Appendix G

Data Validation Reports
(Volume 1 of 2)

Former Gulf States Creosoting Site
Hattiesburg, Mississippi

ENVIRONMENTAL STANDARDS

Setting the Standards for Innovative Environmental Solutions

December 1, 1998

Mr. David Upthegrove
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1100 Poydras Street
New Orleans, LA 70163

Dear Mr. Upthegrove:

Enclosed is the quality assurance review for the samples collected on October 13 and 14, 1998, as part of the Gulf States Creosoting project. The samples were grouped by the laboratory into Sample Delivery Group (SDG) HMS07 and were collectively analyzed for volatile organic compounds, semivolatile organic compounds, and polyaromatic hydrocarbons (PAH).

Overall, the data quality is acceptable. However, a portion of the organic data has been qualified due to calibration issues, low surrogate recoveries, low matrix spike/matrix spike duplicate recoveries, and quantitation of results below the quantitation limit.

If you have any questions/comments, or if I can be of further assistance, please feel free to call.

Sincerely,

Kendra K Grega

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Senior Quality Assurance Chemist II

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Enc.

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Setting the Standards for Innovative
Environmental Solutions

QUALITY ASSURANCE REVIEW OF SAMPLES

COLLECTED FOR GULF STATES CREOSOTING

December 1, 1998

Prepared for:

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TABLE OF CONTENTS

Introduction

This quality assurance review is based upon a rigorous examination of the data generated from the samples collected on October 13 and 14, 1998, as part of the Gulf States Creosoting project. The samples that have undergone the quality assurance review are presented on Table 1.

This review has been performed with guidance from the "National Functional Guidelines for Organic Data Review" (United States Environmental Protection Agency [US EPA], 2/94).

The reported analytical results are presented in Section 2. Data were examined to determine the usability of the analytical results and compliance relative to requirements specified in the analytical methods. Qualifier codes have been placed next to the results so the data user can quickly assess the qualitative and/or quantitative reliability of any result. This critical QA review identifies data quality issues for specific samples and specific evaluation criteria. The data qualifications allow the data end-user to best understand the usability of the analytical results. It should be understood that data not qualified in this report should be considered valid based on the quality control (QC) criteria that have been reviewed. Details of this QA review are presented in the narrative section of this report. This report was prepared to provide a critical review of the laboratory analyses and reported analytical results. Rigorous QA reviews of laboratory-generated data routinely identify various problems associated with analytical measurements, even from the most experienced and capable laboratories.

Section 1 Quality Assurance Review

A. Organic Data

The organic analysis of 21 aqueous samples (including QC samples, dilutions, trip blanks and rinsate blanks) was performed by Lancaster Laboratories, Lancaster, Pennsylvania. Seven samples were analyzed for volatile organic compounds by SW-846 Method 8260B; 20 samples were analyzed for semivolatile organic compounds by SW-846 Method 8270C and PAH by SW-846 8310, as indicated on Table 1. The analytical results are presented in Section 2 of this report.

The findings in this report are based upon a rigorous review of sample holding times, blank analysis results, laboratory control sample (LCS) recoveries, matrix spike and matrix spike duplicate recoveries, sample dilution results, surrogate recoveries, gas chromatography/mass spectroscopy (GC/MS) instrument mass tuning, calibrations, sample preparation, internal standard performance, analytical sequence, surrogate retention time shifts, and the quantitation of positive results. A few deficiencies were identified during the validation of this data set.

In the Data Support Documentation (Section 3) of this report, the data reviewer has included copies of all relevant raw data, QC forms, and other documentation needed to support any changes made to the data package. It should be emphasized that the following items do not necessarily affect data usability. Usability issues are addressed in a subsequent section. This report has been prepared according to sections that provide information that apply to specific analyses performed on the project samples.

Correctable Deficiencies

1. The laboratory incorrectly reported a sample volume of 935mls for sample W19-- (Kerr McGee sample number MW-19) on the Case Narrative for the semivolatiles fraction. According to the associated Organic Extraction Batchlog, the correct sample volume is 985mls. There is no impact on data quality due to this deficiency because the laboratory used the correct volume to calculate all sample results. The Environmental Standards data reviewer has corrected the aforementioned Case Narrative included in the Project Case Narratives and Chain-of-Custody Records (Section 4).

2. In the PAH fraction, the laboratory incorrectly reported on the analysis data sheet and data tables that fluorene was not detected in sample MW-09. According to the associated raw data, fluorene was detected in this sample at a level of 93 µg/L. The Environmental Standards data reviewer has corrected the associated analysis data sheet included in the Organic Data Support Documentation (Section 3). In addition, the data tables have been modified to reflect the correct data (including the associated flagcode).

3. In the PAH fraction, the laboratory incorrectly reported a result of 15.5 µg/L for fluorene for sample MW-06 on the analysis data sheet and data tables. Specifically, the laboratory incorrectly reported the fluorene result from the diluted analysis of this sample instead of the undiluted analysis (i.e., 15.1 µg/L), which is within the calibration range of the instrument. Consequently, the detection limit for fluorene was also incorrectly reported because it was adjusted to reflect the diluted analysis. The Environmental Standards data reviewer has corrected the associated analysis data sheet included in the Organic Data Support Documentation (Section 3). In addition, the data tables have been modified to reflect the correct data (including the associated flagcode).

4. In the PAH fraction, the laboratory incorrectly flagged several surrogate recoveries for samples MW-06, MW-09, and MW-19 with a "D" on the Water Pesticide Surrogate Recovery form (FORM II PEST-1); the "D" flags indicate that the surrogates were diluted out when, in fact, these samples were not analyzed at a dilution. There is no impact on data quality due to this deficiency. The Environmental Standards data reviewer has corrected the aforementioned form included in the Organic Data Support Documentation (Section 3).

5. In the PAH fraction, the laboratory incorrectly reported a surrogate recovery of 0% for nitrobenzene in samples MW-06DL, MW-09DL, and MW-19DL on the Water Pesticide Surrogate Recovery form (FORM II PEST-1). According to the associated raw data, the correct recoveries are 66%, 71%, and 60%, respectively. There is no impact on data quality due to this deficiency because the correct recoveries are within the acceptance criteria. The Environmental Standards data reviewer has corrected the aforementioned form included in the Organic Data Support Documentation (Section 3).

6. In the PAH fraction, the laboratory incorrectly reported several surrogate retention times on the Pesticide Analytical Sequence forms (FORM VIII PESTs) for instrument P1562A. In some instances, no surrogate retention times were reported. The Environmental Standards data reviewer has corrected the aforementioned forms included in the Organic Data Support Documentation (Section 3) and has included several examples of the raw data showing the correct retention times.

7. In the PAH fraction, the laboratory made several errors in reporting the acenapthene and fluorene retention time data on the Pesticide Initial Calibration – Retention Time Summary (FORM VI PEST-1) for the initial calibration performed on 10/20/98 on instrument P1562A. There is no impact on data quality due to this deficiency. The Environmental Standards data reviewer has corrected the aforementioned form included in the Organic Data Support Documentation (Section 3) and has included an example of the raw data reflecting the correct retention times.

8. In the PAH fraction, the laboratory incorrectly reported the "calculated amount" and the "%D" for benzo(k)fluoranthene on the Pesticide Calibration Verification Summary (FORM VII PEST-2) for the calibration verification analyzed on 10/24/98 at 22:17 on instrument P1562B. There is no impact on data quality due to this deficiency. The Environmental Standards data reviewer has corrected the aforementioned form included in the Organic Data Support Documentation (Section 3)

Comments

1. It should be noted that matrix spike and matrix spike duplicate samples were not collected for the volatiles fraction. The laboratory included a matrix spike and matrix spike duplicate which was performed on a non-project sample.

2. It should be noted that the Chain-of-Custody Record specifies that SW-846 Method 8240 be used for analysis of the volatiles fraction. However, the laboratory actually used SW-846 Method 8260B for the analyses, which is the current promulgated SW-846 method for the GC/MS analysis for volatiles.

3. As noted in the Case Narrative for the semivolatiles fraction, reduced volumes were used in the extraction of samples MW-05, MW-09, MW-19, MW-08, MW-06, MW-13, and MW-23 due to insufficient sample volume. In addition, although not noted in the Case Narrative for the PAH fraction, reduced-volume sample extracts were also used in the PAH analyses.

4. As noted in the Case Narratives for the semivolatiles and PAH fractions, samples MW-09, MW-19, and MW-06 were reanalyzed at dilutions due to the presence of target compounds which exceeded the calibration range of the instrument in the initial analyses. The laboratory reported one set of results from all analyses; however, the raw data for all analyses were provided. The laboratory reported the results for target compounds whose concentrations exceeded the calibration range in the initial analyses from the secondary dilution analyses. All target compounds that were within the calibration range in the initial analyses were reported from those analyses. The data reviewer has only qualified the results reported by the laboratory.

5. As noted in the Case Narrative for the PAH fraction, the laboratory quantitated the surrogate compounds using the UV detector, which is indicated as column 1 or as instrument P1562A on the surrogate QC summary forms. Therefore, the surrogate recoveries reported for the alternate detector (indicated as column 2) on the Water Pesticide Surrogate Recovery forms (FORM II PEST-1's) are meaningless and were not used to assess data quality. In addition, the retention times reported for the alternate detector

(indicated as instrument P1562B) on the Pesticide Analytical Sequence forms (FORM VIII PESTs) are meaningless and were not used to assess data quality.

6. As noted in the Case Narrative for the PAH fraction, the triphenylene surrogate recoveries in samples MW-01, MW-05, MW-06, MW-07, MW-08, MW-13 and MW-23 were outside the acceptance criteria. The impact on data quality is addressed in the subsequent data qualifiers section.

7. In the PAH fraction, it should be noted that the initial calibration dates reported on the Pesticide Calibration Verification Summaries (FORM VII PEST-2's) actually reflect the dates that the laboratory updated the retention time windows and not necessarily the dates of the associated initial calibration.

8. In the PAH fraction, it should be noted that the retention time for the surrogate triphenylene in sample MW-11 and the retention times for the surrogate nitrobenzene in samples MW-09DL, MW-19DL, and MW-06DL were outside the established retention time windows. SW-846 Method 8000B (Section 7.6.8) states that whenever the observed retention time of a surrogate is outside the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses. It does not appear that the analyst did this. The Environmental Standards data reviewer reviewed the raw data with expanded retention time windows and verified that no PAH compounds were misidentified or misreported as "not-detected" in these samples due to the retention time shift.

9. Data usability based on the LCS and matrix spike/matrix spike duplicate analyses was evaluated utilizing the laboratory-generated precision and accuracy limits.

10. The laboratory reported "not-detected" results down to the method detection limits (MDLs). In addition, positive results less than the quantitation limit, but greater than the MDL, were qualified by the laboratory as estimated ("J").

With regard to data usability, the principal areas of concern are calibration issues, low surrogate recoveries, low matrix spike/matrix spike duplicate recoveries, and results reported at concentrations below the quantitation limit. Based upon a review of the data package provided, the following data qualifiers are offered. (It should be noted that the following data usability issues represent an interpretation of the QC results obtained for the project samples. Quite often, data qualifications address issues relating to sample matrix problems. Similarly, the validation guidelines routinely specify areas of the data that require qualification, yet the methods used for analysis do not require any corrective action by the laboratory. Accordingly, the following data usability issues should <u>not</u> necessarily be construed as an indication of laboratory performance.)

Organic Data Qualifiers

- The analyses for acetone in all samples in SDG HMS07 are unusable; consequently, the "not-detected" results have been flagged "R" on the data tables. Very low (<0.05) relative response factors (RRFs) were observed for these compounds in the associated initial multipoint calibration standards and continuing calibration standards.

- Although there is no direct reason to question the reported positive result for bis(2-ethylhexyl)phthalate in sample MW-07, this compound is a very common laboratory contaminant. In addition, the reported result for bis(2-ethylhexyl)phthalate in sample MW-07 represents a low-level, on-column detection. Accordingly, extreme caution should be exercised if this result is to be used in a decision-making process, such as risk assessment.

- In the PAH fraction, the reported positive results for benzo(a)pyrene in samples MW-05 and MW-19 should be considered estimated and have been flagged "J' on the data tables. In addition, the actual reporting limits for benzo(a)pyrene in samples MW-01, MW-09, MW-08, MW-12, MW-10, MW-06, and MW-07 and for benzo(g,h,i)perylene in all samples except MW-03 and MW-04 may be higher than reported; consequently, the "not-detected" results have been flagged "UJ" on the data tables. High percent drifts ($15\% < \%D \leq 90\%$) coupled with increases in instrument sensitivity were observed for benzo(g,h,i)perylene and benzo(a)pyrene in the associated calibration verifications. It should be noted that although the reporting limits have been qualified according to protocol, these high percent drifts represent increases in instrument sensitivity. Consequently, the reporting limits may be valid as reported.

- All reported positive results for PAH compounds in samples MW-01, MW-05, MW-06, MW-07, MW-08, MW-13 and MW-23, with the exception of napthalene in sample MW-06, should be considered estimated and have been flagged "J" on the data tables. In addition, the actual reporting limits for all PAH compounds in samples MW-01, MW-05, MW-06, MW-07, MW-08, MW-13 and MW-23 may be higher than reported; consequently, the "not-detected" results have been flagged "UJ" on the data tables. Low percent recoveries ($10\% \leq \%R < 60\%$) were observed the for surrogate compound triphenylene in the analyses of these samples.

- In the PAH fraction, the reported positive result for pyrene in sample MW-04 should be considered estimated and has been flagged "J" on the data tables. A low percent recovery ($10\% \leq \%R < 51\%$) was observed for pyrene in the associated matrix spike and matrix spike duplicate analyses of this sample.

- As per reporting conventions, all positive results reported below the sample-specific reporting limits should be considered estimated and have been flagged "J" on the data tables.

A complete support documentation of this organic data QA review is presented in Section 3 of this report.

B. Conclusions

This QA review has identified several aspects of the analytical data that required qualification. The majority of the data are acceptable. However, a portion of the organic data has been qualified due calibration issues, low surrogate recoveries, low matrix spike/matrix spike duplicate recoveries and results reported at concentrations below the quantitation limit. To confidently use any of the analytical data within these sample sets, the data user should understand the qualifications and limitations of the results.

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TABLE 1

SAMPLES INCLUDED IN THIS QUALITY ASSURANCE REVIEW

Kerr-McGee Corporation Sample Number	Laboratory Sample Number	SDG Number	Date of Sample Collection	Parameter(s) Analyzed
MW-03	3018865	HMS07	10/14/98	SVOA, PAH
MW-01	3018866	HMS07	10/14/98	SVOA, PAH
MW-05	3018867	HMS07	10/14/98	SVOA, PAH
MW-09	3018868	HMS07	10/13/98	SVOA, PAH
MW-09DL (Dilution of MW-09)	3018868DL	HMS07	10/13/98	SVOA, PAH
MW-19	3018869	HMS07	10/13/98	SVOA, PAH
MW-19DL (Dilution of MW-19)	3018869DL	HMS07	10/13/98	SVOA, PAH
MW-08	3018870	HMS07	10/13/98	SVOA, PAH
MW-04	3018871	HMS07	10/14/98	SVOA, PAH
MW-04MS (Matrix Spike)	3018872	HMS07	10/14/98	SVOA, PAH
MW-04MSD (Matrix Spike Duplicate)	3018873	HMS07	10/14/98	SVOA, PAH
MW-12	3018874	HMS07	10/13/98	VOA, SVOA, PAH
MW-10	3018875	HMS07	10/13/98	VOA, SVOA, PAH
MW-06	3018876	HMS07	10/13/98	SVOA, PAH
MW-06DL (Dilution of MW-06)	308876DL	HMS07	10/13/98	SVOA, PAH

TABLE 1 (Cont.)

Kerr-McGee Corporation Sample Number	Laboratory Sample Number	SDG Number	Date of Sample Collection	Parameter(s) Analyzed
MW-07	3018877	HMS07	10/13/98	SVOA, PAH
MW-13	3018878	HMS07	10/14/98	VOA, SVOA, PAH
MW-23	3018879	HMS07	10/14/98	VOA, SVOA, PAH
TB-1 (Trip Blank)	3018880	HMS07	10/14/98	VOA
RB-1 (Rinsate Blank)	3018881	HMS07	10/14/98	VOA, SVOA, PAH
MW-11	3018882	HMS07	10/14/98	VOA, SVOA, PAH

NOTES:

SVOA - Semivolatile organic compounds by SW-846 Method 8270C.
VOA - Volatile organic compounds by SW-846 Method 8260B.
PAH - Polyaromatic hydrocarbons by SW-846 Method 8310.