and prepare at the Corvallis lab. Sigma Aldrich part numbers T2800-25G (OTP, 25g solid) and O504-25G (octacosane, 25g solid).
- 8.4.5.1 In a 100 mL volumetric flask partially filled with methylene chloride add 0.20g of each of the surrogate solid materials. Fill the flask to the mark with methylene chloride and transfer to two VOA vials or similar, log in and label properly.
- 8.4.6 Retention time standard – contains C10, C25 and C36 at 2000 mg/L in methylene chloride. Absolute Standards part # 51174.
- 8.4.7 DRO/RRO Calibration composite standard- a standard at 4000 mg/L diesel #2 /oil composite containing both surrogates at 400 mg/L each.
- 8.4.7.1 Make this standard by combining 10 mL of 20,000 mg/L DRO stock, 10 mL of 20,000 mg/L RRO stock, and 10 mL of the 2000 mg/L surrogate spike mix in a 50 mL class A volumetric flask. Dilute to the mark with methylene chloride.
- 8.4.8 DRO/RRO second source composite standard – a standard created from second source materials at 500 mg/L in methylene chloride containing diesel #2 and a 1:1 mix of 30W and 40W oils. Create this standard by making a 50/50 v/v mix of the two second source standards (8.4.2 & 8.4.4).
- 8.4.9 DRO and RRO QC sample spiking standard – a standard for spiking extraction samples such as blank spikes and matrix spikes.
- 8.4.9.1 Add 5mL of 20,000 mg/L diesel stock (8.4.1) and 5.0 mL of 20,000 mg/L composite oil stock (8.4.3) to a 50 mL volumetric flask partially filled with methylene chloride. Dilute to the mark with methylene chloride. Standard is 2000 mg/L for DRO and RRO analytes.
- 8.5 Helium–Carrier gas, prepurified grade.
- 8.6 Hydrogen--FID gas, prepurified grade.
- 8.7 Air--FID gas, zero grade.

9.0 QA/QC

All reporting limits and acceptance criteria are subject to change on a client specific basis as requested by that client.

- 9.1 Instrument calibration–Six levels at approximately 40, 100, 200, 500, 2000, and 4000 mg/L for DRO and RRO analytes, and at 4.0, 10, 20, 50, 200, and 400 mg/L for both surrogate spikes. Diesel #2 shall be used for the diesel range calibration standard for all tests; a 1:1 mix of 30W and 40W oil will be used for all oil calibrations.
- 9.2 The lowest calibration point in the curve shall be at or below the reporting limit off the GC. Since this point drives the reporting limit, it must always be included in the calibration curve, and may not be discarded. If the low-point is an outlier, the calibration must be repeated. The only point that may be readily removed from a calibration curve is the highest point, if linearity of the detector is exceeded or column overloading occurs. If this is the case, clear documentation of this removal and reason for removal will be written and included with calibration curve raw data.
- 9.2.1 SW-846 methods allow the use of linear and non-linear models for calibration data. The option for non-linear calibration may be necessary to achieve low detection limits or to address specific instrument techniques. However, one shall not allow non-linear calibration to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance.
- 9.2.2 The following steps shall be adhered to when deciding on a calibration fit (refer to SW846 Method 8000B).
- 9.2.2.1 Use the simplest curve fit first, which is linear calibration using average response factor (called ‘Average RF/Amount’ in HP Chemstation software). If RSD for an analyte is > 20% RSD, or prior knowledge of the detector system deems it necessary to utilize another curve, proceed to least squares regression curve fit. [Line equation: $Y = \text{average of (response/amount)} * x$]
- 9.2.2.2 Linear Least squares regression is the next possible choice in curve fit factor (called ‘Linear’ in HP Chemstation software). The correlation coefficient (r) must meet or exceed 0.995 to utilize this curve type. If the curve does not meet

- acceptance criteria and section 9.3.1 is adhered to, then non-linear calibration models may be employed. [Line equation: $Y=mx+b$]
- 9.2.2.3 Non-linear calibration (called Quadratic in the HP Chemstation software, requiring 6 points) models may be used in situations where the analyst knows the instrument response does not follow a linear model over a sufficiently wide working range, or when other approaches above do not meet acceptance criteria. The coefficient of determination (r^2) value must meet or exceed 0.995 to utilize this curve type. Section 9.3.1 must be adhered to in every case. [Line equation: $Y=Ax^2+Bx+C$ for quadratic, 3rd order for cubic.] *NOTE: As a general rule, non-linear curve fits are not necessary for most FID analyses. If this fit is necessary, it is likely that instrument maintenance or a smaller range of standards is needed. Note: The Agilent Chemstation software does not calculate r^2 . It presents the correlation coefficient as r and must be corrected.*
- 9.2.2.4 Project specific criteria will supersede the above criteria on projects.
- 9.2.3 Retention times for an initial calibration will be based on a mid-level calibration standard. As the analytes of interest are calibrated as area sums, this requirement will only affect the surrogate expected retention times.
- 9.2.4 Surrogate standards are calibrated at multiple levels, just as target analytes. The surrogate standards are added to the calibration standards.
- 9.3 Initial calibration verification (ICV) – The ICV is performed any time a new calibration curve is run to ensure the curve was not created from faulty standards. The ICV must be a second source standard and prepared to fall within the normal range of the calibration curve. The ICV must recover between 80-120% to begin sample analysis. If the ICV does not meet acceptance criteria, re-analyze one time to ensure proper injection. If it still fails, the standards used to make the curve may be bad (evaporated, contaminated, etc.). A new primary and second source standard should be ordered to determine the nature of the problem. No samples may be analyzed until a passing ICV is analyzed.
- 9.4 Method detection limit study – Refer to ASL SOP 14 for MDL generation. At the onset of this project an MDL study shall be performed. The MDL needs to be $<1/5^{\text{th}}$ the PQL for ADEQ clients, which calculates to 4 mg/kg for soil and 160 ug/L for water (DRO) and 20 mg/kg or less for oil range organics.
- 9.5 Initial demonstration of capability (IDC) – An IDC study must be performed prior to use of the method by each analyst or after any significant changes to the method. An IDC study consists of four aliquots of reagent water spiked with target analytes and processed through the entire analytical method. For NELAC certification purposes the IDC study may be used to satisfy the yearly training requirement for an analyst or work cell.
- 9.5.1 Prepare and analyze four spiked blank samples at a concentration of 500 ug/mL (on column) following the procedure in Section 10 for a blank spike.
- 9.5.2 Calculate the mean concentration found (X) in mg/L and the standard deviation of the concentration in mg/L for each analyte.
- 9.5.3 For each analyte X should be within the acceptance criteria in table 2. The RSD should be 20% or less. If the results from all analytes meet these criteria then the system and analyst performance are acceptable and results should be signed and stored in the analyst's training binder. If any analyte fails to meet the criteria then investigate and correct the source of the problem and repeat the test.
- 9.5.4 An annual demonstration of capability must be completed and documented in the analyst's training binder. This requirement may be met using a passing PE sample.
- 9.6 Retention Time Standard- At the beginning of every batch or every 24 hours, whichever comes first, a retention time standard containing C10, C25, and C36 will be analyzed to verify the area sum time window has not shifted. The integration start/stop times should be close enough to the RTS peaks to not include or exclude significant area from the quantitation.
- 9.6.1 As an initial step prior to running samples, make three injections of each single component standard over a period of 72 hours (one every 24 hours). Record the retention time of each standard to four decimal places and calculate standard deviation of the peaks (time). The accepted retention time window is 3s (3x the standard deviation). If the standard deviation is zero, or the window is at or below 0.01min, the default value of 0.05min shall be used instead. It should be noted that TPH analysis does not look for

individual peaks other than surrogate spikes, and an analyst must pay close attention to RT shifting of this analysis.

- 9.7 Continuing calibration verification—A mid range primary source standard is used to verify calibration and instrument accuracy. This standard contains both oil and diesel range organics. A calibration verification will be analyzed at the beginning of every batch, every 10 samples (or every 12 hours, whichever comes first), and after the analysis of the last sample. If the concentration varies by more than $\pm 20\%$ from the initial calibration, the CV shall be re-injected one time to verify proper instrument function. If this subsequent injection fails, then instrument recalibration shall be performed and all samples affected by the failing calibration verification re-analyzed. In the event that a calibration check fails in the run and not enough sample is left to re-analyze, the data must be reported with qualifiers and the discrepancy noted on the case narrative. Immediate notification of the project manager is imperative in this instance.
- 9.8 Solvent blank – an injection of methylene chloride prior to any other injections is used as a means to track daily system performance.
- 9.9 Method blank - Analyze laboratory blank samples at a frequency of 1 per extraction batch of up to 20 samples. The acceptance criteria for blank samples is to be less than $< \frac{1}{2}$ the PQL (AFCEE criteria). In any case when a blank exhibits analyte concentration $> \frac{1}{2}$ the PQL, it is recommended to re-extract and re-analyze the associated samples. If the samples associated with this blank are very high in concentration, re-extraction may not be necessary. Contact the acting on-site project manager to determine the proper course of action for blank contamination problems.
- 9.10 Blank spike/laboratory fortified blank (BS/LFB) - Analyze laboratory blank samples at a frequency of 1 in 20 samples extracted/analyzed or 1 per day, whichever is more frequent. The BS is a primary source standard prepared from the same lot number as the calibration curve. The acceptance criteria are listed in table 2. If the BS fails low, re-analyze one time to ensure proper instrument analysis. If the BS still fails low, the entire batch must be re-extracted and analyzed (the source of the low recovery shall be investigated prior to re-extraction and analysis). If a blank spike sample fails high and no client sample has a positive hit, then the data may be reported with a note in the case narrative to this occurrence. Refer to section 9.14 for the reporting of a BS failure.
- 9.11 Matrix spike (MS) - A matrix spike sample is prepared at a rate of 1 set per 20 field samples. MS samples should be chosen randomly from a client batch of samples unless they are pre-selected by the client. Analysts should rotate the client selected for matrix spikes so that recovery data is collected from a wide variety of sample matrices. An MS sample should be processed with each analytical batch if there is sufficient sample. If sufficient sample is not provided then the BS sample results will be used to evaluate analytical batch recovery. This should be noted in the case narrative. Acceptance criteria for the MS are noted in Table 2. Poor recoveries of analytes from a matrix spike sample may indicate matrix interference from the sample or instrument problems. Poor matrix spike recovery or other evidence of method matrix interference should be reported to the client whose sample was used to prepare the MS. If the MS fails, refer to section 9.14 for reporting.

Table 2- BS/BSD or MS/MSD acceptance criteria

| Method | DRO % Rec | DRO % RPD | RRO % Rec | RRO % RPD |
|------------------------------------|--------------|--------------|--------------|--------------|
| AK102/103 – Soil | 75-125% | <20% | 60-120%* | <20%* |
| AK102/103 – Water | 75-125% | <20% | N/A** | N/A |
| AFCEE – Soil | 51-153% | <50% | N/A** | N/A |
| AFCEE – Water | 61-143% | <30% | N/A** | N/A |
| Most stringent criteria - Soil | 75-125% | <20% | 60-120% | <20% |
| Most stringent criteria - Water | 75-125% | <20% | 60-120% | <20% |

*these criteria pertain only to the BS, no MS/MSD limits are assigned by the method.

** No acceptance criteria exist for these methods.

- 9.12 Duplicate - A duplicate sample is prepared at a rate of 1 set per 20 field samples. In all cases, a matrix spike/spike duplicate pair should be chosen if sample volume is sufficient. If sufficient sample is not provided then the BS/BSD sample results will be used to evaluate analytical batch precision. This should be noted in the case narrative. Poor duplicate precision may indicate

- 9.13 problems with the sample composition and should be reported to the client whose sample was selected for the duplicate. If the duplicate fails to meet the criteria in Table 2, refer to section 9.14. Surrogate - O-terphenyl and n-octacosane are used as surrogate compounds in all samples, blanks, blank spike(s), matrix spike(s), duplicates and cal checks. Recovery limits for each test method are listed in Table 3, Surrogate Limits, below. Upon failure of a surrogate, re-analyze the sample one time to ensure proper injection. If the surrogate fails a second time, re-extraction of the sample may be required; if the sample is a QC sample, batch re-extraction may be necessary (refer to project specific requirements for this determination. Before any re-extraction takes place, consult with acting on-site laboratory project manager for instructions. For general failures of surrogate spikes refer to section 9.14.

Table 3- Surrogate Limits

| Method | o-terphenyl % Rec | n-octacosane % Rec |
|---------------------------------|--------------------------------------|---|
| AK102/103 – field sample | 50-150% | 50-150% (using limits for triacontane d ₆₂) |
| AK102/103 – lab sample* | 60-120% | 60-120% (using limits for triacontane d ₆₂) |
| AFCEE – Soil | 47-142% | 25-162% |
| AFCEE – Water | 57-132% | 26-152% |
| Most stringent criteria - Soil | 50-142% Field spl 60-120% Lab spl | 50-150% Field spl 60-120% Lab spl |
| Most stringent criteria - Water | 60-120% | 60-120% |

* These criteria pertain to all laboratory prepared QC samples except calibration check verifications which must meet CV criteria.

- 9.14 If any one of the QC parameters on this list is out of compliance, then the cause will be determined and corrective action taken. Record the out-of-compliance event and remedy in the corrective action logbook for the onsite laboratory. The on-site project manager must be made aware of the situation immediately in order to contact client if applicable. If necessary, rerun all samples analyzed while the system was out of compliance. If insufficient sample is left to re-extract and analyze, then report the data with qualifiers and make a statement of the occurrence on the case narrative. Re-analysis and/or re-sampling and re-analysis will be determined on a case by case basis via the laboratory project manager contacting the project manager for the client.

10.0 PROCEDURE

- 10.1 All volumes, spikes, and ratios herein are based on the following assumptions:
- 10.1.1 The GC is calibrated to 40 mg/L for the DRO and RRO analytes and to 4.0 mg/L for both surrogate spikes.
 - 10.1.2 The nominal sample weight used for all extractions is 10 g soil or 1000 mL water.
 - 10.1.3 The final concentration volume for sample extracts is 2.0 mL.
 - 10.1.4 Any change in these items will change the reporting limits of the method and shall be verified with the acting project manager.
- 10.2 Calibration spiking levels-the calibration standard preparation shall be performed as listed in Table 4 using the composite fuels and surrogate mix in section 8.4.7.

Table 4 – calibration standard levels

| Standard | Spike Volume, fuel mix @ 4000 mg/L with SS @ 400 mg/L | Final Volume | Final conc., DRO-RRO-SS (mg/L) |
|----------|---|--------------|--------------------------------|
| Level 1 | 0.50 mL | 50mL | 40 - 40 - 4.0 |
| Level 2 | 1.25 mL | 50mL | 100 - 100 - 10 |
| Level 3 | 2.50 mL | 50mL | 200 - 200 - 20 |
| Level 4 | 6.25 mL | 50mL | 500 - 500 - 50 |
| Level 5 | 25.0 mL | 50mL | 2000 - 2000 - 200 |
| Level 6 | --- std created at this level --- | 50mL | 4000 - 4000 - 400 |
| ICV | --- std created at this level --- | --- | 500 - 500 - 0 |

- 10.3 Percent solids must be performed on all soil and sediment samples for use in final calculations. Because of the potential for gasoline/diesel or related compounds at high concentrations all drying shall be performed near or in a functioning hood.
- 10.3.1 Open a new solids bench sheet on the computer, print off for use.
- 10.3.2 Label and pre-weigh an aluminum weigh-boat.
- 10.3.3 Weigh out approximately 10 grams of soil, record the weight of sample and weigh boat to the nearest 0.01g on the bench sheet or the computer if available.
- 10.3.4 Dry the sample overnight in a 105°C oven.
- 10.3.5 Remove the sample from the oven and place in a dessicator to cool to room temperature. Weigh the dried sample and record this post weight. Divide the post weight by the initial weight and multiply by 100 to get the % solids of the sample.
- 10.3.6 Record all data on an excel sheet and print daily, keeping in a binder for solids data
- 10.4 Soil Sample Preparation
- 10.4.1 Collect all weights, solvent volumes, spikes added, and anomalies on the bench sheet (figure 1 example at the end of document).
- 10.4.2 EPA method 3550
- 10.4.2.1 Weigh out at least 10 g of homogenized soil into a 40-mL VOA vial, recording the weight to the nearest 0.1 g and add approximately 10 g of sodium sulfate. Mix completely with a metal spatula. Mixture should be grainy; if clumps form, add more sodium sulfate. If adequate homogenization is not achieved, a beaker must be used in place of a VOA vial. Add 20 mL of MeCl₂ to the vial.
- 10.4.2.2 Add the proper surrogate and matrix/blank spikes to the correct samples. See Table 5 for spiking regiment.
- 10.4.2.3 Sonicate sample for 1.0 minute using a horn power setting of 3 (1/2" disrupter horn), with one second on/off pulse rate.
- 10.4.2.4 Prepare a funnel/filter apparatus for each sample by blocking the neck of a glass powder funnel with glass wool. Add approximately 20 g of sodium sulfate to the funnel; set funnel atop a beaker or other collection flask and rinse liberally with MeCl₂. Allow funnel/filter to stand until no more MeCl₂ is draining out, give the funnel a good, quick shake to release any solvent held up in the neck, and remove from waste solvent collection flask. The funnel can now be set on top of the KD flask.
- 10.4.2.5 Add one or two pre-baked Teflon™ boiling chips to the KD flask/concentrating tube apparatus. Collect the sonicated sample into the flask through the funnel/filter.
- 10.4.2.6 Repeat the extraction two more times (total of three aliquots of 20mL added to VOA), adding the extract to the KD flask through the funnel. After the third addition of extract, rinse the funnel/filter with liberal amounts of solvent (collect this rinsing directly into the KD flask). Attach a 3-ball Snyder column. Concentrate the extract to a volume of 5-6mL using a water bath. Rinse the Snyder column with a small aliquot of solvent and collect in the concentrating tube. Remove Snyder column and add a mini Snyder column to the concentrating tube, add another boiling chip, and concentrate the sample down to a final volume of 1.0 mL (sometimes highly contaminated samples may not allow for this level of concentration).

- 10.4.2.7 When samples are done being collected dump the sodium sulfate from the funnels onto a piece of aluminum foil and spread out to let the solvent evaporate in the hood. Dispose of sodium sulfate properly when all solvent is gone.
- 10.4.2.8 Transfer sample to a 2 mL vial with a Teflon™ lined screw cap. Store the extract in a -10°C refrigerator until analyzed.
- 10.4.2.9 After the apparatus has cooled, rinse and then disassemble the concentrator tube from the rest of the apparatus. Bring the solvent volume up to 2.0 mL with methylene chloride.
- 10.4.2.10 Prior to analysis, transfer a portion of the extract to a labeled GC vial with an insert and cap the vial. It is best to do this as close as possible to the analysis date, as sample will not store well in GC vial.

Table 5 – Sample preparation spiking regiment – (2mL final extract volume).

| Sample | DRO/RRO Mix, 2000mg/L (uL) | SS mix, 2000mg/L (uL) | DRO conc., (mg/L) | RRO conc., (mg/L) | SS conc., (mg/L) |
|-------------------------------|----------------------------|-----------------------|-------------------|-------------------|------------------|
| Method blank | --- | 100 | --- | --- | 100 |
| Blank spike/Blank spike dup | 500 | 100 | 500 | 500 | 100 |
| Matrix spike/matrix spike dup | 500 | 100 | 500 | 500 | 100 |

- 10.5 Sample preparation and extraction of waters (SW 3510C) – *no water samples are expected at the Kalakaket creek on-site laboratory.*
- 10.5.1 Determine whether or not the sample contains significant solids (will cause interference with water extraction if >10% total volume). If solids are present, contact acting project manager for advice on proceeding with the extraction.
- 10.5.2 Weigh the sample and container to the nearest 0.1g for volume determination. Pour the sample into a 1000 mL separatory funnel. (Note: Record all information on bench sheet-see figure 1.)
- 10.5.3 Add the proper surrogate and matrix/blank spikes to the correct samples. See Table 4 for spiking regiment.
- 10.5.4 NOTE: NWTPH samples must be pH adjusted with HCl to less than 2 before addition of solvent to the separatory funnel.
- 10.5.5 Add 60 mL methylene chloride into the sample jar. Put lid back on jar and shake the jar to wash off remaining hydrocarbons and then pour the MeCl₂ into the funnel. Stopper the separatory funnel and shake vigorously for one to two minutes, releasing pressure frequently.
- 10.5.6 After the two phases have separated, drain the solvent into a 250 mL Erlenmeyer flask. Be careful not to transfer water into flask. Repeat this extraction process two more times. Reweigh sample container to determine weight extracted. Convert to original sample volume using 1 g/mL as density of water.
- 10.5.7 Add sodium sulfate to the flask to take up any water that might have been drained into the flask.
- 10.5.8 Quantitatively decant the solvent from the flask into a 500 mL K-D flask fitted with a 25 mL concentrator tube through a funnel with glass wool and sodium sulfate to remove any remaining water from the extract. Add pre-rinsed Teflon™ boiling chips and 3-ball Snyder column. Concentrate the sample in a heated water bath to 5 mL. Rinse the Snyder column with a small aliquot of solvent and collect in the concentrating tube. Remove Snyder column and add a mini Snyder column to the concentrating tube, add another boiling chip, and concentrate the sample down to a final volume of 0.5 mL (sometimes highly contaminated samples may not allow for this level of concentration).

- 10.5.9 After the apparatus has cooled, rinse and then disassemble the concentrator tube from the rest of the apparatus. Bring the solvent volume up to 1.0 mL with methylene chloride.
- 10.5.10 Transfer sample to a 2-mL vial with a Teflon™ lined screw cap. Store the extract in the 4°C refrigerator until analyzed.
- 10.6 Extract cleanup
Extract cleanup will be performed at the request of the client and if the sample extracts show obvious signs of biogenic material at a high enough concentration to interfere with sample quantitation (i.e., if samples are non-detect, there is no need to clean the extracts). This silica gel cleanup will be performed following ADEQ technical memo 06-001, “Biogenic Interference and Silica Gel Cleanup”. Any time this is performed on a client sample, the associated QC samples shall also be cleaned in the same manner.
- 10.7 Sample analysis batch - a proper analysis batch will contain the following, in this order:
- 10.7.1 ADEQ instrument blank (methylene chloride only)
 - 10.7.2 Retention time standard
 - 10.7.3 Calibration verification
 - 10.7.4 Laboratory fortified blank / blank spike sample and LFB/BS duplicate
 - 10.7.5 Method blank
 - 10.7.6 Up to 10 samples counting client specified matrix spike, if applicable
 - 10.7.7 Calibration verification
 - 10.7.8 Up to 10 samples
 - 10.7.9 Calibration verification
- 10.8 Calibration batch - A proper calibration sequence will contain the following, in this order:
- 10.8.1 ADEQ instrument blank (methylene chloride)
 - 10.8.2 Retention time standard
 - 10.8.3 All calibration levels for DRO/RRO/SS
 - 10.8.4 ICV (second source verification) for DRO/RRO

10.9 GC Analysis

- 10.9.1 Perform GC analysis on the extract using the suggested GC parameters listed below:

| Parameter | Setting |
|------------------------------------|------------------------------------|
| Head pressure | 17.0psi, |
| Sample Injection Volume | 2uL |
| Initial temp | 50°C hold for 1 minute |
| Temp Ramp | 18°C/min to 320°C, hold for 11 min |
| Injector temp, Detector temp (FID) | 300°C, 300°C |
| Total run time | 27 min |
| Air Flow | 300-400 ml/min |
| Make-up gas flow | ~3ml/min |
| Hydrogen flow | 25-35 ml/min |

- 10.9.2 Capillary Column:
Zebron ZB-5 30 m x 0.32 mm I.D. x 0.25 um film thickness or similar to meet AK102/103 criteria of separating the solvent peak and C10, C24 from C25, and n-C19 from o-terphenyl.
- 10.9.3 Retention time shifting of the surrogate peaks or the retention time standards of 0.05 minutes or greater will be cause for close inspection of all plumbing and any related components for leaks or malfunction. The retention time of all peaks and area sum ranges based on retention time standards should be verified each day and updated as needed from the retention time standard. Any major shift in time will warrant a thorough inspection of the instrument and subsequent recalibration.
- 10.9.4 If the analysis indicates that the results are above the calibration range, dilute the sample extracts such that concentrations fall within the calibration range, at or above the midpoint of the calibration curve. Make sure to add dilution information to the sample information section of the GC sequence table, enter the dilution value in the dilution field and also complete a dilution log to be stored with the data and electronically.
- 10.9.5 Petroleum products are to be identified as follows:

- 10.9.5.1. Diesel and related products are indicated if compounds are detected between decane (C₁₀) and tetracosane (C₂₅).
- 10.9.5.2. Oil (lube, motor oil) and related products are indicated if compounds are detected between tetracosane (C₂₅) and hexatriacontane (C₃₆).
- 10.9.5.3. Matching or fingerprinting of samples is intended to assist in identification of specific compounds.

11.0 DATA REDUCTION

- 11.1 All associated calculations for this method may be found in Table 6 at the end of this document.
- 11.2 Sum the area of all the peaks eluting in the specified carbon ranges. This area is generated by projecting a horizontal baseline between the retention times of the initial and final carbon compounds. The surrogate spike area must be removed from the target area by tangent skimming the peak from the TPH chromatogram.
- 11.3 Sample and standards must be integrated in the same fashion. If the sample area exceeds the calibration standard area, the sample is diluted and reanalyzed. If the sample area is below the reporting limit, the sample is reported as a non-detect.
- 11.4 All samples that are manually integrated (other than surrogate spike tangent skimming, necessary on all samples) must be printed after manual integration clearly showing manual integration; all chromatograms must be signed by the analyst and a peer reviewer. As surrogate spike tangent skimming is part of the method requirements, no manual integration sheet will be included with the AFCEE case narrative. Refer to ASL SOP 26 for further information.
- 11.5 Quantification of the target compounds is based on the integrated areas of the samples in comparison to the integrated areas of the calibration standards for each analyte. The integrator reports the concentrations in mg/L in the extracts. Calculation of the final concentration for each target constituent in the sample is as follows:

$$\text{Conc in } mg / kg = \frac{A \times V_t \times DF}{W_s}$$

$$\text{Conc in } mg / L = \frac{A \times V_t \times DF}{V_w}$$

- Where: A =Amount of target constituent found in the extract in mg/L
V_t =Final volume of solvent in mL (Section 10)
DF =Dilution factor, if required
W_s =Dry weight of the sample added to the VOA vial in grams (Section 10)
V_w =Volume of the sample added to the sep. funnel in mls (Section 10)

12.0 DOCUMENTATION

- 12.1 All raw electronic data and any associated electronic bench sheets will be backed up to CD/DVD on a weekly basis.
- 12.2 Instrument printouts and all raw data are kept in the file cabinet in the instrument lab and filed by date of analysis. This data will be returned to the CH2M HILL Applied Sciences Laboratory to be kept for one year onsite, then archived a total of 7 years time from date of creation.
- 12.3 Extraction logs used herein are printed and kept in a dedicated laboratory binder.
- 12.4 All chromatograms are to be initialed and dated by the analyst. In the case of tangent skimmed peaks (all samples but the RTS), a reviewer needs to initial the integration as being acceptable. There is no “before” manual integration chromatogram printed off for TPH analysis, as all samples need to be tangent skimmed.
- 12.5 Calibration sample chromatograms need to be initialed and dated by the analyst, a peer reviewer, and the acting onsite QA officer.

13.0 REFERENCES

- 13.1 Alaska Department of Environmental Conservation (ADEC) method AK 102 for determination of diesel range organics, Version 04/08/02.
- 13.2 Alaska Department of Environmental Conservation (ADEC) method AK 103 for determination of residual range organics, Version 04/08/02.
- 13.3 Alaska Department of Environmental Conservation (ADEC) technical memorandum 06-001, *Biogenic Interference and Silica Gel Cleanup*, May 18, 2006.
- 13.4 EPA Method 8015B, "Nonhalogenated Volatile Organics using GC/FID," found in EPA SW846, *Test Methods for Evaluating Solid Waste*, 3rd ed., 2nd revision, December, 1996.
- 13.5 This SOP refers to the following methods- SW3510 and SW3550 found in EPA SW846, *Test Methods for Evaluating Solid Waste*, 3rd ed., 2nd revision, December, 1996.
- 13.6 ASL SOP's SVO36, SOP14, SOP32, SOP06 and GEN46.

14.0 DEFINITIONS

- 14.1 Surrogate Standard (SS)—A pure analyte(s), which is extremely unlikely to be found in any sample, and which is added to a sample aliquot in known amount(S) before extraction or other processing and is measured with the same procedures used to measure other sample components. The purpose of the SS is to monitor method performance with each sample.
- 14.2 Laboratory Duplicates—Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of duplicates indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
- 14.3 Field Duplicates—Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedure. Analyses of Duplicates gives a measure of the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.
- 14.4 Laboratory Reagent Blank or Method Blank (WB1, SB1, XB1)—An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The blank is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
- 14.5 Trip Blank (TB)—An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the TB is to determine if method analytes or other interferences are present in the field environment.
- 14.6 Calibration Check Verification (CCV)—A solution of one or more compounds (analytes, surrogates, internal standard, or other test compounds) used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
- 14.7 Blank Spike (BS1W, BS1S)—An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 14.8 Blank Spike Duplicate (BD1W, BD1S)—An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The BD is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.
- 14.9 Matrix Spikes/Spike Duplicates (MS/MSD)—An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

- 14.10 Stock Standard Solution (SSS)—A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.
- 14.11 Primary Standard Solution (PSS)—A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.
- 14.12 Calibration Standard (CAL)—A solution prepared from the primary standard solution or stock standard solution and the internal standards and surrogate analytes. The Cal solutions are used to calibrate the instrument response with respect to analyte concentration.
- 14.13 Initial Calibration Verification (ICV)—A solution of method analytes of known concentrations which is used to fortify an aliquot of WB1 or sample matrix (MS). The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

Table 6

| STATISTIC | SYMBOL | FORMULA | DEFINITION | USES |
|-----------------------------------|-----------|---|---|---|
| 1. Mean | \bar{X} | $\frac{\left(\sum_{i=1}^n X_i\right)}{n}$ | Measure of central tendency | Used to determine the central value of a set of measurements. |
| 2. Standard Deviation | S | $S = \left[\frac{\sum (X_i - \bar{X})^2}{n - 1}\right]^{1/2}$ | Measure of the relative scatter of data | Used in variation of measurements |
| 3. Relative Standard Deviation | RSD | $(S / \bar{X}) \times 100$ | Adjusts for the magnitude of observations when calculating (S) | Used to assess precision for replicate results |
| 4. Percent Difference | %D | $\left[\frac{X_1 - X_2}{X_1}\right] \times 100$ | Measure of the difference of two observations | Used to assess accuracy |
| 5. Relative Percent Difference | RPD | $\left[\frac{(X_1 - X_2)}{(X_1 + X_2)/2}\right] \times 100$ | Measure of variability that adjusts for the magnitude of observations | Used to assess total and analytical precision of duplicate measurements |
| 6. Percent Recovery (Blank spike) | %Rec | $\left[\frac{\text{Experimental Value}}{\text{True value}}\right] \times 100$ | Recovery of spiked compound in pure matrix | Used to assess accuracy |
| 7. Percent Recovery (Sample) | %Rec | $\left[\frac{\text{spiked sample result} - \text{unspiked sample result}}{\text{value of spike added}}\right] \times 100$ | Recovery of spiked compound in sample matrix | Used to assess matrix effects and total precision |

Located at: G:/chemist/forms/calcsheet

Figure 1- Semivolatile extraction log sheet

| CH2M HILL APPLIED SCIENCES SEMI-VOLATILES EXTRACTION LOG | | | | | | | |
|--|-------------------|----------------|----------------|-----------------------------|--------|-------|----------------|
| Project Name: _____ | | | | Analysis Requested: _____ | | | |
| Date Extracted: _____ | | | | Matrix / Ext. Method: _____ | | | |
| Analyst: _____ | | | | Water bath Temp.: _____ | | | |
| LAB ID # | CLIENTS SAMPLE ID | SAMPLE ALIQUOT | SOLVENT VOLUME | FINAL VOLUME | SS VOL | % H2O | COMMENTS NOTES |
| 1 | | | | | | | |
| 2 | | | | | | | |
| 3 | | | | | | | |
| 4 | | | | | | | |
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| 26 | | | | | | | |

| | |
|---------------------------|---------------------|
| EXTRACTION SOLVENT: _____ | DRYING AGENT: _____ |
| LOT # _____ | LOT # _____ |

| | SPIKING COMPOUNDS | CONCENTRATION | VOLUME | LOT # |
|------------------|-------------------|---------------|--------|-------|
| SURROGATE SPIKE: | #1 | _____ | _____ | _____ |
| | #2 | _____ | _____ | _____ |
| | #3 | _____ | _____ | _____ |
| MATRIX SPIKE: | #1 | _____ | _____ | _____ |
| | #2 | _____ | _____ | _____ |
| | #3 | _____ | _____ | _____ |
| INTERNAL STD: | #1 | _____ | _____ | _____ |
| | #2 | _____ | _____ | _____ |
| | #3 | _____ | _____ | _____ |