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Date: September 19, 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 A6D0056

SUMMARY

Level III data validation was performed on the data for 10 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
5237-160401-DC-EMB038G	A6D0056-01	Soil	VOCs, GRO
5237-160401-DC-EMB038	A6D0056-02	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160401-DC-EMB039G	A6D0056-03	Soil	VOCs, GRO
5237-160401-DC-EMB039	A6D0056-04	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160401-DC-EMB046G	A6D0056-05	Soil	VOCs, GRO
5237-160401-DC-EMB046	A6D0056-06	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160401-NDP-EMB002G	A6D0056-07	Soil	VOCs, GRO
5237-160401-NDP-EMB002	A6D0056-08	Soil	SVOCs, PAHs, DRO, Metals, Total CN, Sulfate, TOC, and Ammonia
5237-160401-NDP-EMB003G	A6D0056-09	Soil	VOCs, GRO
5237-160401-NDP-EMB003	A6D0056-10	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
5237-160401-DC-EMB038	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	J	Insufficient calibration

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
		C3-Fluorenes C3-Naphthalenes C3-Phenanthrenes/ Anthracenes		
	NWTPH-Dx	Oil	J	Poor replicate precision
	9013M/ 9014	Total Cyanide	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision
5237-160401-DC-EMB039	8270D Scan	C1-Chrysenes/ Benz(a)anthracenes C1-Fluoranthenes/Pyrenes C1-Phenanthrenes/ Anthracenes	J	Insufficient calibration
	NWTPH-Dx	Oil	J	Poor replicate precision
	9013M/ 9014	Total Cyanide	UJ	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision
5237-160401-DC-EMB046G	8260B	Acetone	UJ	High calibration verification negative bias
5237-160401-DC-EMB046	8270D Scan	All target analytes *except*: C4-Phenanthrenes/ Anthracenes	J	Insufficient calibration
	NWTPH-Dx	Oil	J	Poor replicate precision
	9013M/ 9014	Total Cyanide	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision
5237-160401-NDP-EMB002G	8260B	Acetone	UJ	High calibration verification negative bias

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
5237-160401-NDP-EMB002	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-Phenanthrenes/ Anthracenes C4-Chrysenes/ Benz(a)anthracenes	J	Insufficient calibration
	NWTPH-Dx	Oil	J	Poor replicate precision
	9013M/ 9014	Total Cyanide	J	Poor replicate precision
	SM4500-NH3 Mod	Ammonia as N	J	Low matrix spike recovery and poor replicate precision
5237-160401-NDP-EMB003	8270D	Benzo(a)anthracene Benzo(a)pyrene Benzo(b)fluoranthene Benzo(g,h,i)perylene Chrysene Fluoranthene Indeno(1,2,3-cd)pyrene Pyrene	J	High matrix spike recovery
		4-Nitroaniline	UJ	Low matrix spike recovery
		Aniline 3-Nitroaniline 3,3'-Dichlorobenzidine	R	Very low matrix spike recovery

Sample ID	Method	Analyte	Qualifier	Reason for Qualification
	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	J	Insufficient calibration
	NWTPH-Dx	Oil	J	Poor replicate precision
	9013M/ 9014	Total Cyanide	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 exceptionS.

The sample ID on 1/2 of the methanol preserved VOC vials was EMB**046**, while the label on the 4oz jar had the sample ID EMB0**02G**.

The samples identified as 5237-160401-**NDP**-EMB002G, 5237-160401-**NDP**-EMB002, 5237-160401-**NDP**-EMB003G, and 5237-160401-**NDP**-EMB003 on the COC were identified as 5237-160401-**DC**-EMB002G, 5237-160401-**DC**-EMB002, 5237-160401-**DC**-EMB003G, and 5237-160401-**DC**-EMB003 on the sample containers. The samples were logged in using the sample IDs from the COC.

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

The ICV and/or continuing calibration verification (CCV) %Ds were >25% with positive bias for chloroethane; trichlorofluoromethane; bromoform; bromomethane; 1,2-dibromo-3-chloropropane; dibromochloromethane; hexachlorobutadiene; 1,1,2,2-tetrachloroethane; and viny chloride. 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 ICAL and/or ICV/CCV RRFs were <0.05 but ≥0.01 for pentachlorophenol; 2,4,6-tribromophenol; and benzoic acid. 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 6D05025 was >25% with negative bias for hexachlorocyclopentadiene. The associated samples were QC samples and, therefore, no sample data were qualified.

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 ±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 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

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

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 and trichlorofluoromethane. 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 recoveries were > the laboratory upper acceptance limits for chloroethane, trichlorofluoromethane, and viny chloride. 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 upper acceptance limits for benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(g,h,i)perylene, chrysene, fluoranthene, indeno(1,2,3-cd)pyrene, and pyrene. The results for parent sample 5237-160401-NDP-EMB003 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 lower acceptance limit but ≥10% for 4-nitroaniline. The 4-nitroaniline result for parent sample 5237-160401-NDP-EMB003 was a non-detect and, therefore, was **qualified UJ**. The remaining associated sample results were not qualified based on professional judgment.

The MS recoveries were <10% for aniline, 3-nitroaniline, and 3,3'-dichlorobenzidine. The aniline, 3-nitroaniline, and 3,3'-dichlorobenzidine results for parent sample 5237-160401-NDP-EMB003 were non-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, therefore, were **qualified R**. The remaining associated sample results were not qualified based on professional judgment.

Method 6020

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.

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 NWTPH-Dx

The laboratory duplicate relative percent difference (RPD) was > the laboratory acceptance limit for oil. The associated sample results for were detects and, therefore, were **qualified J**.

Method 9013M/9014

The laboratory duplicate RPD was > the laboratory acceptance limit for total cyanide. The total cyanide result for sample 5237-260401-DC-EMB039 was a non-detect and, therefore, was **qualified UJ**. The remaining 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

The samples 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.

Method 9013M/9014

Sample 5237-160401-DC-EMB038 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#: A6D0056	Laboratory: Apex	Validator: Jeanne Peterson	Validation Date: 06/14/2016
Site: Siltronic - Doane Creek	COC#: NA		Validation Level: ☐ II ☒ III
Matrix: Soil	# of Samples: 10	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 4/1.

Cooler temps OK.

Hahn Level III GCMS Worksheet

SDG: A6D0056	Method: 8260B	Matrix: Soil	Lab Sample IDs: A6D0056-01, -03, -05, -07, -09
Batch #s: 6040059			

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	94.5	✓	NA	179	198	NA	NA	NA			
Trichlorofluoromethane	0.0299**	✓	193	73.0	✓	NA	159	199	NA	NA	NA			
Acetone	✓	✓	-27.8	✓	✓	NA	✓	✓	NA	NA	NA			
Bromoform	✓	✓	✓	46.1	✓	NA	✓	✓	NA	NA	NA			
Bromomethane	✓	✓	✓	43.6	✓	NA	✓	✓	NA	NA	NA			
1,2-Dibromo-3-chloropropane	✓	✓	✓	34.2	✓	NA	✓	✓	NA	NA	NA			
Dibromochloromethane	✓	✓	✓	30.4	✓	NA	✓	✓	NA	NA	NA			
Hexachlorobutadiene	✓	✓	✓	33.2	✓	NA	✓	✓	NA	NA	NA			
1,1,2,2-Tettrachloroethane	✓	✓	✓	43.9	✓	NA	✓	✓	NA	NA	NA			
Vinyl chloride	✓	✓	✓	28.2	✓	NA	✓	138	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
MB, LCS, -09MS; all samples diluted 50X; sample results ND for outliers.
**Alternate curve used
Tune summaries missing entries for m/z 173; found in raw data.

Hahn Level III GCMS Worksheet

SDG: A6D0056	Method: 8270D	Matrix: Soil	Lab Sample IDs: A6D0056-02, -04, -06, -08, -10
Batch #s: 6040070			

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/r^2 ≤30%	SSV %D ±25%	CCV %D ±25%											
ICAL A6C3104															
PCP (Level 3 and 4 only)	0.0315*	✓	✓												
2,4,6-TBP (surr) (L3 & 4)	0.0367*	✓	✓												
Benzoic acid (L5 & 6)	0.0227*	✓	✓												
Sequence 6D05025															
Hexachlorocyclopentadiene	✓	✓	-25.1#												
Sequence 6D06014															
None															
Batch 6040070															
3,3'-Dichlorobenzidine	✓	✓	✓	✓	✓	NA	139	**	NA	NA	✓				
Bezno(g,h,i)perylene	✓	✓	✓	✓	✓	NA	✓	**	NA	NA	28				
Dibenz(a,h)anthracene	✓	✓	✓	✓	✓	NA	✓	**	NA	NA	33				
carbazole	✓	✓	✓	✓	✓	NA	✓	**	NA	NA	27				

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.
6D05025: MB, LCS
6D06014: -02, -02DUP, -04, -06, -08, -10, -10MS
**Refer to attached MS sheet for outliers.
IS summary missing for 6D05025; IS results on Forms Is; raw data checked.
*Alternate curve analyzed; OK
Samples diluted 4X

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY

EPA 8270D

5237-160401-NDP-EMB003

Laboratory: Apex Laboratories
SDG: A6D0056
Client: Hahn and Associates
Project: Siltronic RI-Doane Creek
Matrix: Soil
Batch: 6040070
Laboratory ID: 6040070-MS1
Preparation: EPA 3546
Initial/Final: 15.63 g / 2 mL
Source Sample Name: 5237-160401-NDP-EMB003

COMPOUND	SPIKE ADDED (ug/kg dry)	SAMPLE CONCENTRATION (ug/kg dry)	MS CONCENTRATION (ug/kg dry)	MS % REC. (*=Out)	QC LIMITS REC.
Acenaphthene	673	42.6	717	100	40 - 122
Acenaphthylene	673	18.7	665	96	32 - 132
Anthracene	673	62.6	784	107	47 - 123
Benz(a)anthracene	673	331	1240	135 *	49 - 126
Benzo(a)pyrene	673	459	1490	153 *	45 - 129
Benzo(b)fluoranthene	673	613	1800	177 *	45 - 132
Benzo(k)fluoranthene	673	211	1080	129	47 - 132
Benzo(g,h,i)perylene	673	383	1400	151 *	43 - 134
Chrysene	673	400	1370	144 *	50 - 124
Dibenz(a,h)anthracene	673	73.4	803	108	45 - 134
Fluoranthene	673	530	1570	155 *	50 - 127
Fluorene	673	30.9	718	102	43 - 125
Indeno(1,2,3-cd)pyrene	673	347	1270	137 *	45 - 133
1-Methylnaphthalene	673	ND	655	97	40 - 120
2-Methylnaphthalene	673	13.6	671	98	38 - 122
Naphthalene	673	36.6	649	91	35 - 123
Phenanthrene	673	271	1080	120	50 - 121
Pyrene	673	542	1590	155 *	47 - 127
Carbazole	673	46.2	734	102	50 - 122
Dibenzofuran	673	17.1	692	100	44 - 120
4-Chloro-3-methylphenol	673	ND	705	105	45 - 122
2-Chlorophenol	673	ND	628	93	34 - 121
2,4-Dichlorophenol	673	ND	721	107	40 - 122
2,4-Dimethylphenol	673	ND	723	107	30 - 127
2,4-Dinitrophenol	673	ND	714	106	5 - 137
4,6-Dinitro-2-methylphenol	673	ND	698	104	29 - 132
2-Methylphenol	673	ND	652	97	32 - 122
3+4-Methylphenol(s)	673	ND	693	103	34 - 120
2-Nitrophenol	673	ND	664	99	36 - 123
4-Nitrophenol	673	ND	675	100	30 - 132
Pentachlorophenol (PCP)	673	ND	859	128	25 - 133
Phenol	673	ND	563	84	34 - 120
2,3,4,6-Tetrachlorophenol	673	ND	808	120	44 - 125
2,3,5,6-Tetrachlorophenol	673	ND	775	115	40 - 120
2,4,5-Trichlorophenol	673	ND	706	105	41 - 124
2,4,6-Trichlorophenol	673	ND	745	111	39 - 126
Bis(2-ethylhexyl)phthalate	673	ND	754	112	51 - 133

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY

EPA 8270D

5237-160401-NDP-EMB003

Laboratory: Apex Laboratories
SDG: A6D0056
Client: Hahn and Associates
Project: Siltronic RI-Doane Creek
Matrix: Soil
Batch: 6040070
Laboratory ID: 6040070-MS1
Preparation: EPA 3546
Initial/Final: 15.63 g / 2 mL
Source Sample Name: 5237-160401-NDP-EMB003

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	673	ND	687	102	48 - 132
Diethylphthalate	673	ND	705	105	50 - 124
Dimethylphthalate	673	ND	665	99	48 - 124
Di-n-butylphthalate	673	ND	743	110	51 - 128
Di-n-octyl phthalate	673	ND	727	108	44 - 140
N-Nitrosodimethylamine	673	ND	438	65	23 - 120
N-Nitroso-di-n-propylamine	673	ND	627	93	36 - 120
N-Nitrosodiphenylamine	673	ND	611	91	38 - 127
Bis(2-Chloroethoxy) methane	673	ND	609	90	36 - 121
Bis(2-Chloroethyl) ether	673	ND	515	77	31 - 120
Bis(2-Chloroisopropyl) ether	673	ND	530	79	33 - 131
Hexachlorobenzene	673	ND	657	98	44 - 122
Hexachlorobutadiene	673	ND	599	89	32 - 123
Hexachlorocyclopentadiene	673	ND	389	58	5 - 140
Hexachloroethane	673	ND	454	67	28 - 120
2-Chloronaphthalene	673	ND	627	93	41 - 120
1,2-Dichlorobenzene	673	ND	500	74	33 - 120
1,3-Dichlorobenzene	673	ND	482	72	30 - 120
1,4-Dichlorobenzene	673	ND	477	71	31 - 120
1,2,4-Trichlorobenzene	673	ND	580	86	34 - 120
4-Bromophenyl phenyl ether	673	ND	679	101	46 - 124
4-Chlorophenyl phenyl ether	673	ND	642	95	45 - 121
Aniline	673	ND	21.0 R	3 *	7 - 120
4-Chloroaniline	673	ND	246	37	16 - 120
2-Nitroaniline	673	ND	566	84	44 - 127
3-Nitroaniline	673	ND	48.1 R	7 *	33 - 120
4-Nitroaniline	673	ND	125 J	19 *	35 - 120
Nitrobenzene	673	ND	570	85	34 - 122
2,4-Dinitrotoluene	673	ND	658	98	48 - 126
2,6-Dinitrotoluene	673	ND	671	100	46 - 124
Benzoic acid	1350	ND	2300	171 *	5 - 140
Benzyl alcohol	673	46.0	695	96	29 - 122
Isophorone	673	ND	669	99	30 - 122
Azobenzene (1,2-DPH)	673	ND	689	102	39 - 125
Bis(2-Ethylhexyl) adipate	673	ND	733	109	60 - 121
3,3'-Dichlorobenzidine	1350	ND	ND R	*	22 - 121
1,2-Dinitrobenzene	673	ND	642	95	44 - 120

MATRIX SPIKE / MATRIX SPIKE DUPLICATE RECOVERY

EPA 8270D

5237-160401-NDP-EMB003

Laboratory:	Apex Laboratories	SDG:	A6D0056
Client:	Hahn and Associates	Project:	Siltronic RI-Doane Creek
Matrix:	Soil		
Batch:	6040070	Laboratory ID:	6040070-MS1
Preparation:	EPA 3546	Initial/Final:	15.63 g / 2 mL

Source Sample Name: 5237-160401-NDP-EMB003

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	673	ND	663	98	42 - 127
1,4-Dinitrobenzene	673	ND	607	90	37 - 132
Pyridine	673	ND	372	55	5 - 120

Hahn Level III GCMS Worksheet

SDG: A6D0056	Method: 8270D Scan	Matrix: Soil	Lab Sample IDs: A6D0056-02, -04, -06, -08, -10
Batch #s: 6040070			

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%											
Pyrene NTA	**	**	**	**	✓	NA	✓	185	NA	NA	✓				
Chrysene NTA	**	**	**	**	✓	NA	✓	157	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.

*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.

6D05025: MB, LCS

6D06014: -02, -02DUP, -04, -06, -08, -10, -10MS

IS summary for QC not included in data package. Raw data checked.

Samples diluted 4X

Hahn Level III NWTPH-GX Worksheet

SDG: A6D0056	Matrix: Soil	Lab Sample IDs: A6D0056-01, -03, -05, -07, -09
Method/Batch #s: 6040059		

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, -01, -03, -05, -07, -09
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: A6D0056	Matrix: Soil	Lab Sample IDs: A6D0056-02, -04, -06, -08, -10
Method/Batch #s: 6040068		

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								
Oil	✓	✓	NA	✓	NA	✓	NA	NA	NA	62	

Surrogate Outliers *(50-150%)*

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

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

Hahn Level III Metals Worksheet

SDG: A6D0056	Matrix: Soil	Lab Sample IDs: A6D0056-02, -04, -06, -08, -10
Method: 6020	Batch #s: 6040293	

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*										
Fe	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	59*	NA	✓	NA	NA
Mn	✓	✓	✓	✓	✓	✓	NA	#	#	✓	NA	✓	168*	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**
-02	Sc-45 131**	--	--	None			
-04	SC-45 129**	--	--				
-06	Sc-45 128**	--	--				
-08	Sc-45 125.49**	--	--				
-10	Sc-45 128**	--	--				

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 10X; 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: A6D0056	Matrix: Soil	Lab Sample IDs: A6D0056-02, -04, -06, -08, -10
Method/Batch #s: 9013M/9014 (total CN)/6040257		

(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**										
Total CN	✓	✓	✓	NA	✓	NA	NA	✓	NA	✓	28	NA	NA	25			

Comments: HTs OK. MB, LCS, -02MS, -02DUP

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

Sample -02 diluted 10X

Revised 9/2010

Hahn Level III General Chemistry Worksheet

SDG: A6D0056	Matrix: Soil	Lab Sample IDs: A6D0056-02, -04, -06, -08, -10
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, -02dup, -02MS; all ICAL standards within 10% of true value (see raw data).

TOC: MB, LCS, -10DUP; 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, A6D0013-12DUP, A6D0013-12MS; 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); all sample results >0.05; not qualified.. .

No dilutions