

CONTRACT COMPLIANCE SCREENING FORM FOR HARDCOPY DATA

AMEC Earth & Environmental
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Laboratory Alta

Reviewer K. Shadowlight

Analysis/Method Dioxins

Package ID T711DF39

Task Order 313150010

SDG No. IOA0364

No. of Analyses 1

Date: February 11, 2005

Reviewer's Signature

K. Shadowlight

ACTION ITEMS^a

1. **Case Narrative**
Deficiencies

2. **Out of Scope**
Analyses

3. **Analyses Not Conducted**

4. **Missing Hardcopy**
Deliverables

5. **Incorrect Hardcopy**
Deliverables

6. **Deviations from Analysis**
Protocol, e.g.,

Holding Times

GC/MS Tune/Inst. Performance

Calibration

Method blanks

Surrogates

Matrix Spike/Dup LCS

Field QC

Internal Standard Performance

Compound Identification and

Quantitation

System Performance

Qualifications were assigned for the following:

* Method blank contamination

* Detects below the lower method calibration level

COMMENTS^b

^a Subcontracted analytical laboratory is not meeting contract and/or method requirements.

^b Differences in protocol have been adopted by the laboratory but no action against the laboratory is required.

Data Qualifier Reference Table

Qualifier	Organics	Inorganics
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.	The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.	The associated value is an estimated quantity.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."	Not applicable.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	Not applicable.
UJ	The analyte was not deemed 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.	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.	The data are unusable. (Note: Analyte may or may not be present).

Qualification Code Reference Table

Qualifier	Organics	Inorganics
H	Holding times were exceeded.	Holding times were exceeded.
S	Surrogate recovery was outside QC limits.	The sequence or number of standards used for the calibration was incorrect
C	Calibration %RSD or %D were noncompliant.	Correlation coefficient is <0.995.
R	Calibration RRF was <0.05.	%R for calibration is not within control limits.
B	Presumed contamination from preparation (method) blank.	Presumed contamination from preparation (method) or calibration blank.
L	Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits.	Laboratory Control Sample %R was not within control limits.
Q	MS/MSD recovery was poor or RPD high.	MS recovery was poor.
E	Not applicable.	Duplicates showed poor agreement.
I	Internal standard performance was unsatisfactory.	ICP ICS results were unsatisfactory.
A	Not applicable.	ICP Serial Dilution %D were not within control limits.
M	Tuning (BFB or DFTPP) was noncompliant.	Not applicable.
T	Presumed contamination from trip blank.	Not applicable.
+	False positive – reported compound was not present. Not applicable.	
-	False negative – compound was present but not reported.	Not applicable.
F	Presumed contamination from FB, or ER.	Presumed contamination from FB or ER.
\$	Reported result or other information was incorrect.	Reported result or other information was incorrect.
?	TIC identity or reported retention time has been changed.	Not applicable.
D	The analysis with this flag should not be used because another more technically sound analysis is available.	The analysis with this flag should not be used because another more technically sound analysis is available.
P	Instrument performance for pesticides was poor.	Post Digestion Spike recovery was not within control limits.
DNQ	The compound was detected between the MDL and the RL and, by definition, is considered an estimated value.	The compound was detected between the MDL and the RL and, by definition, is considered an estimated value.

*#

Unusual problems found with the data that have been described in Section 2.#, "Data Validation Findings." The number following the asterisk (*) will indicate the subsection where a description of the problem can be found (eg. *1 would indicate a sample was not within temperature limits).

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DATA VALIDATION REPORT

NPDES Monitoring

ANALYSIS: DIOXINS/FURANS

SAMPLE DELIVERY GROUPS: IOA0364

Prepared by

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1. INTRODUCTION

Task Order Title: NPDES Monitoring
Contract Task Order #: 313150010
Sample Delivery Group #: IOA0364
Project Manager: B. McIlvaine
Matrix: Water
Analysis: Dioxins/Furans
QC Level: Level IV
No. of Samples: 1
No. of Reanalyses/Dilutions: 0
Reviewer: K. Shadowlight
Date of Review: February 11, 2005

The samples listed in Table 1 were validated based on the guidelines outlined in the *AMEC Data Validation Procedure for Dioxins and Furans (DVP-19, Rev. 1)*, *EPA Method 1613*, and the *National Functional Guidelines For Chlorinated Dioxin/Furan Data Review (8/02)*. Any deviations from these procedures and guidelines are documented herein. Qualifiers were applied in cases where the data did not meet the required QC criteria or where special consideration by the data user is required. Data qualifiers were placed on Form Is with the associated qualification codes. Analytes that were rejected for any reason are denoted on the Form I as having only the "R" data qualifier and associated qualification code(s) denoting the reason for rejection. Any additional problems with the data that may have resulted in an estimated value were not denoted by a qualification code since the data had already been rejected.

Table 1. Sample Identification

Client ID	Laboratory ID (Del Mar)	Laboratory ID (Pace)	Matrix	COC Method
AMB	IOA0364-01	105965001	water	1613

2. DATA VALIDATION FINDINGS

2.1 SAMPLE MANAGEMENT

Following are findings associated with sample management:

2.1.1 Sample Preservation, Handling, and Transport

The sample in this SDG was received at Del Mar Analytical within the temperature limits of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The sample in this SDG was received at Pace Analytical Services below the temperature limits of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$; however, as the sample was noted to have been damaged, no qualifications were required. The sample was received in good condition at both laboratories. No qualifications were required.

2.1.2 Chain of Custody

The COC and transfer COC were signed by the appropriate field and laboratory personnel, and accounted for the analysis presented in this SDG. As the sample was couriered directly to the laboratory (Del Mar Analytical), custody seals were not required. There was no information regarding custody seals upon receipt at Pace. No qualifications were required.

2.1.3 Holding Times

The sample was extracted and analyzed within a year of collection. No qualifications were required.

2.2 INSTRUMENT PERFORMANCE

Following are findings associated with instrument performance:

2.2.1 GC Column Performance

A column performance standard was combined with the daily calibration verification and analyzed at the beginning of each analytical sequence. The GC column performance was acceptable with the chromatographic separation of 2,3,7,8-TCDD and other TCDD isomers resolved with a valley of $\leq 25\%$. No qualifications were required.

2.2.2 Mass Spectrometer Performance

The mass spectrometer performance could not be evaluated as the laboratory did not provide selected ion current profiles for the lock-mass ions. No qualifications were required.

2.3 CALIBRATION

2.3.1 Initial Calibration

There was one initial calibration, analyzed 11/29/04 on Instrument 10MSHR05. The calibration consisted of five concentration level standards (CS1 through CS5) analyzed to verify instrument linearity. The initial calibration was acceptable with %RSDs $\leq 20\%$ for the 15 native compounds (calibration by isotope dilution) and $\leq 35\%$ for the 2 native and all labeled compounds (calibration by internal standard). The relative retention times and ion abundance ratios were within the QC limits listed in Method 1613 for all standards. A representative number of %RSDs were verified from the raw data, and no calculation or transcription errors were noted. No qualifications were required.

2.3.2 Continuing Calibration

Calibration verification (VER) consisted of a mid-level standard (CS3) analyzed at the beginning of each analytical sequence. The VER was acceptable with the concentrations within the acceptance criteria listed in the Table 6 of the EPA Method 1613. The ion abundance ratios and relative retention times were within the method QC limits. A representative number of %Ds were verified from the raw data, and no calculation or transcription errors were noted. No qualifications were required.

2.4 BLANKS

One method blank (Blank-6202) was extracted and analyzed with this SDG. Target compounds 1,2,3,4,6,7,8-HpCDD, total HpCDD, OCDF, and OCDD were reported in the method blank. Any detects for the aforementioned target compounds reported at concentrations $< 5\times$ the concentrations reported in the method blank were qualified as estimated nondetects "UJ," at the levels of interference in the sample of this SDG. A review of the method blank raw data and chromatograms indicated no false negatives or false positives. No further qualifications were required.

2.5 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

One LCS/LCSD pair (LCS-6203/LCSD-6204) was extracted and analyzed with this SDG. All recoveries were within the acceptance criteria listed in Table 6 of the Method 1613. There were no QC limits established for RPDs. The reported RPDs were within $\pm 20\%$. No qualifications were required.

2.6 MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD analyses were not performed in this SDG. Evaluation of method accuracy and precision was based on the LCS/LCSD results. No qualifications were required.

2.7 FIELD QC SAMPLES

Following are findings associated with field QC:

2.7.1 Field Blanks and Equipment Rinsates

The samples in this SDG had no associated field QC samples. No qualifications were required.

2.7.2 Field Duplicates

No field duplicate samples were identified for this SDG.

2.8 INTERNAL STANDARDS

The labeled standard recoveries were within the acceptance criteria listed in Table 7 of Method 1613. No qualifications were required.

2.9 COMPOUND IDENTIFICATION

The laboratory analyzed for polychlorinated dioxins/furans by EPA Method 1613. The compound identifications were verified from the raw data and no false negatives or positives were noted. No qualifications were required.

2.10 COMPOUND QUANTIFICATION AND REPORTED DETECTION LIMITS

Compound quantitation was verified from the raw data. The laboratory calculated and reported compound-specific detection limits. Any detects below the lower method calibration limit (MCL) were qualified as estimated, "J." Any reported EMPC was qualified as an estimated nondetect, "UJ." No further qualifications were required.

Method 1613B Analysis Results

Client - Del Mar Analytical

Client's Sample ID I0A0364-01
Lab Sample ID 105965001
Filename F50127B_06
Injected By MRO
Total Amount Extracted 1010 mL
% Moisture NA
Dry Weight Extracted NA
ICAL Date 11/29/2004
CCal Filename(s) F50127A_13
Method Blank ID BLANK-6202

Matrix Water
Dilution NA
Collected 01/07/2005
Received 01/11/2005
Extracted 01/24/2005
Analyzed 01/28/2005 01:08

Raw Qual	Qual Lab	Native Isomers	Conc pg/L	EMPC pg/L	LOD pg/L	Internal Standards	ng's Added	Percent Recovery
u		2,3,7,8-TCDF	ND	----	3.4	2,3,7,8-TCDF-13C	2.00	49
u		Total TCDF	ND	----	3.4	2,3,7,8-TCDD-13C	2.00	62
						1,2,3,7,8-PeCDF-13C	2.00	82
u		2,3,7,8-TCDD	ND	----	3.5	2,3,4,7,8-PeCDF-13C	2.00	79
		Total TCDD	ND	----	3.5	1,2,3,7,8-PeCDD-13C	2.00	91
						1,2,3,4,7,8-HxCDF-13C	2.00	74
u		1,2,3,7,8-PeCDF	ND	----	1.8	1,2,3,6,7,8-HxCDF-13C	2.00	101
u		2,3,4,7,8-PeCDF	ND	----	2.0	2,3,4,6,7,8-HxCDF-13C	2.00	95
u		Total PeCDF	ND	----	1.9	1,2,3,7,8,9-HxCDF-13C	2.00	83
						1,2,3,4,7,8-HxCDD-13C	2.00	71
u		1,2,3,7,8-PeCDD	ND	----	2.9	1,2,3,6,7,8-HxCDD-13C	2.00	96
u		Total PeCDD	ND	----	2.9	1,2,3,4,6,7,8-HpCDF-13C	2.00	83
						1,2,3,4,7,8,9-HpCDF-13C	2.00	78
u		1,2,3,4,7,8-HxCDF	ND	----	1.6	1,2,3,4,6,7,8-HpCDD-13C	2.00	100
u		1,2,3,6,7,8-HxCDF	ND	----	1.5	OCDD-13C	4.00	97
u		2,3,4,6,7,8-HxCDF	ND	----	1.2			
u		1,2,3,7,8,9-HxCDF	ND	----	2.1	1,2,3,4-TCDD-13C	2.00	NA
u		Total HxCDF	ND	----	1.6	1,2,3,7,8,9-HxCDD-13C	2.00	NA
u		1,2,3,4,7,8-HxCDD	ND	----	1.9	2,3,7,8-TCDD-37Cl4	0.20	58
u		1,2,3,6,7,8-HxCDD	ND	----	1.6			
u		1,2,3,7,8,9-HxCDD	ND	----	1.6			
J	DNQ	Total HxCDD	2.4	----	1.7 J			
J	DNQ	1,2,3,4,6,7,8-HpCDF	5.5	----	1.8 J			
u		1,2,3,4,7,8,9-HpCDF	ND	----	2.4			
J	DNQ	Total HpCDF	15.0	----	2.1 J			
u	B	1,2,3,4,6,7,8-HpCDD	9.2	----	1.8 BJ			
J	DNQ	Total HpCDD	20.0	----	1.8 J			
u	B	OCDF	11.0	----	2.8 BJ			
u	B	OCDD	45.0	----	3.3 BJ			

Conc = Concentration (Totals include 2,3,7,8-substituted isomers).
EMPC = Estimated Maximum Possible Concentration
LOD = Limit of Detection. Totals are averages of individual isomer LODs.
D = Result obtained from analysis of diluted sample
B = Less than 10 times higher than method blank level
P = Recovery outside of method 1613 control limits
J = Concentration detected is below the calibration range
Nn = Value obtained from additional analysis

I = Interference
E = PCDE Interference
ND = Not Detected
NA = Not Applicable
NC = Not Calculated
* = See Discussion

Report No.....105965

REPORT OF LABORATORY ANALYSIS

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LEVEL IV