

**Date:** July 13, 2016

**To:** Rob Ede  
Hahn and Associates Inc.

**From:** Jeanne Peterson  
Sr. Data Validator, AQA

**Subject:** Data Validation  
Siltronic RI - Doane Creek  
Fremont Analytical Work Order # 1604078 (A6C1076)

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## SUMMARY

Level III data validation was performed on the data for four soil samples prepared and analyzed with approved procedures using the Method for the Determination of Volatile Petroleum Hydrocarbons (VPH) Fractions or the Method for the Determination of Extractable Petroleum Hydrocarbons (EPH) Fractions. The samples were submitted to Fremont Analytical (Fremont) for analysis. Data were reported for all requested analytes.

The analytical data were evaluated in accordance with the *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (October 1999) and the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (February 1994) (NFG).

In general, most of the data are valid as reported. No sample data were rejected. Other qualifiers were applied to the data as specified in the Data Qualifiers section below.

See attached data validation spreadsheets for supporting documentation on the data review and validation.



## SAMPLES

The samples included in this validation are listed below.

Sample ID	Fremont Laboratory ID	APEX Laboratory ID	Analysis
5237-160328-DC-SED075G	1604078-001	A6C1076-11	VPH
5237-160328-DC-SED075	1604078-002	A6C1076-12	EPH
5237-160328-DC-SED087G	1604078-003	A6C1076-21	VPH
5237-160328-DC-SED087	1604078-004	A6C1076-22	EPH

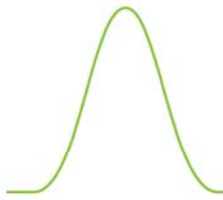
**DATA QUALIFIERS** (see following sections for detailed explanations)

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
5237-160328-DC-SED075G	VPH	All target analytes	UJ	Analyzed beyond the holding time
5237-160328-DC-SED075	EPH	Aliphatic hydrocarbon (C16-C21)	J	Low surrogate recovery
		Aliphatic hydrocarbon (C8-C10)	UJ	Low surrogate recovery
		Aliphatic hydrocarbon (C10-C12)		
		Aliphatic hydrocarbon (C12-C16)		
Aliphatic hydrocarbon (C21-C34)				
5237-160328-DC-SED087G	VPH	All target analytes	UJ	Analyzed beyond the holding time
5237-160328-DC-SED087	EPH	Aliphatic hydrocarbon (C16-C21)	J	Low surrogate recovery
		Aliphatic hydrocarbon (C8-C10)	UJ	Low surrogate recovery
		Aliphatic hydrocarbon (C10-C12)		
		Aliphatic hydrocarbon (C12-C16)		
Aliphatic hydrocarbon (C21-C34)				

## DISCUSSION

### Sample Shipping/Receiving

All COC, analysis request, and sample receipt documentation was complete and correct.



### **Holding Times and Preservation**

The samples were properly preserved and analyzed within the prescribed holding times with the following exceptions.

#### **Method VPH**

The samples were analyzed beyond, but within 2X, the method specified holding time. The associated sample results were non-detects and, therefore, were **qualified UJ**.

### **Calibration**

All initial and continuing calibration acceptance criteria were met with the following exceptions.

#### **Method VPH**

The initial continuing calibration verification (ICV) and/or continuing calibration verification (CCV) percent differences (%Ds) were >20% with positive bias for aliphatic hydrocarbon (C5-C6), aliphatic hydrocarbon (C10-C12), aromatic hydrocarbon (C8-C10), aromatic hydrocarbon (C10-C12), and aromatic hydrocarbon (C12-C13). The associated sample results were non-detects and not affected by the high bias and, therefore, were not qualified based on professional judgment.

### **Blanks**

No target analytes were detected in the method blanks.

### **Surrogates**

All surrogate recoveries were within laboratory QC acceptance criteria with the following exceptions.

#### **Method EPH**

The 1-chlorooctadecane recoveries were < the lower acceptance limit but  $\geq 10\%$  for samples 5237-160328-DC-SED075 and 5237-160328-DC-SED087. The associated sample results that were detects were **qualified J**, and the associated sample results that were non-detects were **qualified UJ**.

### **Laboratory Control Sample (LCS)**

The LCS analysis met all laboratory acceptance criteria with the following exception.



### **Method VPH**

The LCS recovery was > the upper acceptance limit for aromatic hydrocarbon (C10-C12). The associated sample results were non-detects and not affected by the high bias and, therefore, were not qualified based on professional judgment.

### **Matrix Spike (MS)/Matrix Spike Duplicate (MSD)**

The MS/MSD analysis met all QC acceptance criteria with the following exceptions.

### **Method VPH**

The MS/MSD recoveries were > the upper acceptance limit for aromatic hydrocarbon (C10-C12). The associated sample results were non-detects and not affected by the high bias and, therefore, were not qualified based on professional judgment.

The MSD recovery was > the upper acceptance limit for aromatic hydrocarbon (C12-C13). The associated sample results were non-detects and not affected by the high bias and, therefore, were not qualified based on professional judgment.

### **Laboratory Duplicate**

The laboratory duplicate analysis met all QC acceptance criteria with the following exception.

### **Method VPH**

The relative percent difference (RPD) between the MS and MSD results was > the acceptance limit for aromatic hydrocarbon (C12-C13). Because the parent sample was from another work order and the duplicate sample performed on a sample from this work order was within acceptance criteria, no sample data were qualified based on professional judgment.

### **Method EPH**

The RPD between the sample and duplicate results was > the acceptance limit for aliphatic hydrocarbon (C16-C21). Because the parent sample was from another work order and all other QC criteria were met, no sample data were qualified based on professional judgment.

### **Field Duplicate**

A field duplicate was not collected with the samples in this work order.



**Reporting Limits**

All reporting limits were properly reported. The samples were not diluted.

**Other QC**

No other specific issues that affect data quality were identified.

## Hahn Data Validation Summary Worksheet

SDG#: 1604078_A6C1076	Laboratory: Fremont	Validator: Jeanne Peterson	Validation Date: 06/13/2016
Site: Siltronic - Doane Creek	COC#: NA		Validation Level: <input type="checkbox"/> II <input checked="" type="checkbox"/> III
Matrix: Soil	# of Samples: 4	Tracking docs present: See sample receipt and log-in documentation	
COCs present: Yes	COCs signed: Yes	COCs dated: Yes	Sample Container Integrity: OK
Analyses: <input type="checkbox"/> VOCs <input type="checkbox"/> SVOCs <input type="checkbox"/> PAHs <input type="checkbox"/> GRO <input type="checkbox"/> DRO <input type="checkbox"/> Pests <input type="checkbox"/> PCBs <input type="checkbox"/> Metals <input type="checkbox"/> Gen Chem <input type="checkbox"/> Cyanide <input checked="" type="checkbox"/> Other: VPH, EPH			

Requested Analyses Not Reported			
Client Sample ID	Lab Sample ID	Analysis	Comments
None			

Hold Time/Preservation Outliers								
Client Sample ID	Lab Sample ID	Analysis	Pres.	Collection Date	Preparation Date	Analysis Date	Analysis <3X HT	Analysis ≥3X HT
5237-160328-DV-SED075G	1604078-001	VPH	4°C	3/28/2016	4/11/2016	4/12/2016	Yes	No
5237-160328-DV-SED087G	1604078-003	VPH	4°C	3/28/2016	4/11/2016	4/12/2016	Yes	No

Comments: Samples collected 3/28.  
Cooler temps OK.

## Hahn Level III VPH Worksheet

SDG: 1604078	Matrix: Soil	Lab Sample IDs: 1604078-001, -003
Method/Batch #: VPH/13429		

*Lab-established control limits may be used in place of these:*

Analyte (outliers)	Calibration			Method Blank	5X Blank	LCS %R <i>70-130%</i>	LCSD %R <i>70-130%</i>	LCSD/ LCSD RPD <i>≤25%</i>	MS %R <i>70-130%</i>	MSD % <i>70-130%</i>	MS/D RPD <i>≤25%</i>
	RSD/r <i>≤20%/</i> <i>≥0.99</i>	ICV %D <i>±20%</i>	CCV %D <i>±20%</i>								
Aliphatic hydrocarbon (C5-C6)	✓	149	✓	✓	NA	✓	NA	NA	✓	✓	✓
Aliphatic hydrocarbon (C10-C12)	✓	122	✓	✓	NA	✓	NA	NA	✓	✓	✓
Aromatic hydrocarbon (C8-C10)	✓	123	✓	✓	NA	✓	NA	NA	✓	✓	✓
Aromatic hydrocarbon (C10-C12)	✓	127	171/127	✓	NA	149	NA	NA	204	226	✓
Aromatic hydrocarbon (C12-C13)	✓	✓	150	✓	NA	✓	NA	NA	✓	153	30

**Surrogate Outliers** (*60-140% or laboratory-established control limits*)

Sample ID	Surrogate	%R	Sample ID	Surrogate	%R	Sample ID	Surrogate	%R
None								

Comments: HTs out. MB, LCS, 1604081-001AMS/MSD, 1604078-003ADUP

NOTE: ICV had detects, but run log shows cleanout runs in after the CCV and LCS prior to the method blank and samples. The method blank had no detects; therefore, the ICV detects were not applicable.

## Hahn Level III EPH Worksheet

SDG: 1604078	Matrix: Soil	Lab Sample IDs: 1604078-002, -004
Method/Batch #: EPH/13403		

*Lab-established control limits may be used in place of these:    70-130%                      70-130%                      ≤25%*

Analyte (outliers)	Calibration			Method Blank	5X Blank	LCS %R	MS %R	MSD %R	MS/D RPD	Lab Dup RPD		
	RSD/r ≤20%/ ≥0.99	ICV/CCV %D ±20%	Daily RT Windows									
Aliphatic Hydrocarbon (C16-C21)	✓	✓	NA	✓	NA	✓	NA	NA	NA	47.4		

### Surrogate Outliers (60-140% or laboratory-established control limits)

Sample ID	Surrogate	%R	Sample ID	Surrogate	%R	Sample ID	Surrogate	%R
1604081-004ADUP	1-CIOD	53.6						
-002	1-CIOD	24.5						
-004	1-CIOD	40.7						

### IS Outliers (-50% to +100% of CCV)

Sample ID	Area	RT	Area	RT	Area	RT	Area	RT	Area	RT	Area	RT
NA												

Comments: HT OK. MB, LCS, 1604081-004ADUP.

NOTE: ICV had detects, but run log shows ICV was run much earlier in the day than the samples, and the method blank extracted with and run right before the samples had no detects; therefore, the ICV detects were not applied to the sample results.