**Hazardous Waste Support Section**
**SOP NO. HW-2c Revision 15**
**Mercury and Cyanide Data Validation**

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## NOTICE

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This document can be obtained from the USEPA's Region 2 Quality Assurance website at:

http://www.epa.gov/region2/qa/documents.htm

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## ACRONYMS

| | |
|---|---|
| **ASB** | Analytical Services Branch |
| **CCB** | Continuing Calibration Blank |
| **CCS** | Contract Compliance Screening |
| **CCV** | Continuing Calibration Verification |
| **CLP** | Contract Laboratory Program |
| **CO** | Contracting Officer |
| **CRQL** | Contract Required Quantitation Limit |
| **DF** | Dilution Factor |
| **DQO** | Data Quality Objective |
| **EDD** | Electronic Data Deliverable |
| **EDM** | EXES Data Manager |
| **ESAT** | Environmental Services Assistance Team |
| **EXES** | Electronic Data eXchange and Evaluation System |
| **HWSS** | Hazardous Waste Support Section |
| **ICB** | Initial Calibration Blank |
| **ICP** | Inductively Coupled Plasma |
| **ICP-AES** | Inductively Coupled Plasma - Atomic Emission Spectroscopy |
| **ICP-MS** | Inductively Coupled Plasma - Mass Spectrometry |
| **ICS** | Interference Check Sample |
| **ICV** | Initial Calibration Verification |
| **LCS** | Laboratory Control Sample |
| **MDL** | Method Detection Limit |
| **NIST** | National Institute of Standards and Technology |
| **OSRTI** | Office of Superfund Remediation and Technology Innovation |
| **OSWER** | Office of Solid Waste and Emergency Response |
| **PE** | Performance Evaluation |
| **%D** | Percent Difference |
| **%R** | Percent Recovery |
| **%S** | Percent Solids |
| **PO** | Project Officer |
| **QA** | Quality Assurance |
| **QAPP** | Quality Assurance Project Plan |
| **QC** | Quality Control |
| **RPD** | Relative Percent Difference |
| **RSCC** | Regional Sample Control Center Coordinator |
| **SDG** | Sample Delivery Group |
| **SMO** | Sample Management Office |
| **SOP** | Standard Operating Procedure |
| **SOW** | Statement of Work |
| **TR/COC** | Traffic Report/Chain of Custody Documentation |
| **USEPA** | United States Environmental Protection Agency |

## TARGET ANALYTE LIST

**CN** Cyanide
**Hg** Mercury

## INTRODUCTION

This document is designed to offer the data reviewer guidance in determining the validity of analytical data generated through the USEPA Contract Laboratory Program (CLP) Statement of Work (SOW) ISM01.X Inorganic Superfund Methods (Multi-Media, Multi-Concentration), hereinafter referred to as the ISM01.2 SOW, and any future editorial revisions of ISM01.2. This guidance is somewhat limited in scope and is intended to be used as an aid in the formal technical review process.

The guidelines presented in the document will aid the data reviewer in establishing (a) if data meets the specific technical and QC criteria established in the SOW, and (b) the validity and extent of bias of any data not meeting the specific technical and QC criteria established in the SOW. It must be understood by the reviewer that acceptance of data not meeting technical requirements is based upon many factors, including, but not limited to site-specific technical requirements, the need to facilitate the progress of specific projects, and availability for re-sampling.

The reviewer should note that while this document is to be used as an aid in the formal data review process, other sources of guidance and information, as well as **professional judgment**, should also be used to determine the ultimate validity of data, especially in those cases where all data does not meet specific technical criteria.

## DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process.

| | |
|---|---|
| **U** | The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit. |
| **J** | The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. |
| **J+** | 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. |
| **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. |
| **UJ** | The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise. |

## DATA PACKAGE INSPECTION

For data obtained through the Contract Laboratory Program (CLP), the EXES Data Manager (EDM) is a useful tool in the data review process. For more information about EDM, please refer to the following Sample Management Office (SMO) website:

https://epasmoweb.fedcsc.com/help/guides/Submit%20and%20Inspect%20Data%20Quick%20Guide%20%28EXES%29.pdf

EDM will identify any missing and/or incorrect information in the data package. The CLP laboratory may submit a reconciliation package for any missing items or to correct data.
If there are any concerns regarding the data package, contact the CLP Project Officer (CLP PO) from the Region where the samples were taken. For personnel contact information, please refer to the following CLP website:

http://www.epa.gov/superfund/programs/clp/contacts.htm

## HWSS DATA VALIDATION PROCESS

After downloading the data package from EDM, the data validator will use the recommendations in this SOP as well as their own professional judgment to validate the data.

The data will be saved in the following location, under the appropriate case number folder:

G:\DESADIV\HWSS\DATA VALIDATION

The file naming conventions will consist of

A. case number i.e., 12345
B. SDG name i.e., MBXY12
C. level of validation performed i.e., S2BVE

Examples: **12345_MBXY12_S2BVE.xls**

**12345_MBXY12_S2BVEM.xls**

When data validation is completed, the data package is uploaded for the client to download from the HWSS data delivery website:

https://epaqpx.rtp.epa.gov/hwssclpdeliverables

The completed data package includes the Executive Narrative (see Appendix B for template), the Sample Summary Report (see Appendix C for example), and the Electronic Data Deliverable (EDD) (see Appendix D for a list of the column headers included in this document).

## PRELIMINARY REVIEW

This document is for the review of analytical data generated through the ISM01.2 SOW and any future editorial revisions of ISM01.2. To use this document effectively, the reviewer should have an understanding of the analytical method and a general overview of the Sample Delivery Group (SDG) or sample Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in the analysis are essential information.

It is suggested that an initial review of the data package be performed, taking into consideration all information specific to the sample data package [e.g., Modified Analysis requests, Traffic Report/Chain of Custody (TR/COC) documentation, SDG Narratives, etc.].

The reviewer should also have a copy of the Quality Assurance Project Plan (QAPP) or similar document for the project for which the samples were analyzed. The reviewer should contact the appropriate Regional Contract Laboratory Program Project Officer (CLP PO) to obtain copies of the QAPP and relevant site information. This information is necessary in determining the final usability of the analytical data.

The SDGs or Cases routinely have unique samples that require special attention from the reviewer. These include field blanks and trip blanks, field duplicates, and Performance Evaluation (PE) samples which must be identified in the sampling records. The sampling records (e.g., TR/COC records, field logs, and/or contractor tables) should identify:

1. The Region where the samples were taken, and
2. The complete list of samples with information on:
    a. Sample matrix;
    b. Field blanks*;
    c. Field duplicates*;
    d. Field spikes*;
    e. PE samples*;
    f. Shipping dates;
    g. Preservatives;
    h. Types of analysis; and
    i. Laboratories involved.

    * If applicable.

The TR/COC documentation includes sample descriptions and date(s) of sampling. The reviewer must consider lag times between sampling and start of analysis when assessing technical sample holding times.

The laboratory's SDG Narrative is another source of general information. Notable problems with matrices, insufficient sample volume for analysis or reanalysis, samples received in broken containers, preservation, and unusual events should be documented in the SDG Narrative. The reviewer should also inspect any email or telephone/communication logs detailing any discussion of sample or analysis issues between the laboratory, the CLP Sample Management Office (SMO), and the USEPA Region.

**An Example Analytical Sequence for Mercury**

S0
S0.2
S1.0
S2.0
S5.0
S10.0
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

**An Example Analytical Sequence for Cyanide**

S0
S10
S50
S100
S200
S400
ICV
ICB
CCV
CCB
samples
CCV
CCB
samples
CCV
CCB, etc.

## Mercury Preservation and Holding Times

**Action:**

**NOTE:** Apply the action to each sample for which the preservation or holding time criteria was not met.

1. If the pH of aqueous/water metal samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH of the sample to ≤ 2 at the time of sample receipt. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of the metal(s) of interest. Qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-), and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data, based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-), and qualify non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

**Table 1. Technical Holding Time Actions for Mercury Analysis**

| Preservation & Holding Time Results | Action for Samples |
|---|---|
| Aqueous/water mercury samples received with pH > 2 and pH not adjusted | Use professional judgment<br>Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as unusable (R) |
| Technical Holding Time exceeded: Aqueous/water samples > 28 days | Use professional judgment<br>Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as unusable (R) |
| Technical Holding Time exceeded: Soil/sediment samples > 28 days | Use professional judgment<br>Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as unusable (R) |

## Cyanide Preservation and Holding Times

**Action:**

**NOTE:** Apply the action to each sample for which the preservation or holding time criteria was not met.

1. If oxidizing agents are detected in aqueous/water cyanide samples at the time of sample preparation, qualify results that are ≥ Method Detection Limit (MDL) as estimated low (J-) and non-detects as unusable (R). If sulfides are detected in aqueous/water cyanide samples at the time of sample preparation and there is no evidence that the laboratory removed the sulfides (using precipitation and filtration), qualify results that are ≥ MDL as estimated (J) and non-detects as unusable (R). If the pH of aqueous/water cyanide samples is < 12 at the time of sample receipt, use professional judgment to qualify the samples based on the pH of the sample. Qualify results that are ≥ MDL as estimated low (J-) and qualify non-detects as unusable (R).
2. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples are properly preserved. The expected bias would be low. Qualify results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R).
3. Due to limited information concerning holding times for soil/sediment samples, it is left to the discretion of the data reviewer whether to apply aqueous/water holding time criteria to soil/sediment samples. If they are applied, it must be clearly documented in the Data Review Narrative.
4. When the holding times are exceeded, the reviewer should comment in the Data Review Narrative on any possible consequences for the analytical results.
5. When holding times are grossly exceeded, note it for Contract Laboratory Program Project Officer (CLP PO) action.

**Table 2. Technical Holding Time Actions for Cyanide Analysis**

| Preservation & Holding Time Results | Action for Samples |
|---|---|
| Aqueous/water cyanide samples received with oxidizing agents present | Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as unusable (R) |
| Aqueous/water cyanide samples received with sulfides present, and sulfides are not removed | Qualify results that are ≥ MDL as estimated (J)<br>Qualify non-detects as unusable (R) |
| Aqueous/water cyanide samples received with pH < 12 | Use professional judgment<br>Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as unusable (R) |
| Technical Holding Time exceeded: Cyanide > 14 days | Use professional judgment<br>Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as unusable (R) |

## Mercury Calibration

**Table 3. Acceptance Criteria for Mercury ICVs and CCVs**

| Analytical Method | Inorganic Analytes | ICV/CCV Low Limit (% of True Value) | ICV/CCV High Limit (% of True Value) |
|---|---|---|---|
| Cold Vapor AA | Mercury | 85 | 115 |

**Action:**

**NOTES:** For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.
For CCVs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical run.

**NOTE:** The data validator shall verify the correlation coefficient by calculating it using the standard concentrations and the corresponding instrument response.

1. If the instrument was not calibrated daily and each time the instrument was set up or an ICV standard was not analyzed before field and QC samples, qualify the data as unusable ®. If the instrument was not calibrated with a blank and at least 5 calibration standards, or if the instrument was not calibrated with standards prepared at the same time as the samples, use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J), and non-detects as estimated (UJ). If the calibration curve does not include standards at required concentrations (e.g., a blank and at least one standard at or below CRQL), use professional judgment to qualify results that are ≥ MDL as estimated (J), and non-detects as estimated (UJ).
2. If the correlation coefficient is < 0.995, percent differences are outside the ±30% limit, or the y-intercept ≥ CRQL, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ). If the correlation coefficient is < 0.990, qualify results that are ≥ MDL as estimated (J) and non-detects as unusable (R).
3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
    a. If the ICV or CCV %R is < 70%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as unusable (R).
    b. If the ICV or CCV %R falls within the range of 70-84%, qualify sample results that are ≥ MDL as estimated low (J-), and qualify non-detects as estimated (UJ).
    c. If the ICV or CCV %R falls within the range of 116-130%, qualify sample results that are ≥ MDL as estimated high (J+).
    d. If the ICV or CCV %R is within the range of 116-130%, non-detects should not be qualified.
    e. If the ICV or CCV %R is > 130%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.

f. If the %R is > 165%, qualify all results that are ≥ MDL as unusable (R).

4. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
5. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
6. If calibration criteria are grossly exceeded, note this for CLP Project Officer (CLP PO) action.

**NOTE:** For critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

**Table 4. Calibration Actions for Mercury Analysis**

| Calibration Result | Action for Samples |
|---|---|
| Calibration not performed | Qualify all results as unusable (R) |
| Calibration incomplete | Use professional judgment<br>Qualify results that are ≥ MDL as estimated (J)<br>Qualify non-detects as estimated (UJ) |
| Not at least one calibration standard at or below the CRQL for each analyte | Qualify results that are ≥ MDL but < 2x the CRQL as estimated (J)<br>Qualify non-detects as estimated (UJ) |
| Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL | Qualify results that are ≥ MDL as estimated (J)<br>Qualify non-detects as estimated (UJ) |
| Correlation coefficient < 0.990 | Qualify results that are ≥ MDL as estimated (J)<br>Qualify non-detects as unusable (R) |
| ICV/CCV %R < 70% | Qualify results that are ≥ MDL as unusable (R)<br>Qualify all non-detects as unusable (R) |
| ICV/CCV %R 70-84% | Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as estimated (UJ) |
| ICV/CCV %R 116-130% | Qualify results that are ≥ MDL as estimated high (J+) |
| ICV/CCV %R > 130% | Qualify results that are ≥ MDL as estimated high (J+) |
| ICV/CCV %R > 165% | Qualify results that are ≥ MDL as unusable (R) |

## Cyanide Calibration

**Table 5. Acceptance Criteria for Cyanide ICVs and CCVs**

| Analytical Method | Inorganic Analytes | ICV/CCV Low Limit (% of True Value) | ICV/CCV High Limit (% of True Value) |
|---|---|---|---|
| Colorimetric | Cyanide | 85 | 115 |

**Action:**

**NOTES:** For initial calibrations or ICVs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.
For CCVs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical run.

**NOTE:** The data validator shall verify the correlation coefficient by calculating it using the standard concentrations and the corresponding instrument response.

1. If the instrument was not calibrated daily and each time the instrument was set up or an ICV standard was not analyzed before field and QC samples, qualify the data as unusable (R). If the instrument was not calibrated with a blank and at least 5 calibration standards, or if the instrument was not calibrated with standards prepared at the same time as the samples, use professional judgment to qualify results that are ≥ Method Detection Limit (MDL) as estimated (J), and non-detects as estimated (UJ). If the calibration curve does not include standards at required concentrations (e.g., a blank and at least one standard at or below CRQL), use professional judgment to qualify results that are ≥ MDL as estimated (J), and non-detects as estimated (UJ).
2. If the correlation coefficient is < 0.995, percent differences are outside the ±30% limit, or the y-intercept ≥ CRQL, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ). If the correlation coefficient is < 0.990, qualify results that are ≥ MDL as estimated (J) and non-detects as unusable (R).
3. If the standards, the ICV, or the CCVs are not distilled for cyanide, qualify sample results that are ≥ MDL as estimated (J).
4. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
    a. If the ICV or CCV %R is < 70%, qualify non-detects as unusable (R). Use professional judgment to qualify all results that are ≥ MDL as unusable (R).
    b. If the ICV or CCV %R falls within the range of 70-84%, qualify sample results that are ≥ MDL as estimated low (J-), and qualify non-detects as estimated (UJ).
    c. If the ICV or CCV %R falls within the range of 116-130%, qualify sample results that are ≥ MDL as estimated high (J+).
    d. If the ICV or CCV %R is within the range of 116-130%, non-detects should not be qualified.

e. If the ICV or CCV %R is > 130%, use professional judgment to qualify results that are ≥ MDL as estimated high (J+). Non-detects should not be qualified.
f. If the %R is > 165%, qualify all results that are ≥ MDL as unusable (R).

5. If the laboratory failed to provide adequate calibration information, the Region's designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgment to assess the data.
6. Note the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
7. If calibration criteria are grossly exceeded, note this for CLP Project Officer (CLP PO) action.

**NOTE:** For critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

**Table 6. Calibration Actions for Cyanide Analysis**

| Calibration Result | Action for Samples |
|---|---|
| Calibration not performed | Qualify all results as unusable (R) |
| Calibration incomplete | Use professional judgment<br>Qualify results that are ≥ MDL as estimated (J)<br>Qualify non-detects as estimated (UJ) |
| Not at least one calibration standard at or below the CRQL for each analyte | Qualify results that are ≥ MDL but < 2x the CRQL as estimated (J)<br>Qualify non-detects as estimated (UJ) |
| Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL | Qualify results that are ≥ MDL as estimated (J)<br>Qualify non-detects as estimated (UJ) |
| Correlation coefficient < 0.990 | Qualify results that are ≥ MDL as estimated (J)<br>Qualify non-detects as unusable (R) |
| Standards and QC not distilled | Qualify results that are ≥ MDL as estimated (J) |
| ICV/CCV %R < 70% | Qualify results that are ≥ MDL as unusable (R)<br>Qualify all non-detects as unusable (R) |
| ICV/CCV %R 70-84% | Qualify results that are ≥ MDL as estimated low (J-)<br>Qualify non-detects as estimated (UJ) |
| ICV/CCV %R 116-130% | Qualify results that are ≥ MDL as estimated high (J+) |
| ICV/CCV %R > 130% | Qualify results that are ≥ MDL as estimated high (J+) |
| ICV/CCV %R > 165% | Qualify results that are ≥ MDL as unusable (R) |

## Mercury/Cyanide Calibration/Preparation Blanks

**Action:**

**NOTES:** For ICBs that do not meet the technical criteria, apply the action to all samples reported from the analytical run.
For CCBs that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical run.
For Preparation Blanks that do not meet the technical criteria, apply the action to all samples prepared in the same preparation batch.

**NOTES:** The preparation blank for mercury is the same as the calibration blank.
Convert soil sample result to mg/kg on wet weight basis to compare with the soil preparation result on Form III.
Associated samples are all samples digested with the preparation blank.

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
    a. Any blank (including Preparation Blanks) reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank (including Preparation Blanks) reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
    b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. Specific "method" actions include:
    a. If the absolute value of an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the affected samples were not reanalyzed, report non-detects and results that are ≥ MDL, but ≤ CRQL as CRQL-U. For results that are > CRQL but < Blank Result, report the results at the level of the blank with a "U" qualifier. Use professional judgment to qualify results that

are > Blank Result. Note this situation for CLP PO action and record it in the Data Review Narrative.

b. If the absolute value of the concentration of the Preparation Blank is ≤ CRQL, report non-detects and results that are ≥ MDL but ≤ CRQL as CRQL-U. Use professional judgment to quality results that are > CRQL.
c. If the mercury concentration in the Preparation Blank is > CRQL, the lowest concentration of mercury in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redigested and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redigestion (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redigest and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.
d. If the cyanide concentration in the Preparation Blank is > CRQL, the lowest concentration of cyanide in the associated samples must be 10x the Preparation Blank concentration. Otherwise, all samples associated with that blank with concentrations < 10x the Preparation Blank concentration and > CRQL should be redistilled and reanalyzed. Raise the CRQL to the concentration found in the Preparation Blank and report those samples that do not require redistillation (that are ≥ MDL but ≤ CRQL) as CRQL-U. Note for CLP PO action and record in the Data Review Narrative if the laboratory failed to redistill and reanalyze the affected samples. The reviewer shall then use professional judgment to assess the data.

**Table 7. Calibration/Preparation Blank Actions for Mercury/Cyanide Analysis**

| Blank Type | Blank Result | Sample Result | Action for Samples |
|---|---|---|---|
| ICB/CCB | ≥ MDL but ≤ CRQL | Non-detect | No action |
| | | ≥ MDL but ≤ CRQL | Report CRQL value with a "U" |
| | | > CRQL | Use professional judgment |
| ICB/CCB | > CRQL | ≥ MDL but ≤ CRQL | Report CRQL value with a "U" |
| | | > CRQL but < Blank Result | Report at level of Blank Result with a "U" |
| | | > Blank Result | Use professional judgment |
| ICB/CCB | ≤ (-MDL) but ≥ (-CRQL) | ≥ MDL, or non-detect | Use professional judgment |
| ICB/CCB | < (-CRQL) | < 10x the CRQL | Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ) |
| Preparation Blank | > CRQL | ≥ MDL but ≤ CRQL | Report CRQL value with a "U" |
| | | > CRQL but < 10x the Blank Result | Qualify results as estimated high (J+) |
| | | ≥ 10x the Blank Result | No action |
| Preparation Blank | ≥ MDL but ≤ CRQL | Non-detect | No action |
| | | ≥ MDL but ≤ CRQL | Report CRQL value with a "U" |
| | | > CRQL | Use professional judgment |
| Preparation Blank | < (-CRQL) | < 10x the CRQL | Qualify results that are ≥ CRQL as estimated low (J-) Qualify non-detects as estimated (UJ) |

## Mercury/Cyanide Laboratory Duplicate Sample Analysis

**Action:**

**NOTE:** For a duplicate sample analysis that does not meet the technical criteria, apply the action to only the field sample used to prepare the duplicate sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

**NOTE:** Delete "*" from Form IAs.
If one value is > CRQL and the other value is non-detect, calculate the absolute difference between the value > CRQL and the MDL and use this difference to qualify sample results.
If more than one lab duplicate sample was analyzed for an SDG, then qualify the associated samples based on the worst lab duplicate analysis.

1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for CLP Project Officer (CLP PO) action.
2. If the results from a duplicate analysis for mercury/cyanide fall outside the control limits for > 5x the CRQL, qualify aqueous sample results that are ≥ CRQL as estimated (J) if the RPD is between 20% - 100% and as unusable (R) if the RPD is > 100%. Qualify soil/sediment sample results that are ≥ CRQL as estimated (J) if the RPD is between 35% - 120% and as unusable (R) if the RPD is > 120%.
3. If the results from a duplicate analysis for mercury/cyanide fall outside the control limits for ≤ 5x the CRQL, qualify those results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
4. If a field blank or PE sample was used for the duplicate sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
5. Note the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

**Table 8. Duplicate Sample Actions for Mercury/Cyanide Analysis**

| Duplicate Sample Results | Action for Samples |
|---|---|
| *Aqueous:*<br>Both original sample and duplicate sample > 5x the CRQL and 20% < RPD < 100% | Qualify those results that are ≥ CRQL as estimated (J) |
| *Aqueous:*<br>Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 100% | Qualify those results that are ≥ CRQL as unusable (R) |
| *Soil/Sediment:*<br>Both original sample and duplicate sample > 5x the CRQL and 35% < RPD < 120% | Qualify those results that are ≥ CRQL as estimated (J) |
| *Soil/Sediment:*<br>Both original sample and duplicate sample > 5x the CRQL and RPD ≥ 120% | Qualify those results that are ≥ CRQL as unusable (R) |
| Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL | Qualify those results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ) |

## Mercury Spike Sample Analysis

**Action:**

**NOTE:** For a Matrix Spike that does not meet the technical criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

**NOTE:** The final spike concentrations required for mercury are presented in the methods described in the Statement of Work (SOW).

**NOTE:** When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

**NOTES:** Disregard the out of control spike recoveries for analytes whose unspiked concentrations are ≥ 4x the spike added.
Delete "N" from Form IAs.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike %R is < 30%, qualify affected results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R).
4. If the Matrix Spike %R falls within the range of 30-74% and the sample results are ≥ MDL, qualify the affected data as estimated low (J-).
5. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
6. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.
7. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, qualify the affected data as estimated high (J+).
8. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

**Table 9. Spike Sample Actions for Mercury Analysis**

| Spike Sample Results | Action for Samples |
|---|---|
| Matrix Spike %R < 30% | Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R) |
| Matrix Spike %R 30-74% | Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ) |
| Matrix Spike %R > 125% | Qualify affected results that are ≥ MDL as estimated high (J+) Non-detects are not qualified |

## Cyanide Spike Sample Analysis

**Action:**

**NOTE:** For a Matrix Spike that does not meet the technical criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

**NOTE:** The final spike concentrations required for cyanide are presented in the methods described in the Statement of Work (SOW).

**NOTE:** When the sample concentration is < Method Detection Limit (MDL), use SR = 0 only for the purpose of calculating the %R. The actual spiked sample results, sample results, and %R (positive or negative) shall still be reported on Forms VA-IN and VB-IN.

**NOTES:** Disregard the out of control spike recoveries for analytes whose unspiked concentrations are ≥ 4x the spike added.
Delete "N" from Form IAs.

1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. Note the situation in the Data Review Narrative, and for Contract Laboratory Program Project Officer (CLP PO) action.
2. If a field blank or PE sample was used for the spiked sample analysis, note this for CLP PO action. All of the other Quality Control (QC) data must then be carefully checked and professional judgment exercised by the data reviewer when evaluating the data.
3. If the Matrix Spike recovery does not meet the evaluation criteria and a required post-distillation spike was not performed, note this for CLP PO action.
4. If the Matrix Spike %R is < 30%, verify that a post-distillation spike was analyzed if required. If the post-distillation spike %R is < 75% or is not performed, qualify sample results that are ≥ MDL as estimated low (J-) and non-detects as unusable (R). If the post-distillation spike %R is ≥ 75%, qualify sample results that are ≥ MDL as estimated (J) and non-detects as estimated (UJ).
5. If the Matrix Spike %R falls within the range of 30-74% and the sample results are ≥ MDL, verify that a post-distillation spike was analyzed if required. If the %R for the post-distillation spike is also < 75% or not performed, qualify the affected data as estimated low (J-). If the %R for the post-distillation spike is ≥ 75%, qualify the affected data as estimated (J).
6. If the Matrix Spike %R falls within the range of 30-74% and the sample results are non-detects, qualify the affected data as estimated (UJ).
7. If the Matrix Spike %R is > 125% and the reported sample results are non-detects, the sample data should not be qualified.
8. If the Matrix Spike %R is > 125% and the sample results are ≥ MDL, verify that a post-distillation spike was analyzed if required. If the %R for the post-distillation spike is also

> 125% or is not performed, qualify the affected data as estimated high (J+). If the %R for the post-distillation spike is ≤ 125%, qualify the affected data as estimated (J).

9. Note the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

**Table 10. Spike Sample Actions for Cyanide Analysis**

| Spike Sample Results | Action for Samples |
|---|---|
| Matrix Spike %R < 30%<br>Post-distillation spike %R < 75% | Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R) |
| Matrix Spike %R < 30%<br>Post-distillation spike %R ≥ 75% | Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ) |
| Matrix Spike %R 30-74%<br>Post-distillation Spike %R < 75% | Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as estimated (UJ) |
| Matrix Spike %R 30-74%<br>Post-distillation spike %R ≥ 75% | Qualify affected results that are ≥ MDL as estimated (J) and affected non-detects as estimated (UJ) |
| Matrix Spike %R > 125%<br>Post-distillation spike %R > 125% | Qualify affected results that are ≥ MDL as estimated high (J+) |
| Matrix Spike %R > 125%<br>Post-distillation spike %R ≤ 125% | Qualify affected results that are ≥ MDL as estimated (J) |
| Matrix Spike %R < 30%<br>No post-distillation spike performed | Qualify affected results that are ≥ MDL as estimated low (J-) and affected non-detects as unusable (R) |
| Matrix Spike %R 30-74%<br>No post-distillation spike performed | Qualify affected results that are ≥ MDL as estimated low (J-) and non-detects as estimated (UJ) |
| Matrix Spike %R > 125%<br>No post-distillation spike performed | Qualify affected results that are ≥ MDL as estimated high (J+)<br>Non-detects are not qualified |

## Mercury/Cyanide Field Duplicates

**Action:**

**NOTES:** For field duplicates that do not meet the technical criteria, apply the action to only the field sample and its duplicate. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.
Check the Sampling Trip Report for the field duplicate pair.
Substitute MDL for CRQL when MDL > CRQL.
Do not calculate RPD when both values are non-detects.
If one value is > the CRQL and the other value is non-detect, calculate the absolute difference between the value > the CRQL and the MDL, and use this criteria to qualify the results.

1. If a field duplicate pair was collected and analyzed, calculate and report the RPD when the sample and its field duplicate values are both ≥ 5x the CRQL. Calculate and report the absolute difference when at least one value (sample or duplicate) is < 5x the CRQL.
2. When aqueous sample and duplicate values are both ≥ 5x the CRQL, and the RPD is > 20%, qualify the sample and its duplicate as estimated (J).
3. When aqueous sample and/or the duplicate value is < 5x the CRQL, and the absolute difference is > the CRQL, qualify results > the MDL as estimated (J) and non-detects as estimated (UJ).
4. When soil/sediment sample and duplicate values are both ≥ 5x the CRQL, and the RPD is > 50%, qualify the sample and its duplicate as estimated (J).
5. When soil/sediment sample and/or the duplicate value is < 5x the CRQL, and the absolute difference is > 2x the CRQL, qualify results > the MDL as estimated (J) and non-detects as estimated (UJ).

**Table 11. Field Duplicate Actions for Mercury/Cyanide Analysis**

| Sample Type | Field Duplicate Result | Action for Samples |
|---|---|---|
| Aqueous | Sample and its field duplicate ≥ 5x the CRQL and RPD > 20% | Qualify sample and its duplicate as estimated (J) |
| | Sample and/or its field duplicate < 5x the CRQL and absolute difference > the CRQL | Qualify results > the MDL as estimated (J)<br>Qualify non-detects as estimated (UJ) |
| Soil/Sediment | Sample and its field duplicate ≥ 5x the CRQL and RPD > 50% | Qualify sample and its duplicate as estimated (J) |
| | Sample and/or its field duplicate < 5x the CRQL and absolute difference > 2x the CRQL | Qualify results > the MDL as estimated (J)<br>Qualify non-detects as estimated (UJ) |

## Mercury/Cyanide Field/Rinsate/Trip Blanks

**Action:**

**NOTE:** Designate "Field Blank" as such on Form IA.
Field Blank results previously rejected due to other criteria cannot be used to qualify field samples.
Do not use Rinsate Blank associated with soils to qualify water samples and vice versa.
If the MDL is > the CRQL, substitute CRQL with 2x the MDL.

1. If the appropriate blanks were not analyzed with the correct frequency, the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should then be recorded in the Data Review Narrative, and noted for Contract Laboratory Program Project Officer (CLP PO) action.
2. Action regarding unsuitable blank results depends on the circumstances and origin of the blank. The reviewer should note that in instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
3. Some general "technical" review actions include:
    a. Any blank reported with a negative result, whose value is ≤ (-MDL) but ≥ (-CRQL), should be carefully evaluated to determine its effect on the sample data. The reviewer shall then use professional judgment to assess the data. For any blank reported with a negative result, whose value is < (-CRQL) qualify results that are ≥ CRQL as estimated low (J-) and non-detects as estimated (UJ).
    b. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form I-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form III-IN. The reviewer may find it easier to work with the raw data.
4. If the absolute value of mercury/cyanide in a Field/Rinsate/Trip Blank is > the CRQL, then the CRQL shall be raised to the level in the Field/Rinsate/Trip Blank and the associated sample data below this level shall be reported as CRQL-U.
5. Sample results > the Field/Rinsate/Trip Blank value but < 10x the Field/Rinsate/Trip Blank value shall be qualified as estimated (J).
6. Sample results ≥ the MDL but ≤ the CRQL shall be reported at the CRQL value with a "U".

**Table 12. Field/Rinsate/Trip Blank Actions for Mercury/Cyanide Analysis**

| Blank Result | Sample Result | Action for Samples |
|---|---|---|
| > CRQL | ≥ MDL but ≤ CRQL | Report CRQL value with a "U" |
| | > CRQL but < Blank Result | Report at level of Blank Result with a "U" |
| | > Blank Result but < 10x the Blank Result | Use professional judgment to qualify results as estimated (J) |

## Mercury/Cyanide Linear Ranges

**Action:**

1. If any sample result was higher than the highest calibration standard for mercury/cyanide and the sample was not diluted to obtain the result reported on Form I, qualify the affected results ≥ MDL as estimated (J).

## Mercury/Cyanide Percent Solids of Sediments

**Action:**

1. If the percent solids in sediment for a sample are < 50%, qualify the affected results ≥ MDL as estimated (J) and the non-detects as estimated (UJ).

## Regional Quality Assurance (QA) and Quality Control (QC)

**Action:**

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Contract Laboratory Program Project Officer (CLP PO) action.

## Overall Assessment

**Action:**

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the QC criteria previously discussed.
2. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Note any discrepancies between the data and the Sample Delivery Group (SDG) Narrative for Contract Laboratory Program Project Officer (CLP PO) action. If sufficient information on the intended use and required quality of the data is available, the reviewer should include an assessment of the data usability within the given context.
3. If any discrepancies are found, the laboratory may be contacted by the Region's designated representative to obtain additional information for resolution. If a discrepancy remains unresolved, the reviewer may determine that qualification of the data is warranted.

## Calculations for Mercury

**Aqueous/Water Samples:**

$$Hg\ Concentration\ \left(\frac{\mu g}{L}\right) = C \times DF$$

**Where,**

C = Instrument value in µg/L from the calibration curve

DF = Dilution Factor

**Soil/Sediment Samples:**

$$Hg\ Concentration\ \left(\frac{mg}{kg}\right) = C \times \frac{1}{W \times S} \times \frac{DF}{10}$$

**Where,**

C = Instrument value in µg/L from the calibration curve

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D of ISM01.2 - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

**Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:**

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, multiply the value of the MDL (µg/L) or CRQL (µg/L) by the Dilution Factor (DF). Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

$$Adjusted\ Concentration\ \left(\frac{mg}{kg}\right) = C \times \frac{W_M}{W \times S} \times DF$$

**Where,**

C = MDL or CRQL (mg/kg)

$W_M$ = Minimum method required aliquot amount (g) (0.50 g)

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D of ISM01.2 - Introduction to Analytical Methods, Section 1.6).

DF = Dilution Factor

## Calculations for Cyanide

**Aqueous/Water Sample Concentration:**

$$CN\ Concentration\ \left(\frac{\mu g}{L}\right) = C \times \frac{V_f}{V} \times DF$$

**Where,**

C = Instrument value in µg/L CN from the calibration curve

$V_f$ = Final prepared (absorbing solution) volume (mL)

V = Initial aliquot amount (mL)

DF = Dilution Factor

**Soil/Sediment Sample Concentration:**

$$CN\ Concentration\ \left(\frac{mg}{kg}\right) = C \times \frac{V_f}{W \times S} \times \frac{DF}{1000}$$

**Where,**

C = Instrument value in µg/L CN from the calibration curve

$V_f$ = Final prepared (absorbing solution) volume (mL)

W = Initial aliquot amount (g)

S = % Solids/100 (see Exhibit D of ISM01.2 - Introduction to Analytical Methods, Section 1.6)

DF = Dilution Factor

**Adjusted Method Detection Limit (MDL)/Adjusted Contract Required Quantitation Limit (CRQL) Calculation:**

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, follow the instructions in Exhibit D of ISM01.2 – Data Analysis and Calculations, Section 11.1.1. Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

$$Adjusted\ Concentration\ \left(\frac{mg}{kg}\right) = C \times \frac{W_M}{W \times S} \times DF$$

**Where,**

| | | |
|---|---|---|
| C | = | MDL or CRQL (mg/kg) |
| $W_M$ | = | Minimum method required aliquot amount (g) (1.00 g for Midi or 0.50 g for Micro) |
| W | = | Initial aliquot amount (g) |
| S | = | % Solids/100 (see Exhibit D of ISM01.2 - Introduction to Analytical Methods, Section 1.6) |
| DF | = | Dilution Factor |

## APPENDIX A: GLOSSARY

**Analyte** -- The element of interest, ion, or parameter an analysis seeks to determine.
**Analytical Services Branch (ASB)** -- Directs the Contract Laboratory Program (CLP) from within the Office of Superfund Remediation and Technical Innovation (OSRTI) in the Office of Solid Waste and Emergency Response (OSWER).
**Analytical Sample** -- Any solution or media introduced into an instrument on which an analysis is performed excluding instrument calibration, Initial Calibration Verification (ICV), Initial Calibration Blank (ICB), Continuing Calibration Verification (CCV), and Continuing Calibration Blank (CCB). Note that the following are all defined as analytical samples: undiluted and diluted samples (USEPA and non-USEPA); Matrix Spike samples; duplicate samples; serial dilution samples, analytical (post-digestion/post-distillation) spike samples; Interference Check Samples (ICSs); Laboratory Control Samples (LCSs); and Preparation Blanks.
**Associated Samples** -- Any sample related to a particular Quality Control (QC) analysis. For example, for Initial Calibration Verification (ICV), all samples run under the same calibration curve. For duplicates, all Sample Delivery Group (SDG) samples digested/distilled of the same matrix.
**Blank** -- A sample designed to assess specific sources of contamination. See individual definitions for types of blanks.
**Calibration** -- The establishment of an analytical curve based on the absorbance, emission intensity, or other measured characteristic of known standards. The calibration standards are to be prepared using the same type of reagents or concentration of acids as used in the sample preparation.
**Calibration Blank** -- A blank solution containing all of the reagents in the same concentration as those used in the analytical sample preparation. This blank is not subject to the preparation method.
**Calibration Curve** -- A plot of instrument response versus concentration of standards.
**Calibration Standards** -- A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). The solutions may or may not be subjected to the preparation method, but contain the same matrix (i.e., the same amount of reagents and/or preservatives) as the sample preparations to be analyzed.
**Case** -- A finite, usually predetermined number of samples collected over a given time period from a particular site. Case numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).
**Continuing Calibration Blank (CCB)** -- A reagent water sample that is run 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN) and designed to detect any carryover contamination.
**Contract Compliance Screening (CCS)** -- A screening of electronic and hardcopy data deliverables for completeness and compliance with the contract. This screening is performed under USEPA direction by the Contract Laboratory Program (CLP) Sample Management Office (SMO) contractor.
**Continuing Calibration Verification (CCV)** -- A single parameter or multi-parameter standard solution prepared by the analyst and used to verify the stability of the instrument calibration with time, and the instrument performance during the analysis of samples. The CCV can be one of the calibration standards. However, all parameters being measured by the particular system must be

represented in this standard and the standard must have the same matrix (i.e., the same amount of reagents and/or preservatives) as the samples. The CCV should have a concentration in the middle of the calibration range and shall be run every 2 hours (ICP-AES, ICP-MS) or every hour (Hg, CN).

**Contract Laboratory Program (CLP)** -- Supports the USEPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known quality. This program is directed by the Analytical Services Branch (ASB) of the Office of Superfund Remediation and Technical Innovation (OSRTI) of USEPA.

**Contract Laboratory Program Project Officer (CLP PO)** -- The Regional USEPA official responsible for monitoring laboratory performance and/or requesting analytical data or services from a CLP laboratory.

**Contract Required Quantitation Limit (CRQL)** -- Minimum level of quantitation acceptable under the contract Statement of Work (SOW).

**Duplicate** -- A second aliquot of a sample that is treated the same as the original sample in order to determine the precision of the method.

**Field Blank** -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

**Field Duplicate** -- A duplicate sample generated in the field, not in the laboratory.

**Holding Time** -- The maximum amount of time samples may be held before they are processed.

**Contractual** -- The maximum amount of time that the Contract Laboratory Program (CLP) laboratory may hold the samples from the sample receipt date until analysis and still be in compliance with the terms of the contract, as specified in the CLP Analytical Services Statement of Work (SOW). These times are the same or less than technical holding times to allow for sample packaging and shipping.

**Technical** -- The maximum amount of time that samples may be held from the collection date until analysis.

**Initial Calibration** -- Analysis of analytical standards for a series of different specified concentrations to define the quantitative response, linearity, and dynamic range of the instrument to target analytes.

**Initial Calibration Blank (ICB)** -- The first blank standard run to confirm the calibration curve.

**Initial Calibration Verification (ICV)** -- Solution(s) prepared from stock standard solutions, metals, or salts obtained from a source separate from that utilized to prepare the calibration standards. The ICV is used to verify the concentration of the calibration standards and the adequacy of the instrument calibration. The ICV should be traceable to National Institute of Standards and Technology (NIST) or other certified standard sources when USEPA ICV solutions are not available.

**Internal Standard** -- A non-target element added to a sample at a known concentration after preparation but prior to analysis. Instrument responses to internal standards are monitored as a means of assessing overall instrument performance.

**Interference Check Sample (ICS)** -- Verifies the contract laboratory's ability to overcome interferences typical of those found in samples.

**Laboratory Control Sample (LCS)** -- A control sample spiked at known level(s). LCSs are processed using the same sample preparation, reagents, and analytical methods employed for the USEPA samples received.
**Matrix** -- The predominant material of which the sample to be analyzed is composed. For the purposes of this document, the matrices are aqueous/water, soil/sediment, wipe, and filter.
**Matrix Spike** -- Introduction of a known concentration of analyte into a sample to provide information about the effect of the sample matrix on the digestion and measurement methodology (also identified as a pre-distillation/digestion spike).
**Method Detection Limit (MDL)** -- The concentration of a target parameter that, when a sample is processed through the complete method, produces a signal with 99 percent probability that it is different from the blank. For 7 replicates of the sample, the mean value must be 3.14s above the blank, where "s" is the standard deviation of the 7 replicates.
**Narrative (SDG Narrative)** -- Portion of the data package which includes laboratory, contract, Case, Sample Number identification, and descriptive documentation of any problems encountered in processing the samples, along with corrective action taken and problem resolution.
**Office of Solid Waste and Emergency Response (OSWER)** – The USEPA office that provides policy, guidance, and direction for the USEPA's solid waste and emergency response programs, including Superfund.
**Percent Difference (%D)** -- As used in this document and the Statement of Work (SOW), is used to compare two values. The difference between the two values divided by one of the values.
**Performance Evaluation (PE) Sample** -- A sample of known composition provided by USEPA for contractor analysis. Used by USEPA to evaluate Contractor performance.
**Post Digestion Spike** -- The addition of a known amount of standard after digestion or distillation (also identified as an analytical spike).
**Preparation Blank** -- An analytical control that contains reagent water and reagents, which is carried through the entire preparation and analytical procedure.
**Relative Percent Difference (RPD)** -- As used in this document and the Statement of Work (SOW) to compare two values, the RPD is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).
**Regional Sample Control Center Coordinator (RSCC)** -- In USEPA Regions, coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.
**Relative Standard Deviation (RSD)** -- As used in this document and the Statement of Work (SOW), the mean divided by the standard deviation, expressed as a percentage.
**Sample** -- A single, discrete portion of material to be analyzed, which is contained in single or multiple containers and identified by a unique Sample Number.
**Sample Delivery Group (SDG)** -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

a. Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case; or
b. Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

c. Scheduled at the same level of deliverable.

In addition, all samples and/or sample fractions assigned to an SDG must be scheduled under the same contractual turnaround time. Preliminary Results have **no impact** on defining the SDG. Samples may be assigned to SDGs by matrix (i.e., all soil/sediment samples in one SDG, all aqueous/water samples in another) at the discretion of the laboratory.

**Sample Management Office (SMO)** -- A contractor-operated facility operated under the SMO contract, awarded and administered by the USEPA. Provides necessary management, operations, and administrative support to the Contract Laboratory Program (CLP).

**Serial Dilution** -- The dilution of a sample by a factor of five. When corrected by the Dilution Factor (DF), the diluted sample must agree with the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents [Inductively Coupled Plasma (ICP) only].

**Statement of Work (SOW)** -- A document which specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

**Tune** -- Analysis of a solution containing a range of isotope masses to establish Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) mass-scale accuracy, mass resolution, and precision prior to calibration.

## APPENDIX B: INORGANIC DATA EXECUTIVE NARRATIVE TEMPLATE

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

**EXECUTIVE NARRATIVE**

**Case No.**:
**Site**:

**SDG No.**:
**Laboratory**:

**QAPP**
HWSS #:
Contractor #:

**Number of Samples**:
**Sampling date**:

**SUMMARY:**

**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 have been qualified "J" estimated.

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

**Critical Findings:** None

**Major Findings:** None

**Minor Findings:** None

**COMMENTS:**

**Validator's Signature:** **Date:**
**Name:**
**Affiliation:**

**Approver's Signature:** **Date:**
**Name:**
**Affiliation:**

## APPENDIX C: SAMPLE INORGANIC DATA SAMPLE SUMMARY

| | | | |
|---|---|---|---|
| Case No: 00001 | Contract: XYZ1234 | SDG No: XY123 | Lab Code: ABCD |
| Sample Number: XY123 | Method: ICP_AES | Matrix: FLUFF | MA Number: DEFAULT |
| Sample Location: SOMEWHERE OUT THERE | pH: 15 | Sample Date: 13322059 | Sample Time: 24:03:00 |
| % Moisture : | | % Solids : | |

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable | Validation Level |
|---|---|---|---|---|---|---|---|
| Aluminum | 400 | ug/L | 1 | | | Yes | S2BVEM |
| Antimony | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Arsenic | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Barium | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Beryllium | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Cadmium | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Calcium | 400 | ug/L | 1 | | | Yes | S2BVEM |
| Chromium | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Cobalt | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Copper | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Iron | 40 | ug/L | 1 | J | U | Yes | S2BVEM |
| Lead | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Magnesium | 400 | ug/L | 1 | | | Yes | S2BVEM |
| Manganese | 400 | ug/L | 1 | | | Yes | S2BVEM |
| Nickel | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Potassium | 400 | ug/L | 1 | | | Yes | S2BVEM |
| Selenium | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Silver | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Sodium | 400 | ug/L | 1 | | | Yes | S2BVEM |
| Thallium | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Vanadium | 40 | ug/L | 1 | U | U | Yes | S2BVEM |
| Zinc | 40 | ug/L | 1 | U | U | Yes | S2BVEM |

## APPENDIX D: ELECTRONIC DATA DELIVERABLE TEMPLATE

| | | |
|---|---|---|
| DATA_PROVIDER | LAB_MATRIX_CODE | RESULT_UNIT |
| SYS_SAMPLE_CODE | ANAL_LOCATION | DETECTION_LIMIT_UNIT |
| SAMPLE_NAME | BASIS | TIC_RETENTION_TIME |
| SAMPLE_MATRIX_CODE | CONTAINER_ID | RESULT_COMMENT |
| SAMPLE_TYPE_CODE | DILUTION_FACTOR | QC_ORIGINAL_CONC |
| SAMPLE_SOURCE | PREP_METHOD | QC_SPIKE_ADDED |
| PARENT_SAMPLE_CODE | PREP_DATE | QC_SPIKE_MEASURED |
| SAMPLE_DEL_GROUP | LEACHATE_METHOD | QC_SPIKE_RECOVERY |
| SAMPLE_DATE | LEACHATE_DATE | QC_DUP_ORIGINAL_CONC |
| SYS_LOC_CODE | LAB_NAME_CODE | QC_DUP_SPIKE_ADDED |
| START_DEPTH | QC_LEVEL | QC_DUP_SPIKE_MEASURED |
| END_DEPTH | LAB_SAMPLE_ID | QC_DUP_SPIKE_RECOVERY |
| DEPTH_UNIT | PERCENT_MOISTURE | QC_RPD |
| CHAIN_OF_CUSTODY | SUBSAMPLE_AMOUNT | QC_SPIKE_LCL |
| SENT_TO_LAB_DATE | SUBSAMPLE_AMOUNT_UNIT | QC_SPIKE_UCL |
| SAMPLE_RECEIPT_DATE | ANALYST_NAME | QC_RPD_CL |
| SAMPLER | INSTRUMENT_ID | QC_SPIKE_STATUS |
| SAMPLING_COMPANY_CODE | COMMENT | QC_DUP_SPIKE_STATUS |
| SAMPLING_REASON | PRESERVATIVE | QC_RPD_STATUS |
| SAMPLING_TECHNIQUE | FINAL_VOLUME | BREAK_2 |
| TASK_CODE | FINAL_VOLUME_UNIT | SYS_SAMPLE_CODE |
| COLLECTION_QUARTER | CAS_RN | LAB_ANL_METHOD_NAME |
| COMPOSITE_YN | CHEMICAL_NAME | ANALYSIS_DATE |
| COMPOSITE_DESC | RESULT_VALUE | TOTAL_OR_DISSOLVED |
| SAMPLE_CLASS | RESULT_ERROR_DELTA | COLUMN_NUMBER |
| CUSTOM_FIELD_1 | RESULT_TYPE_CODE | TEST_TYPE |
| CUSTOM_FIELD_2 | REPORTABLE_RESULT | TEST_BATCH_TYPE |
| CUSTOM_FIELD_3 | DETECT_FLAG | TEST_BATCH_ID |
| COMMENT | LAB_QUALIFIERS | CASE |
| BREAK_1 | VALIDATOR_QUALIFIERS | CONTRACT_NUM |
| SYS_SAMPLE_CODE | INTERPRETED_QUALIFIERS | SCRIBE_SAMPLE_ID |
| LAB_ANL_METHOD_NAME | ORGANIC_YN | SAMPLE_TIME |
| ANALYSIS_DATE | METHOD_DETECTION_LIMIT | FRACTION |
| TOTAL_OR_DISSOLVED | REPORTING_DETECTION_LIMIT | PH |
| COLUMN_NUMBER | QUANTITATION_LIMIT | DATA_VAL_LABEL |
| TEST_TYPE | | |