Deepwater Horizon – Data Validation Report
PAH & Alkylated Homologs by GC/MS-SIM

SDG: 1005013	**Matrix:** Water	**Number of Samples:** 16
Laboratory: Alpha Analytical Services		**Method/SOP:** Alpha SOP/0-008 (Issue 6)
Validation Level: Stage 4 Validation		**Validation Criteria Table:** MC252-PAH, Rev. 0
Date of Report: July 23, 2010		**Approved for Release:** cn=Linda Bohannon, o=EcoChem, Inc., ou, email=lbohannon@ecochem.net, c=US 2010.07.23 10:00:38 -07'00'

Refer to the SAMPLE INDEX for a list of validated samples.

Refer to the DATA VALIDATION PLAN for validation approach, Criteria Tables, qualifier and reason code definitions.

The quality control (QC) elements that were reviewed are listed below.

√	Data Package Completeness	**1**	Sample Duplicate Analysis
√	Verification of EDD to Hardcopy Data Package	**2**	Blank Spike/Blank Spike Duplicate Sample Analyses
1	Chain-of-Custody and Sample Receipt	**1**	Reference Material Analysis
√	Holding Times	√	Internal Standards
√	Instrument Tuning	√	Detection Limits
√	Initial Calibration	√	Target Analyte List
√	Initial Calibration Verification	**2**	Compound Quantitation
√	Continuing Calibration	√	Compound Identification/Spectral Match (Stage 4 only)
2	Method Blank Analysis	**1**	Mass Discrimination (Stage 4 only)
√	Surrogate Compound Recovery	**1**	Calculation Verification (Stage 4 only)

√ **Stated method quality objectives (MQO) and QC criteria have been met. No outliers are noted or discussed.**

1 Quality control results are discussed below, but no data were qualified.

2 Quality control outliers that impact the reported data were noted. Data qualifiers were issued as discussed in this Data Validation Report.

Overall Assessment

Data were qualified as not detected or estimated based on method blank contamination. Data were also estimated due to blank spike/blank spike duplicate recovery outliers and matrix interferences.

All data, as qualified, are acceptable for use.

Data Package Completeness

The laboratory followed adequate corrective action processes and all anomalies were discussed in the case narrative. The laboratory submitted all required deliverables.

Verification of EDD to Hardcopy Data Package

Sample results and related quality control data were received in both an electronic and hardcopy format. Electronic data were verified against the laboratory report; no errors were found.

Chain-of-Custody and Sample Receipt

Sample identification (ID) numbers listed on the chain-of-custody record are consistent with the sample ID reported in the laboratory electronic data deliverable (EDD) and hardcopy data package, with one exception. Sample JF.REF.BLANK.20100510.N007 (laboratory sample number 1005013-04) was mistyped. The sample name was corrected in the database.

With the exceptions noted below, samples were received within the advisory temperature range of 2° to 6°C (Analytical Quality Assurance Plan (AQAP), Section 3.1). The laboratory noted all sample conditions on the sample receipt form.

The laboratory received several of the sample coolers with temperatures greater than the upper advisory limit, ranging to 9.6°C. These outliers did not impact data quality and no qualifiers were required. All discrepancies and reconciliation were discussed in the analytical report narrative.

Holding Times

Samples were analyzed within the holding time specified in the Analytical AQAP, Section 3.1, and documented in the Validation Criteria Table.

Instrument Tuning

Instrument tuning was performed at the required frequency and met all criteria as specified in the referenced laboratory analytical SOP.

Initial Calibration

Initial calibration (ICAL) standards were analyzed at the required frequency and the percent difference (%RSD) values were within the control limits specified in the AQAP, Table 6.1a and documented in the Validation Criteria Table.

Initial Calibration Verification

Initial calibration verification (ICV) standards were analyzed required frequency and the percent recovery (%R) values were within the control limits specified in the AQAP, Table 6.1a and documented in the Validation Criteria Table. The ICV was a separate standard prepared from a second source.

Continuing Calibration

Continuing calibration (CCAL) standards were analyzed at the required frequency and the percent difference (%D) values were within the control limits specified in the AQAP, Table 6.1a and documented in the Validation Criteria Table.

Method Blank Analysis

To assess the impact of each blank contaminant on the reported sample results, two action levels are established at two (2x) and five times (5x) the concentration reported in the blank (ten times for common laboratory contaminants). If a contaminant is reported in an associated field sample and the concentration is less than the lower (2x) action level, the result is qualified as not detected (U-7). If a contaminant is reported in an associated field sample and the concentration is less than the higher (5x) action level, the result is qualified as estimated (J-7). If the result is also less than the reporting limit, then the result is elevated to the reporting limit. No action is taken if the sample result is greater than the higher action level, or for non-detected results.

The following table summarizes the method blank contamination and action levels:

Blank ID	Analyte	Result (ng/L)	Action Limits (ng/L)	
			2x	5x
SW051710B01	naphthalene	0.226	0.452	1.13
	fluorene	0.214	0.428	1.07
	phenanthrene	0.182	0.364	0.910

Surrogate Compound Recovery

The percent recovery (%R) values for surrogates were within the control limits of 40% – 120% (control limits for d12-perylene are 10% – 120%).

Sample Duplicate Analysis

There was no sample duplicate analyzed with this analytical batch. Precision was evaluated using the blank spike duplicate analysis.

Blank Spike/Blank Spike Duplicate Sample Analyses

One set of blank spike/blank spike duplicate (BS/BSD) samples (for each analytical batch of 20 or fewer samples) was extracted and analyzed. The percent recovery (%R) and relative percent difference (RPD) values were calculated and evaluated.

With the exceptions noted below, the %R values were within the criteria of 50% – 130%.

Analyte	BS %R	BSD %R	Potential Bias
Benzo(a)anthracene	133	133	High
Benzo(a)pyrene	137	137	High
Chrysene/Triphenylene	127	131	High

Analyte	BS %R	BSD %R	Potential Bias
Indeno(1,2,3-cd)pyrene	126		High
Naphthalene		128	High

For the indeno(1,2,3-cd)pyrene and naphthalene outliers, no action was taken as the recovery for the corresponding BS or BSD was acceptable. Benzo(a)pyrene was not detected in the field samples; no action was necessary. Positive results for benzo(a)anthracene, chrysene/triphenylene, and alkylated chrysenes were estimated (J-10) to indicate a potential high bias.

All RPD values were less than the control limit of ≤30%.

Reference Material Analysis

The reference material SRM 1582 was analyzed. All recoveries were within established limits.

An aliquot of North Slope Crude was analyzed. All recovery values were within the laboratory defined criteria of 65% – 135%.

Internal Standards

The percent recovery (%R) values for internal standards (IS) were within the control limits of 50% – 200% of the area in the associated CCAL.

Compound Quantitation

The laboratory applied a J-flag to all results between the quantitation limit (QL) and the method detection limit (MDL). During validation, results less than the MDL were qualified as "found" (F).

A number of individual analytes (such as 4-methyldibenzothiophene) were not included in the calibration mixture. These analytes use the response factor of a designated 'parent' compound (such as dibenzothiophene) for calculation of the analyte concentration. Because the response factor of the parent compound can be significantly different than that of the individual analyte, all positive results for non-calibrated individual analytes are estimated (J-24).

The laboratory flagged the results for several analytes with a "G", indicating matrix interference that affected quantitation. These "G" flagged results were estimated (J-14).

The C1-dibenzothiophene result for Sample JF.4KM.150FT.WD.20100512.N104 was incorrectly calculated. The laboratory recalculated the result and submitted a revised concentration on 7/20/10. The result in the EDD was corrected by the validator.

Mass Discrimination

The ratio for the raw areas of benzo[g,h,i]perylene to phenanthrene (calculated for the ICAL and CCAL) was ≥ 0.70.

Calculation Verification

Stage 4 validation was performed on this SDG. No transcription or calculation errors were found.

Attachment 1: Sample Index - SDG 1005013
Aliphatic Hydrocarbons by GC/FID

Sample ID	Lab ID	Date Collected
JF.REF.MID.WD.20100511.N023	1005013-01	5/10/2010
JF.REF.SURF.DIWD.20100511.N009	1005013-02	5/10/2010
JF.REF.SURF.WD.20100510.N028	1005013-03	5/10/2010
JF.REF.BLANK.20100510.N007	1005013-04	5/10/2010
JF.REF.BLANK.20100510.N008	1005013-05	5/10/2010
JF.REF.BLANK.20100510.N006	1005013-06	5/10/2010
JF.REF.BLANK.DIWD.20100511.N039	1005013-07	5/11/2010
JF.REF.DEEP.WD.20100511.N015	1005013-08	5/11/2010
JF.REF.MIX.WD.20100511.N031	1005013-09	5/11/2010
JF.REF.8KM.DEEP.WD.20100512.N047	1005013-10	5/12/2010
JF.8KM.300FUZZ.WD.20100512.N055	1005013-11	5/12/2010
JF.8KM.MIX45.WD.20100512.N063	1005013-12	5/12/2010
JF.4KM.SURF.DIWD.20100512.N116	1005013-13	5/12/2010
JF.4KM.360FT.WD.20100512.N096	1005013-14	5/12/2010
JF.4KM.150FT.WD.20100512.N104	1005013-15	5/12/2010
JF.4KM.240FT.WD.20100512.N100	1005013-16	5/12/2010