

## MEMORANDUM

DATE: August 7, 2002

FROM: Mark Woodke, START-Chemist, E & E, Seattle, WA

SUBJ: **Dioxin/Furan Data Quality Assurance Review, Poles Inc. Dioxin/Furan Sampling, Oldtown, Idaho**

REF: TDD: 02-05-0014 PAN: 001281.0183.01SF

The data quality assurance review of one product, one water, and five soil samples collected from the Poles Inc. Dioxin/Furan site located in Oldtown, Idaho, has been completed. Polychlorinated Dibenzop-Dioxin (PCDD)/Polychlorinated Dibenzofuran (PCDF) analysis (EPA Method 8290) was performed by Southwest Laboratories of Oklahoma, Broken Arrow, Oklahoma.

The samples were numbered:

Soil	02040001	02040002	02040003	02040004	02060006
Product	02060005				
Water	02040007				

### Data Qualifications:

#### **1. Holding Times, Storage, and Preservation: Acceptable.**

The samples were maintained at 4°C ( $\pm 2^\circ\text{C}$ ) from collection until extraction. The samples were collected on June 18, 2002, and were extracted and analyzed by July 16, 2002, therefore meeting QC criteria of less than 30 days between collection and extraction and less than 45 days between extraction and analysis.

#### **2. GC/MS Performance Check: Acceptable.**

Mass spectrometer (MS) resolution of  $\geq 10,000$  was demonstrated at the beginning and end of each 12-hour analytical sequence.

#### **3. Initial Calibration: Acceptable.**

For all calibration solutions, the RTs of the isomers were within the appropriate RT windows established by the WDM analysis and the S/N ratios were  $\geq 10.0$ . The %RSD of the five RRFs were less than 35%.

**4. Calibration Verification: Satisfactory.**

The CS3 was analyzed at the beginning and end of each 12-hour analysis sequence. The ion abundance ratio criteria were met. The RRFs of the unlabeled analytes were within 20% and the labeled analytes were within 30% except the <sup>13</sup>C-OCDD recovery in the July 3, 2002, calibration; no action was taken based on this internal standard calibration outlier alone.

**5. Method Blank Analysis: Satisfactory.**

Method blanks were prepared at the required frequency of every time samples were extracted for each matrix and concentration or every 20 samples (whichever is greater). The following analytes were detected in blanks:

Analyte	Concentration	Associated Sample(s)
1,2,3,4,6,7,8-HpCDD	1.213 ng/kg	02060001, 02060002, 02060003
OCDD	26.24 ng/kg	
1,2,3,4,6,7,8-HpCDF	0.491 ng/kg	
OCDF	8.04 ng/kg	
1,2,3,4,6,7,8-HpCDD	8.943 pg/L	02060007
OCDD	134.3 pg/L	
OCDF	26.23 pg/L	
OCDD	19.62 ng/kg	02060005
OCDF	3.391 ng/kg	

Results less than 5 times associated blank contamination were qualified as not detected (U).

**7. Internal Standards: Satisfactory.**

C-13 Labeled internal standards - The recoveries of Method 8290 C-13 labeled PCDD and PCDF isomers were within 40 % to 135 % except 1,2,3,4,6,7,8-HpCDD and OCDD in sample 02060006re and PeCDD in sample 02060007, each with high recoveries. Associated sample results were qualified as estimated quantities (J or UJ).

Instrument Recovery internal standards - The sum of the area counts of two masses for each of the two instrument recovery internal standards for samples, blanks, and standards was within a factor of four (- 25 % to + 400 %) from the sum of the associated average areas from the five initial calibration standards.

**8. Compound Identification: Satisfactory.**

The signals for the two exact m/z's being monitored maximized within 2 seconds of one another. The signal-to-noise ratio (S/N) of each of the two exact m/z's was greater than or equal to 2.5 for sample extracts and greater than or equal to 10 for calibration standards. The ratio of the integrated ion currents (EICPs) of both the exact m/z's monitored was within method limits. The relative retention time (RRT) of the peaks representing an unlabeled 2,3,7,8 substituted PCDD or PCDF was within method limits. The retention time (RT) of peaks representing non-2,3,7,8-substituted PCDDs or PCDFs was within the method RT windows. The positive TCDF result for sample 02060005 was not confirmed on a secondary column, so the result was rejected (R). Analytes that didn't meet all QC requirements, including matrix interferences and/or ratio outliers,

were rejected (R).

**9. Laboratory Control Sample (LCS): Satisfactory.**

A LCS was analyzed for each 20 samples per matrix. All results were within QC limits except the soil LCS OCDD recovery (256 % vs the QC limits of 40% to 135%). The laboratory indicated that the high result was due to interference from the previous high level sample analysis. An additional soil LCS was analyzed with all results within QC limits, therefore no action was taken based on the LCS outlier.

**10. Performance Evaluation Samples: Not Provided.**

Performance evaluation samples were not provided to the laboratory.

**11. Error Determination: Not Performed.**

Samples necessary for bias and precision determination were not provided to the laboratory. All samples were flagged RND (Recovery Not Determined) and PND (Precision Not Determined), although the flags are not found on the Form I's.

**12. Laboratory Contact: Required or Not Required.**

No laboratory contact was required.

**13. Overall Assessment**

Samples 02060004 and 02060006 were reextracted and reanalyzed due to QC outliers. The reanalyses were reported in this validation memorandum.

The laboratory used EPA Toxicity Equivalent Factors (TEFs) in TEQ calculation, but EPA Region 10 uses World Health Organization (1997) TEFs. The reviewer changed the appropriate calculations on the Form Is for PeCDD, OCDD, and OCDF as appropriate.

The overall usefulness of the data is based on the criteria outlined in the "EPA Region 10 SOP For the Validation of Polychlorinated Dibenzodioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Data", January 31, 1996, and the analytical method. Based upon the information provided, the data are acceptable for use with the above stated data qualifications.

Data Qualifiers and Definitions

- U - The analyte was analyzed for, but was not detected above the Sample Quantitation Limit (SQL).
- J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- R - The sample results are unusable. The analyte may or may not be present in the sample.