

APPENDIX D  
DATA VALIDATION REPORT  
(MACTEC DATA)

**TO:** Debbie Leibensberger, Mactec, E & C, Inc.

March 18, 2004

**FROM:** Donna Breaux, DataVal, Inc.

Mactec Project No. 56286 080107

**QUALITY CONTROL SUMMARY REPORT FOR THE FORT ORD PRESCRIBED  
BURN SITE – REVISED FROM PREVIOUS REPORT DATED JANUARY 10, 2004**

**LABORATORIES:** DataChem Laboratories, Inc., Cincinnati, OH  
Frontier Analytical Laboratory, El Dorado Hills, CA  
Air Toxics, Ltd., Folsom, CA

**SAMPLING DATES:** October 24, 25 and 26, 2003 and November 12, 2003

Data validation of Levels III and IV laboratory data packages was performed according to the following guidelines: the U. S. Environmental Protection Agency Analytical Operations/Data Quality Center National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, August, 2002; U. S. Environmental Protection Agency Contract Laboratory Program National Functional Guidelines for Organic Data Review, October, 1999; and the U. S. Environmental Protection Agency Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, July, 2002. The analysis methods provided additional guidance during the data validation effort.

The data were reviewed for compliance with analytical holding times, GC/MS tunes, initial calibrations, continuing calibration verification standards (CCV), internal standard and ICP interference check standard acceptance criteria. The data were also reviewed for compliance to accuracy limits for surrogate recoveries, laboratory control samples (LCS), matrix spikes (MS) and matrix spike duplicate (MSD) recoveries. Matrix duplicate samples and co-located field samples were evaluated to assess compliance with precision requirements. Laboratory method blank and field blank results were reviewed for evidence of contamination and potential impacts on the project sample results. Compound identification and quantitation were evaluated in the samples that received full (Level IV) data validation.

Table 1 presents a cross reference of the site samples, laboratory sample IDs, sampling dates, and level of data review. Table 2a presents a summary of data qualifiers applied to target analytes. Table 2b presents a summary of qualifiers applied to non-target analytes. Table 3 presents the precision results for the co-located project samples.

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*Debbie Leibensberger*

The following paragraphs highlight the essential findings of the data validation effort:

I. **Polychlorinated Dibenzodioxins and Dibenzofurans (TO-9A)**

Overall, the data are usable as reported. Qualification was not required. The dioxin and furan samples were analyzed by Frontier Analytical Laboratory.

A. Reporting Limits

The laboratory established reporting limits were used for this analysis method.

B. Holding Times

Technical holding time criteria were met for all project samples. All samples were prepared within 7 days from the collection date, and all extracts were analyzed within 40 days from the extraction date.

C. Blanks

Target analytes were not observed in any laboratory method blanks associated with the project samples.

Target analytes were not observed in any field blanks associated with the project samples.

D. Mass Calibration Tunes

All HRGC-HRMS tunes met QC acceptance criteria and were analyzed at the frequency required by the analytical method.

E. Initial Calibration

Initial calibration criteria were met for all calibration standards associated with the project samples.

F. Column Performance Check Standards

All column performance check standards met QC acceptance criteria and were analyzed at the frequency required by the analytical method.

G. Continuing Calibration

Continuing calibration criteria were met for all continuing calibration standards associated with the project samples.

H. Laboratory Control Samples

All QC criteria were met for the laboratory control samples associated with the project samples, with the following exception:

1. The percent recovery for 1,2,3,4,7,8,9-HpCDF was outside the 70%-130% QC acceptance criteria in laboratory control sample 2301-001-LCS at 139%. The associated samples were non-detect for 1,2,3,4,7,8,9-HpCDF, and qualification was not required.

- I. Matrix Spike/Matrix Spike Duplicate  
A matrix spike and matrix spike duplicate were not prepared and analyzed with the project samples.
  - J. Internal Standards  
Internal standard recoveries met QC acceptance criteria for all project samples.
  - K. Surrogate Recoveries  
All pre-spike surrogates met a 70%-130% QC acceptance criteria.
  - L. Compound Identification and Quantitation  
For the samples that received full (Level IV) data validation, the sample results were recalculated with the proper dilution factors, weights and volumes used to calculate the sample results. The samples were correctly quantitated and reported by the laboratory.
- II. Acrolein by GC/MS (TO-14A)  
Overall, the data are usable as reported. Qualification was not required. The acrolein samples were analyzed by Air Toxics, Ltd.
- A. Reporting Limits  
A 0.5 ppbv reporting limit was requested on the chain-of-custody documents. Most samples were analyzed at two-fold dilutions. The reporting limits were raised by the dilution factors.
  - B. Holding Times  
The project samples were evaluated using a 30-day analysis holding time. All project samples were analyzed within thirty days from the collection date. The laboratory case narratives stated that some samples were analyzed past day fourteen. A 14-day sample holding time is used by the laboratory as standard operating procedure. Samples analyzed past day fourteen were considered passing, and were not considered outliers.
  - C. Blanks  
Target analytes were not observed in any laboratory method blanks associated with the project samples.  
  
Target analytes were not observed in any field blanks associated with the project samples.
  - D. GC/MS Tunes  
All QC criteria were met for the GC/MS tunes associated with the project samples.
  - E. Initial Calibration  
Initial calibration criteria were met for all calibration standards associated with the project samples.

- F. Continuing Calibration  
Continuing calibration criteria were met for all continuing calibration standards associated with the project samples.
- G. Laboratory Control Samples  
All QC criteria were met for the laboratory control samples associated with the project samples.
- H. Matrix Spike/Matrix Spike Duplicate  
A matrix spike and matrix spike duplicate were not prepared and analyzed with the project samples.
- I. Matrix Duplicate Samples  
All QC criteria were met for the matrix duplicate samples associated with the project samples.
- J. Surrogate Recoveries  
Surrogate spike recoveries met QC acceptance criteria for all project samples, with the following exception:
  - 1. The percent recovery for surrogate 4-bromofluorobenzene was outside the 70%-130% project acceptance criteria in sample PS1P25315 (0310533B-24A) at 131%. Acrolein was non-detect in this sample, and qualification was not required.
- K. Internal Standards  
Internal standard areas and retention times met QC acceptance criteria for all project samples.
- L. Compound Identification and Quantitation  
For the samples that received full (Level IV) data validation, the sample results were recalculated with the proper dilution factors, weights and volumes used to calculate the sample results. The samples were correctly quantitated and reported by the laboratory.

### III. Aldehydes (TO-11A)

Overall, the data are usable as reported with any added qualifiers. Qualifications were required for the reasons noted in Sections C and E. The aldehyde samples were analyzed by DataChem Laboratories, Inc.

- A. Reporting Limits  
The laboratory established reporting limits were used for this analysis method.
- B. Holding Times  
All samples were prepared within 14 days from the date of collection and analyzed within 30 days from the date of extraction.

C. Blanks

The method of analysis does not require preparation and analysis of method blanks within the laboratory QC batch.

Target analytes were not observed in any field blanks associated with the project samples.

Two non-target analytes were detected in field blanks submitted to the laboratory for analysis:

1. Field blank B2763-7 had a detected level of valeraldehyde at 1.6 ug/sample. The associated samples were non-detect for valeraldehyde, and qualification was not required.
2. Field blank B2763-24 had a detected level of non-target compound, n-butyraldehyde, at 1.5 ug/sample. Ten project samples had detected levels of n-butyraldehyde less than five times the blank amount. The results for n-butyraldehyde in these samples should be considered non-detect (U) due to blank contamination.

Since neither of the compounds detected in the field blanks were chemicals of concern for this project, the field blank detections have no impact on the quality or usability of the data associated with the chemicals of concern, which are formaldehyde and acetaldehyde. Table 2a presents a summary of the qualified data for non-target compounds.

D. Initial Calibration

Initial calibration criteria were met for all calibration standards associated with the project samples.

E. Continuing Calibration

Continuing calibration criteria were met for all target compound continuing calibration standards associated with the project samples.

Several non-target compounds reported by the laboratory were associated with continuing calibration standards that failed to meet the acceptance criteria:

1. One continuing calibration standard had three non-target compounds with percent differences (%D) outside the +/-15% difference acceptance criteria, n-butyraldehyde, isovaleraldehyde and m & p-tolualdehyde. The detected and non-detected results for these compounds in the associated samples were qualified as estimated (J+/J-/UJ).
2. One continuing calibration standard had four non-target compounds with %D < -15%, isovaleraldehyde, valeraldehyde, m & p-tolualdehyde and hexaldehyde. The detected and non-detected results for these compounds in the associated samples were qualified as estimated (J-/UJ).

3. One continuing calibration standard had three non-target compounds with %D < -15%, n-butyraldehyde, benzaldehyde and isovaleraldehyde. The detected and non-detected results for these compounds in the associated samples were qualified as estimated (J-/UJ).
4. One continuing calibration standard had one non-target compound with %D < -15%, p-tolualdehyde (-41%). The detected and non-detected results for p-tolualdehyde in the associated samples were qualified as estimated (J-/UJ).

Since the failing compounds noted above were not chemicals of concern for this project, the failures have no impact on the quality or usability of the data associated with the chemicals of concern, which are formaldehyde and acetaldehyde. Qualified data for non-target analytes are presented in Table 2b.

- F. Laboratory Control Samples  
All QC criteria were met for the laboratory control samples associated with the project samples.
- G. Matrix Spike/Matrix Spike Duplicate  
A matrix spike and matrix spike duplicate were not prepared and analyzed with the project samples.
- H. Surrogate Recoveries  
The project samples were not prepared and analyzed with surrogate standards.
- I. Compound Identification and Quantitation  
For the samples that received full (Level IV) data validation, the sample results were recalculated with the proper dilution factors, weights and volumes used to calculate the sample results. The samples were correctly quantitated and reported by the laboratory.

#### IV. Metals (EPA Method 12, NIOSH 6009 and NIOSH 7300)

Overall, the data are usable as reported with any added qualifiers. Qualifications were required for the reason noted in Section C. The metals samples were analyzed by DataChem Laboratories, Inc. The method of analysis used for metals collected on Teflon filters was NIOSH Method 7300. The method of analysis used for metals collected on quartz filters was EPA Method 12. The metals reported for both NIOSH Method 7300 and EPA Method 12 were aluminum, antimony, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, molybdenum, nickel and zinc. Mercury was analyzed by NIOSH Method 6009 for samples collected on both Teflon and quartz filters.

- A. Reporting Limits  
The laboratory established reporting limits were used for this analysis method.

B. Holding Times

Technical holding time criteria were met for all project samples.

C. Blanks

Target analytes were not observed in any laboratory method blanks associated with the project samples, with the following exception:

1. Laboratory method blank, 'Prep Blank (10/29/03)', was analyzed for metals using EPA Method 12. This method blank had a detected level of aluminum at 350 ug/filter. Samples BA2PQ0116287 (03-31479), BA2PQ0116283 (03-31480), BA2CQ0116284 (03-31481), BA1PQ0116278 (03-31486), BA2PQ0116285 (03-31488) and BA2CQ0116286 (03-31489) had reported detections of aluminum that were less than five times the amount detected in the method blank. The reported values for aluminum in these field samples were qualified as non-detected as a result of the laboratory method blank contamination. Qualified data for all target analytes is presented in Table 2a.

Target analytes were not observed in any field blanks associated with the project samples.

D. Initial and Continuing Calibrations

All initial and continuing calibration standards associated with the project samples met QC acceptance criteria.

E. Laboratory Control Samples

All QC criteria were met for the laboratory control samples associated with the project samples.

F. Matrix Spike/Matrix Spike Duplicate

All QC criteria were met for the matrix spikes and matrix spike duplicates associated with the project samples.

G. ICP Interference Check Samples

All QC criteria were met for the ICP interference check samples associated with the project samples.

H. ICP Serial Dilution

ICP serial dilutions were not required by the analysis method used for the project samples.

I. Compound Identification and Quantitation

For the samples that received full (Level IV) data validation, the sample results were recalculated with the proper dilution factors, weights and volumes used to calculate the sample results. The samples were correctly quantitated and reported by the laboratory.

**V. Particulate Matter (PM-10 and TSP)**

Overall, the data are usable as reported. Qualification was not required. The particulate matter samples were analyzed by DataChem Laboratories, Inc.

**A. Compound Identification and Quantitation**

For the samples that received full (Level IV) data validation, the TSP and PM-10 results were checked against the raw data and verified to be correct as reported.

**CO-LOCATED FIELD SAMPLES**

**The following paragraphs highlight the essential findings of the co-located field samples:**

Duplicate precision was evaluated by calculating the relative percent difference (RPD) between the detected results in the primary sample and its associated co-located sample. The control limit used for co-located samples was a relative percent difference less than or equal to 50 percent. Or, for those analytes that were at or near the detection limit, an acceptance limit of the absolute difference of the two sample results must be less than the analyte reporting limit. Table C3 presents the results of the co-located project samples. All samples met a 50% RPD acceptance limit, with the following exceptions:

1. The RPD between the dioxin/furan TEQ values failed the 50% acceptance limit at 200% in the co-located sample pair BA2PPUFF5 (2301-002-SA) and BA2CPUFF7 (2301-003-SA).
2. The RPD failed the 50% acceptance limit in co-located sample pair BA1PQ0142653 (03-33621) and BA1CQ0116295 (03-33622) for copper (70%) and zinc (200%). Review of the sample results within the sample delivery group shows a possible sample mix-up as the cause of the RPD failures.

The analysis of co-located samples is a measure of both field and analytical precision. The imprecision in the results in the co-located pair listed above may be due to the sample matrix, sampling or laboratory technique, or method defects. With the exceptions noted above, the results between the co-located pairs matched very well. Since the effect on the quality of the data is not known, data is not qualified for co-located duplicate failure.

**VALIDATION SUMMARY AND DATA USABILITY**

The quality control criteria were reviewed, and other than those discussed above, all criteria were met and the data are considered acceptable. Estimated sample results (J/UJ) are usable only for limited purposes. Based upon the cursory and full validation all other results are considered valid and usable for all purposes. In general, the absence of rejected data indicates high usability.

**VALIDATION QUALIFIERS IDENTIFICATION**

The definitions of the following qualifiers are prepared according to the document, "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October, 1999.

- U      The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
  
- J      The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
  
- N      The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
  
- NJ     The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
  
- UJ     The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
  
- R      The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.