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ANALYTICAL DATA ASSESSMENT AND VALIDATION  
HOOKER-RUCO OU-1 SAMPLING  
MILLER SPRINGS REMEDIATION MANAGEMENT, INC.  
HICKSVILLE, NEW YORK  
JUNE 2004

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## 1.0 INTRODUCTION

One groundwater sample was collected from well E-1 at the former Hooker Ruco Site in Hicksville, New York (Site) in support of Operable Unit-1 (OU-1) monitoring program. Analytical services were performed by H2M Labs, Inc., in Melville, New York (H2M). A summary of the sampling and analysis scheme is presented in Table 1.

A summary of the analytical data is presented in Table 2. The sample was analyzed for the Volatile Organic Compound (VOC) tetrachloroethene by Contract Laboratory Protocol (CLP) Method SOW OLM03.2 and iron and zinc by CLP Method ILM04. The methods were referenced from "USEPA Contract Laboratory Program Statement of Work For Analysis of Low Concentration Organics", OLC03.2 and "USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration", ILM 4.0 EPA/540/R95/121.

The Quality Assurance/Quality Control (QA/QC) criteria by which these data have been assessed are outlined in the analytical methods. Additional validation guidelines were referenced from the following documents:

- i) "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review", EPA 540/R-94-012, February 1994;
- ii) "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review", EPA 540/R-94-013, February 1994; and
- iii) Quality Assurance Project Plan , OU-3 Predesign Activities, Hooker/Ruco Site, Hicksville, New York, November 2001.

Full CLP-equivalent raw data deliverables were provided by the laboratory. The data quality assessment and validation presented in the following subsections were performed based on the sample results and supporting QA/QC provided.

## 2.0 SAMPLE HOLDING TIMES

The method-specific holding time criteria are summarized in Table 5.1 of the Quality Assurance Project Plan (QAPP). All sample extractions and/or analyses were performed within the specified holding times.

All samples were properly preserved and cooled to 4°C ( $\pm 2^\circ\text{C}$ ) after collection. All samples were received by the laboratory in good condition.

### 3.0 GAS CHROMATOGRAPH/MASS SPECTROMETER (GC/MS) TUNING AND MASS CALIBRATION - VOCs

Prior to analysis, GC/MS instrumentation is tuned to ensure optimization over the mass range of interest. To evaluate instrument tuning, the VOC method requires the analysis of the specific tuning compound bromofluorobenzene (BFB). The resulting spectra must meet the criteria cited in the method before analysis is initiated. Analysis of the tuning compound must then be repeated every 12 hours throughout sample analysis to ensure the continued optimization of the instrument.

Instrument tuning data were reviewed. Tuning compounds were analyzed at the required frequency throughout the VOC analysis period. All tuning criteria were met for the analyses, indicating proper optimization of the instrumentation.

### 4.0 INSTRUMENT CALIBRATION

#### 4.1 CC/MS CALIBRATION - VOCs

##### 4.1.1 TUNING AND MASS CALIBRATION

Prior to analysis, GC/MS instrumentation is tuned to ensure optimization over the mass range of interest. To evaluate instrument tuning, the VOC method require the analysis of the specific tuning compounds BFB. The resulting spectra must meet the criteria cited in the method before analysis is initiated. Analysis of the tuning compound must then be repeated every 12 hours throughout sample analysis to ensure the continued optimization of the instrument.

Instrument tuning data were reviewed. Tuning compounds were analyzed at the required frequency throughout the VOC analysis period. All tuning criteria were met for the analyses, indicating proper optimization of the instrumentation.

##### 4.1.2 INITIAL CALIBRATION

To quantify compounds of interest in samples, calibration of the GC/MS over a specific concentration range must be performed. Initially, a five-point calibration curve containing all compounds of interest is analyzed to characterize instrument response for



each analyte over a specific concentration range. Linearity of the calibration curve and instrument sensitivity are evaluated against the following criteria:

- i) all relative response factors (RRFs) must be greater than or equal to 0.05; and
- ii) for average response factors are employed, percent relative standard deviation (%RSD) values must not exceed 30 percent.

The initial calibration data for VOCs were reviewed and met the above criteria for linearity and sensitivity for all compounds of interest.

#### **4.1.3 CONTINUING CALIBRATION**

To ensure that instrument calibration is acceptable throughout the sample analysis period, continuing calibration standards must be analyzed and compared to the initial calibration curve every 12 hours.

The following criteria were employed to evaluate continuing calibration data:

- i) all RRF values must be greater than or equal to 0.05; and
- ii) percent difference (%D) values must not exceed 25 percent.

Calibration standards were analyzed at the required frequency and the results met the above criteria for instrument sensitivity and linearity.

### **4.3 INSTRUMENTAL CALIBRATION - METALS**

#### **4.3.1 INITIAL CALIBRATION**

Initial calibration of the instruments ensures that they are capable of producing satisfactory quantitative data at the beginning of a series of analyses. For trace inductively coupled plasma (ICP) analysis, a calibration blank and at least one standard must be analyzed at each wavelength to establish the analytical curve.

After calibration, an initial calibration verification (ICV) standard must be analyzed to verify the analytical accuracy of the calibration curves. All analyte recoveries from the analyses of the ICVs must be within control limits of 90-110 percent.

Upon review of the data, it was determined that all inorganic calibration curves and ICVs were analyzed at the proper frequencies and that all of the above-specified criteria were met. The laboratory effectively demonstrated that instrumentation used for these analyses were properly calibrated prior to sample analyses.

#### **4.3.2 CONTINUING CALIBRATION**

To ensure that instrument calibration is acceptable throughout the sample analysis period, continuing calibration verification (CCV) standards are analyzed on a regular basis. Each CCV is deemed acceptable if all analyte recoveries are within the control limits specified above for the ICVs. If some of the CCV analyte recoveries are outside the control limits, samples analyzed before and after the CCV, up until the previous and proceeding CCV analyses, are affected.

For this study, CCVs were analyzed at the proper frequency. All analyte recoveries reported for the CCVs were within the specified limits.

#### **4.3.3 CONTRACT REQUIRED DETECTION LIMIT (CRDL) STANDARD ANALYSES**

To verify the linearity of the ICP calibration near the detection limit, a standard is analyzed which contains the ICP analytes at specified concentrations. This standard must be analyzed at the beginning and end of each sample analysis run or a minimum of twice per 8-hour working shift.

Control limits of 80-120 percent were used to evaluate the data. The CRDL recoveries were acceptable

### **5.0 SURROGATE COMPOUND ANALYSES - VOCs**

In accordance with the methods employed, all samples, blanks, and standards analyzed for VOCs are spiked with surrogate compounds prior to sample analysis. Surrogate recoveries provide a means to evaluate the effects of individual sample matrices on analytical efficiency and are assessed against method control limits.

Surrogates were added to all samples, blanks, and QC samples prior to analysis. Surrogate recoveries met the acceptance criteria for all samples demonstrating acceptable analytical accuracy in this sample matrix.

## **6.0 INTERNAL STANDARD RECOVERIES - VOCs**

To ensure that changes in GC/MS response and sensitivity do not affect sample analysis results, internal standard compounds are added to all samples, blanks, and spike samples prior to VOC analysis. All results are calculated as a ratio of the internal standard response. The criteria by which the internal standard results are assessed are as follows:

- i) internal standard area counts must not vary by more than a factor of two (-50 percent to +100 percent) from the associated calibration standard; and
- ii) the retention time of the internal standard must not vary more than  $\pm 30$  seconds from the associated calibration standard.

The sample IS recoveries met the above criteria and were used to calculate all positive sample results.

## **7.0 MATRIX SPIKE (MS) AND DUPLICATE ANALYSES - METALS**

To evaluate the effects of sample matrices on the digestion, measurement procedures, and accuracy of a particular analysis, samples are spiked with a known concentration of the analyte of concern and analyzed as MS samples. The established control limits for inorganic matrix spike recoveries are 75 to 125 percent. Spike recoveries are not assessed for samples having original concentrations significantly greater than the spike concentration (>four times).

Analytical precision is evaluated based on the analysis of duplicate samples. Laboratory duplicate results are assessed against a maximum RPD of 20 percent. Metals sample results less than five times the CRDL are evaluated based on the difference between the sample and duplicate results, which should not exceed two times the CRDL.

Matrix spikes and duplicate analyses were performed at the required frequency for metals. The results showed acceptable accuracy and precision on this sample matrix.



## 8.0 LABORATORY CONTROL SAMPLE (LCS) ANALYSES

The LCS serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation. LCSs are analyzed using the same sample preparation, analytical methods, and QA/QC procedures employed for the investigative samples.

LCSs were prepared and analyzed for metals of interest. The LCS results showed good overall analytical accuracy.

## 9.0 METHOD BLANK ANALYSES

Method blanks are prepared from deionized water and analyzed with investigative samples to determine the existence and magnitude of sample contamination introduced during the procedures. Additionally, continuing calibration blanks (CCBs) are routinely analyzed after each CCV for the inorganic parameters.

For this study, method blanks were analyzed at a minimum frequency of one per analytical batch and CCBs were analyzed for inorganic parameters after each CCV. The data were non-detect with the exception of metals analysis. Upon review of the initial calibration blanks, continuing calibration blanks, and preparation blanks, it was noted that some metal concentrations were detected above the instrument detection limit (IDL) in the calibration blanks associated with the samples collected for this project.

The concentrations of these metals in the investigative sample were significantly greater than the blank concentrations and would not have been impacted by the level of contamination detected.

## 10.0 ICP SERIAL DILUTION

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix. A minimum of one per 20 investigative samples is analyzed at a five-fold dilution. For samples yielding analyte concentrations greater than 50 times the IDL, the serial dilution results must agree within 10 percent of the original results.

Serial dilutions were performed at the required frequency and the results were acceptable.

## 11.0 ICP INTERFERENCE CHECK SAMPLE ANALYSIS (ICS)

To verify that proper inter-element and background correction factors have been established by the laboratory, ICSs are analyzed. These samples contain high concentrations of aluminum, calcium, magnesium, and iron and are analyzed at the beginning and end of each sample analysis period.

ICS analysis results were evaluated for all samples. The ICS recoveries were within the established control limits of 80 to 120 percent.

## 12.0 CONCLUSION

Based on the preceding assessment, the data summarized in Table 2 are acceptable without qualification.

TABLES

**TABLE 1**  
**SAMPLE AND ANALYSIS SUMMARY**  
**HOOKER-RUCO OU-1 SAMPLING**  
**MILLER SPRINGS REMEDIATION MANAGEMENT, INC.**  
**HICKSVILLE, NEW YORK**  
**JUNE 2004**

<i>Sample ID</i>	<i>Location ID</i>	<i>Collection Date (mm/dd/yy)</i>	<i>Collection Time (hr:min)</i>	<i>Analysis/Parameters</i>	
				<i>VOCs-PCE</i>	<i>Fe &amp; Zinc</i>
GW-6883-61504-BTF-EI	MW-E1	06/15/04	12:30	X	X

Notes:

Fe Iron

PCE Tetrachloroethene.

VOCs Volatile Organic Compounds.



TABLE 2  
ANALYTICAL RESULTS SUMMARY  
HOOKER-RUCO OU-1 SAMPLING  
MILLER SPRINGS REMEDIATION MANAGEMENT, INC.  
HICKSVILLE, NEW YORK  
JUNE 2004

*Sample Location:* E-1  
*Sample ID:* GW-6883-61504-BTF-E1  
*Sample Date:* 6/15/2004

<i>Parameters</i>	<i>Units</i>	
<i>Volatile Organic Compounds</i>		
Tetrachloroethene	µg/L	3
<i>Metals</i>		
Iron	µg/L	6900
Zinc	µg/L	45.2