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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 A6D0013

SUMMARY

Level III data validation was performed on the data for 12 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
5237-160331-NDP-SED003G	A6D0013-01	Soil	VOCs, GRO
5237-160331-NDP-SED003	A6D0013-02	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160331-NDP-SED002G	A6D0013-03	Soil	VOCs, GRO
5237-160331-NDP-SED002	A6D0013-04	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160331-NDP-SED001G	A6D0013-05	Soil	VOCs, GRO
5237-160331-NDP-SED001	A6D0013-06	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160331-NDP-SED005G	A6D0013-07	Soil	VOCs, GRO
5237-160331-NDP-SED005	A6D0013-08	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160331-NDP-SED004G	A6D0013-09	Soil	VOCs, GRO
5237-160331-NDP-SED004	A6D0013-10	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160331-NDP-EMB001G	A6D0013-11	Soil	VOCs, GRO
5237-160331-NDP-EMB001	A6D0013-12	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
5237-160331-NDP-SED003	8270D	Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Chrysene Dibenz(a,h)anthracene Fluoranthene Indeno(1,2,3-cd)pyrene Phenanthrene Pyrene Pentachlorophenol	J	Poor replicate precision
	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes	J	Insufficient calibration
		C1-Fluoranthenes/Pyrenes C2-Phenanthrenes/ Anthracenes	J	Insufficient calibration
	6020	Aluminum Zinc	J	High matrix spike recovery
		Vanadium	J	High matrix spike recovery and poor replicate precision
		Manganese	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	High initial calibration positive bias, low matrix spike recovery, and poor replicate precision
5237-160331-NDP-SED002G	8260B	Acetone	UJ	High calibration verification negative bias
5237-160331-NDP-SED002	8270D	Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Chrysene Dibenz(a,h)anthracene Fluoranthene Indeno(1,2,3-cd)pyrene Phenanthrene	J	Poor replicate precision

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
		Pyrene		
		Pentachlorophenol	UJ	Poor replicate precision
	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes	J	Insufficient calibration
	6020	Aluminum Zinc	J	High matrix spike recovery
		Vanadium	J	High matrix spike recovery and poor replicate precision
		Manganese	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision
5237-160331-NDP-SED001	8270D	Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Chrysene Dibenz(a,h)anthracene Fluoranthene Indeno(1,2,3-cd)pyrene Phenanthrene Pyrene	J	Poor replicate precision
		Pentachlorophenol	UJ	Poor replicate precision
	6020	Aluminum Zinc	J	High matrix spike recovery
		Vanadium	J	High matrix spike recovery and poor replicate precision
		Manganese	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
5237-160331-NDP-SED005	8270D	Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Chrysene Dibenz(a,h)anthracene Fluoranthene Indeno(1,2,3-cd)pyrene Phenanthrene Pyrene	J	Poor replicate precision
		Pentachlorophenol	UJ	Poor replicate precision
	8270D Scan	C1-Fluoranthenes/Pyrenes	J	Insufficient calibration
	6020	Aluminum Zinc	J	High matrix spike recovery
		Vanadium	J	High matrix spike recovery and poor replicate precision
		Manganese	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision
5237-160331-NDP-SED004G	8260B	Acetone	UJ	High calibration verification negative bias
5237-160331-NDP-SED004	8270D	Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(g,h,i)perylene Chrysene Dibenz(a,h)anthracene Fluoranthene Indeno(1,2,3-cd)pyrene Phenanthrene Pyrene	J	Poor replicate precision
		Pentachlorophenol	UJ	Poor replicate precision

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Fluorenes C1-Phenanthrenes/ Anthracenes C2-Chrysenes/ Benz(a)anthracenes C2-Naphthalenes C3-Chrysenes/ Benz(a)anthracenes C3-Naphthalenes C4-Naphthalenes	J	Insufficient calibration
	6020	Aluminum Zinc	J	High matrix spike recovery
		Vanadium	J	High matrix spike recovery and poor replicate precision
		Manganese	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision
5237-160331-NDP-EMB001	8270D	Acenaphthene Anthracene Carbazole	J	Low matrix spike recovery
		Benzo(k)fluoranthene Dibenz(a,h)anthracene Phenanthrene	J	Low matrix spike recovery and poor replicate precision
		Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(g,h,i)perylene Chrysene Fluoranthene Indeno(1,2,3-cd)pyrene Pyrene Pentachlorophenol	J	Poor replicate precision
	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Phenanthrenes/ Anthracenes	J	Insufficient calibration

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
		C2-Chrysenes/ Benz(a)anthracenes C2-Phenanthrenes/ Anthracenes C3-Chrysenes/ Benz(a)anthracenes C3-Phenanthrenes/ Anthracenes C4-Chrysenes/ Benz(a)anthracenes		
	6020	Aluminum Zinc	J	High matrix spike recovery
		Vanadium	J	High matrix spike recovery and poor replicate precision
		Manganese	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision

DISCUSSION

Sample Shipping/Receiving

All COC, analysis request, and sample receipt documentation was complete and correct with the following exception.

The collection time on one of the two VOC vials preserved with methanol was 11:20. The collection time on the COC and remaining sample containers was 11:00.

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 8260B

The initial calibration (ICAL) relative response factors (RRFs) were <0.05 but ≥0.01 for chloroethane and trichlorofluoromethane. Quadratic equations were used to calculate the sample results; therefore no sample data were qualified based on professional judgment.

The initial calibration verification (ICV) and/or continuing calibration verification (CCV) percent differences (%Ds) were >25% with positive bias for chloroethane, trichlorofluoromethane, bromoform, bromomethane, and 1,1,2,2-tetrachloroethane. The associated sample results were non-detects and not affected by the high bias, therefore, were not qualified based on professional judgment.

The ICV %D was >25% with negative bias for acetone. The associated sample results were non-detects and, therefore, were **qualified UJ**.

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 %D associated with sequence 6D07009 was >25% with positive bias 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 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 6020

The CCV recovery associated with sequence 6D11028 was > the acceptance limit for selenium. The associated sample results were non-detects and not affected by the high bias and, therefore, were not qualified based on professional judgment.

Method SM 5310B Mod

The recalculated TOC ICAL standard was not within ±10% of the true value for ICAL level 1. The 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 ±10% of the true value for ICAL level 1. The ammonia as N result for sample 5237-160331-NDP-SED003 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.

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

Chromium was detected in one the calibration blanks associated with sequence 6D11028. The associated sample results were detects >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 8260B

The LCS recoveries were > the laboratory upper acceptance limits for chloroethane, trichlorofluoromethane, and bromomethane. The associated sample results were non-detects and not affected by the high bias, therefore, were not qualified based on professional judgment.

Method 8270D

The LCS recovery was > the laboratory upper acceptance limit 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.

Matrix Spike (MS)

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

Method 8260B

The MS was performed on a project sample from work order A6C1134, and the recoveries for several analytes were > the laboratory upper acceptance limits. The associated sample results were non-detects and not affected by the high bias, therefore, were not qualified based on professional judgment.

Method 8270D

The MS recoveries were < the laboratory lower acceptance limit for acenaphthene, anthracene, benzo(k)fluoranthene, dibenz(a,h)anthracene, phenanthrene, and carbazole. The results for parent sample 5237-160331-NDP-EMB001 were detects and, therefore, were **qualified J**. The remaining associated sample results were not qualified based on professional judgment.

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, therefore, were not qualified based on professional judgment.

Method 8270D Scan

The MS recoveries were < the laboratory lower acceptance limit for pyrene and chrysene. The parent sample concentrations were >4X the spike amounts and, therefore, no sample data were qualified.

Method 6020

The MSs were performed on project samples from work order A6C1076, and the MS recoveries associated with parent sample 5237-160328-DC-SED063 were > the laboratory upper acceptance limits for vanadium, aluminum, and zinc. The associated vanadium, aluminum, and zinc results were detects and, therefore, were **qualified J**.

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

Method SM4500-NH3 Mod

The MS recovery was < the laboratory lower acceptance limit but ≥30% for ammonia as N. The associated sample results were detects and, therefore, were **qualified J**.

Methods 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.

Laboratory Duplicate

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

Method 8270D

The laboratory duplicate relative percent differences (RPDs) were > the laboratory acceptance limits for 12 target analytes (refer to data validation worksheet). The associated sample results that were detects were **qualified J**, and the associated sample results that were non-detects were **qualified UJ**.

Method 8270D Scan

The laboratory duplicate RPDs or absolute values were > the laboratory acceptance limits for C1-fluoranthenes/pyrenes, C1-phenanthrenes/anthracenes, C2-phenanthrenes/anthracenes, and C3-phenanthrenes/anthracenes. Precision was not expected due to the method used to quantitate the results, and the analytes that were detects were already qualified for insufficient calibration; therefore, the sample results were not further qualified based on professional judgment.

Method 6020

The laboratory duplicate RPDs were > the laboratory acceptance limit for iron, vanadium, and manganese. All associated sample results were detects and, therefore, were **qualified J**.

Method SM4500-NH3 Mod

The laboratory duplicate RPD was > the laboratory acceptance limit for ammonia as N. 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

Sample 5237-160331-NDP-EMB001 was diluted 10X, and all samples *except* 5237-160331-NDP-EMB001were 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 5X dilutions. In addition, samples 5237-160331-NDP-SED003 and 5237-160331-NDP-SED002 were analyzed at 50X dilutions. RLs were adjusted accordingly and may not have met the project-specified RLs and/or project quantitation limit goals.

Method SM4500-NH3 Mod

Sample 5237-160331-NDP-SED005 was diluted 10X. RLs were adjusted accordingly and may not have met the project 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#: A6D0013	Laboratory: Apex	Validator: Jeanne Peterson	Validation Date: 06/14/2016
Site: Siltronic - Doane Creek	COC#: NA		Validation Level: ☐ II ☒ III
Matrix: Sediment	# of Samples: 12	Tracking docs present: See sample receipt and log-in documentation	
COCs present: Yes	COCs signed: Yes	COCs dated: Yes	Sample Container Integrity: OK

Analyses:
☒ VOCs ☒ SVOCs ☒ PAHs ☒ GRO ☒ DRO ☐ Pests ☐ PCBs ☒ Metals ☒ Gen Chem ☒ Cyanide
☐ 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 3/31.

Cooler temps OK.

Hahn Level III GCMS Worksheet

SDG: A6D0013	Method: 8260B	Matrix: Sediment	Lab Sample IDs: A6D0013-01, -03, -05, -07, -09, -11
Batch #s: 6040011			

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/r²** ≤30% ≥0.990	**ICV %D** ±25%	**CCV %D** ±25%										
Chloroethane	0.0201**	✓	268	105	✓	NA	203	*	NA	NA	*			
Trichlorofluoromethane	0.0299**	✓	193	84.8	✓	NA	185	*	NA	NA	*			
Acetone	✓	✓	-27.6	✓	✓	NA	✓	*	NA	NA	*			
Bromoform	✓	✓	✓	36.8	✓	NA	✓	*	NA	NA	*			
Bromomethane	✓	✓	✓	57.2	✓	NA	137	*	NA	NA	*			
1,1,2,2-Tetrachloroethane	✓	✓	✓	36.6	✓	NA	✓	*	NA	NA	*			

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
A6C0904/6040011: MB, LCS, all samples; all samples diluted 50X
*Lab dup and MS performed on project sample from A6C1134; Dup OK, MS recoveries high, but only parent sample qualified.
**Alternate curve used
Tune summaries missing entries for m/z 173; found in raw data.

Hahn Level III GCMS Worksheet

SDG: A6D0013	Method: 8270D	Matrix: Sediment	Lab Sample IDs: A6D0013-02, -04, -06, -08, -10, -12
Batch #s: 6040143			

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%											
ICAL A6C3104															
PCP (Level 3 and 4 only)	0.0315*	✓	✓												
2,4,6-TBP (surr) (L 3&4)	0.0367*	✓	✓												
Sequence 6D06014															
None (MB and LCS only)															
Sequence 6D07009															
3,3'-Dichlorobenzidine	✓	✓	✓	25.4											
Batch 6040143															
3+4-Methylphenol	✓	✓	✓	✓	✓	NA	✓	**	NA	NA	**				
3,3'-Dichlorobenzidine	✓	✓	✓	✓	✓	NA	157	**	NA	NA	**				
Benzoic acid	✓	✓	✓	✓	✓	NA	✓	**	NA	NA	**				

Surrogate Recovery Outliers *(lab limits)*

Sample ID	2-Fluorophenol	Phenol-d5	2,4,6-TBP	Nitrobenzene-d5	2-Fluorobiphenyl	Terphenyl-d14
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. -Same ICAL and ICV/CCV as 8270D Scan; ICAL and SSV raw data included with 8270D Scan section.
MB, LCS, -02, -02DUP, -04, -06, -08, -10, -12, -12MS
**Refer to attached MS and Dup sheet for outliers.
IS summary missing for 6C31036; IS results on Forms Is; raw data checked.
*Alternate curve analyzed; OK
Sample -12 diluted 10X; all others diluted 4X

DUPLICATES

EPA 8270D

5237-160331-NDP-SED003

Laboratory: Apex Laboratories
Client: Hahn and Associates
Matrix: Sediment
Batch: 6040143
Preparation: EPA 3546
Source Sample Name: 5237-160331-NDP-SED003

SDG: A6D0013
Project: Siltronic RI-Doane Creek
Laboratory ID: 6040143-DUP2
Lab Source ID: A6D0013-02RE1
Initial/Final: 15.53 g / 2 mL
% Solids: 72.22

ANALYTE	CONTROL LIMIT	SAMPLE CONCENTRATION (ug/kg dry)	C	DUPLICATE CONCENTRATION (ug/kg dry)	C	RPD %	Q	METHOD
Acenaphthene abs diff <RL	30	10.9		ND				EPA 8270D
Acenaphthylene	30	16.7		16.9		1		EPA 8270D
Anthracene abs diff <RL	30	19.6		ND				EPA 8270D
Benz(a)anthracene	30	145		35.9		121	*	EPA 8270D
Benzo(a)pyrene	30	231		68.3		109	*	EPA 8270D
Benzo(b)fluoranthene	30	286		72.4		119	*	EPA 8270D
Benzo(k)fluoranthene	30	98.2		24.9		119	*	EPA 8270D
Benzo(g,h,i)perylene	30	204		70.8		97	*	EPA 8270D
Chrysene	30	171		45.5		116	*	EPA 8270D
Dibenz(a,h)anthracene	30	32.4		9.22		111	*	EPA 8270D
Fluoranthene	30	272		65.4		122	*	EPA 8270D
Fluorene abs diff <RL	30	9.33		ND				EPA 8270D
Indeno(1,2,3-cd)pyrene	30	184		55.5		107	*	EPA 8270D
1-Methylnaphthalene	30	ND		ND				EPA 8270D
2-Methylnaphthalene	30	ND		ND				EPA 8270D
Naphthalene	30	ND		ND				EPA 8270D
Phenanthrene	30	96.9		24.6		119	*	EPA 8270D
Pyrene	30	291		105		94	*	EPA 8270D
Carbazole abs diff <RL	30	23.5		ND				EPA 8270D
Dibenzofuran	30	ND		ND				EPA 8270D
4-Chloro-3-methylphenol	30	ND		ND				EPA 8270D
2-Chlorophenol	30	ND		ND				EPA 8270D
2,4-Dichlorophenol	30	ND		ND				EPA 8270D
2,4-Dimethylphenol	30	ND		ND				EPA 8270D
2,4-Dinitrophenol	30	ND		ND				EPA 8270D
4,6-Dinitro-2-methylphenol	30	ND		ND				EPA 8270D
2-Methylphenol	30	ND		ND				EPA 8270D
3+4-Methylphenol(s)	30	ND		ND				EPA 8270D
2-Nitrophenol	30	ND		ND				EPA 8270D
4-Nitrophenol	30	ND		ND				EPA 8270D
Pentachlorophenol (PCP)	30	98.8		623		145	*	EPA 8270D
Phenol	30	ND		ND				EPA 8270D

DUPLICATES

EPA 8270D

Laboratory: Apex Laboratories
Client: Hahn and Associates
Matrix: Sediment
Batch: 6040143
Preparation: EPA 3546
Source Sample Name: 5237-160331-NDP-SED003

SDG: A6D0013
Project: Siltronic RI-Doane Creek
Laboratory ID: 6040143-DUP2
Lab Source ID: A6D0013-02RE1
Initial/Final: 15.53 g / 2 mL
% Solids: 72.22

ANALYTE	CONTROL LIMIT	SAMPLE CONCENTRATION (ug/kg dry)	C	DUPLICATE CONCENTRATION (ug/kg dry)	C	RPD %	Q	METHOD
2,3,4,6-Tetrachlorophenol	30	ND		ND				EPA 8270D
2,3,5,6-Tetrachlorophenol	30	ND		ND				EPA 8270D
2,4,5-Trichlorophenol	30	ND		ND				EPA 8270D
2,4,6-Trichlorophenol	30	ND		ND				EPA 8270D
Bis(2-ethylhexyl)phthalate	30	ND		ND				EPA 8270D
Butyl benzyl phthalate	30	ND		ND				EPA 8270D
Diethylphthalate	30	ND		ND				EPA 8270D
Dimethylphthalate	30	ND		ND				EPA 8270D
Di-n-butylphthalate	30	ND		ND				EPA 8270D
Di-n-octyl phthalate	30	ND		ND				EPA 8270D
N-Nitrosodimethylamine	30	ND		ND				EPA 8270D
N-Nitroso-di-n-propylamine	30	ND		ND				EPA 8270D
N-Nitrosodiphenylamine	30	ND		ND				EPA 8270D
Bis(2-Chloroethoxy) methane	30	ND		ND				EPA 8270D
Bis(2-Chloroethyl) ether	30	ND		ND				EPA 8270D
Bis(2-Chloroisopropyl) ether	30	ND		ND				EPA 8270D
Hexachlorobenzene	30	ND		ND				EPA 8270D
Hexachlorobutadiene	30	ND		ND				EPA 8270D
Hexachlorocyclopentadiene	30	ND		ND				EPA 8270D
Hexachloroethane	30	ND		ND				EPA 8270D
2-Chloronaphthalene	30	ND		ND				EPA 8270D
1,2-Dichlorobenzene	30	ND		ND				EPA 8270D
1,3-Dichlorobenzene	30	ND		ND				EPA 8270D
1,4-Dichlorobenzene	30	ND		ND				EPA 8270D
1,2,4-Trichlorobenzene	30	ND		ND				EPA 8270D
4-Bromophenyl phenyl ether	30	ND		ND				EPA 8270D
4-Chlorophenyl phenyl ether	30	ND		ND				EPA 8270D
Aniline	30	ND		ND				EPA 8270D
4-Chloroaniline	30	ND		ND				EPA 8270D
2-Nitroaniline	30	ND		ND				EPA 8270D
3-Nitroaniline	30	ND		ND				EPA 8270D
4-Nitroaniline	30	ND		ND				EPA 8270D

DUPLICATES

EPA 8270D

Laboratory: Apex Laboratories
Client: Hahn and Associates
Matrix: Sediment
Batch: 6040143
Preparation: EPA 3546
Source Sample Name: 5237-160331-NDP-SED003

SDG: A6D0013
Project: Siltronic RI-Doane Creek
Laboratory ID: 6040143-DUP2
Lab Source ID: A6D0013-02RE1
Initial/Final: 15.53 g / 2 mL
% Solids: 72.22

ANALYTE	CONTROL LIMIT	SAMPLE CONCENTRATION (ug/kg dry)	C	DUPLICATE CONCENTRATION (ug/kg dry)	C	RPD %	Q	METHOD
Nitrobenzene	30	ND		ND				EPA 8270D
2,4-Dinitrotoluene	30	ND		ND				EPA 8270D
2,6-Dinitrotoluene	30	ND		ND				EPA 8270D
Benzoic acid	30	ND		ND				EPA 8270D
Benzyl alcohol	30	ND		ND				EPA 8270D
Isophorone	30	ND		ND				EPA 8270D
Azobenzene (1,2-DPH)	30	ND		ND				EPA 8270D
Bis(2-Ethylhexyl) adipate	30	ND		ND				EPA 8270D
3,3'-Dichlorobenzidine	30	ND		ND				EPA 8270D
1,2-Dinitrobenzene	30	ND		ND				EPA 8270D
1,3-Dinitrobenzene	30	ND		ND				EPA 8270D
1,4-Dinitrobenzene	30	ND		ND				EPA 8270D
Pyridine	30	ND		ND				EPA 8270D
1,4-Dichlorobenzene-d4 (ISTD)		2000		2000				EPA 8270D

* Values outside of QC limits

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY

EPA 8270D

5237-160331-NDP-EMB001

Laboratory: Apex Laboratories
SDG: A6D0013
Client: Hahn and Associates
Project: Siltronic RI-Doane Creek
Matrix: Sediment
Batch: 6040143
Laboratory ID: 6040143-MS1
Preparation: EPA 3546
Initial/Final: 15.24 g / 2 mL

4X spike amount = 3024

Source Sample Name: 5237-160331-NDP-EMB001

COMPOUND	SPIKE ADDED (ug/kg dry)	SAMPLE CONCENTRATION (ug/kg dry)	MS CONCENTRATION (ug/kg dry)	MS % REC. (*=Out)	QC LIMITS REC.
Acenaphthene	756	631	870	32 *	40 - 122
Acenaphthylene	756	49.7	730	90	32 - 132
Anthracene	756	713	1040	43 *	47 - 123
Benz(a)anthracene	756	3930 >4X	2640	-171 *	49 - 126
Benzo(a)pyrene	756	5390	3500	-250 *	45 - 129
Benzo(b)fluoranthene	756	6790	4320	-328 *	45 - 132
Benzo(k)fluoranthene	756	2640	2110	-70 *	47 - 132
Benzo(g,h,i)perylene	756	3620 >4X	2820	-106 *	43 - 134
Chrysene	756	4420 >4X	2940	-196 *	50 - 124
Dibenz(a,h)anthracene	756	856	1100	33 *	45 - 134
Fluoranthene	756	5610 >4X	3720	-250 *	50 - 127
Fluorene	756	333	835	66	43 - 125
Indeno(1,2,3-cd)pyrene	756	3640 >4X	2780	-114 *	45 - 133
1-Methylnaphthalene	756	ND	729	97	40 - 120
2-Methylnaphthalene	756	55.2	743	91	38 - 122
Naphthalene	756	120	735	81	35 - 123
Phenanthrene	756	2940	2060	-116 *	50 - 121
Pyrene	756	5350 >4X	3560	-237 *	47 - 127
Carbazole	756	716	1050	45 *	50 - 122
Dibenzofuran	756	164	761	79	44 - 120
4-Chloro-3-methylphenol	756	ND	770	102	45 - 122
2-Chlorophenol	756	ND	784	104	34 - 121
2,4-Dichlorophenol	756	ND	758	100	40 - 122
2,4-Dimethylphenol	756	ND	791	105	30 - 127
2,4-Dinitrophenol	756	ND	722	96	5 - 137
4,6-Dinitro-2-methylphenol	756	ND	764	101	29 - 132
2-Methylphenol	756	ND	833	110	32 - 122
3+4-Methylphenol(s)	756	ND	868	115	34 - 120
2-Nitrophenol	756	ND	690	91	36 - 123
4-Nitrophenol	756	ND	780	103	30 - 132
Pentachlorophenol (PCP)	756	ND	815	108	25 - 133
Phenol	756	ND	749	99	34 - 120
2,3,4,6-Tetrachlorophenol	756	ND	829	110	44 - 125
2,3,5,6-Tetrachlorophenol	756	ND	829	110	40 - 120
2,4,5-Trichlorophenol	756	ND	742	98	41 - 124
2,4,6-Trichlorophenol	756	ND	774	102	39 - 126
Bis(2-ethylhexyl)phthalate	756	ND	976	129	51 - 133

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY

EPA 8270D

5237-160331-NDP-EMB001

Laboratory:	Apex Laboratories	SDG:	A6D0013
Client:	Hahn and Associates	Project:	Siltronic RI-Doane Creek
Matrix:	Sediment		
Batch:	6040143	Laboratory ID:	6040143-MS1
Preparation:	EPA 3546	Initial/Final:	15.24 g / 2 mL

Source Sample Name: 5237-160331-NDP-EMB001

COMPOUND	SPIKE ADDED (ug/kg dry)	SAMPLE CONCENTRATION (ug/kg dry)	MS CONCENTRATION (ug/kg dry)	MS % REC. (*=Out)	QC LIMITS REC.
Butyl benzyl phthalate	756	ND	743	98	48 - 132
Diethylphthalate	756	ND	783	104	50 - 124
Dimethylphthalate	756	ND	723	96	48 - 124
Di-n-butylphthalate	756	ND	817	108	51 - 128
Di-n-octyl phthalate	756	ND	885	117	44 - 140
N-Nitrosodimethylamine	756	ND	543	72	23 - 120
N-Nitroso-di-n-propylamine	756	ND	774	102	36 - 120
N-Nitrosodiphenylamine	756	ND	776	103	38 - 127
Bis(2-Chloroethoxy) methane	756	ND	668	88	36 - 121
Bis(2-Chloroethyl) ether	756	ND	630	83	31 - 120
Bis(2-Chloroisopropyl) ether	756	ND	676	90	33 - 131
Hexachlorobenzene	756	ND	693	92	44 - 122
Hexachlorobutadiene	756	ND	621	82	32 - 123
Hexachlorocyclopentadiene	756	ND	270	36	5 - 140
Hexachloroethane	756	ND	550	73	28 - 120
2-Chloronaphthalene	756	ND	683	90	41 - 120
1,2-Dichlorobenzene	756	ND	654	87	33 - 120
1,3-Dichlorobenzene	756	ND	604	80	30 - 120
1,4-Dichlorobenzene	756	ND	620	82	31 - 120
1,2,4-Trichlorobenzene	756	ND	623	82	34 - 120
4-Bromophenyl phenyl ether	756	ND	685	91	46 - 124
4-Chlorophenyl phenyl ether	756	ND	660	87	45 - 121
Aniline	756	ND	547	72	7 - 120
4-Chloroaniline	756	ND	497	66	16 - 120
2-Nitroaniline	756	ND	672	89	44 - 127
3-Nitroaniline	756	ND	522	69	33 - 120
4-Nitroaniline	756	ND	610	81	35 - 120
Nitrobenzene	756	ND	730	97	34 - 122
2,4-Dinitrotoluene	756	ND	680	90	48 - 126
2,6-Dinitrotoluene	756	ND	722	96	46 - 124
Benzoic acid	1510	ND	2450	162 *	5 - 140
Benzyl alcohol	756	ND	758	100	29 - 122
Isophorone	756	ND	734	97	30 - 122
Azobenzene (1,2-DPH)	756	ND	766	101	39 - 125
Bis(2-Ethylhexyl) adipate	756	ND	829	110	60 - 121
3,3'-Dichlorobenzidine	1510	ND	1630	108	22 - 121
1,2-Dinitrobenzene	756	ND	673	89	44 - 120

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY

EPA 8270D

5237-160331-NDP-EMB001

Laboratory:	Apex Laboratories	SDG:	A6D0013
Client:	Hahn and Associates	Project:	Siltronic RI-Doane Creek
Matrix:	Sediment		
Batch:	6040143	Laboratory ID:	6040143-MS1
Preparation:	EPA 3546	Initial/Final:	15.24 g / 2 mL

Source Sample Name: 5237-160331-NDP-EMB001

COMPOUND	SPIKE ADDED (ug/kg dry)	SAMPLE CONCENTRATION (ug/kg dry)	MS CONCENTRATION (ug/kg dry)	MS % REC. (*=Out)	QC LIMITS REC.
1,3-Dinitrobenzene	756	ND	681	90	42 - 127
1,4-Dinitrobenzene	756	ND	644	85	37 - 132
Pyridine	756	ND	578	77	5 - 120

Hahn Level III GCMS Worksheet

SDG: A6D0013	Method: 8270D Scan	Matrix: Sediment	Lab Sample IDs: A6D0013-02, -04, -06, -08, -10, -12
Batch #s: 6040143			

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** *(≤25%)*				
	RF ≥0.05	**RSD/r²** ≤30%	**SSV %D** ±25%	**CCV %D** ±25%											
1,6,7-Trimethylnaphthalene	**	**	**	**	✓	NA	✓	✓	NA	NA	✓				
1-Methylphenanthrene	**	**	**	**	✓	NA	✓	✓	NA	NA	✓				
C1-Fluoranthenes/Pyrenes	**	**	**	**	✓	NA	✓	✓	NA	NA	26				
C1-Phenanthrenes/Anthrace	**	**	**	**	✓	NA	✓	✓	NA	NA	|102|				
C2-Phenanthrenes/Anthrace	**	**	**	**	✓	NA	✓	✓	NA	NA	130				
C3-Phenanthrenes/Anthrace	**	**	**	**	✓	NA	✓	✓	NA	NA	|214|				
Pyrene NTA	**	**	**	**	✓	NA	✓	-301*	NA	NA	96				
Chrysene NTA	**	**	**	**	✓	NA	✓	-242*	NA	NA	|101|				

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.

*Parent sample conc >4SX spike amount.

**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, -02DUP, -04, -06, -08, -10, -12, -12MS

IS summary for QC not included in data package. See 8270D IS summaries.

Sample -12 diluted 10X; all others diluted 4X

Hahn Level III NWTPH-GX Worksheet

SDG: A6D0013	Matrix: Sediment	Lab Sample IDs: A6D0013-01, -03, -05, -07, -09, -11
Method/Batch #s: 6040011		

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	**ICV/CCV %D** ±20%	**RT Windows** ±20%								
None								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.
A6C0904/6040011: MB, LCS, samples
*Lab dup and MS performed on project sample from A6C1134; Dup and MS recoveries OK
Tune summaries missing entries for m/z 173; found in raw data. ICV/CV surrogates checked as %Rs. All OK.
All samples diluted 50X

Hahn Level III NWTPH-DX Worksheet

SDG: A6D0013	Matrix: Sediment	Lab Sample IDs: A6D0013-02, -04, -06, -08, -10, -12
Method/Batch #s: 6040064		

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** ±15%	**RT Windows**								
None											

Surrogate Outliers *(50-150%)*

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

Comments: HTs OK.
6040064: MB, LCS, -02, -02DUP, -04, -06, -08, -10, -12
Lab use avg RFs; all RSDs <15%. ICV/CV surrogates checked as %Rs. All OK.
No dilutions

Hahn Level III Metals Worksheet

SDG: A6D0013	Matrix: Sediment	Lab Sample IDs: A6D0013-02, -04, -06, -08, -10, -12
Method: 6020	Batch #s: 6040194	

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 (-02)**	**MS %R (-22)**	**Lab Dup RPD** *≤20%*	**PS %R**	**Ser. Dil. %D** ≤10%
	r	**ICV**	**CCV**	**CRI**	**ICB**	**CCB** ug/L	**10X CCB** *mg/kg*										
6D11020																	
None								#	#								
6D11028																	
Se	✓	✓	126	✓	✓	✓	NA	#	#								
Cr	✓	✓	✓	✓	✓	1.31	6.55	#	#								
6D12019																	
Fe	✓	✓	✓	✓	✓	✓	NA	#	#								
6040194																	
Fe	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	22*	165*	47	NA	NA
V	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	111	✓	44	NA	NA
Mn	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	-187*	774*	53	NA	NA
Al	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	139	✓	✓	NA	NA
Zn	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	128	✓	✓	NA	NA
Ba	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	✓	126*	✓	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				Beginning CCV	Li-6 77.30	Sc-45 76.58	Ge-74 76.85
					Rh-103 75.65	Tb-159 77.57	
				Ending CCB	Li-6 79.49	Sc-45 79.87	Ge-74 78.89
					Rh-103 79.37	Tb-159 79.90	

Comments: HTs OK. CRI ≤ CRDL, not at 2X CRDL. See A6C1076-02MS, -02Dup, and -22MS.
6D11020: MB, LCS
6D11028: -02, -04, -06, -08, -10, -12
6D12019: -02RE, -04RE
*Parent sample conc >4X spike amount.
#All samples diluted 5X; samples -02 and -04 also diluted 50X; nominals < ICS spike amounts for all samples.
Na sample and dup results <5X RL, and abs diff <RL; not qualified for RPD >20%.
**SC-45: Na, Mg, Al, K, CA; no associated results were reported from this analytical sequence.

Hahn Level III Cyanide Worksheet

SDG: A6D0013	Matrix: Sediment	Lab Sample IDs: A6D0013-02, -04, -06, -08, -10, -12
Method/Batch #s: 9013M/9014 (total CN)/6040256		

(80-120%) (75-125%) (≤20%)

Analyte (outliers)	*(85-115%)* **Calibration**							**MB**	**5X MB**	**LCS %R**	**MS %R**	**MSD %R**	**MS/D RPD**	**DUP RPD**			
	r ≥0.995	**ICV**	**CCV**	**Dist. ICV**	**ICB**	**CCB** (ug/L)	**5X CB**										
None																	

Comments: HTs OK. MB, LCS, -12MS, -12DUP

ICAL results not on ICAL summary; results found in raw data.

No dilutions

Revised 9/2010

Hahn Level III General Chemistry Worksheet

SDG: A6D0013	Matrix: Sediment	Lab Sample IDs: A6D0013-02, -04, -06, -08, -10, -12
Method/Batch #s: 9056 (sulfate)/6040311; SM 5310B Mod (TOC)/6040241; SM4500-NH3 Mod (NH3)/6040054		

(80-120%) (75-125%) ≤20%

Analyte (outliers)	*(90-110%)* **Calibration**						**Method Blank**	**5X MB**	**LCS %R**	**MS %R**	**Lab Dup RPD**						
	r ≥0.995	**ICV**	**CCV**	**ICB**	**CCB**	**5X CB**											
NH3	See below	✓	✓	✓	✓	NA	✓	NA	✓	54	44						

Comments: HTs OK.

SO4: MB, LCS, -08dup, -08MS; all ICAL standards within 10% of true value (see raw data).

TOC: MB, LCS, -08DUP; No ICAL summary for TOC; results found in raw data. ICV not reported and no sequence provided for ICAL. All ICAL standards within 10% of true value except 20 ugC (see raw data), all sample results >50ugC standard (see raw data), not qualified.

NH3: MB, LCS, -12DUP, -12MS; ICAL summary incomplete; Correlation Coef blacked out in raw data; ICAL calculated and all ICAL standards within 10% of true value except lowest 0.02 ppm - positive bias (see recalcs); -02 nominal <0.05; qualified J; all others OK . -8 10X.