



**APPENDIX F**  
**Laboratory Analytical Data Quality Assessment**  
**Summary**

## APPENDIX F LABORATORY ANALYTICAL DATA QUALITY ASSESSMENT SUMMARY

### DATA QUALITY ASSESSMENT SUMMARY

TOTAL PETROLEUM HYDROCARBONS BY NWTPH-Gx  
VOLATILE ORGANIC COMPOUNDS BY SW 8260B/SIM

Anatek Laboratory SDG	Samples Validated (Bold indicates the sample was qualified)
120417034 (soil samples)	SVE-1 (5), SVE-1 (10), SVE-1 (20), SVE-2 (15), SVE-2 (25), SVE-2 (35), MP-1 (15), MP-1 (25), MP-1 (35), MP-2 (15), MP-2 (25), AS-1 (10), AS-1 (15), AS-1 (25), AS-1 (35), MW-16 (13.5), B-6 (25), MW-17 (35), TRIP BLANK
120418006 (soil samples)	MSV-18 (20), MW-19 (35), MW-19 (40), TRIP BLANK
111202043 (water samples from direct push)	DP-26-112911, DP-27-112911, <b>DP-28-112911</b> , DP-29-112911, DEV H2O MW-13-14-15,
111202043 (soil samples)	DP-26 (22-23), DP-27 (13-14), DP-27 (15-16), DP-28 (11-12), DP-28 (15-16), DP-28 (17-18), DP-29 (11-12), DP-29 (14-15), DP-30 (33-35), DP-31 (34-35), DP-32 (27-28), DP-32 (36-40), DP-33 (27-28), DP-33 (35-37), DP-34 (7-8), DP-35 (19-20), DP-36 (19-20), DP-37 (19-20), DP-37 (27-28), DP-38 (7-8), DP-38 (15-16), DP-39 (10-12), DP-40 (7-8), TRIP BLANK, MeOH TRIP
120822017 (water sample from domestic water well)	DOMESTIC WELL 6608/6609-0821, TRIP BLANK
120731002 (water sample from domestic water well)	WELL 6610 NEW EAST, WELL 6610 NEW WEST, WELL 6606, WELL6608/6609, TRIP BLANK
120822019 (ground water samples from monitoring wells)	MW-7-082112, <b>MW-10-082112</b> , MW-11-082112, MW-12-082112, MW-15-082112, MW-16-082112, MW-17-082112, MW-18-082112, <b>DUPLICATE-1-082112</b>

This report documents the results of an EPA level 2a data validation of analytical data from the analyses of soil samples and the associated laboratory and field quality control (QC) samples. The review included the following:

- Chain of Custody
- Holding Times
- Surrogates
- Method and Trip Blanks
- Laboratory Control Samples
- Matrix Spikes/Matrix Spike Duplicates
- Laboratory and Field Duplicates

## I. DATA PACKAGE COMPLETENESS

Anatek Labs, Inc., located in Spokane, Washington, analyzed the samples evaluated as part of this data validation review. The laboratory provided all required deliverables for the validation according to the National Functional Guidelines. The laboratory followed adequate corrective action processes and all identified anomalies were discussed in the case narrative.

The following sections discuss the data.

### OBJECTIVE

The objective of the data validation was to review laboratory analytical procedures and QC results to evaluate whether:

- The samples were analyzed using well-defined and acceptable methods that provide detection limits below applicable regulatory criteria;
- The precision and accuracy of the data are well defined and sufficient to provide defensible data; and
- The quality assurance/quality control (QA/QC) procedures utilized by the laboratory meet acceptable industry practices and standards.

The environmental samples were analyzed by one or more of the analytical methods listed in the title of this appendix.

### DATA QUALITY ASSESSMENT SUMMARY

The results for each of the QC elements are summarized below. The data assessment was performed using guidance in the US Environmental Protection Agency (USEPA) Contract Laboratory Program *National Functional Guidelines for Inorganic Data Review* (USEPA 2002) and USEPA Contract Laboratory Program *National Functional Guidelines for Organic Data Review* (USEPA 2008).

#### Chain-of-Custody Documentation

Chain-of-custody (COC) forms were provided with the laboratory analytical reports. There were no anomalies noted on the COC forms; proper COC protocols appear to have been followed for this sampling event.

#### Holding Times

The holding time is defined as the time that elapses between sample collection and sample analysis. Maximum holding time criteria exist for each analysis to help ensure that the analyte concentrations found at the time of analysis reflect the concentration present at the time of sample collection. The holding times for all laboratory analyses were met with one exception:

**SDG 111202043:** (BTEX Compounds) Sample DP-28-112911 was initially analyzed within the holding time of 14 days. However, the positive result for benzene exceeded the linear range of the instrument. For this reason, the sample was diluted and re-analyzed several times, analyzing

benzene outside of the holding time of 14 days. The positive result for benzene was qualified as estimated (J) in this sample.

### Surrogate Recoveries

A surrogate compound is a compound that is chemically similar to the analytes of interest, but unlikely to be found in any environmental sample. Surrogates are used for organic analyses and are added to all samples, standards, and blanks to serve as an accuracy and specificity check of each analysis. The surrogates are added at a known concentration and percent recoveries are calculated following analysis. All surrogate recoveries for field samples were within the laboratory control limits.

### Method and Trip Blanks

Method blanks are analyzed to ensure that laboratory procedures and reagents do not introduce measurable concentrations of the analytes of interest. Method blanks were analyzed with each batch of samples, at a frequency of one per twenty samples. For all sample batches, method blanks for all applicable methods were analyzed at the required frequency.

If a compound was found at a measurable concentration in the method blank, an “action level” for this compound was assigned to the associated batch samples by multiplying the concentration by five. This action level is then multiplied by any dilutions the sample may have gone through in the laboratory extraction process.

Two Trip Blanks, mentioned below were carried with the field sampler to and from the site, and these samples were analyzed to ensure that the transportation environment did not introduce measurable concentrations of the analytes of interest. Trip Blanks are usually analyzed at the frequency of one per every sample cooler.

**SDG 120417034:** (Volatiles) The trip blank acquired on 4/12/12 reported no positive results for any target analytes.

**SDG 120418006:** (Volatiles) The trip blank acquired on 4/17/12 reported no positive results for any target analytes.

**SDG 111202043:** (Volatiles) The trip blank acquired on 11/29/11 reported no positive results for any target analytes. Also, the field blank sample named MeOH TRIP acquired on 12/10/11 reported no positive results for any target analytes.

**SDG 120731002:** (Volatiles) The trip blank acquired on 7/27/12 reported no positive results for any target analytes.

**SDG 120822017:** (Volatiles) The trip blank acquired on 8/21/12 reported no positive results for any target analytes.

### Matrix Spikes/Matrix Spike Duplicates (MS/MSD)

Because the actual analyte concentration in an environmental sample is not known, the accuracy of a particular analysis is usually inferred by performing a matrix spike (MS) analysis. One aliquot

of sample is analyzed in the normal manner, and then a second aliquot of the sample is spiked with a known amount of analyte concentration and analyzed. From these analyses, a percent recovery (%R) is calculated. Matrix spike duplicates (MSD) analyses are generally performed for organic analyses as a precision check. For some organic analytical methods, such as NWTPH-Dx, a laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) sample set is performed in lieu of a MS/MSD analysis.

For inorganics methods, the matrix spike (referred to as a “spiked sample”) is typically followed by a post spike sample if any element recoveries were outside the control limits in the “spiked sample”.

Matrix spike analyses should be performed once per analytical batch or every twenty field samples, whichever is more frequent. The recovery criteria for matrix spikes and laboratory control samples are specified in the laboratory documents as are the relative percent difference values. The frequency requirements were met for all analyses and the %R/RPD (need define RPD) values were within the proper control limits.

#### **Laboratory Control Samples/ Laboratory Control Sample Duplicates (LCS/LCSD)**

A laboratory control sample (LCS) is essentially a blank sample that is spiked with a known amount of analyte concentration and analyzed. It is to be treated much like a MS, without the possibility for matrix interference. As there is no actual sample matrix in the analysis, the analytical expectations for accuracy and precision are usually more rigorous and qualification would apply to all samples in the batch, instead of the parent sample only.

LCS analyses should be performed once per analytical batch or every twenty field samples, whichever is more frequent. The recovery criteria for laboratory control samples are specified in the laboratory documents as are the relative percent difference values. The frequency requirements were met for all analyses, and the %R/RPD values were within the proper control limits.

#### **Field Replicates/Duplicates**

Field duplicate samples were collected and analyzed along with the reviewed sample batches. The duplicate samples were analyzed for the same parameters as the associated parent samples. As mentioned above for the laboratory duplicates the RPD is used as the criteria for assessing precision, unless one or more of the samples used has a concentration greater than five times the reporting limit for that sample, the absolute difference is used instead of the RPD.

**SDG 120822019:** (Volatiles) One set of field duplicates, MW-10-082112 & DUPLICATE-1-082112, was submitted with this SDG. The RPD/absolute difference values for ethylbenzene, o-xylene, and m,p-xylene were greater than the control limits. The positive results for these compounds were qualified as estimated (J) in both samples.

## OVERALL ASSESSMENT

As was determined by this data validation, the laboratory followed the specified analytical methods. Accuracy was acceptable, as demonstrated by the surrogate, LCS/LCSD, and MS/MSD %R values. Precision was acceptable, as demonstrated by the laboratory duplicate, LCS/LCSD and MS/MSD RPD and absolute difference values.

Data was qualified because of a holding time outlier and field duplicate precision outliers.

The data are acceptable for use as qualified.