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NCBC GULFPORT
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LETTER REGARDING HISTORY OF ASH RESAMPLING AT HERBICIDE ORANGE SITE A
NCBC GULFPORT MS
3/22/1996
ABB ENVIRONMENTAL



1.3 8.4

March 22, 1996

Commanding Officer
Southern Division
Naval Facilities Engineering Command
2155 Eagle Drive
North Charleston, SC 29418

Attention: Mr. Dan Owens

Subject: History of Ash Resampling at
Herbicide Orange Site A
N62467-89-D-0317/CTO 92

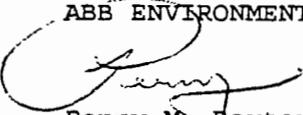
Dear Dan:

Enclosed is a writeup that Bob Fisher did for me that details the history of problems we have had in executing the ash sampling. I hope that this is what you are looking for.

If you have any questions, please call me at (423) 531-1922.

Sincerely,

ABB ENVIRONMENTAL SERVICES, INC.



Penny M. Baxter
Senior Project Manager

attachment

cc: file

[8504.027]

ABB Environmental Services Inc.

To: Penny Baxter

From: Bob Fisher

RE: Ash Sampling Issues
Site 8, NCBC Gulfport
Gulfport, Mississippi

The dioxin data associated with the most recent ash sampling effort (August 1994) has been rejected. This action became necessary when the data was scrutinized prior to its use as part of the Delisting Petition, due to the State of Mississippi Department of Environmental Quality by the end of February, 1996. This memo will provide the chronology, the events that led up to the rejection of the data, and provide suggestions for resolution and procedures to assure that this issue does not arise again.

Purpose for Sampling In 1987 and 1988, dioxin contaminated soil at Site 8, NCBC Gulfport was incinerated. The resulting ash was stored back upon the original site. Delisting of the ash was considered to be a matter of course following the incineration and verification that all dioxin contaminated soils above 1 part per billion were incinerated. As it turns out, however, delisting became considerably more difficult due to more stringent EPA and State standards regarding dioxin compounds. As a result, a more formal delisting of the ash would be required. This delisting petition required sampling of the ash piles left behind on Site 8 before they could be considered for reclassification as non-hazardous waste. This environment makes the results of these ash samples critical to delisting and requires that the ash be analyzed by a strict 8290 USEPA Method (Dioxins) so that the data is defensible even under intense scrutiny, which would surely come from the State and EPA.

Sampling History The first ash samples collected for this delisting effort were collected in April 1994 and analyzed for dioxins by Canviro Labs for Method 8290. The sample data went to an independent validator (Validata) for validation. The data was rejected as not useable by Validata for numerous reasons (attached). Basically, because GC performance checks and mass resolution checks were not performed, all positive sample results were invalidated.

Canviro reviewed the validators comments and agreed to pay for resampling and reanalysis of all ash samples. The resampling was performed in August 1995. Due to problems at their Waterloo, Ont. Laboratory, Canviro was not able to run the samples until November 1995. While this delay alone would not invalidate the data, it would require that all positive sample results be flagged as estimations. The data from the August samples were again sent to Validata for validation. ABB received Validata case narrative in January, 1995. We noted some irregularities in their discussion of blanks and QA/QC results. Validata resubmitted the validation report with amended comments. Otherwise, their case narrative stated that the data were useable with the exception of sample runs outside of holding times. When the completed PARCC report arrived, ABB

included the ash samples in a letter report in June 1995 as a precursor to the delisting petition.

On February 11, 1996, I reviewed the data again for comparability between the hits (detections) tables and concentration reports from the laboratory before the data was used in the delisting petition. I noted some irregularities in the reporting of the concentration reports; specifically that OCDD and OCDF were not reported in samples G14, D11, and A14. I soon discovered that these compounds were not reported because the laboratory could not have identified these compounds during this analytical run because their internal standards for these compounds were recovering at 0%. I sent the data package to an independent validator (Heartland, Inc) so that they could review this problem. The result of Heartland's review was that the data should have been reanalyzed by the laboratory when the percent recovery for OCDD/OCDF was 0%. This reanalysis is required by both the laboratory's internal QA/QC and by the 8290 Method. Also, Heartland stated that the original data validator (Validata) should have rejected the data. Validata's case narrative clearly indicates that they missed this problem with the internal standards (see attached).

Resolution I contacted Canviro Labs on February 13, 1996 and informed them of the problem. I spoke to Glenna Royal (Dioxin Manager) and Jeff Pike (Lab Manager) and they agreed the data should have been reanalyzed based on the recovery problem alone, and that the data should not have left the laboratory in the condition.

On February 14, 1996 Jeff Pike agreed in principle to pay for another resampling of the ash and sample analysis, but expressed that Canviro felt that Validata should pay for any additional validation since they clearly were negligent in not discovering the problem as part of their validation process.

Canviro has recently proven their ability to run a strict 8290 Method analysis that should provide defensible data, and for the sake of the delisting effort, resampling should begin on February 19, 1996 in combination with a rapid turn around cycle on the samples. Messages have been left with Validata, but they have not responded at this time.

Rejection of Initial Dioxin and Furan Sampling

- No GC Column Performance Check for each 12 hour run.
- No Mass Resolution Check at the end of each 12 hour run.
- No Mass Verification Check at the end of each 12 hour run.
- No Calibration and Verification Check at the end of each 12 hour run.
- Instrument total cycle time was greater than 1.0 second.
- Ion Abundance Ratios were outside QC limits.

ACCORDING TO USEPA SW-846 METHOD 8290 (SECTION 8.3.2.4) DEVIATIONS FROM CRITERIA SPECIFIED FOR THE GC PERFORMANCE CHECK OR FOR THE MASS RESOLUTION CHECK INVALIDATE ALL ASSOCIATED POSITIVE SAMPLE DATA.

DATA QUALIFICATION SUMMARY

CANVIRO Analytical Laboratories Ltd. - R8537 2,3,7,8-substituted PCDD's and PCDF's

SAMPLES: GPT-R-F15, GPT-R-E12, GPT-R-G14, GPT-R-D11, GPT-R-A14

X NOVAK

2,3,7,8-SUBSTITUTED PCDD'S AND PCDF'S

I.) Holding Times:

The holding times for the following samples exceeded the QC limits:

Client Sample #:	Holding Time to Extraction	QC Limit (days)	Holding Time to Analyzed	QC Limit (days)
All samples	47	30	82	45

According to EPA 8290, all samples must be extracted within 30 days and completely analyzed within 45 days of sample collection. Since there were no data supporting the longer holding times for these samples, all associated samples results were flagged as estimated (J) and (UJ).

II.) HRGC/HRMS System Performance:

GC Column Performance:

All criteria were met, so no action was taken.

HRMS Resolution:

All criteria were met, so no action was taken.

Mass Verification:

All criteria were met, so no action was taken.

MS Data Acquisition:

All criteria were met, so no action was taken.

III.) Calibration:

Calibration Range:

All criteria were met, so no action was taken.

Initial Calibration:

All criteria were met, so no action was required.

Calibration Verifications:

All criteria were met, so no action was taken.

IV.) Blanks

Method Blanks:

No 2,3,7,8-substituted PCDD's or PCDF's were detected in the water method blanks, so no action was required.

Equipment Blanks:

~~No equipment blank was analyzed.~~ OCDD detected at 11.3 ppb for sample BS-C1R-RJ. Result flagged EmPC due to an incorrect ion ratio. No action required.

Field Blanks:

~~No field blank was analyzed for this batch.~~ No 2,3,7,8-substituted PCDD's or PCDF's detected in field blanks, so no action required.

V.) Internal Standards Performance:

All criteria were met, so no action was taken.

VI.) Spike/Spike Duplicates:

The Percent Recovery (%R) of OCDF for the method spike was 817%, which exceeded the 60-140% QC limits. There were no positive results for this analyte in the associated samples, so no action was required.

No field sample spike/spike duplicate were analyzed for this batch.

VII.) Duplicates:

~~No field duplicates were analyzed for this batch.~~ See SDGR3517 narrative. Duplicates were GPT-R-F7 and GPT-R-F7D. RD's were within 60% so no action taken.

VIII.) PCDD/PCDF Identifications:

Retention Times:

All criteria were met, so no action was taken.

Ion Abundance:

Due to a HRMS computer hard disk failure, the direct comparison between ion ratio and the QC limits

KRATOS MACH3 PEAK AREA QUANTITATION:- CONCENTRATION REPORT:

Date: 4 Nov 94 Internal Standard method.

Sample: GPT-R-A14 K750_0023 Library: 1613

GPT-R-A14

Run Title: 9408182-05

Sample Dry Weight (grams) = 2.117

Average response factor calibration method

Analytes	pg/g	EDL	TE
2,3,7,8-TCDD	-----	uJ 0.18	
2,3,7,8-TCDF	-----	0.07	
1,2,3,7,8-PeCDD	-----	0.42	
1,2,3,7,8-PeCDF	-----	0.17	
2,3,4,7,8-PeCDF	-----	0.17	
1,2,3,4,7,8-HxCDD	-----	0.44	
1,2,3,6,7,8-HxCDD	-----	0.50	
1,2,3,7,8,9-HxCDD	-----	0.57	
1,2,3,4,7,8-HxCDF	-----	0.35	
1,2,3,6,7,8-HxCDF	-----	0.32	
2,3,4,6,7,8-HxCDF	-----	0.50	
1,2,3,7,8,9-HxCDF	-----	0.47	
1,2,3,4,6,7,8-HpCDD	-----	0.85	
1,2,3,4,6,7,8-HpCDF	-----	0.88	
1,2,3,4,7,8,9-HpCDF	-----	0.70	
OCDD			
OCDF			

Clean-Up Standard	pg/ul	% Rec.
37C14-2,3,7,8-TCDD	2.96	73.9

Internal Standards % Recovery

13C-2,3,7,8-TCDD	107 %
13C-2,3,7,8-TCDF	82.5 %
13C-1,2,3,7,8-PeCDD	100 %
13C-1,2,3,7,8-PeCDF	84.7 %
13C-2,3,4,7,8-PeCDF	85.8 %
13C-1,2,3,4,7,8-HxCDD	128 %
13C-1,2,3,6,7,8-HxCDD	104 %
13C-1,2,3,4,7,8-HxCDF	114 %
13C-1,2,3,6,7,8-HxCDF	126 %
13C-2,3,4,6,7,8-HxCDF	93.7 %
13C-1,2,3,7,8,9-HxCDF	123 %
13C-1,2,3,4,6,7,8-HpCDD	42.5 %
13C-1,2,3,4,6,7,8-HpCDF	56.3 %
13C-1,2,3,4,7,8,9-HpCDF	110 %
13C-OCDD	0.0 %