**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**
**REGION 2**
**DESA/HWSB/HWSS**
**2890, Woodbridge Avenue, Edison, NJ 08837**

## EXECUTIVE NARRATIVE

**Case No.:** 46255                                    **SDG No.:** MBDJE8
**Site:** Lower Hackensack River                       **Laboratory:** CHEMTECH
**Number of Samples:** 18 (Sediment) and 2 (Water)     **Sampling dates**: 7/15/2016 – 7/21/2016
**Analysis:** Metals and Hg                            **Validation SOP:** HW-3a and -3c (Rev 0)


**QAPP:**
Not Available


## SUMMARY OF DEFINITIONS:

**Critical:** Results have an unacceptable level of uncertainty and should not be used for making decisions. Data have been qualified "R" rejected.

**Major:** A level of uncertainty exists that may not meet the data quality objectives for the project. A bias is likely to be present in the results. Data has been qualified "J" estimated. "J+" and "J-" represent Likely direction of the bias.

**Minor:** The level of uncertainty is acceptable. No significant bias in the data was observed.

**Critical Findings:**
None

**Major Findings:**
Samples MBDJG3, MBDJG0, MBDJG1, MBDJF7, MBDJF9, MBDJG9, MBDJH8, MBDJH7, MBDJH6, MBDJH4, MBDJH3 and MBDJH1 have analytes that have been qualified J, J+ or J-.

**Minor Findings:**
None

> **COMMENT:** None


**Reviewer Name(s):  Jianwei Huang**

**Approver's Signature:**                              **Date: 08/31/2016**

**Name:**                Narendra Kumar

**Affiliation: USEPA/R2/HWSB/HWSS**

| Data Qualifier Definitions (National Functional Guidelines) | | | |
|---|---|---|---|
| **Qualifier Symbol** | **Explanation** | | |
| | **INORGANICS** | **ORGANICS** | **CHLORINATED DIOXIN/FURAN** |
| U | The analyte was analyzed for, but was not detected above the level of the reported quantitation limit. | The analyte was analyzed for, but was not detected at a level greater than or equal to the level of the adjusted Contract Required Quantitation Limit (CRQL) for sample and method | The analyte was analyzed for but not detected. The value preceding the "U" may represent the adjusted Contract Required Quantitation Limit (see DLM02.X, Exhibit D, Section 1.2 and Table 2), or the sample specific estimated detection limit (EDL, see Method 8290A, Section 11.9.5). |
| J | The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. | The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to the quality of the data generated because certain quality control criteria were not met, or the concentration of the analyte was below the CRQL. | The analyte was positively identified and the associated numerical value is the approximate concentration of the analyte in the sample (due either to an issue with the quality of the data generated because certain QC criteria were not met, or the concentration of the analyte was below the adjusted CRQL). |
| J+ | The result is an estimated quantity, but the result may be biased high. | The result is an estimated quantity, but the result may be biased high. | |
| J– | The result is an estimated quantity, but the result may be biased low. | The result is an estimated quantity, but the result may be biased low. | |
| UJ | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. | The analyte was not detected at a level greater than or equal to the adjusted CRQL. However, the reported adjusted CRQL is approximate and may be inaccurate or imprecise. | The analyte was not detected (see definition of "U" flag, above). The reported value should be considered approximate. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample. | The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample. | The sample results are unusable due to the quality of the data generated because certain criteria were not met. The analyte may or may not be present in the sample. |
| N | | The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification". | |
| NJ | | The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration. | |
| C | | This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatograph/Mass Spectrometer (GC/MS). | |
| X | | This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful. | |

# DATA ASSESSMENT

## ANALYSIS: METALS ICP-AES

The current SOP HW-3a (Revision 0) July 2015, USEPA Region II for the evaluation of ICP-AES metals generated through Statement of Work ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi-Automated Screening Results Report.

## 1. HOLDING TIME AND PRESERVATION

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time or pH (aqueous samples are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (180 days) or pH ($\leq 2$) have not been met, will be qualified as estimated, "J"; the non-detects will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

## 2. CALIBRATION

Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for the metals on the Inorganic Target Analyte List (TAL). Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

## A) INITIAL CALIBRATION

A blank and at least five calibration standards shall be used to establish each analytical curve. At least one of these standards shall be at or below the CRQL. The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The curve must have a correlation coefficient $\geq 0.995$. The percent differences calculated for all of the non-zero standards must be within $\pm 30\%$ of the true value of the standard. The y-intercept of the curve must be less than the CRQL. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

## B) INITIAL AND CONTINUING CALIBRATION VERIFICATION

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s).
The CCV standard shall be analyzed at a frequency of every two hours during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 90 – 110%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

## 3. BLANK CONTAMINATION

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Calibration blanks (ICB and CCB) are used to ensure a stable instrument baseline before and during the analysis of analytical samples. The preparation blank is used to assess the level of contamination introduced to the analytical samples throughout the sample preparation process. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

The following samples have analyte results less than or equal to CRQLs. The associated CCB analyte results are less than or equal to CRQLs. Detects are qualified as U. Sample results are reported at CRQLs.

**Thallium** MBDJG0, MBDJH1

4. **INTERFERENCE CHECK SAMPLE**

The Interference Check Sample (ICS) verifies the analytical instrument's ability to overcome interferences typical of those found in samples. The laboratory should have analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. Results for the analysis of ICS Solution must fall within the control limits of ± 20% or $\pm$CRQL (whichever is greater) of the true value for the analytes and interferents included in the solution. If results that are ≥ MDL are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is ≥ MDL, the possibility of false negatives in the samples exists. In general, ICP sample data can be accepted if the concentrations of Al, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

5. **SPIKE SAMPLE ANALYSIS**

The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.

The following sample is associated with Matrix Spike sample that has spike analyte %R within 30 - 74% and Post-digestion spike analyte %R greater than or equal to 75%. Detects are qualified as J. Non-detects are qualified as UJ.

**Antimony and Selenium** MBDJH6

6. **DUPLICATE SAMPLE ANALYSIS**

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 35% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required

Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.

No problems were found for this criterion.

### 7. FIELD DUPLICATE

**Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 35% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and it's duplicate.**

The following Duplicate and original soil sample results are greater than or equal to 5x the CRQL and RPD is greater than 50%. Detected analytes are qualified J.

**Chromium and Copper** MBJF4 (SDG MBDJD4), MBJF5 (this SDG)

### 8. LABORATORY CONTROL SAMPLE

**The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and Quality Assurance/Quality Control (QA/QC) procedures as employed for the samples. All LCS Percent Recoveries (%R) must fall within the control limits of 70-130%, except for Sb and Ag which must fall within the control limits of 50-150%. Qualifications were applied to the samples and analytes as shown below.**

No problems were found for this criterion.

### 9. ICP SERIAL DILUTION

**The serial dilution of samples quantitated by Inductively Coupled Plasma determines whether or not significant physical or chemical interferences exist due to sample matrix. If the analyte concentration is sufficiently high [concentration in the original sample is > 50 times (50x) the Method Detection Limit (MDL)], the Percent Difference (%D) between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 15. For a serial dilution analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the serial dilution sample.**

No problems were found for this criterion.

### 10. PERCENT SOLIDS

**The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. All results of a sample with percent solids less than 50% are qualified estimated, "J". Qualifications were applied to the samples and analytes as shown below.**

The following samples are with percent solids less than 50%. Detects are qualified as estimated J. Non-detects are qualified as estimated UJ.

MBDJG3, MBDJG0, MBDJG1, MBDJF7, MBDJF9, MBDJG9, MBDJH8, MBDJH7, MBDJH6, MBDJH4, MBDJH3, MBDJH1

## ANALYSIS: MERCURY

**The current SOP HW-3c (Revision 0) July 2015, USEPA Region II for the evaluation of Mercury generated through Statement of Work ISOM02.2 has been applied. Data have been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi-Automated Screening Results Report.**

**1. HOLDING TIME AND PRESERVATION**

**The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time, pH (aqueous samples), or cooler temperature are not within the acceptable range, the data may not be valid. Those analytes detected in the samples whose holding time (28 days) and pH (≤2) have not been met, will be qualified as estimated, "J"; the non-detects (sample quantitation limits) will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.**

No problems were found for this criterion.

**2. CALIBRATION**

**Method requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data for mercury. Initial Calibration Verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing Calibration Verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.**

**A) INITIAL CALIBRATION**

**A blank and at least five calibration standards shall be employed to establish the analytical curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curves for mercury shall possess a correlation coefficient of ≥ 0.995 to ensure the linearity over the calibrated range. The percent differences calculated for all of the non-zero standards must fall within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL. All sample results shall be reported from an analysis within the calibrated range. Qualifications were applied to the samples and analytes as shown below.**

No problems were found for this criterion.

**B) INITIAL AND CONTINUING CALIBRATION VERIFICATION**

**Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for mercury by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every hour during an analytical run. The CCV standard shall also be analyzed at the beginning of the run, and again after the last analytical sample. The percent recovery**

**acceptable limits for ICV/CCV are 85 – 115%. Qualifications were applied to the samples and analytes as shown below.**

No problems were found for this criterion.

### 3. BLANK CONTAMINATION

**Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.**

No problems were found for this criterion.

### 4. SPIKE SAMPLE ANALYSIS

**The spiked sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike Percent Recovery (%R) shall be within the established acceptance limits of 75 – 125%. However, spike recovery limits do not apply when the sample concentration is ≥ 4x the spike added. For a matrix spike analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the matrix spike sample.**

No problems were found for this criterion.

### 5. DUPLICATE SAMPLE ANALYSIS

**The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. For a duplicate sample analysis that does not meet the technical criteria, the action was applied to only the field sample used to prepare the duplicate sample.**

No problems were found for this criterion.

### 6. FIELD DUPLICATE

**Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ five times (5x) the Contract Required Quantitation Limit (CRQL). A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and it's duplicate.**

No problems were found for this criterion.

### 7. PERCENT SOLIDS

**The laboratory is required to perform the percent solids determination prior to sample preparation and analysis. Qualifications were applied to the samples and analytes as shown below.**

The following samples are with percent solids less than 50%. Detects are qualified as estimated J. Non-detects are qualified as estimated UJ.

MBDJG3, MBDJG0, MBDJG1, MBDJF7, MBDJF9, MBDJG9, MBDJH8, MBDJH7, MBDJH6, MBDJH4, MBDJH3, MBDJH1