



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
Apex Laboratories, LLC SDG A6C1124

SUMMARY

Level III data validation was performed on the data for 16 soil samples prepared and analyzed with approved procedures using methods SW846 8260B (volatile organic compounds [VOCs]), SW846 8270D (semivolatile organic compounds [SVOCs] and polynuclear aromatic hydrocarbon [PAH] homologues), NWTPH-Gx (gasoline range organics [GRO]), NWTPH-Dx (diesel range organics [DRO]), SW846 6020 (total metals by ICPMS), SW846 9013M/9014 (total cyanide), SW846 9056A (sulfate by IC), SM 5310B Mod (total organic carbon [TOC]), and/or SM4500-NH3 (ammonia as N). 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	Laboratory ID	Matrix	Analysis
5237-160329-DC-EMB010G	A6C1124-01	Soil	VOCs, GRO
5237-160329-DC-EMB010	A6C1124-02	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160329-DC-EMB005G	A6C1124-03	Soil	VOCs, GRO
5237-160329-DC-EMB005	A6C1124-04	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160329-DC-EMB004G	A6C1124-05	Soil	VOCs, GRO
5237-160329-DC-EMB004	A6C1124-06	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160329-DC-EMB001G	A6C1124-07	Soil	VOCs, GRO
5237-160329-DC-EMB001	A6C1124-08	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160329-DC-EMB013G	A6C1124-09	Soil	VOCs, GRO
5237-160329-DC-EMB013	A6C1124-10	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160329-DC-EMB014G	A6C1124-11	Soil	VOCs, GRO
5237-160329-DC-EMB014	A6C1124-12	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160329-DC-EMB017G	A6C1124-13	Soil	VOCs, GRO
5237-160329-DC-EMB017	A6C1124-14	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160329-DC-EMB020G	A6C1124-15	Soil	VOCs, GRO
5237-160329-DC-EMB020	A6C1124-16	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia



DATA QUALIFIERS (see following sections for detailed explanations)

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
5237-160329-DC-EMB010	8270D	Benzoic acid	UJ	High calibration verification negative bias
	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Phenanthrenes/ Anthracenes C2-Chrysenes/ Benz(a)anthracenes C2-Phenanthrenes/ Anthracenes C3-Chrysenes/ Benz(a)anthracenes C3-Naphthalenes C3-Phenanthrenes/ Anthracenes C4-Chrysenes/ Benz(a)anthracenes	J	Insufficient calibration
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	Poor replicate precision
5237-160329-DC-EMB005	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes	J	Insufficient calibration
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	Poor replicate precision
5237-160329-DC-EMB004	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Phenanthrenes/ Anthracenes C2-Chrysenes/ Benz(a)anthracenes	J	Insufficient calibration



Sample ID	Method	Analyte	Qualifier	Reason for Qualification
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	Poor replicate precision
	SM4500-NHS Mod	Ammonia as N	J	High initial calibration positive bias
5237-160329-DC-EMB001	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Phenanthrenes/ Anthracenes C2-Chrysenes/ Benz(a)anthracenes C2-Phenanthrenes/ Anthracenes	J	Insufficient calibration
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	Poor replicate precision
5237-160329-DC-EMB013	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes	J	Insufficient calibration
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	Poor replicate precision
5237-160329-DC-EMB014	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes	J	Insufficient calibration
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	Poor replicate precision



Sample ID	Method	Analyte	Qualifier	Reason for Qualification
5237-160329-DC-EMB017	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Phenanthrenes/ Anthracenes C2-Chrysenes/ Benz(a)anthracenes	J	Insufficient calibration
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	Poor replicate precision
5237-160329-DC-EMB020	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Phenanthrenes/ Anthracenes	J	Insufficient calibration
	6020	Aluminum	J	Low matrix spike recovery and poor replicate precision
	SM 5310B Mod	TOC	J	High initial calibration negative bias and poor replicate precision

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.

Instrument Tune

All instrument tune requirements were met.



Calibration

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

Method 8270D

The ICAL and/or ICV/CCV RRFs were <0.05 but ≥ 0.01 for pentachlorophenol and 2,4,6-tribromophenol. Quadratic equations were used to calculate the sample results; therefore no sample data were qualified based on professional judgment.

The CCV percent difference (%D) associated with sequence 6D01020 was $>25\%$ with negative bias for benzoic acid. The associated sample result was a non-detect and, therefore, was **qualified UJ**.

The CCV %Ds associated with sequence 6C31036 were much $>25\%$ with positive bias for benzo(g,h,i)perylene, dibenz(a,h)anthracene, and indeno(1,2,3-cd)pyrene. The associated samples were QC samples (method blank and LCS) that met QC criteria and, therefore, no sample data were qualified.

The CCV %D associated with sequence 6C31036 was $>25\%$ with negative bias for benzoic acid. The associated samples were QC samples (method blank and LCS) and, therefore, no sample data were qualified.

The CCV percent recoveries (%Rs) associated with sequence 6D08012 was $>25\%$ with positive bias for 3,3'-dichlorobenzidine. The associated sample results were non-detects and not affected by the high bias, therefore, were not qualified based on professional judgment.

Method 8270D Scan

The SVOC analyses were scanned for the quantitative ions corresponding to 15 PAH homologue groups. Full calibration of the target groups was not performed; therefore, all sample results that were detects were **qualified J** based on professional judgment.

Method SM 5310B Mod

The recalculated TOC ICAL standard was not within $\pm 10\%$ of the true value for ICAL level 1. The ammonia as N result for sample 5237-160329-DC-EMB020 was a detect whose nominal was $<$ the concentration of the ICAL Level 2 standard and, therefore, was **qualified J**. The remaining associated sample results were detects greater than the concentration of ICAL level 2 and not affected by the high bias demonstrated by the lower standard and, therefore, were not qualified based on professional judgment.



Method SM4500-NH3 Mod

The recalculated ammonia as N ICAL standard was not within $\pm 10\%$ of the true value for ICAL level 1. The ammonia as N result for sample 5237-160329-DC-EMB004 was a detect whose nominal was $<$ the concentration of the ICAL Level 2 standard and, therefore, was **qualified J**. The remaining associated sample results were detects greater than the concentration of ICAL level 2 and not affected by the high bias demonstrated by the lower standard and, therefore, were not qualified based on professional judgment.

Reporting Limit Verification

All CRI recoveries met QC acceptance criteria.

ICP Interference Check Samples (ICS A and ICS AB)

The ICS A and ICS AB analyses were not applicable to all samples because concentrations of the interferents (aluminum, calcium, iron and magnesium) in the samples at their lowest dilutions were $<$ those in the ICS solutions. The ICS recoveries met all QC acceptance criteria.

Blanks

Methods 8260B, 8270D, 8270D Scan, NWTPH-Gx, NWTPH-Dx, 9013M/9014, 9056A, SM 5310B Mod, and SM4500-NHS Mod

No target analytes were detected in the calibration blanks and/or method blanks.

Method 6020

Iron was detected in one the calibration blanks. The associated sample results were $> 10X$ the calibration blank value and, therefore, were not qualified.

Surrogates

All surrogate recoveries met laboratory QC acceptance criteria.

Laboratory Control Sample

The LCS analyses met laboratory acceptance criteria with the following exceptions.



Method 8270D

The LCS recoveries were > the laboratory upper acceptance limits for 3+4-methylphenol and 3,3'-dichlorobenzidine. The associated sample results were non-detects and not affected by the high bias, therefore, were not qualified based on professional judgment.

Matrix Spike (MS)

The MS analyses met laboratory acceptance criteria with the following exceptions.

Methods 8260B, NWTPH-Gx, NWTPH-Dx, and SM 5310B Mod

An MS was not analyzed with the samples in this work order; therefore, matrix-specific accuracy data were not available.

Method 8270D

The MS recovery was > the laboratory upper acceptance limit for benzoic acid. The associated sample results were non-detects and not affected by the high bias and, therefore, were not qualified based on professional judgment.

Method 6020

The MS recovery was < the laboratory lower acceptance limit but $\geq 30\%$ for aluminum. The associated sample results were detects and, therefore, were **qualified J**.

MS recoveries were outside of acceptance limits for iron and manganese. The parent sample concentrations were >4X the spike amounts and, therefore, no sample data were qualified.

Laboratory Duplicate

The laboratory duplicate analyses met all QC acceptance criteria with the following exceptions.

Method 6020

The laboratory duplicate RPD was > the laboratory acceptance limit for aluminum. The associated sample results were detects and, therefore, were **qualified J**.

Method SM 5310B Mod

The laboratory duplicate RPD was > the laboratory acceptance limit for TOC. All associated sample results were detects and, therefore, were **qualified J**.

Internal Standards

All required internal standards met QC acceptance criteria.

ICPMS Serial Dilution

A serial dilution analysis was not performed with the samples in this work order.

Reporting Limits (RLs)

All reporting limits (RLs) were properly reported.

Methods 8260B and NWTPH-Gx

The samples were analyzed as mid-level soils with a 50X dilution factor. RLs were adjusted accordingly and may not have met the project-specified RLs and/or project quantitation limit goals.

Methods 8270D and 8270D Scan

Samples 5237-160329-DC-EMB010, 5237-160329-DC-EMB004, 5237-160329-DC-EMB013, and 5237-160329-DC-EMB014 were diluted 4X. RLs were adjusted accordingly and may not have met the project RLs and/or project quantitation limit goals.

Method 6020A

The samples were analyzed at 10X dilutions. RLs were adjusted accordingly and may not have met the project-specified RLs and/or project quantitation limit goals.

Other QC

QC summary forms were either incomplete or not submitted in the data package for some analyses. In these cases, the results were either found in the raw data or were calculated for validation purposes (refer to the Comments sections of the data validation spreadsheets).

No other specific issues that affect data quality were identified.

Hahn Data Validation Summary Worksheet

SDG#: A6C1124	Laboratory: Apex	Validator: Jeanne Peterson	Validation Date: 06/14/2016
Site: Siltronic - Doane Creek	COC#: NA		Validation Level: <input type="checkbox"/> II <input checked="" type="checkbox"/> III
Matrix: Soil	# of Samples: 16	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 checked="" type="checkbox"/> VOCs <input checked="" type="checkbox"/> SVOCs <input checked="" type="checkbox"/> PAHs <input checked="" type="checkbox"/> GRO <input checked="" type="checkbox"/> DRO <input type="checkbox"/> Pests <input type="checkbox"/> PCBs <input checked="" type="checkbox"/> Metals <input checked="" type="checkbox"/> Gen Chem <input checked="" type="checkbox"/> Cyanide <input type="checkbox"/> Other:			

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
None								

Comments: Samples collected 3/29.
Cooler temps OK.

Hahn Level III GCMS Worksheet

SDG: A6C1124	Method: 8260B	Matrix: Soil	Lab Sample IDs: A6C1124-01, -03, -05, -07, -09, -11, -13, -15
--------------	---------------	--------------	---------------------------------------------------------------

Batch #: 6031000

Tuning: Pass Fail TICs Required? Yes No (lab limits) (lab limits)

Analyte (outliers)	Calibration				Method Blank	5X (10X) Method Blank	LCS %R	MS %R	MSD %R	MS/D RPD	Lab Dup RPD				
	RF ≥0.05	RSD/r ² ≤30% ≥0.990	ICV %D ±25%	CCV %D ±25%											
None (BTEX only)															

Surrogate Recovery Outliers (method/lab limits)									
Sample ID	DBFM	1,4-DFB	Tol-d8	4-BFB	Sample ID	DBFM	1,4-DCB	Tol-d8	4-BFB
None									

IS Outliers (-50% to +100% of CCV)												
Sample ID	Area	RT	Area	RT	Area	RT	Area	RT	Area	RT	Area	RT
None												

Comments: HTs OK. ICAL A6C0904
 MB, LCS, -01 Dup; All samples diluted 50X

Hahn Level III GCMS Worksheet (cont)

SDG: A6C1124	Method: 8270D	Matrix: Soil	Lab Sample IDs: A6C1124-02, -04, -06, -08, -10, -12, -14, -16
Batch #s: 6031018			

Comments: HTs OK. -Same ICAL and ICV/CCV as 8270D Scan; ICAL and SSV raw data included with 8270D Scan section.

MB, LCS, -02 DUP, -16 MS

ICAL A6C0702; CCV: 6D01020 - -02, -02DUP; CCV:6D08012 - -08, -04, -14, -16

ICAL A6C3104; CCV: 6C31036 - MB, LCS; CCV: 6D04009 - -6, -12, -10, -16MS

IS summary missing for 6C31036; IS results on Forms Is; raw data checked.

*Alternate curve analyzed; OK

Samples -02, -06, -10, and -12 diluted 4X

Revised 9/2010

Hahn Level III GCMS Worksheet

SDG: A6C1124	Method: 8270D Scan	Matrix: Soil	Lab Sample IDs: A6C1124-02, -04, -06, -08, -10, -12, -14, -16
--------------	--------------------	--------------	---------------------------------------------------------------

Batch #s: 6031018

Tuning: Pass Fail TICs Required? Yes No (lab limits) (lab limits)

Analyte (outliers)	Calibration				Method Blank	5X (10X) Method Blank	LCS %R	MS %R	MSD %R	MS/D RPD	Lab Dup RPD				
	RF ≥0.05	RSD/r ² ≤30%	SSV %D ±25%	CCV %D ±25%											
1,6,7-Trimethylnaphthalene	*	*	*	*	✓	NA	✓	✓	NA	NA	✓				
1-Methylphenanthrene	*	*	*	*	✓	NA	✓	✓	NA	NA	✓				

Surrogate Recovery Outliers (lab limits)

Sample ID	Acenaphthene-d8	Benzo(a)pyrene-d12	Sample ID	Acenaphthene-d8	Benzo(a)pyrene-d12
None					

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

Sample ID	Area	RT	Area	RT	Area	RT	Area	RT	Area	RT	Area	RT
None												

Comments: HTs OK. - All detects qualified J due to use of scan mode instead of full calibration.
 *Same ICAL and ICV/CCV as 8270D. ICAL and ICV/CCV summaries in 8270D form section do not have extra compounds (2,6-dimethylnaphthalene, 1,6,7-trimethylnaphthalene, and 1-methylphenanthrene). ICAL and ICV/CCV raw data have results for 2,6-DMN, but only CCV raw data has 1,6,7-TMP and 1-MP results.
 MB, LCS, -02 DUP, -16 MS
 ICAL A6C0702; CCV: 6D01020 - -02, -02DUP; CCV:6D08012 - -08, -04, -14, -16
 ICAL A6C3104; CCV: 6C31036 - MB, LCS; CCV: 6D04009 - -6, -12, -10, -16MS
 IS summary missing for 6C31036; IS results on Forms Is; raw data checked.
 Samples -02, -06, -10, and -12 diluted 4X

Hahn Level III NWTPH-GX Worksheet

SDG: A6C1124	Matrix: Soil	Lab Sample IDs: A6C1124-01, -03, -05, -07, -09, -11, -13, -15
Method/Batch #s: 6031000		

Tuning: Pass Fail TICs Required? Yes No (lab limits) (lab limits)

Analyte (outliers)	Calibration			Method Blank	5X Blank	LCS %R	MS %R	MSD %R	MS/D RPD	Lab Dup RPD
	r ² ≥0.990 ±20%	ICV/CCV %D ±20%	RT Windows							
None							NA	NA	NA	

Surrogate Outliers (50-150%)

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

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

Area	RT	Area	RT	Area	RT	Area	RT	Area	RT	Area	RT
None											

Comments: HTs OK. MB, LCS, -01dup
 Lab used avg RFs; RSD <15%. ICV/CV surrogates checked as %Rs. All OK.
 All samples diluted 50X

Hahn Level III NWTPH-DX Worksheet

SDG: A6C1124	Matrix: Soil	Lab Sample IDs: A6C1124-02, -04, -06, -08, -10, -12, -14, -16
Method/Batch #s: 6031026, 6031010		

Analyte (outliers)	Calibration			Method Blank	5X Blank	LCS %R	MS %R	MSD %R	MS/D RPD	Lab DUP RPD
	r^2 ≥ 0.990 $\pm 20\%$	ICV/CCV %D $\pm 15\%$	RT Windows							
None										

Surrogate Outliers (50-150%)

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

Comments: HTs OK.
 6031026: MB, LCS, -02, -04, -04 DUP, -06
 6031010: MB, LCS, -08, -10, -13, -14, -16
 Lab use avg RFs; all RSDs <15%. ICV/CV surrogates checked as %Rs. All OK.
 No dilutions

Hahn Level III Metals Worksheet

SDG: A6C1124	Matrix: Soil	Lab Sample IDs: A6C1124-02, -04, -06, -08, -10, -12, -14, -16
Method: 6020	Batch #: 6040228	

ICPMS Mass Cal: Pass Fail NA ICPMS %RSD: Pass Fail NA (80-120%) (75-125%)

Analyte (outliers)	(90-110%) Calibration							ICS A <IDL	ICS AB %R ±20%	MB mg/kg	10X MB mg/kg	LCS %R	MS %R	MSD %R	Lab Dup RPD ≤20%	PS %R	Ser. Dil. %D ≤10%
	r	ICV	CCV	CRI	ICB	CCB ug/L	10X CCB mg/kg										
Al	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	74	NA	47	NA	NA
Fe	✓	✓	✓	✓	✓	48.2	48.2	#	#	✓	NA	✓	14*	NA	✓	NA	NA
Mn	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	-365*	NA	✓	NA	NA

IS Outliers (Samples 60-125%; CCV/CCB 80-120%)				IS Outliers (Samples 60-125%; CCV/CCB 80-120%)			
Sample ID	%Recovery	%Recovery	%Recovery	CCV/CCB ID	%Recovery	%Recovery	%Recovery
None		--	--	None			

Comments: HTs OK. CRI ≤ CRDL, not at 2X CRDL.
 MB, LCS< -06DUP, -06MS
 *Parent sample conc >4X spike amount.
 #All samples diluted 10X; nominals < ICS spike amounts for all samples.
 Na sample and dup results <5X RL, and abs diff <RL; not qualified for RPD >20%.

