

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: METALS

SAMPLE DELIVERY GROUP: IOC0456

Prepared by

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1. INTRODUCTION

Task Order Title: NPDES Monitoring
Contract Task Order #: 313150010
SDG#: IOC0456
Project Manager: B. McIlvaine
Matrix: Water
Analysis: Metals
QC Level: Level IV
No. of Samples: 1
No. of Reanalyses/Dilutions: 0
Reviewer: P. Meeks
Date of Review: April 06, 2005

The samples listed in Table 1 were validated based on the guidelines outlined in the *AMEC Data Validation Procedure for Levels III and IV ICP-MS Metals, (DVP-5-A, Rev.0)*, *AMEC Data Validation Procedure for Levels III and IV ICP Metals (DVP-5, Rev. 0)*, *SW-846 Method 6020B for Inductively Coupled Plasma – Mass Spectrometry*, *SW-846 Method 6010B for Inductively Coupled Plasma*, *SW-846 Method 7471A for Mercury (Manual Cold-Vapor Technique)*, and validation guidelines outlined in the *USEPA CLP National Functional Guidelines for Inorganic Data Review (2/94)*. 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	EPA ID	Laboratory ID	Matrix	COC Method
Ambient	Ambient	IOC0456-01	water	ILM04

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 the laboratory below the temperature limits of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$, at 1°C ; however, as the sample was not noted to be frozen, no qualifications were required. No sample preservation, handling, or transport problems were noted, and no qualifications were necessary.

2.1.2 Chain of Custody

The COC was signed and dated by field and laboratory personnel and accounted for the sample and analyses presented in this SDG. A duplicate aliquot was submitted for the sample in this SDG; however, duplicate analyses were not required. No sample qualifications were required.

2.1.3 Holding Times

The date of collection recorded on the COC and the dates of analyses recorded in the raw data, documented that the sample analyses were performed within the specified holding times of six months for the ICP and ICP/MS metals and 28 days for mercury. No qualifications were required.

2.2 ICP-MS TUNING

A precalibration routine must be completed prior to calibrating the instrument, which consists of analyzing a tuning solution to verify resolution, mass calibration, and thermal stability. The solution must be analyzed a minimum of five times and must contain isotopes representing all mass regions of interest. All %RSDs were less than 5%. The mass calibrations were within 0.1 amu of the true mass and the instrument resolutions were less than 0.75 amu at 5 percent peak height for all analytes in the tune solution. No site sample qualifications were required.

2.3 CALIBRATION

The ICV and CCV results showed acceptable recoveries, 90-110% for ICP/MS metals and 80-120% for mercury. Antimony was not recovered in the 0.2 ppb reporting limit check standard and was recovered below the control limit in the 1.0 ppb reporting limit check standard; therefore, nondetected antimony in sample Ambient was qualified as estimated, "UJ." The remaining reporting limit check standards were recovered within the AMEC control limits of 70-130%. No further sample qualifications were required.

2.4 BLANKS

There were detects reported in the method blanks and CCBs associated with the site sample; however, none required samples qualifications

2.5 ICP INTERFERENCE CHECK SAMPLE (ICS A/AB)

ICSA and ICSAB analyses were included in the raw data for the ICP-MS analyses. Results were not provided for spiked interferents sulfur, phosphorus, carbon, and chloride, and antimony, selenium, thallium, and lead were not spiked into the ICSAB solution. The results for potassium were above the calibration range of the instrument in all the ICSA and ICSAB analyses and aluminum was below the calibration range in the ICSA and ICSAB analyses; however, as these analytes were not reported in the site sample, no qualifications were required. Copper and cadmium were detected above the applicable reporting limit in the ICSA. The validator reviewed the raw data for the site sample ICP/MS analyses for the level of reported interferents, Al, Ca, Fe, and Mg, and determined that the level of reported interferents were not high enough to cause matrix affects. No assessment could be made with respect to possible interference from sulfur, phosphorus, carbon, and chloride.

ICSA and ICSAB analyses were included in the raw data for the ICP analyses, but were not run on the days the site sample was analyzed. The recoveries for the interferents and the other spiked analytes were within the control limits of 80-120%. In the ICSA analyses there was a result for nickel that was above the reporting limit. The validator reviewed the raw data for the site sample ICP analyses for the level of reported interferents, Al, Ca, Fe, and Mg, and determined that the level of reported interferents were not high enough to cause matrix affects. No qualifications were required.

2.6 BLANK SPIKES AND LABORATORY CONTROL SAMPLES

The ICP/MS LCS sample was identified as 5C08106-BS1 and the ICP LCS sample was identified as 5C09086-BS1. The mercury LCS sample was identified as 5C09050-BS1. The LCS results on the summary forms and in the raw data were within the laboratory-established ICP, ICP/MS, and mercury control limits of 85-115%. No qualifications were required.

2.7 LABORATORY DUPLICATES

MS/MSD analyses were performed on sample Ambient for mercury only. The RPD was within the control limit of 20% and no qualifications were required.

2.8 MATRIX SPIKE

MS/MSD analyses were performed on sample Ambient for mercury only. The recoveries were within the AMEC control limits of 75-125% and no qualifications were required. ICP and ICP/MS method accuracy was evaluated based on LCS results.

2.9 FURNACE ATOMIC ABSORPTION QC

Furnace atomic absorption was not utilized for the analysis of this sample; therefore, furnace atomic absorption QC is not applicable.

2.10 ICP/MS AND ICP SERIAL DILUTION

No serial dilution analyses were performed in association with the sample in this SDG; therefore, no assessment was made with respect to this criterion.

2.11 INTERNAL STANDARDS PERFORMANCE

The ICP-MS internal standard recoveries for the site sample and associated QC sample analyses were within the 60-125% control limits and no qualifications were required.

2.12 SAMPLE RESULT VERIFICATION

A Level IV review was performed for the sample in this data package. Calculations were verified, and the sample results reported on the Form Is were verified against the raw data. No transcription errors or calculation errors were noted. Analytes detected below the reporting limit were qualified as estimated, "J."

Antimony was reported by the laboratory as a nondetect at the reporting limit. The raw data review indicated that the sample result was actually $-0.47 \mu\text{g/L}$. Due to this negative result and the poor reporting limit check standard recoveries, the reviewer raised the antimony MDL to $1.0 \mu\text{g/L}$. No further qualifications were required.

2.13 FIELD QC SAMPLES

Field QC samples are evaluated, and if necessary, qualified based only on laboratory blanks. Any remaining detects are used to evaluate the associated sample.

2.13.1 Field Blanks and Equipment Rinsates

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

2.13.2 Field Duplicates

There were no field duplicate analyses performed in association with the site sample.

