Memorandum

To: Karen Vetrano

From: Paula DiMattei

CC: Elizabeth Denly

Date: April 8, 2015

Subject: Dioxin Data Validation Review: Montgomery County/Dickerson, MD: Laboratory Work Order: 7304

SUMMARY

Limited validation was performed on the data for three air samples and two field blank samples collected at the Montgomery County site in Dickerson, Maryland. The samples were collected from February 3, 2015 through March 4, 2015. Samples were submitted to Cape Fear Analytical (CFA) for analysis. The samples were analyzed for polychlorinated dibenzodioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) using EPA Method TO-9A. CFA reported the results under laboratory work order 7304.

The sample results were assessed using the "USEPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dioxin/Furan Data Review," September 2011, modified for the non-CLP methodology used.

In general, the data appear valid as reported and may be used for decision-making purposes. The following issues were noted which have a minor impact on the data usability:

- The results for 1,2,3,4,6,7,8-HpCDD, 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,7,8,9-HpCDF, total HpCDD and total HpCDF in samples B'ville-Dioxins-PS-4-Primary, B'ville-Dioxins-PS-1-Collocate, and Lucketts-Dioxins-PS-5-Primary were qualified as estimated due to elevated field spike recoveries; these results may be biased high.
- Select congeners and/or total homologs in samples were reported by the laboratory as Estimated Maximum Possible Concentrations (EMPCs) since all identification criteria were not met. These results were qualified as estimated (J) during data validation.
- Select results were reported which were below the lowest calibration standard; these results were qualified as estimated (J).

SAMPLES

Samples included in this review are listed below:

B'ville-Dioxins-PS-4-Primary	B'ville-Dioxins-PS-1-Collocate[1]	B'ville Blank Dioxins
Lucketts-Dioxins-PS-5-Primary	Lucketts Blank Dioxins	

[1]Field duplicate of B'ville-Dioxins-PS-4-Primary

REVIEW ELEMENTS

Sample data were reviewed for the following parameters:

- Agreement of analyses conducted with chain-of-custody requests
- Data completeness
- Holding times and sample preservation
- Initial and continuing calibrations
- Blanks
- Labeled compound recoveries
- Matrix spike/matrix spike duplicate (MS/MSD) results
- Laboratory control sample (LCS)/Laboratory control sample duplicate (LCSD) results
- Field spike results
- Field duplicate results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analyses Conducted with Chain-of-Custody Requests

Sample reports were checked to verify that the results corresponded to analytical requests as designated on the chain-of-custody (COC). No discrepancies were noted.

Data Completeness

The data packages were found to be complete as received from the laboratory.

Holding Times and Sample Preservation

All samples were prepared and analyzed within the method-specified holding times. The cooler temperatures were within the acceptance criteria upon sample receipt.

Initial and Continuing Calibrations

All chromatographic resolution criteria were met for the initial and continuing calibrations.

All initial and continuing calibration standards met the ion abundance ratio criteria specified in the method.

The percent relative standard deviations of all target congeners were within the acceptance criteria for the initial calibrations. The percent differences of all target congeners were within the acceptance criteria for the continuing calibrations.

Blanks

The following tables list the concentrations of target congeners detected in the laboratory method blanks and field blanks associated with the samples in this data set and the resulting validation actions. Media certification checks were not performed. However, all media used for the air samples, field blanks and

method blanks were from the same lot# (8964); therefore, data were not adversely impacted. It should be noted that the laboratory used a volume of 8700 m^3 for all method blanks and field blanks to allow a more direct comparison with sample results.

Compound	Blank Concentration (pg/m^3)	Blank ID: Associated Samples	Validation Action
1,2,3,7,8-PeCDD	0.000446 J	MB (Batch 28366): All samples	Qualification was not required for the associated samples since 1,2,3,7,8-PeCDD was present at a concentration greater than 5x the blank concentration.
1,2,3,7,8-PeCDF	0.000359 J		Qualification was not required for the associated samples since 1,2,3,7,8-PeCDF was present at a concentration greater than 5x the blank concentration.
2,3,4,7,8-PeCDF	0.00029 J		Qualification was not required for the associated samples since 2,3,4,7,8-PeCDF was present at a concentration greater than 5x the blank concentration.
OCDD	0.00449 J	B'ville Blank-Dioxins: B'ville-Dioxins-PS-4-Primary B'ville-Dioxins-PS-1-Collocate	Qualification was not required for the associated samples since OCDD was present at a concentration greater than 10x the blank concentration.
OCDD	0.00246 J	Lucketts-Blank-Dioxins: Lucketts—Dioxins-PS-5-Primary	Qualification was not required for the associated sample since OCDD was present at a concentration greater than 10x the blank concentration.
1,2,3,4,6,7,8-HpCDF	0.000818 J		Qualification was not required for the associated sample since 1,2,3,4,6,7,8-HpCDF was present at a concentration greater than 5x the blank concentration.

Qualification of the data was performed as follows:

- Sample results > the estimated detection limit (EDL) were qualified as nondetect (U) at the sample concentration if the result was < the blank concentration.
- Sample results ≥ the EDL and < 10x the blank concentration for OCDD and OCDF and <5x the blank concentration for all other congeners were qualified as estimated (J).
- Sample results ≥ the EDL and > 10x the blank concentration for OCDD and OCDF and >5x the blank concentration for all other congeners were accepted without qualification.

It should be noted that sample total homologue results were not assessed with regards to blank contamination. The total homologues are determined by summing the 2378-isomers and non-2378-isomers at each level of chlorination. As is typical, the individual peaks included in the total homologues are not identified by their specific compound name. Consequently, the total homologues in the method blanks could be comprised of different individual components than the associated samples and thus are not evaluated with respect to blank contamination.

Labeled Compound Recoveries

The labeled compound recoveries were within the acceptance criteria for all sample analyses.

MS/MSD Results

MS/MSD analyses were not associated with the air matrix.

LCS/LCSD Results

An LCS and LCSD were prepared with each extraction batch. All criteria were met.

Field Spike Results

All media were spiked with 13C-labeled compounds prior to sample collection. These compounds are identified as sampling standards (SS) by the laboratory. All percent recoveries (%Rs) were within the 50-120% recovery criteria with the following exceptions.

Sample ID	Surrogate Compound (SS)	%R	Validation Action
B'ville-Dioxins-PS-4-Primary	13C-1,2,3,4,7,8,9-HpCDF	127	Professional judgment was used to apply data validation actions based on the level of chlorination. Therefore, the positive results for 1,2,3,4,6,7,8-HpCDD, 1,2,3,4,6,7,8-HpCDF, 1,2,3,4,7,8,9-HpCDF, total HpCDD and total HpCDF in the affected samples were qualified as estimated (J). Nondetect results were not affected.
B'ville-Dioxins-PS-1-Collocate	13C-1,2,3,4,7,8,9-HpCDF	125	
Lucketts-Dioxins-PS-5-Primary	13C-1,2,3,4,7,8,9-HpCDF	124	
Lucketts-Blank-Dioxins	13C-1,2,3,4,7,8,9-HpCDF	130	

Field Duplicate Results

Samples B'ville-Dioxins-PS-4-Primary and B'ville-Dioxins-PS-1-Collocate were submitted as the field duplicate pair with this sample data set. The following table summarizes the relative percent differences (RPDs) of the detected congeners and total homologs in the field duplicate pair. The results for some RPDs were not calculable (NC) since the affected congener was not detected in one of the two samples; qualification of the data was not required on this basis. All RPD criteria were met.

Compound	QL (pg/m3)	B'ville-Dioxins-PS-4-Primary (pg/m3)	B'ville-Dioxins-PS-1-Collocate (pg/m3)	RPD
2,3,7,8-TCDD	0.00226	0.00226 U	0.00114 J	NC
1,2,3,4,7,8-HxCDD	0.0113	0.00636 J	0.00511 J	22
1,2,3,6,7,8-HxCDD	0.0113	0.0095 J	0.00915 J	3.7
1,2,3,7,8,9-HxCDD	0.0113	0.0109 J	0.0123	12
1,2,3,4,6,7,8-HpCDD	0.0113	0.122	0.112	8.5
OCDD	0.0226	0.416	0.344	19
2,3,7,8-TCDF	0.00226	0.00168 J	0.00173 U	NC
2,3,4,7,8-PeCDF	0.0113	0.00299 J	0.00373 J	22
1,2,3,4,7,8-HxCDF	0.0113	0.00403 J	0.00403 J	0
1,2,3,6,7,8-HxCDF	0.0113	0.00373 J	0.0038 J	1.9

Compound	QL (pg/m3)	B'ville-Dioxins-PS-4-Primary (pg/m3)	B'ville-Dioxins-PS-1-Collocate (pg/m3)	RPD
2,3,4,6,7,8-HxCDF	0.0113	0.00467 J	0.00399 J	16
1,2,3,7,8,9-HxCDF	0.0113	0.00265 J	0.00204 J	26
1,2,3,4,6,7,8-HpCDF	0.0113	0.018	0.018	0
1,2,3,4,7,8,9-HpCDF	0.0113	0.0031 J	0.00306 J	1.3
OCDF	0.0226	0.0197 J	0.0159 J	2.1
Total TCDD	0.00226	0.0142	0.0192	29.9
Total PeCDD	0.0113	0.0441	0.0463	4.9
Total HxCDD	0.0113	0.127	0.135	6.6
Total HpCDD	0.0113	0.269	0.254	5.7
Total TCDF	0.00226	0.0674	0.0715	5.9
Total PeCDF	0.0113	0.0478	0.0521	8.6
Total HxCDF	0.0113	0.0485	0.0418	15
Total HpCDF	0.0113	0.0353	0.0352	0.28
Criteria: When both results are ≥ 5x the QL, RPDs must be <30%. When both results are < 5x the QL, RPDs must be <60%.				

Quantitation Limits and Sample Results

Nondetect results were reported to the EDL as stipulated in the method.

Select results were reported which were below the lowest calibration standard level and QL. These results were qualified as estimated (J) by the laboratory.

2378-TCDF confirmation analysis was performed for results detected above the quantitation limit. Results detected at concentrations below the QL are qualified as estimated by the laboratory and are not confirmed. Further qualification of these data was not required.

Several congeners in the select samples were reported by the laboratory as EMPCs since all identification criteria were not met. These results were qualified as estimated (J) during data validation.

No dilutions were performed on samples in this data set.

Memorandum

To: Karen Vetrano

From: Paula DiMattei

CC: Elizabeth Denly

Date: April 13, 2015

Subject: Inorganic Data Validation Review: Montgomery County, Dickerson, MD site: Alpha Analytical Project Nos: L1403022, L1403198 and L1404480 and Brooks Rand Laboratory Project Nos: 1406018, 1407008 and 1410010

SUMMARY

Limited validation was performed on the data for 17 air samples, five field blanks and one field spike collected at the Montgomery County site in Dickerson, Maryland. The samples were collected on January 29, 2014, February 6-7, 2014 and February 26-27, 2014. Samples (PM-10 filters) were submitted to Alpha Analytical in Mansfield, Massachusetts for select metals analysis using SW-846 Methods 6020A/7471B; Alpha Analytical reported the results under laboratory project numbers L1403022, L1403198 and L1404480. Samples (iodated carbon traps) were also submitted to Brooks Rand Laboratory in Seattle, Washington for low-level mercury analysis using EPA Method 1631; Brooks Rand reported the results under laboratory project numbers 1406018, 1407008 and 1410010.

The sample results were assessed using the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review," August 2014, modified for the non-CLP methodologies utilized.

In general, the data appear valid as reported and may be used for decision-making purposes. The positive and nondetect results for low-level mercury were qualified as estimated (J/UJ) in samples B'ville-Hg-1-PRI and B'ville-Hg-1-COL due to field duplicate variability and a potential sample switching error in the laboratory; caution should be used with these results for decision-making purposes. The following other issues were noted which may have a minor impact on the data usability:

- The positive results for cadmium in samples B'ville-Metals-1-PRI and B'ville-Metals-1-COL were qualified as estimated (J) due to laboratory duplicate variability.
- The results for cadmium and lead in samples B'ville-Metals-1-PRI and B'ville-Metals-1-COL were qualified as estimated (J) due to field duplicate variability.
- Potential uncertainty exists for select low-level mercury results which were below the reporting limit (RL), but greater than or equal to the method detection limit (MDL).

SAMPLES

Samples included in this review are listed below:

L1403022

B'ville-Metals-1-PRI	B'ville-Metals-1-COL[1]	Luck-Metals-1-PRI	FBB-Metals-1

[1]Field duplicate of B'ville-Metals-1-PRI

L1403198

B'ville-Metals-2-PRI	B'ville-Metals-2-COL[2]	Luck-Metals-2-PRI	FBB-Metals-2

[2]Field duplicate of B'ville-Metals-2-PRI

L1404480

B'ville-Metals-3-PRI	B'ville-Metals-3-COL[3]	Luck-Metals-3-PRI	FBB-Metals-3

[3]Field duplicate of B'ville-Metals-3-PRI

1406018

B'ville-Hg-1-COL[4]	B'ville-Hg-1-PRI	Lucketts-Hg-1	B'ville-Hg-Spike

[4]Field duplicate of B'ville-Hg-1-PRI

1407008

B'ville-Hg-2-COL[5]	B'ville-Hg-2-PRI	FBB-Hg-1	Lucketts-Hg-2

[5]Field duplicate of B'ville-Hg-2-PRI

1410010

B'ville-Hg-3-PRI	B'ville-Hg-3-COL[6]	FBB-Hg-2

[6]Field duplicate of B'ville-Hg-3-PRI

REVIEW ELEMENTS

Sample data were reviewed for the following parameters:

- Agreement of analyses conducted with chain-of-custody requests
- Data completeness
- Holding times
- Initial and continuing calibrations
- Blanks
- Matrix spike (MS) results
- Field spike results
- Laboratory control sample (LCS) results
- Laboratory duplicate results
- Field duplicate results
- ICP serial dilution results
- Reporting limits and sample results

DISCUSSION

Agreement of Analyses Conducted with Chain-of-Custody Requests

Sample reports were checked to verify that the results corresponded to analytical requests as designated on the chain-of-custody (COC) and any correspondence between TRC and the laboratory. The following discrepancy was noted.

- The sample collection dates were not noted on the COC for samples reported in laboratory report L1403022. The laboratory did report sample collection dates on the sample data summary forms. These collection dates were confirmed with the TRC project manager.

Data Completeness

The data package was found to be complete as received from the laboratory.

Holding Times

All samples were prepared and analyzed within the method-specified holding times.

Initial and Continuing Calibrations

All initial calibration and initial and continuing calibration verifications were within criteria for the low-level mercury analyses. There were no calibration issues noted in the case narratives for the select metals laboratory reports; thus, data qualification was not required.

Blanks

Target analytes were not detected in the laboratory blanks or field blanks associated with the samples in this data set with one exception in which the end data user should be aware. Low-level mercury was detected at a concentration of 37.6 ng/tube in the field blank FBB-Hg-1 (in laboratory report 1407008). Although in a different laboratory report (1406018), the field duplicate pair B'ville-Hg-1-PRI/B'ville-Hg-1-COL was prepared and analyzed in the same batch as this field blank. Sample B'ville-Hg-PRI yielded a low-level mercury concentration of 22.1 ng/tube and low-level mercury in the associated field duplicate sample (B'ville-Hg-1-COL) was found to be not detected. It appears that the field blank and field duplicate sample B'ville-Hg-1-COL may have been switched during sample preparation. However, the sample preparation requires removal of the iodated carbon (IC) from the glass trap; therefore, the original trap and plastic bag were not preserved and the switch could not be confirmed. No data validation actions were taken on this basis. The results of the field blank were not used to qualify sample data due to this issue.

Media certification results are not available for the select metals analyses. Data are not adversely affected since the laboratory method blanks were performed using the same lot as the field samples and target analytes were not detected in any laboratory method blanks.

Trap blanks were prepared using an unused trap to demonstrate that the IC material used in sample collection was free from low-level mercury contamination. It should be noted that a trap blank was not prepared for the samples reported in laboratory report 1410010 (B'ville-Hg-3-PRI, B'ville-Hg-3-COL6 and FBB-Hg-2). No data validation actions were taken on this basis.

MS Results

MS analyses were performed as summarized in the following table. All criteria were met.

Sample ID	MS Analyses
B'ville-Metals-1-PRI	All select metals except mercury
B'ville Metals-2-PRI	Mercury only
B'ville-Metals-3-COL	Mercury only
B'ville Metals-3-PRI	All select metals except mercury

A post-digestion spike analysis was also performed on sample B'ville-Hg-3-PRI for low-level mercury; all criteria were met.

Field Spike Results

One field spike sample (B'ville-Hg-Spike) was analyzed in association with samples in this data set. This field spike was associated with samples B'ville-Hg-1-PRI and B'ville-Hg-1-COL. This IC trap was pre-spiked by the laboratory with a known amount of low-level mercury and sampled in the field in the same manner as the original and field duplicate sample. Since the results between the original and field duplicate sample showed a significant degree of variability and a potential sample switching error (see Blanks section), the actual percent recovery (%R) was not verifiable. If the original sample result is used as the true value in the source sample, the %R is 49% and if the field duplicate concentration is used as the true value in the source sample, the %R is 94%. No validation action was taken on this basis as these results were subsequently qualified as estimated (J/UJ) due to field duplicate variability.

LCS Results

All criteria were met.

Laboratory Duplicate Results

Laboratory duplicate analyses were performed as summarized in the following table:

Sample ID	Laboratory Duplicate Analyses
B'ville-Hg-3-PRI	Low-level mercury
B'ville-Metals-2-PRI	Mercury only
B'ville Metals-1-PRI	All select metals except mercury
B'ville-Metals-3-COL	Mercury
B'ville-Metals-3-PRI	All select metals except mercury

The following table summarizes the relative percent differences (RPDs) which were outside of the acceptance criteria in the laboratory duplicate analyses.

Sample ID	Analyte	RPD (%)	QC Limits	Associated Samples	Validation Action
B'ville-Metals-1-PRI	Cadmium	70	20	B'ville-Metals-1-PRI B'ville-Metals-1-COL	The positive results for cadmium in samples B'ville-Metals-1-PRI and B'ville-Metals-1-COL were qualified as estimated (J).

Field Duplicate Results

Samples B'ville-Hg-1-PRI/B'ville-Hg-1-COL, B'ville-Hg-2-PRI/B'ville-Hg-2-COL, B'ville-Hg-3-PRI/B'ville-Hg-3-COL, B'ville-Metal-1-PRI/B'ville-Metals-1-COL, B'ville-Metals-2-PRI/B'ville-Metals-2-COL and B'ville-Metals-3-PRI/B'ville-Metals-3-COL were submitted as the field duplicate pairs with this data set. Field duplicate samples B'ville-Hg-1-PRI and B'ville-Hg-1-COL were not assessed for field duplicate precision due to the uncertainty of the results reported for B'ville-Hg-1-COL as noted in the Blanks section above. However, due to this uncertainty, the positive and nondetect results for low-level mercury in these samples were qualified as estimated (J/UJ). Low-level mercury was not detected in field duplicate pair B'ville-Hg-2-PRI/B'ville-Hg-2-COL; precision was deemed acceptable in this case.

The following tables summarize the RPDs of the detected analytes in the remaining field duplicate pairs. The results for cadmium and lead in samples B'ville-Metals-1-PRI and B'ville-Metals-1-COL were qualified as estimated (J) due to field duplicate variability. The RPD was not calculable (NC) for the cadmium results in samples B'ville-Metals-3-PRI and B'ville-Metals-3-COL due to a nondetect result in one of the two samples; qualification of the data on this basis was not required. The calculated RPDs were within the acceptance criteria for all remaining results.

Analyte	B'ville-Metals-1-PRI (µg/filter)	B'ville-Metals-1-COL (µg/filter)	RPD (%)
Arsenic	0.981	1.11	12
Cadmium	0.351	3.04	158
Lead	2.79	4.10	38
Nickel	1.38	1.71	21
Criteria : For results ≥ 5x RL , RPD <30 For results <5x RL, the difference between the results must be <RL			

Analyte	B'ville-Metals-2-PRI (µg/filter)	B'ville-Metals-2-COL (µg/filter)	RPD (%)
Arsenic	1.20	1.10	8.7
Lead	3.96	3.88	2.0
Nickel	1.31	1.34	2.3
Criteria : For results ≥ 5x RL , RPD <30 For results <5x RL, the difference between the results must be <RL			

Analyte	B'ville-Metals-3-PRI (µg/filter)	B'ville-Metals-3-COL (µg/filter)	RPD (%)
Arsenic	0.609	0.719	17

Analyte	B'ville-Metals-3-PRI (µg/filter)	B'ville-Metals-3-COL (µg/filter)	RPD (%)
Cadmium	0.180 U	0.233	NC
Lead	3.22	3.39	5.1
Nickel	1.91	1.67	14
Criteria : For results ≥ 5x RL , RPD <30 For results <5x RL, the difference between the results must be <RL			

Analyte	B'ville-Hg-3-PRI (ng/trap)	B'ville-Hg-3-COL (ng/trap)	RPD (%)
Low-level mercury	1.3 J	3.1 J	82
Criteria : For results ≥ 5x RL , RPD <30 For results <5x RL, the difference between the results must be <RL			

ICP Serial Dilution Results

No issues were noted in the laboratory case narratives regarding ICP serial dilutions; thus, no data validation actions were required on this basis.

Reporting Limits and Sample Results

Select low-level mercury results were reported which were below the below the RL, but greater than or equal to the MDL. These results were qualified by the laboratory as "B". The "B" qualifier was changed to "J" during validation to indicate the result is estimated.

Two-fold diluted analyses were performed for all select metals with the exception of mercury. RLs were adjusted accordingly. The RLs for chromium (45 µg/filter) were significantly higher than expected (0.8 µg/filter) due to inherent chromium in the PM-10 filters at about 20 µg/filter; the RLs for chromium were elevated to account for the background in the matrix.

It should be noted that the results reported for low-level mercury by Brooks Rand Laboratory were incorrectly reported in units of ng/m3. Upon questioning during validation, it was discovered that the laboratory had not corrected the results for the sample volumes and the units were corrected to ng/trap.