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Date: September 18, 2019

To: Rob Ede

Hahn and Associates Inc.

**From**: Jeanne Peterson

Project Manager, AQA

**Subject**: Data Validation

Gasco Mult 802 Decommissioning

Apex Laboratories, LLC Work Order A9E0582

#### **SUMMARY**

Level II (i.e., EPA Stage 2A) data validation was performed on the data for one solid sample prepared and analyzed using approved procedures for methods SW846 8260C (VOCs), SW846 8270D (SVOCs), NWTPH-Gx (gasoline range organics [GRO]), NWTPH-Dx (diesel and oil), SW846 6020A (metals by ICPMS), and D7511-12 (total cyanide). 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, collectively), and the applicable methods.

In general, the data are valid as reported. No 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 sample included in this validation is listed below.

Sample ID	APEX Sample ID	Analysis	Matrix
2708-190515-005	A9E0582-01	VOCs, SVOCs, GRO, DRO, Total Metals, Total CN	Solid

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

Sample ID	Method	Analyte	Qualifier	Qualifier Code	Reason for <b>Qualification</b>
	8260C	Methylene chloride	UJ	10	Low laboratory control sample recovery
	8270D	Acenaphthene 2-Methylnaphthalene Naphthalene	J	10	High laboratory control sample recovery
2708-190515-005	C020 A	Vanadium	J	8,9	Low matrix spike recovery and poor duplicate precision
	6020A	Calcium Aluminum Zinc	J	9	Poor duplicate precision

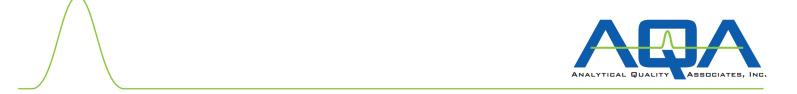
## **DISCUSSION**

## Sample Shipping/Receiving

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

The sample receipt section of the COCs was not completed; the information was documented on the Cooler Receipt Form.

The date and time the sample was relinquished to the laboratory was documented in the "Received by" block of the COC.



## **Holding Times and Preservation**

The sample was properly preserved and analyzed within the prescribed holding times.

## **Blanks**

#### Methods 8260C, NWTPH-Gx, NWTPH-Dx, 6020A, and D7511-12

No target analytes were detected in the method blank. Field blanks were not collected with the sample in this work order.

#### Method 8270D

Acenaphthene, fluorene, 1-methylnaphthalene, 2-methylnaphthalene, naphthalene, phenanthrene, dibenzofuran, and phenol were detected in the method blank. The associated sample results were either non-detects or detects >10X the method blank value and, therefore, were not qualified.

## **Surrogates**

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

#### Method 8270D

The surrogates were diluted out of samples 2708-190515-005 (5000X) and 2708-190515-005 DUP (10000X). No sample results were qualified.

#### Method NWTPH-Dx

The surrogates were diluted out of samples 2708-190515-005 (100X) and 2708-190515-005 DUP (100X). No sample results were qualified.

## Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD)

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

### Method 8260C

The LCS recoveries were > the upper acceptance limit for bromoform; carbon tetrachloride; dichlorofluoromethane; and 2,2-dichloropropane. The associated sample results were non-detects and not affected by the high bias and, therefore, were not qualified based on professional judgment.



The LCS recovery was < the lower acceptance limit but  $\geq 30\%$  for methylene chloride. The associated sample result was a non-detect and, therefore, was **qualified UJ**.

#### Method 8270D

The LCS recoveries were > the upper acceptance limit for acenaphthene; 2-methylnaphthalene; naphthalene; and 3,3'-dichlorobenzidine. The 3,3'-dichlorobenzidine result for sample 2708-190515-005 was a non-detect and not affected by the high bias and, therefore, was not qualified based on professional judgment. The remaining associated sample results were detects and, therefore, were qualified J.

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

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

#### Method 8260C

The MS analysis was performed on a non-project sample.

#### Methods 8270D, NWTPH-Gx, and NWTPH-Dx

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

#### Method 6020A

The MS recovery was < the lower acceptance limit but  $\ge 10\%$  for vanadium. The associated sample result was a detect and, therefore, was **qualified J**.

The MS recoveries were outside of the acceptance limits for iron and manganese. The parent sample concentration was >4X the spike amount; therefore, no sample results were qualified based on professional judgment.

## Method D7511-12

The MS/MSD recoveries were outside of the acceptance limits for total cyanide. The parent sample concentration was >4X the spike amount; therefore, no sample results were qualified based on professional judgment. It should be noted that the MS/MSD analyses were performed on a project sample from another SDG.





## **Laboratory Duplicate**

The laboratory duplicate analyses (LCS/LCSD, MS/MSD, and/or sample/duplicate) were within laboratory QC acceptance criteria with the following exceptions.

#### Method 6020A

The duplicate sample relative percent differences (RPDs) and/or absolute differences were >the acceptance limit for calcium, aluminum, vanadium, and zinc. The associated sample results were detects and therefore, were **qualified J**.

#### Method 8260C

It should be noted that the laboratory duplicate analysis was performed on a non-project sample.

#### Method NWTPH-Gx

It should be noted that the laboratory duplicate analyses were performed on non-project samples.

#### Methods NWTPH-Dx and D7511-12

It should be noted that the laboratory duplicate analysis was performed on a project sample from another SDG.

#### **Field Duplicate**

A field duplicate was not collected with the sample in this data package.

#### **Reporting Limits**

All reporting limits (RLs) were properly reported.

Sample 2708-190515-005 was diluted 20000X for VOCs and GRO, 5000X for SVOCs, 100X for DRO, and 10X for total metals and total cyanide. Reporting limits were adjusted accordingly.

## Other QC

#### Method 8270D

The laboratory noted that peak separation of structural isomers was insufficient for accurate quantification of benzo(b)fluoranthene and benzo(k)fluoranthene for sample 2708-190515-005. Because this could not be verified with a Level II data package, the sample results were not qualified by the validator; however, the end user of the results should be aware that the results were considered to be estimated.





## Method NWTPH-Dx

The laboratory noted that no fuel pattern was detected for sample 2708-190515-005. The diesel result represents carbon range C12 to C24, and the oil result represents >C24 to C40. Because this could not be verified with a Level II data package, the sample results were not qualified by the validator; however, the end user of the results should be aware that the results were considered to be estimated.

No other specific issues that affect data quality were identified.

# Hahn Data Validation Summary Worksheet

SDG#: A9E0582	Laboratory:	Apex		Valida	tor: Jeanne Pete	rson	Validation D	Date: 08/27/2019	)
Site: Mult 802 Decommissioning	COC#: 1		·				Validation L	evel: 🛛 II	
Matrix: Solid	# of Sample	s: 1		Trackii	ng docs present:	See sample receip	ot and log-in do	cumentation	
COCs present: Yes	COCs signe	d: Yes		COCs	dated: Yes		Sample Conta	ainer Integrity:	OK
Analyses:  ⊠ VOCs ⊠ SVOCs □ PAHs ⊠  □ Other: VPH/EPH	GRO 🛭 DRO	Pests	] РСВ	s 🛛	Metals 🗌 G	en Chem 🛭 C	yanide		
		Requested		ses Not	t Reported				
•	Sample ID	Analysis	<b>S</b>			Con	nments		
None									
		Hold Time	/Prese	rvatio					T
Client Sample ID Lab S	ample ID	Analysis	Pro	es.	Collection Date	Preparation Date	Analysis Date	Analysis <2X HT	Analysis ≥2X HT
None									
Comments: Samples collected 05/15/2019; Temp and containers not completed on COC Date and Time samples relinquished docum		*	`orm.						

# Hahn Level III GCMS Worksheet

SDG: A9E0582	Method: 826	50C	Matrix:	Solid		Lab Samp	ole ID:	A9E058	82-01						
Seq/Batch #s:/905109	)2		,		1										
Tuning: Pass Fa	il	TIC	Cs Required?	Yes	No No	1			(lab	limits)		(lab lim	its)		
			Calil	oration								2.501			
Analyte (outliers)		<b>RF</b> ≥0.05	RSD/r <sup>2</sup> ≤30% ≥0.990	ICV <sup>1</sup> %D ±25%	CCV %D ±25%	) Blan	ba   M	(10X) lethod Blank	LCS %R	MS %R	MSD %R	MS/ MSD RPD	LAB DUP RPD	ТВ	
Bromoform						✓		NA	129	✓	NA	NA	✓	NA	
CCl4						✓		NA	123	✓	NA	NA	✓	NA	
Dichlorofluoromethane						✓		NA	122	✓	NA	NA	✓	NA	
2,2-Dichloropropane						✓		NA	125	✓	NA	NA	✓	NA	
MeCl2						✓		NA	71	✓	NA	NA	✓	NA	
													<u> </u>		
				Surrogat	te Reco	very Outli	ers (met	hod/lab	limits)						
Sample ID	DBFM		1,4-DCB	Tol-d8	4	4-BFB	S	ample I	D	DBFM	1	1,4-DCB	Т	ol-d8	4-BFB
None															
				IS (	Outlier	<b>rs</b> (-50% to	+100%	of CCV,	)						
Sample ID	Area	RT	Area	RT	A	rea	RT	A	Area	RT	A	rea	RT	Area	RT
NA															

Comments: HTs OK.

MB, LCS, -01, unknown Dup, unknown MS

Sample -01 diluted 20000X

# **Hahn Level III GCMS Worksheet**

SDG: A9E0582	Method: 8	270D		Matrix: S	olid	L	ab Sampl	e IDs:	A9E0	582-01							
Seq/Batch #s:/905106	5					•											
Tuning: Pass Fa	il	Ί	TCs Requ	uired?	Yes 🖂	No			(lab li	mits)		а	ab limits)				
			Cali	bration			5X				LCS/				Lab		
Analyte (outliers)		<b>RF</b> ≥0.05	RSD/r <sup>2</sup> ≤30%	ICV %D ±25%	CCV %D ±25%	Method Blank	(10X) Method Blank		CS 6R	LCSD %R	D RPD	MS %R	MSD %R	MS/D RPD	Dup RPD		
Acenaphthene						148	(148)	1	28	NA	NA	NA	NA	NA	✓		
Fluorene						26.2	(262)		✓	NA	NA	NA	NA	NA	✓		
1-Methylnaphthalene						108	(1080)		✓	NA	NA	NA	NA	NA	✓		
2-Methylnaphthalene						223	(2230)	1	39	NA	NA	NA	NA	NA	✓		
Naphthalene						1070	(10700)	) 2	.73	NA	NA	NA	NA	NA	✓		
Phenanthrene						27.7	(277)		✓	NA	NA	NA	NA	NA	✓		
Dibenzofuran						46.6	(466)		✓	NA	NA	NA	NA	NA	✓		
Phenol						23.4	(234)		✓	NA	NA	NA	NA	NA	✓		
3,3'-Dichlorobenzidine						✓	NA	2	:77	NA	NA	NA	NA	NA	✓		
																	_
					Surroga	te Reco	very Outli	iers (la	b limit:	s)							
Sample ID	Nitrobenz	zene-d	5	2-Fluoro	biphenyl		Phenol-d6		<b>p-</b>	Terphenyl-	d14	2-Fl	uorophen	ol	2,4,6-Tr	ibromoph	nenol
-01 5000X	D	O		D	0		DO			DO			DO			DO	
Dup 10000X	D	O		D	O		DO			DO			DO			DO	
					IS Out	liers <i>(-3</i>	50% to +1	00% of	CCV)								
Sample ID	Acen-d1	0	RT	Chry-d12	RT	Pe	r-d12	RT	1	Dibenz-d1	14	RT	Area	RT	1	Area	RT
NA																	

Comments: HTs OK. DO = Diluted out

MB, LCS, -01, A9E0582-05 Dup

Sample -01 diluted 5000X for all target compounds

 $Sample - 01: The \ benzo(b) fluoranthene \ and \ benzo(k) fluoranthene \ results \ are \ estimated; \ peak \ separation \ for \ structural \ isomers \ is \ insufficient \ for \ accurate \ quantification.$ 

# Hahn Level III NWTPH-GX Worksheet

SDG: A9E0582	M	latrix:	Solid		Lab S	Sample ID	s: A9E05	582-0	1								
Seq./Batch #s:/90	51092																
Tuning: Pass	☐ Fa	il									(lab limits	s) (lab lim	its)				
					Calib	ration										[ a.b.	
Analyte (outliers)			≥0	r <sup>2</sup> .990 20%	۱ ،	V/CCV <b>%D</b> -20%	RT Windov		Method Blank	5X Blank	LCS %R	MS %R	MSD %R		S/D I	Lab Dup1 RPD	
None												NA	NA	N	NΑ		
							Surroga	te Ou	tliers (50	-150%)							
Sample ID		Surre	ogate	%R		San	nple ID		Surroga	te %R	R	Sample II	)	Sı	ırrogate		%R
None																	
										% of CCV)		_				1	
Area	RT		Area		RT	Aı	rea	R	Γ	Area	RT	Area	]	RT	Area	1	RT
NA																	

Comments: HT OK for unpreserved samples (pH of samples unknown).

MB, LCS, -01, unknown Dup1, unknown Dup2

Sample -01 diluted 20000X

## Hahn Level III NWTPH-DX Worksheet

SDG: A9E0582	Matrix: Solid	Lab Sample IDs: A9E0582-01
Seq./Batch #s:/90510	067	

(lab limits) (lab limits)

		Calibration				I CC/					
Analyte (outliers)	r <sup>2</sup> ≥0.990 ±20%	ICV/CCV %D ±15%	RT Windows	Method Blank	5X Blank	LCS/ LCSD %R	MS %R	MSD %R	MS/D RPD	LCSD %R	LAB RPD
None							NA	NA	NA	NA	

# Surrogate Outliers (50-150%)

Sample ID	Surrogate	%R	Sample ID	Surrogate	%R	Sample ID	Surrogate	%R
-01 (100X)	o-Terphenyl	DO						
Dup (100X)	o-Terphenyl	DO						

Comments: HTs OK. DO = Diluted out

MB, LCS, -01, A9E0508-05 Dup

Sample -01 diluted 100X

Sample -01: F17 No fuel pattern was detected. The diesel result represents carbon range C12 to C24, and the oil result represents >C24 to C40.

## Hahn Level III Metals Worksheet

SDG: A9E058	2				Matrix:	Solid		Lat	Sample	IDs: A9	E0582-0	01						
Method: 6020	A		Seq/E	atch #:	/9051	056		·										
ICPMS Mass Cal	: 🔲 P	ass [	Fail 🛛	NA IC	PMS %	RSD: 🗌	Pass [	]Fail 🛚	NA		(80-	-120%)		(75-125%	<b>)</b>			
		(:	90-110%)	Calil	bration				ICS		10X		Dup			MS/		Ser.
Analyte (outliers)	r	ICV	CCV <sup>1</sup>	CRI	ICB	CCB ug/L	5X CCB	ICS A <idl<sup>1</idl<sup>	<b>AB</b> % <b>R</b> <sup>1</sup> ±20%	MB ug/L	MB ug/L	LCS %R	RPD ≤40%	MS %R	MSD %R	MSD RPD ≤40%	PS %R	Dil. %D ≤10%
Al										✓	NA	✓	43	✓	NA	NA	NA	NA
As										✓	NA	✓	56#	✓	NA	NA	NA	NA
Ca										✓	NA	✓	44/ 240	✓	NA	NA	NA	NA
Mg										✓	NA	✓	54#	✓	NA	NA	NA	NA
V										✓	NA	✓	43	60	NA	NA	NA	NA
Zn										✓	NA	✓	33	✓	NA	NA	NA	NA
Fe										✓	NA	✓	✓	214*	NA	NA	NA	NA
Mn										✓	NA	✓	✓	231*	NA	NA	NA	NA
I	S Outli	iers	(Samples	60-125%	s; CCV/C	CB 80-120	)%)				IS Outli	ers	(Samples 6	0-125%; C	CCV/CCB	80-120%)		
Sample ID	Li6 %	6R	Sc45 %R	Ge74	1 %R	Rh103 %	6R T	b159 %R	CCV/C	CB ID	Li6 %	6R Sc	15 %R	Ge74 %	R R	h103 %R	Tb1	59 %R
NA									NA									

Comments: HTs OK.

MB, LCS, -01, A9E0582-01 Dup, A9E0582-01 MS

#Parent and dup sample conc <5\*RL and abs diff <RL; OK

Sample -01 diluted 10X

<sup>\*</sup>Parent sample conc >4X spike amount

*(≤47%)* 

# Hahn Level III Cyanide Worksheet

SDGs: A9E0582 Matrix: Solid Lab Sample IDs: A9E0582-01

Method/Seq/Batch #s: D7511-12 (Total CN)/--/9051027

r

≥0.995

	(85-115	%)	Calib	ration					LCS/	LCCD	MC	MCD	MS/	D	CNI
	ICV	CCV	Dist. ICV	ICB (ug/L)	CCB (ug/L)	5X CB (mg/L)	MB	5X MB	D %R	LCSD RPD	MS %R	MSD %R	MSD RPD	Dup RPD	CN- 1
							✓	NA	✓	NA	-95*	-266*	✓	NA	0.11
_															

(80-120%)

(≤20%)

(75-125%)

Comments: HTs OK.

Analyte

(outliers)

Total CN

Tot CN: MB, LCS1, LCS2, A9E0508-05, A9E0508-05 MS/MSD

\*Parent sample conc >4X spike amount Sample -01 diluted 10X for total CN

Revised 9/2010