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NATIONAL FUNCTIONAL GUIDELINES FOR CHEMICAL DATA REVIEW - (USED AS A REFERENCE IN OU3 RI/FS/PP)

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GUIDELINES

NATIONAL FUNCTIONAL GUIDELINES FOR CHEMICAL DATA REVIEW

Prepared for the

HAZARDOUS SITE EVALUATION DIVISION
U.S. ENVIRONMENTAL PROTECTION AGENCY

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INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation. In some aspects, it is equivalent to a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. These Guidelines have been updated to include all requirements in the 7/87 Statement of Work (SOW) for Inorganics, Amendment 1 and December 1987 Revisions.

Those areas where specific SOPs are possible are primarily areas in which definitive performance requirements are established. These requirements are concerned with specifications that are not sample dependent; they specify performance requirements on matters that should be fully under a laboratory's control. These specific areas include blanks, calibration standards, calibration verification standards, laboratory control standards, and interference check standards. In particular, mistakes such as calculation and transcription errors must be rectified by resubmission of corrected data sheets.

This document is intended for technical review. Some areas of overlap between technical review and Contract Compliance Screening (CCS) exist; however, determining contract compliance is not intended to be a goal of these guidelines. It is assumed that the CCS is available and can be utilized to assist in the data review procedure.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does not constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to rerun or resubmit data even if the previously submitted data have been utilized due to urgent program needs; data which do not meet specified requirements are never fully acceptable. The only exception to this requirement is in the area of requirements for individual sample analysis; if the nature of the sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

All data reviews must have, as a cover sheet, the Inorganic Regional Data Assessment (IRDA) form. (A copy is attached at the end of this document.) If mandatory actions are required, they should be specifically noted on this form. In addition, this form is to be used to summarize overall deficiencies requiring attention, as well as general laboratory performance and any discernible trends in the quality of the data. (This form is not a replacement for the data review.) Sufficient supplementary documentation must accompany the form to clearly identify the problems associated with a Case. The form and any attachments must be submitted to the Contract Laboratory Program Quality Assurance Coordinator (CLP QAC), the Regional Deputy Project Officer (DPO), and the Environmental Monitoring Systems Laboratory in Las Vegas (EMSL/LV).

It is the responsibility of the data reviewer to notify the Regional DPO concerning problems and shortcomings with regard to laboratory data. If there is an urgent requirement, the DPO may be contacted by telephone to expedite corrective action. It is recommended that all items for DPO action be presented at one time. In any case, the Inorganic Regional Data Assessment form must be completed and submitted.

PRELIMINARY REVIEW

In order to use this document effectively, the reviewer should have a general overview of the Case at hand. The exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved in their analysis are essential information. Background information on the site is helpful but often this information is very difficult to locate. The site project officer is the best source for answers or further direction.

CCS is a source of a large quantity of summarized information. It can be used to alert the reviewer of problems in the Case or what may be sample-specific problems. This information may be utilized in data validation. If CCS is unavailable, those criteria affecting data validity must be addressed by the data reviewer.

Cases routinely have unique samples which require special attention by the reviewer. Field blanks, field duplicates, and performance audit samples need to be identified. The sampling records should provide:

1. Project Officer for site
2. Complete list of samples with notations on
   - a) sample matrix
   - b) blanks*
   - c) field duplicates*
   - d) field spikes*
   - e) QC audit sample*
   - f) shipping dates
   - g) labs involved

   * If applicable

The chain-of-custody record includes sample descriptions and date of sampling. Although sampling date is not addressed by contract requirements, the reviewer must take into account lag time between sampling and shipping while assessing sample holding times.

INORGANICS PROCEDURE

The requirements to be checked in validation are listed below. ("CCS" indicates that the contractual requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.)

I. Holding Times (CCS - Lab holding times only)

II. Calibration

- o Initial (CCS)
- o Initial and Continuing Calibration Verification (CCS)

III. Blanks (CCS)

IV. ICP Interference Check Sample (CCS)

V. Laboratory Control Sample (CCS)

VI. Duplicate Sample (CCS)

VII. Matrix Spike Sample (CCS)

VIII. Furnace Atomic Absorption QC (CCS)

IX. ICP Serial Dilution (CCS)

X. Sample Result Verification (CCS - 10%)

XI. Field Duplicates

XII. Overall Assessment of Data for a Case

I. HOLDING TIMES

A. Objective

The objective is to ascertain the validity of results based on the holding time of the sample from time of collection to time of analysis.

Note: The holding time is based on the date of collection, rather than verified time of sample receipt, and date of digestion/distillation. It is a technical evaluation rather than a contractual requirement.

B. Criteria

Technical requirements for sample holding times have only been established for water matrices. The following holding time and preservation requirements were established under 40 CFR 136 (Clean Water Act) and are found in Volume 49, Number 209 of the Federal Register, page 43260, issued on October 26, 1984.

METALS:	6 months; preserved to pH < 2
MERCURY:	28 days; preserved to pH < 2
CYANIDE:	14 days; preserved to pH > 12

C. Evaluation Procedure

Actual holding times are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of analysis found in the laboratory raw data (digestion logs and instrument run logs). Examine the digestion and/or distillation logs to determine if samples were preserved at the proper pH.

Analyte Holding Time (Days) = Analysis Date - Sampling Date

D. Action

1. If 40 CFR 136 criteria for holding times and preservation are not met, qualify all results > Instrument Detection Limit (IDL) as estimated (J) and results < IDL as estimated (UJ).

2. If holding times are exceeded, the reviewer must use professional judgment to determine the reliability of the data and the effects of additional storage on the sample results. The expected bias would be low and the reviewer may determine that results < IDL are unusable (R).

3. Due to limited information concerning holding times for soil samples, it is left to the discretion of the data reviewer whether to apply water holding time criteria to soil samples. If the data are qualified when water holding time criteria are applied to soil samples, it must be clearly documented in the review.

II. CALIBRATION

A. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

B. Criteria

1. Initial Calibration

Instruments must be calibrated daily and each time the instrument is set up.

a. ICP Analysis

A blank and at least one standard must be used in establishing the analytical curve.

b. Atomic Absorption Analysis (AA)

1) A blank and at least three standards, one of which must be at the Contract Required Detection Limit (CRDL), must be used in establishing the analytical curve.

2) The correlation coefficient must be ≥0.995.

Note: The correlation coefficient of 0.995 is a technical criterion and not contractual.

c. Mercury Analysis

1) A blank and at least four standards must be used in establishing the analytical curve.

2) The correlation coefficient must be ≥0.995.

d. Cyanide Analysis

1) A blank and at least three standards must be used in establishing the analytical curve.

2) A midrange standard must be distilled.

3) A correlation coefficient ≥0.995 is required for photometric determination.

2. Initial and Continuing Calibration Verification (ICV and CCV)

a. Analysis results must fall within the control limits of 90 -110 percent Recovery (%R) of the true value for all analytes except mercury and cyanide.

b. Analysis results for mercury must fall within the control limits of 80-120%R.

c. Analysis results for cyanide must fall within the control limits of 85-115%R.

C. Evaluation Procedure

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank.

2. Verify that the correlation coefficient is ≥0.995

3. Check the distillation log and verify that the midrange CN standard was distilled.

4. Recalculate one or more of the ICV and CCV %R per type of analysis (ICP, GFAA, etc.) using the following equation and verify that the recalculated value agrees with the laboratory reported values on Form IIA. Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows (e.g., 89-111%).

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Where,

Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution

True = concentration (in ug/L) of each analyte in the ICV or CCV source

D. Action

1. If the minimum number of standards as defined in section B were not used for initial calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).

2. If the correlation coefficient is <0.995, qualify results > IDL as estimated (J), and results < IDL as estimated (UJ).

Note: For critical samples, further evaluation of the calibration curve may be warranted to determine if qualification is necessary.

3. If the midrange CN standard was not distilled, qualify all associated results 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 falls outside the acceptance windows but within the ranges of 75-89% or 111-125% (CN, 70-84% or 116-130%; Hg, 65-79% or 121-135%), qualify results > IDL as estimated (J).

   b. If the ICV or CCV %R is within the range of 111-125% (CN, 116-130%; Hg, 121-135%), results < IDL are acceptable.

   c. If the ICV or CCV %R is 75-89% (CN, 70-84%; Hg, 65-79%), qualify results < IDL as estimated (UJ).

   d. If the ICV or CCV %R is <75%, (CN, <70%; Hg, <65%), qualify all positive results as unusable (R).

   e. If the ICV or CCV %R is >125%, (CN >130%; Hg >135%), qualify results > IDL as unusable (R); results < IDL are acceptable.

III. BLANKS

A. Objective

The assessment of blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples. If problems with any blank exist, all data associated with the Case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the Case, or if the problem is an isolated occurrence not affecting other data.

B. Criteria

No contaminants should be in the blank(s).

C. Evaluation Procedures

Review the results reported on the Blank Summary (Form III) as well as the raw data (ICP printouts, strip charts, printer tapes, bench sheets, etc.) for all blanks and verify that the results were accurately reported.

D. Action

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Sample results > IDL but <5 times the amount in any blank should be qualified as (U).

Any blank with a negative result whose absolute value is > IDL must be carefully evaluated to determine its effect on the sample data.

Note: The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results reported on Form I will not be on the same basis (units, dilution) as the calibration blank data reported on Form III. The reviewer may find it easier to work from the raw data when applying 5X criteria to soil sample data/calibration blank data.

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 a contaminant. The results must not be corrected by subtracting any blank value.

IV. ICP INTERFERENCE CHECK SAMPLE (ICS)

A. Objective

The ICP Interference Check Sample verifies the contract laboratory's interelement and background correction factors.

B. Criteria

1. An ICS must be run at the beginning and end of each sample analysis run (or a minimum of twice per 8 hour working shift, whichever is more frequent).

2. Results for the ICS solution AB analysis must fall within the control limits of ± 20% of the true value.

C. Evaluation Procedure

1. Recalculate from the raw data (ICP printout) one or more of the recoveries using the following equation (%R) and verify that the recalculated value agrees with the laboratory reported values on Form IV.

$$\text{ICS } \%R = \frac{\text{Found Solution AB}}{\text{True Solution AB}} \times 100$$

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Where,

Found Solution AB = concentration (in ug/L) of each analyte measured in the analysis of solution AB

True Solution AB = concentration (in ug/L) of each analyte in solution AB

2. Check ICS raw data for results with an absolute value > IDL for those analytes which are not present in the ICS solution.

D. Action

1. For samples with concentrations of Al, Ca, Fe, and Mg which are comparable to or greater than their respective levels in the Interference Check Sample:

   a. If the ICS recovery for an element is >120% and the sample results are < IDL, this data is acceptable for use.

   b. If the ICS recovery for an element is >120% and the sample results are > IDL, qualify the affected data as estimated (J).

   c. If the ICS recovery for an element falls between 50 and 79% and the sample results are > IDL, qualify the affected data as estimated (J).

   d. If sample results are < IDL, and the ICS recovery for that analyte falls within the range of 50-79%, the possibility of false negatives may exist. Qualify the data for these samples as estimated (UJ).

   e. If ICS recovery results for an element fall <50%, qualify the affected data as unusable (R).

   Note: If possible, indicate the bias for the estimated results in the review.

2. If results > IDL are observed for elements which are not present in the EPA provided ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected elements should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS (false positives), qualify sample results > IDL as estimated (J).

3. If negative results are observed for elements that are not present in the EPA ICS solutions, and their absolute value is > IDL, the possibility of false negatives in the samples may exist. If the absolute value of the negative results is > IDL, an evaluation of the associated sample data should be made. For samples with comparable or higher levels of interferents, qualify results for the affected analytes < IDL as estimated (UJ).

4. In general, the 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. If these elements are present at concentrations greater than the level in the ICS, or other elements are present in the sample at >10 mg/L, the reviewer should investigate the possibility of other interference effects by using Table 2 given on page D-22 of the 7/87 SOW. These analyte concentration equivalents presented in the Table should be

considered only as estimated values, since the exact value of any analytical system is instrument specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is >2X CRDL and also greater than 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

V. LABORATORY CONTROL SAMPLE (LCS)

A. Objective

The laboratory control sample serves as a monitor of the overall performance of all steps in the analysis, including the sample preparation.

B. Criteria

1. All aqueous LCS results must fall within the control limits of 80-120%R, except Sb and Ag which have no control limits. 787/E-12

2. All solid LCS results must fall within the control limits established by the EPA. This information is available from EMSL/LV. 787/E-12

C. Evaluation Procedure

1. Review Form VII and verify that results fall within the control limits.

2. Check the raw data (ICP printout, strip charts, bench sheets) to verify the reported recoveries on Form VII. Recalculate one or more of the recoveries (%R) using the following equation:

$$\text{LCS \%R} = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Where,

LCS Found = concentration (in ug/L for aqueous; mg/kg for solid) of each analyte measured in the analysis of LCS solution

LCS True = concentration (in ug/L for aqueous; mg/kg for solid) of each analyte in the LCS source

D. Action

1. Aqueous LCS

a. If the LCS recovery for any analyte falls within the range of 50 - 79% or >120%, qualify results > IDL as estimated (J).

b. If results are < IDL and the LCS recovery is greater than 120%, the data are acceptable.

c. If results are < IDL and the LCS recovery falls within the range of 50-79%, qualify the data for the affected analytes as estimated (UJ).

d. If LCS recovery results are <50%, qualify the data for these samples as unusable (R).

2. Solid LCS

a. If the solid LCS recovery for any analyte falls outside the EPA control limits, qualify all sample results > IDL as estimated (J).

b. If the LCS results are higher than the control limits and the sample results are < IDL, the data are acceptable.

c. If the LCS results are lower than the control limits, qualify all sample results < IDL as estimated (UJ).

VI. DUPLICATE SAMPLE ANALYSIS

A. Objective

Duplicate analyses are indicators of laboratory precision based on each sample matrix.

B. Criteria

1. Samples identified as field blanks cannot be used for duplicate sample analysis.

2. A control limit of ± 20% (35% for soil) for the Relative Percent Difference (RPD) shall be used for sample values >5X CRDL.

3. A control limit of ±CRDL (±2X CRDL for soil) shall be used for sample values <5X CRDL, including the case when only one of the duplicate sample values is <5X CRDL.

C. Evaluation Procedure

1. Review Form VI and verify that results fall within the control limits.

2. Check the raw data and recalculate one or more RPD using the following equation to verify that results have been correctly reported on Form VI.

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

Where,

S = First Sample Value (original)
D = Second Sample Value (duplicate)

3. Verify that the field blank was not used for duplicate analysis.

D. Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same matrix as estimated (J).

2. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgment exercised when evaluating the data.

   Note: This information must be included on the IRDA form.

VII. MATRIX SPIKE SAMPLE ANALYSIS

A. Objective

The matrix spike sample analysis provides information about the effect of each sample matrix on the digestion and measurement methodology.

B. Criteria

1. Samples identified as field blanks cannot be used for spiked sample analysis.

2. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

C. Evaluation Procedure

1. Review Form V and verify that results fall within the specified limits.

2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported on Form V.

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

   Where,

   SSR = Spiked Sample Result
   SR = Sample Result
   SA = Spike Added

3. Verify that the field blank was not used for spike analysis.

D. Action

1. If the spike recovery is >125% and the reported sample results are < IDL, the data is acceptable for use.

2. If the spike recovery is >125% or <75% and the sample results are > IDL, qualify the data for these samples as estimated (J).

3. If the spike recovery falls within the range of 30-74% and the sample results are < IDL, qualify the data for these samples as estimated (UJ).

4. If spike recovery results fall <30% and the sample results are < IDL, qualify the data for these samples as unusable (R).

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5. If the field blank was used for matrix spike analysis, all other QC data must be carefully checked and professional judgment exercised when evaluating the data.

   Note: This information must be included on the IRDA form.

Note: If the matrix spike recovery does not meet criteria (except in Ag), a post digestion spike is required for all methods except furnace, but this data is not used to qualify sample results. However, this information must be included in the IRDA report.

VIII. FURNACE ATOMIC ABSORPTION QC

A. Objective

Duplicate injections and furnace post digestion spikes establish the precision and accuracy of the individual analytical determinations.

B. Criteria

1. For sample concentrations > CRDL, duplicate injections must agree within ±20% Relative Standard Deviation (RSD), (or Coefficient of Variation (CV)), otherwise the sample must be rerun once (at least two additional injections).

2. Spike recovery must be ≥85% and ≤115%.

3. The Furnace Atomic Absorption Scheme must be followed as described in the 7/87 SOW, p. E-15.

C. Evaluation Procedure

1. Check raw data to verify that duplicate injections agree within ±20% RSD (or CV) for sample concentrations > CRDL.

2. Review Furnace AA raw data to verify that the Furnace Atomic Absorption Scheme has been followed.

D. Action

1. If duplicate injections are outside the ±20% RSD (or CV) limits and the sample has not been rerun once as required, qualify the data as estimated (J).

2. If the rerun sample results do not agree within ±20% RSD (or CV), qualify the data as estimated (J).

3. If the post digestion spike recovery is <40%, qualify results > IDL as estimated (J).

4. If the post digestion spike recovery is ≥10%, but <40%, qualify results < IDL as estimated (UJ).

5. If the post digestion spike recovery is <10%, qualify results < IDL as unusable (R).

6. If sample absorbance is <50% of the post digestion spike absorbance then:

   a. If the furnace post digestion spike recovery is not within 85-115%, qualify the sample results > IDL as estimated (J).

   b. If the furnace post digestion spike recovery is not within 85-115%, qualify the sample results < IDL as estimated (UJ).

7. If Method of Standard Additions (MSA) is required but has not been done, qualify the data as estimated (J).

8. If any of the samples run by MSA have not been spiked at the appropriate levels, qualify the data as estimated (J).

9. If the MSA correlation coefficient is <0.995, qualify the data as estimated (J).

IX. ICP SERIAL DILUTION

A. Objective

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix.

B. Criteria

If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL), an analysis of a 5-fold dilution must agree within 10% Difference (%D) of the original results.

C. Evaluation Procedures

1. Check the raw data and recalculate the %D using the following equation to verify that the dilution analysis results agree with results reported on Form IX.

$$\%D = \frac{|I-S|}{I} \times 100$$

Where,

I = Initial Sample Result
S = Serial Dilution Result (Instrument Reading x 5)

2. Check the raw data for evidence of negative interference, i.e., results of the diluted sample are significantly higher than the original sample.

D. Action

1. When criteria are not met, qualify the associated data as estimated (J).

2. If evidence of negative interference is found, use professional judgment to qualify the data.

X. SAMPLE RESULT VERIFICATION

A. Objective

The objective is to ensure that the reported quantitation results are accurate.

B. Criteria

Analyte quantitation must be calculated according to the appropriate SOW.

C. Evaluation Procedures

The raw data should be examined to verify the correct calculation of sample results reported by the laboratory. Digestion and distillation logs, instrument printouts, strip charts, etc. should be compared to the reported sample results.

1. Examine the raw data for any anomalies (i.e., baseline shifts, negative absorbances, omissions, legibility, etc.).

2. Verify that there are no transcription or reduction errors (e.g., dilutions, percent solids, sample weights) on one or more samples.

3. Verify that results fall within the linear range of the ICP (Form XIII) and within the calibrated range for the non-ICP parameters.

4. Verify that sample results are >5X ICP IDL, if ICP analysis results are used for As, Tl, Se, or Pb.

Note: When the laboratory provides both ICP and furnace results for an analyte in a sample and the concentration is > ICP IDL, the results can assist in identifying quantitation problems.

D. Action

If there are any discrepancies found, the laboratory may be contacted by the designated representative to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer may determine qualification of the data is warranted.

XI. FIELD DUPLICATES

A. Objective

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than lab duplicates which measure only lab performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field samples.

B. Criteria

There are no review criteria for field duplicate analyses comparability.

C. Evaluation Procedures

Samples which are field duplicates should be identified using EPA Sample Traffic Reports or sample field sheets. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference (RPD), if appropriate.

D. Action

Any evaluation of the field duplicates should be provided with the reviewer's comments.

XII. OVERALL ASSESSMENT OF DATA FOR A CASE

It is appropriate for the data reviewer to make professional judgments and express concerns and comments on the validity of the overall data for a Case. This is particularly appropriate when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner, but the reviewer has a responsibility to inform the user concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. If qualifiers other than those used in this document are necessary to describe or qualify the data, it is necessary to thoroughly document/explain the additional qualifiers used. The data reviewer would be greatly assisted in this endeavor if the data quality objectives were provided. The cover form and supplementary documentation must be included with the review.

GLOSSARY A

Data Qualifier Definitions

For the purposes of this document the following code letters and associated definitions are provided.

U - 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 associated value is an estimated quantity.

R - The data are unusable. (Note: Analyte may or may not be present.)

UJ - The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

GLOSSARY B

Additional Terms

Associated Samples	Any sample related to a particular QC analysis. For example: - For ICV, all samples run under the same calibration curve. - For duplicate RPD, all SDG samples digested/distilled of the same matrix.
AA	Atomic Absorption
Calibration Curve	A plot of absorbance versus concentration of standards
Case	A finite, usually predetermined number of samples collected in a given time period for a particular site. A Case consists of one or more Sample Delivery Groups.
CCB	Continuing Calibration Blank - a deionized water sample run every ten samples designed to detect any carryover contamination.
CCS	Contract Compliance Screening - process in which SMO inspects analytical data for contractual compliance and provides EMSL/LV, laboratories, and the Regions with their findings.
CCV	Continuing Calibration Verification - a standard run every ten samples designed to test instrument performance.
CLP	Contract Laboratory Program
CRDL	Contract Required Detection Limit
CV	Coefficient of Variation
DPO	Deputy Project Officer
EMSL/LV	Environmental Monitoring System Laboratory/ Las Vegas (P.O. Box 15027, Las Vegas, Nevada 89114)
Field Blank	Field blanks are intended to identify contaminants that may have been introduced in the field. Examples are trip blanks, travel blanks, rinsate blanks, and decontamination blanks.

Field Duplicate	A duplicate sample generated in the field, not in the laboratory.
Holding Time	The time from sample collection to laboratory analysis.
ICB	Initial Calibration Blank - first blank standard run to confirm the calibration curve.
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample
ICV	Initial Calibration Verification - first standard run to confirm the calibration curve.
Initial Calibration	The establishment of a calibration curve with the appropriate number of standards and concentration range. The calibration curve plots absorbance or emission versus concentration of standards.
IRDA	Inorganic Regional Data Assessment
LCS	Laboratory Control Sample - supplied by EPA
MS	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.
MSA	Method of Standard Addition
Post digestion Spike	The addition of a known amount of standard after digestion. (Also identified as analytical spike, or spike, for furnace analyses.)
QAC	Quality Assurance Coordinator
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center
RSD	Relative Standard Deviation
Serial Dilution	A sample run at a specific dilution to determine whether any significant chemical or physical interferences exist due to sample matrix effects. (ICP only)

SDG	Sample Delivery Group - defined by one of the following, whichever occurs first: - case of field samples - each twenty field samples in a Case - each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the SDG.
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work

SDG	Sample Delivery Group - defined by one of the following, whichever occurs first: - case of field samples - each twenty field samples in a Case - each 14-day calendar period during which field samples in a Case are received, beginning with receipt of the first sample in the SDG.
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work

Region _____

INORGANIC REGIONAL DATA ASSESSMENT

CASE NO. _____ SITE _____

LABORATORY _____ NO. OF SAMPLES/ MATRIX _____

SDG# _____ REVIEWER (IF NOT ESD) _____

SOW# _____ REVIEWER'S NAME _____

DPO: ACTION _____ FYI _____ COMPLETION DATE _____

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES				
2.	CALIBRATIONS				
3.	BLANKS				
4.	ICS				
5.	LCS				
6.	DUPLICATE ANALYSIS				
7.	MATRIX SPIKE				
8.	MSA				
9.	SERIAL DILUTION				
10.	SAMPLE VERIFICATION				
11.	OTHER QC				
12.	OVERALL ASSESSMENT				

O = Data had no problems/or qualified due to minor problems.
M = Data qualified due to major problems.
Z = Data unacceptable.
X = Problems, but do not affect data.

ACTION ITEMS: _____

AREAS OF CONCERN: _____

NOTABLE PERFORMANCE: _____

Lucy Guzman

7601

REGION I

LABORATORY DATA VALIDATION

FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSES

Prepared for the

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PREFACE

The Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses (June 1988 revision) have been modified for use within Region I. The modifications were performed to delineate regionally required actions that were not specified in the June 1988 revision. Formats and procedures relating to information transfer within Region I have also been incorporated. By presenting additional guidance, this document will assist the reviewer in using professional judgement to make more informed decisions and in better serving the needs of the data users.

SCOPE OF THE CONTRACT LABORATORY PROGRAM ROUTINE ANALYTICAL SERVICES

Samples from hazardous waste sites are generally unpredictable, and may contain high concentrations of contaminants, complex mixtures of contaminants, and constituents that interfere with the analytical method. The Routine Analytical Services (RAS) Statement of Work (SOW) for the Contract Laboratory Program (CLP) is a contractual step-by-step method intended to obtain characterization of the contaminants present at the highest concentration and identification of samples that may need special methods for proper analysis. The CLP SOW requires a substantial amount of quality control and complete documentation of all steps employed during the analysis. This information is necessary for a reliable validation process, and is essential for litigation.

A major driving force of the CLP is to provide routine analysis of a large number of samples. For the majority of samples, valid, usable data are obtained. For those difficult samples, however, CLP provides sufficient documentation to indicate the nature of the problem. CLP Special Analytical Services (SAS) can then be employed to target the analysis problem. The risk of obtaining unusable data is highest when little site information is known or when interferences are present. EPA recognizes that the quality of the analytical results obtained by the same method may not be consistent for all samples.

As presented in this document, the reviewer assumes that the precision and accuracy of the CLP meets or exceeds the Data Quality Objectives (DQOs) for the sampling event. It is beyond the scope of this document to compare site-specific DQOs to the data quality obtained. However, the DQO Summary Form enclosed with each data package should enable the data reviewer to assess site-specific data usability for particular data sets.

<u>Summary</u>

1. The CLP analyzes large numbers of potentially complex environmental samples.

2. The SOW requires that analysis be attempted via a rigid protocol. Under normal circumstances, proper analysis is achieved. When complex or difficult samples are analyzed, the SOW focuses the laboratory's effort on the constituents present at the highest concentrations. For these samples, the SOW does not require that minimum detection limits be achieved.

3. The SOW requires a substantial amount of quality control and documentation as part of the analysis.

4. The required documentation permits full-scale data review by EPA.

ROLE OF THE REVIEWER

Upon completion of analysis, the CLP laboratory sends a replicate data package to each of the following: the Region requesting the analysis, EPA's Sample Management Office (SMO), and the Environmental Monitoring Systems Laboratory (EMSL-LV). It is SMO's responsibility to determine whether the laboratory was contractually compliant with the SOW through Contract Compliance Screening (CCS) as part of the government's right of inspection. On the other hand, the Regional data reviewer takes the results from the Statement of Work (SOW) analysis, receives input from the user as to objectives of the sampling effort, and attempts to determine if the data meet the user's needs. The driving force for Regional data validation is that contractually compliant data are not always technically useful and that contractually non-compliant data are sometimes very useful.

Throughout this document, data validation criteria are discussed from four perspectives: technical objective of the criteria, the criteria itself, evaluation of the criteria, and actions resulting from the review. Once the entire data set has been reviewed, a narrative report indicating the data quality and identifying specific problem areas is written for the user.

Summary

Data review requires that four objectives be simultaneously considered:

1. To assess and summarize the quality and reliability of CLP data for the user (usability);

2. To document (for the historical record of the site) factors affecting usability:

   a. discrepancies in the data,

   b. poor laboratory practice not regulated in the SOW, and

   c. site locations which are difficult to analyze;

3. To assist regional DPOs in monitoring CLP laboratory performance and maintaining good lab practices; and

4. To provide program personnel with information concerning the effectiveness of SOW methods and the CLP, and to identify problems requiring resolution by headquarters.

LABORATORY DATA VALIDATION
FUNCTIONAL GUIDELINES FOR EVALUATING INORGANICS ANALYSES

1.0 INTRODUCTION

This document is designed to offer guidance in laboratory data evaluation and validation. In some aspects, it is equivalent to a Standard Operating Procedure (SOP). In other, more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples. These Guidelines have been updated to include all requirements in the 7/87 Statement of Work (SOW) for Inorganics.

Those areas where specific SOPs are possible are primarily areas in which definitive performance requirements are established. These areas also correspond to specific requirements in Agency contracts and these requirements are concerned with specifications that are not sample dependent; they specify performance requirements on matters that should be fully under a laboratory's control. These specific areas include blanks, calibration standards, performance evaluation standard materials, and tuning. In particular, mistakes such as calculation and transcription errors must be rectified by resubmission of corrected data sheets.

This document is intended to provide guidance for technical data review. Some areas of overlap exist between technical review and Contract Compliance Screening (CCS); however, contract compliance is not intended to be a goal of these guidelines. It is assumed that the CCS is available and can be utilized to assist in the data review procedure.

Some requirements are not identical for every Case or batch of samples. Requirements for frequency of Quality Control (QC) actions are dependent on the number of samples, sample preparation technique, time of analysis, etc. Specific Case requirements and the impact of non-conformance must be addressed on a case by case basis; no specific guidance is provided.

At times, there may be an urgent need to use data which do not meet all contract requirements and technical criteria. Use of these data does <u>not</u> constitute either a new requirement standard or full acceptance of the data. Any decision to utilize data for which performance criteria have not been met is strictly to facilitate the progress of projects requiring the availability of the data. A contract laboratory submitting data which are out of specification may be required to re-run or submit data even if the previously submitted data have been utilized due to urgent program needs; data which do not meet specified requirements are never fully acceptable.

The only exception to this requirement is in the area of requirements for individual sample analysis; if the nature of the

sample itself limits the attainment of specifications, appropriate allowances must be made. The overriding concern of the Agency is to obtain data which are technically valid and legally defensible.

All data reviews must have, as a cover sheet, the inorganic regional data assessment (IRDA) form (see Appendix D, Figure 9). If mandatory actions are required, they should be specifically noted on this form. In addition, this form is to be used to summarize overall deficiencies requiring attention, as well as general laboratory performance and any discernible trends in the quality of the data. (This form is not a replacement for the data review.) Sufficient supplementary documentation must accompany the form to clearly identify the problems associated with a Case. The form and any attachments must be submitted to the Contract Laboratory Program Quality Assurance Officer (CLP QAO), the Regional Deputy Project Officer (DPO), and the Environmental Monitoring Systems Laboratory in Las Vegas (EMSL/LV).

It is the responsibility of the data reviewer to notify the Regional DPO concerning problems and shortcomings with regard to laboratory data in writing. If there is an urgent requirement, the DPO may be contacted by telephone to expedite corrective action. It is recommended that all items regarding a specific case needing DPO action be presented at one time. In any case, the inorganic regional data assessment form must be completed and submitted.

1.1 Overview of the Data Review Process

In Region I, the CLP DPO provides all data validation training and oversight of contractor work. The DPO approves data reviewer qualifications, presents workshops to teach step-by-step data validation and to introduce new regional guidelines and procedures, and audits data validation packages for technical content, format, and accuracy.

Appendix D, Figures 1 and 2 illustrate the normal flow of the data review process. Sources of information are noted, as well as communication channels and key decision points in the review process.

1.2 Information Available to the Reviewer

1.2.1 The Data Package

A. Objective

The CLP data package is designed to provide all necessary documentation to verify compliance to the Statement of Work (SOW) and to enable verification of the accuracy and reliability of the reported results.

B. Requirements

Items examined during CCS and contractual quality control requirements are presented in Appendix A and B of this document, respectively. A list of the required deliverables is contained in the SOW.

C. Evaluation Procedure

Procedures for the evaluation of specific deliverables are referenced in Sections 3 of this document.

D. Action

When contract-required information necessary for data validation is missing from the data package, the reviewer should call the laboratory and request the omitted data according to the procedure referenced in Section 1.3. Only authorized regional personnel may contact the laboratory.

1.2.2 Information Supplied by the Sampling Team

In order to use this document effectively, the reviewer should have a general overview of the case at hand; the exact number of samples, their assigned numbers, their matrix, and the number of laboratories involved for the analysis are essential information. Additional useful information includes the types of waste disposal, the overall purpose and goals of the sampling investigation, sample locations/descriptions (particularly identification of any residential wells within the sample set), and knowledge of any positive results from prior on-site sampling efforts. Background information on the site is very helpful and an effort should be made to obtain these data. The EPA site project officer or contractor site project manager are the best sources for answers or further direction.

1.2.2.1 Field QA/QC Samples

A. Objective

Field QA/QC samples, such as trip blanks, equipment blanks, and field duplicates, enable data reviewers to evaluate sampling conditions, techniques, and precision.

B. At a minimum, equipment blanks and duplicates must be included at a frequency of five percent.

C. Evaluation Procedure

The reviewer should evaluate the contamination found in the blanks as part of the lab method blank review, and the field duplicate precision concurrent with lab duplicate precision evaluation.

D. Action

1. If the laboratory has not provided results for any of the samples that were shipped, the reviewer should check the case narrative for a possible explanation (broken sample, insufficient sample for re-analysis, etc.). If no explanation is found, the RSCC should be contacted to further investigate and resolve the issue.

2. The sampler should be called if any of the following problems are noted:

   a. equipment blanks or field duplicates not identified; and

   b. anomalies such as traffic report numbers listed twice, etc.

1.2.2.2 Sample Descriptions

A. Objective

Sample descriptions/locations are necessary information for preparing the data summary tables and for the evaluation of holding times (see Section 3.2). (In addition, sample descriptions are useful as supplementary information for the consideration and discussion of matrix problems and chemical constituents identified in particular samples.)

B. Requirements

Copies of the traffic reports (see Appendix D, Figure 3) are mandatory deliverables in the laboratory data package. In addition, a copy of the chain-of-custody form (COC) (see Appendix D, Figure 4) may be provided in the data package or may be available to the reviewer from the sampling team. Both forms contain the date of sampling as well as the sampling locations.

C. Evaluation Procedure

Traffic reports and the COC form must be compared for

consistency with respect to the designation of quality control samples (blanks and field duplicates) and the identification numbers for field samples.

D. Action

1. If discrepancies are identified, the sampler should be contacted for resolution.

2. If information is illegible (sample descriptions or sampling date), the sampler should also be contacted to provide a legible copy of this information.

3. If traffic reports are missing, the laboratory should be contacted for this required deliverable.

1.2.3 Laboratory Contract Compliance Screening (CCS)

A. Objective

CCS provides a rapid, high volume assessment of deliverables for technical compliance to contract requirements. The primary application is to enable a determination of payment recommendation. Because of this direct link to payment, CCS fosters a somewhat timely resolution of contractual problems.

B. Requirements

Items examined by CCS are listed in Appendix A. The Sample Management Office (SMO) performs CCS on 100 percent of data packages submitted. Laboratories are required to submit all missing data, and resubmit or explain all data identified as non-compliant during CCS.

C. Evaluation Procedure

CCS must be used by the reviewer, when available, to evaluate those technical criteria that are also contractual criteria and to determine the completeness of the data package. The CCS results should be previewed to determine important compliance issues. The reviewer should compare the findings of the CCS to the laboratory data package in the course of data validation. A list of defect statements utilized by CCS is contained in Appendix B.

D. Action

1. If the CCS is not provided with the data package,

it should be requested through the RSCC. However, data review can still be initiated without CCS results, provided the reviewer assesses non-compliance issues which may affect the usability of the data.

2. If errors are noted on the CCS, they should be flagged and forwarded to the attention of the Region I DPO.

3. When a contract-required re-analysis or deliverable was noted as missing by CCS, the reviewer should call the laboratory to find out the expected delivery date.

1.2.4 Performance Evaluation Samples

The reviewer should contact the sampler to determine if any PE Samples were sent with the case. If they were, the reviewer should obtain all pertinent identification information (i.e. Traffic Report Nos., EPA identification no., etc.). Once this information is obtained, the data reviewer should call Cheryl O'Halloran at (617) 860-4614 to obtain the true values of the sample for comparison to the laboratory results.

1.3 The Regional/Laboratory Communication Network

A. Objective

In January 1983, the National Program Office established a system of direct communication between the regions and contract laboratories as a routine method for regional data review staff to obtain answers to technical questions concerning program data in the timeliest and most direct manner possible.

B. Requirements

The ground rules for this system are as follows:

1. Regional contact of laboratories is permissible only after laboratory data submission.

2. All logistical questions involving data delivery, contractual requirements, procedural recommendations, and other general matters are to be referred to the RSCC, to SMO, or to program management (i.e., DPO) as appropriate. In addition, re-analysis requests originating from the data reviewer must be channeled through the RSCC or the Region I DPO.

3. Only authorized regional personnel may contact laboratories and they may contact only the specified laboratory personnel.

C. Procedure

1. All conversations between the regions and the laboratories are recorded by both the laboratory and the regional contact on the CLP telephone record log (Appendix D, Figure 5).

2. One copy of the telephone record log is forwarded by the reviewer to each of the following:

   - SMO
   - the Region 1 laboratory DPO
   - the laboratory
   - RSCC

D. Action

1. When requesting information from the laboratory, the insistence on short deadlines by the data reviewer during the initial contact has been shown to be the primary factor in minimizing the time required to get an answer.

2. The four types of problems that require direct contact between the reviewer and the laboratory for resolution are illustrated in the attached flow chart (Appendix D, Figure 2) and are described below:

   a. In the case of missing or illegible deliverables, the reviewer should call the laboratory to establish and record the expected due date for the requested deliverable.

   b. When a contract-required re-analysis necessary for data validation is missing, the reviewer should check to see if the problem was noted by the CCS. If so, the reviewer should call the laboratory to find out the expected due date. If the problem was not noted by CCS, the reviewer, in conjunction with the site project manager, must decide whether initiation of a re-analysis request would provide usable data (weighing a consideration of holding times, etc.). To initiate a re-analysis request, the reviewer must contact the RSCC or the Region I laboratory DPO.

c. Clarification of discrepancies or errors in the reported data usually requires correction and re-submission of results by the laboratory. If the laboratory does not verify the error, then the reviewer should doublecheck his work to insure the accurate reporting and qualification of data.

d. In some cases, it may be necessary to have the laboratory provide certain explanations or details of the conditions of analysis that do not correspond to any of the SOW required deliverables. In such cases, a verbal answer is all that is required of the laboratory.

1.4 The DPO Communication Network

Similar to the above, DPO communications involve contact with project officers, laboratories, SMO, and data reviewers. The DPO receives numerous reports from SMO and EMSL-LV. Those which relate directly and specifically to data review will be forwarded to reviewers as appropriate. The DPOs will also provide updates to SOW protocols as they are received.

Inter-regional questions or problems with laboratory performance are referred to DPOs for resolution. For instance, if Region 1 data reviewers uncovered a possible contamination problem in a laboratory assigned to Region 2, the problem is first referred to the Region 1 DPO who then contacts the DPO in Region 2.

It is recommended that the DPO be notified of all problems and requirements for a case at one time. If there is an urgent requirement, the DPO may be contacted by phone to expedite corrective action. A data validation report with the ORDA form as a cover page must be submitted to the DPO to provide documentation of the data review, and for resolution of inter-regional problems.

1.5 The Data Validation Report

A. Objective

The findings of the review are distributed to readers for three distinct applications: site assessment, oversight of Contract Laboratory Program (CLP) laboratory and method performance, and oversight of the data review contractors performance. For individuals involved in site-related decisions, it is imperative that the data validation report present a clear explanation of those issues affecting the application

of the data to the site investigation. The report must provide the data users with an overview of data quality, but should also explain the qualitative confidence and quantitative error associated with all individual results. In addition, the data users require a condensed form of the analytical results which includes all positive sample results, detection limits, and associated qualifier codes.

On the other hand, the EPA individuals responsible for management and oversight of CLP laboratory and method performance require a presentation of issues related to laboratory non-compliance, poor laboratory practice not regulated in the SOW, and any unusual method or analytical problems.

For both contractual issues and problems affecting the usability of the data for site assessment, support documentation must be sufficient to allow EPA to perform a full-scale review of the data validation in order to substantiate the report's conclusions.

B. Requirements

In order to meet the needs of the data users, a four-part data validation report and an Inorganic Regional Data Assessment form are the required data validation deliverables.

1. DQO Summary Form
The DQO Summary Form submitted with the one-month projections must be included in the data validation report. An example of a blank form and a completed form is contained in Appendix D, Figure 6 and Appendix C, respectively.

2. Memo

This should briefly identify the scope of the analytical effort, provide a general overview of data quality, and list and interpret specific problem areas that affect the usability of the data.

All memos must be addressed to the RSCC and the Region I DPO must be copied.

The following parameters must be covered in the memo:

- A brief summary of the type and number of samples analyzed, the case and SDG numbers, the

site, the laboratory that performed the analysis, the type of analysis performed, whether routine or non-routine protocols were followed, the parameters evaluated, and the type and number of field QC samples included in the sample set.

- A balanced perspective of the data quality must be provided by summarizing the usable aspects of the data and areas of compliant laboratory performance, as well as qualifying problems as having either a major or minor impact on data usability.

- For each result which has been qualified, the memo must identify the associated analytical problem or quality control criterion which was not met, and explain the resultant effect on the data. The memo must clearly differentiate problems affecting the confidence concerning the presence of a compound versus those involving quantitative error. The message to the user should be conveyed in simple, concise language that an individual without an extensive background in analytical chemistry could understand. The narrative should also list or reference all changes that the reviewer has made to the laboratory's reported data, whether due to misidentification or errors in transcription or calculation. Lastly, the narrative should identify support documentation attachments and should include the reviewer's name and signature. The format of the memo, and a brief explanation of how it is to be completed follows:

3. Data Summary

Separate tables in Lotus 1,2,3 have been developed for soil and water analyses and for organics and inorganics analyses. These tables must include all positive sample results, IDLs and CRDLs and associated qualifier codes. Traffic report numbers, sample locations/descriptions, matrix, concentration units, site name, case number, and SDG number should be clearly identified. Examples of the data summary tables are provided in Appendix D, Figure 7 and Appendix C. Only codes defined by this document are permitted to qualify data. Should it be necessary to include other codes, prior approval must be obtained through the Region I DPO. If approval is given, complete definitions must be supplied in the key for data summary tables. The glossary of standard codes for use in qualifying data as per this SOP is located in Glossary A.

4. Standard Worksheets

These worksheets must be utilized to perform the data review. Examples of blank and completed worksheets can be found in Appendix D, Figure 8 and Appendix C, respectively.

5. Inorganic Regional Data Assessment Form (IRDA)

The IRDA delineates those issues relating to laboratory non-compliance, poor laboratory practice, and any unusual method or analytical problems resulting in unacceptable or qualified data. The form contains a checklist of areas verified during the review with notations on data qualification in each area. DPO Action items must be detailed and documented, with notations indicating which issues require special attention or follow-up action. Specific recommended actions should be noted on the IRDA. An example of a blank form and a completed form is contained in Appendix D, Figure 9 and Appendix E, respectively. Appendix E also contains guidance for completing the IRDA.

6. Support Documentation

All problems identified either in the narrative report or on the IRDA should be documented by the attachment of laboratory forms, raw data, or reviewer-prepared tabulations that substantiate the findings and conclusions presented in the text. (On the other hand, it is not

necessary to attach support documentation to substantiate compliant laboratory performance in cases where there is no problem with data usability.) It is recommended that support documentation attachments be numbered or labeled and referenced accordingly in the text of the narrative report and on the IRDA. In addition, it is helpful if the reviewer circles the specific items of concern located on these attachments.

C. Procedure

Sections 2, 3 and 4 of this document specify the procedures for the preparation of all sections of the data validation report. As a final step in this process, it is important that a senior data reviewer check all outgoing reports for accuracy and completeness, due to the complexity of the data validation and the importance of an accurate final assessment of data quality.

D. Distribution

1. A. One copy of the complete four-part validation report (DQO Summary Form, Memo, Summary Tables, and Standard Worksheets) is distributed to:

   - the Region I RSCC
   - the Region I DPO

   B. One copy of the Inorganic Regional Data Assessment Form with memo and Support Documentation is distributed to each of the following:

   - the Region I DPO (without memo);
   - the DPO for the laboratory that performed the analysis;
   - the EPA HQ Quality Assurance officer; and
   - the designated recipient at EMSL-LV.

   (Names and addresses will be forwarded to the reviewer and periodically updated by the Region I DPO.)

2. The cognizant EPA contract manager may require the data reviewer to send a preliminary copy of the completed data summary, glossary, and sample location map to the EPA site project officer immediately upon completion of the reviewer's evaluation (but prior to typing and submission of the narrative with the rest of the sampling investigation report).

E. Action

EPA will review and comment upon contractor-prepared data validation reports. Resubmission of data validation reports may be required in cases where the required format and procedures were not followed, or when clarifications or corrections are needed.

2.0 PRELIMINARY REVIEW

2.1 The Data Summary

A. Objective

The purpose of the data summary is to provide a simple, condensed form of the analytical results to the user, which enables a quick evaluation and comparison of the constituents identified at the various sampling locations.

B. Requirements

Requirements have been addressed above under Section 1.5.

C. Procedure

Transcribe the results from the Form Is onto the data summary tables (for samples of data summary sheets see Appendix D, Figure 7). Do not transcribe the qualification codes used by the laboratory. The reviewer will fill in the usability qualifiers which have been determined through the validation process.

D. Action

As appropriate, information will be added to or deleted from the data summary during the course of data validation.

2.2 Usage of Qualifier Codes on the Data Summary

A. Objective

The data qualifier codes presented in Glossary A identify the degree of confidence concerning the presence (or absence) of reported analytes and identify results that are considered to be quantitatively inaccurate. These codes have been regionally standardized to insure that contractors throughout the region all employ the same set of simple, concise definitions that are understandable to personnel within the various EPA branches.

B. Requirements

1. Only codes defined in Glossary A are permitted to qualify data. Should it be necessary to include other codes, prior approval must be obtained through the Region I DPO.

2. In general, only one qualifier code is permitted with each reported result. The following hierarchy has been developed to insure that only the most important code is used in situations where more than one quality control problem is associated with an analytical result:

   a. Codes relating to identification take precedence over codes related to quantitation. Thus, whenever a positive result is qualified with an R, the J code will not be used. Also, whenever a not-detected result is qualified with an R, the code UJ will not be used. The qualifier R should replace any numerical value that the laboratory reported.

   b. Within each of the two categories of codes, the code that indicates a more serious problem with the data takes precedence.

3. The above restriction on the use of multiple qualifiers for a single result is applicable only to the data summary and not to the narrative report. (The narrative should mention major, as well as minor, problems associated with individual results, using appropriate emphasis.)

C. Procedure/Action

Section 3 of this document addresses the individual situations requiring use of particular qualifier codes. A summary of qualifier codes is presented in Glossary A. Upon completion of the data validation, the reviewer should doublecheck the data summary for accuracy and completeness to insure that the appropriate qualifier codes were added according to the requirements listed herein.

3.0 INORGANIC DATA VALIDATION PROCEDURE

The requirements to be checked in validation are listed below: ("CCS" indicates that the contractual requirements for these items will also be checked by CCS; CCS requirements are not always the same as the data review criteria.)

I. Data Completeness

II. Holding Times (CCS checks Lab holding times only)

III. Calibration

- Initial (CCS)
- Continuing (CCS)

IV. Blanks (CCS)

V. ICP Interference Check Sample (CCS)

VI. Matrix Spike Sample Analysis (CCS)

VII. Laboratory Duplicates Sample Analysis (CCS)

VIII. Field Duplicate Sample Analysis

IX. Laboratory Control Sample Analysis (CCS)

X. Furnace Atomic Absorption Analysis (CCS)

XI. ICP Serial Dilution Analysis (CCS)

XII. Detection Limits (CCS)

XIII. Sample Result Verification (CCS 10%)

XIV. Overall Assessment of Data for a Case

I. DATA COMPLETENESS

Check that the data package is complete and contains all the required deliverables listed in SOW 7/87.

If any of the required data are missing, contact the laboratory to request the information and prepare a Telephone Record Log. Review CCS results to see if a request was made for missing information and find out the expected delivery date.

II. HOLDING TIMES

A. Objective

The objective is to ascertain the validity of results based on the holding time of samples from time of collection to time of analysis or sample preparation, as appropriate.

B. Criteria

Technical requirements for sample holding times (HT) have only been established for water matrices. The holding times for soils are currently under investigation. Until the results are available, the holding times for water matrices should be applied to soil matrices. The following holding time and preservative requirements were established under 40 CFR 136 (Clean Water Act) and are found in Volume 49, Number 209 of the Federal Register, page 43260, issued October 26, 1984. Preservation refers to waters only.

METALS: 6 months; preserved at pH<2

MERCURY: 28 days; preserved at pH<2

CYANIDE: 14 days; preserved at pH>12

C. Evaluation Procedure

Actual holding times are established by comparing the sampling date on the EPA Sample Traffic Report with the dates of analysis, these dates can be found in the laboratory raw data. Examine the sample records to determine if samples were properly preserved.

METALS: HT (Days)= Analysis Date - Sampling Date

MERCURY: HT (Days) = Analysis Date - Sampling Date

CYANIDE: HT (Days) = Analysis Date - Sampling Date

NOTE: **Holding times for Cyanide are calculated based on the date of analysis and not on distillation date.** If the samples were distilled within holding times and analyzed a couple days later it may be acceptable to use the data unqualified. However,

if several days pass between distillation and analysis, the data may be questionable and need to be qualified. Professional judgement must be used.

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D. Action

1. If 40 CFR 136 criteria for holding times and preservation are not met, qualify all positive results as estimated (J) and results below the Instrument Detection limit (IDL) as estimated (UJ).

2. If holding times are grossly exceeded, the reviewer must use professional judgement to determine the reliablity of the data and the effects of additional storage on the sample results. The reviewer may determine that non-detect data are unusable (R).

III. CALIBRATION

A. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run, and continuing calibration verification documents that the initial calibration is still valid.

B. Criteria

1. Initial Calibration

Instruments must be calibrated daily, and each time the instrument is set up.

a. ICP Analysis

A blank and at least one standard must be used in establishing the analytical curve.

b. Atomic Absorption Analysis (AA)

A blank and at least three standards, one of which must be at the Contract Required Detection Limit (CRDL), must be used in establishing the calibration curve.

The correlation coefficient must be $\geq$0.995.

c. Mercury Analysis

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A blank and at least four standards must be used in establishing the analytical curve.

The correlation coefficient must be ≥ 0.995.

d. Cyanide Analysis

A blank and at least three standards must be used in establishing the analytical curve.

A correlation coefficient ≥ 0.995 is required for photometric determination.

At least one mid-point standard must be distilled before analysis.

2. Initial and Continuing Calibration Verification

a. An EPA certified standard must be used for the Initial Calibration Verification (ICV) and must be analyzed for each wavelength used for analysis.

b. Analysis results must fall within the control limits of 90-110%R of the true value for all analytes except mercury and cyanide.

c. Analysis results for mercury must fall within the control limits of 80-120%R.

d. Analysis results for cyanide must fall within the control limits of 85-115%R.

e. A CCV must be analyzed every ten samples or every 2 hours whichever is more frequent.

f. To verify linearity near the CRDL for ICP analysis, the contractor must analyze a standard at 2 times the CRDL or 2 times the IDL whichever is greater. There are no specific acceptance criteria for the standard at the present time. Professional judgement must be used in qualifying the data.

C. Evaluation Procedure

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and a blank.

2. Verify that a standard at the CRDL was used in the

AA calibration curve or that a standard at the CRDL was analyzed at the beginning of the analysis run.

3. Verify the correlation coefficient is $\geq$0.995 for all AA, Hg, and CN analyses.

4. Check the distillation log to verify that a mid-range standard was distilled for cyanide analysis.

5. Verify that an EPA certified standard was used for the ICV for all analyses.

6. Verify that all ICV and CCV recoveries fall within the required windows.

7. Check the raw data to verify that the calibration standard values were transcribed correctly on to Form II. Recalculate one or more of the ICV and CCV percent recoveries (%R) using the following equation and verify that the recalculated value agrees with the laboratory reported values on Form IIA. Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows.

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Where,

Found = concentration (in ug/L) of each analyte measured in the analysis of the ICV or CCV solution.

True = concentration (in ug/L) of each analyte in the ICV or CCV source.

8. Verify that a CCV was analyzed every 10 samples or every 2 hours.

9. Verify that a standard at 2xCRDL or 2xIDL, whichever is greater, was analyzed to verify linearity near the IDL for ICP analysis.

D. **Action**

1. If the **appropriate number of standards** were not used for the initial calibration or if the instrument was **not calibrated daily** and each time the instrument was setup, professional judgement must be used to qualify the data. It may be necessary to qualify the data as unusable **(R)**.

2. If a standard at the CRDL was not used in establishing the calibration curve for AA, positive results up to 2xCRDL and non-detected results may have to be estimated. Examine the recoveries of any low level standards analyzed during the analysis scheme in order to make judgements on the accuracy of the calibration curve at the low end.

3. If the correlation coefficient is <0.995 for AA, Hg, or CN, qualify results >IDL as estimated (J), and non-detected results as estimated (UJ).

4. If a mid range standard for cyanide was not distilled before analysis or did not meet the 10% criteria, use professional judgement to qualify the data. It may be necessary to estimate results if it appears the distillation procedure has a significant impact on sample results.

5. If an EPA standard was not used for the ICV, check the source of the standard used. The ICV must be from a different source than the CCV or the data is questionable.

6. If the ICV or CCV %R falls outside the acceptance windows, use professional judgement to qualify all associated data. The following guidelines are recommended:

   a. If the ICV or CCV %R falls outside the acceptance windows but within the ranges of 75-89%, or 111-125% (CN, 70-84% or 116-130%; Hg, 65-79% or 121-135%), qualify results >IDL as estimated (J).

   b. If the ICV or CCV %R fall outside the acceptance windows but is within the range of 111-125% (CN, 116-130%; Hg, 121-135%), non-detected results are not qualified.

   c. If the ICV or CCV %R is 75%-89% (CN, 70-84%; Hg, 65-79%), qualify non-detected results as estimated (UJ).

   d. If the ICV or CCV %R falls outside the range of 75-125% (CN,70-130%; Hg, 65-135%), qualify all associated results as unusable (R).

7. If the CCV was not analyzed at the required intervals, use professional judgement to qualify the data. The data may be unaffected.

8. If the 2xCRDL standard for ICP is not within ±20% results near the CRDL are questionable. Estimate (J) positive results less than 3xCRDL and (UJ) non-detected results.

IV. BLANKS

The assessment of blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples. If problems with any blank exist,(ie. prep blank, calibration blank, equip[ment blank, or trip blank) all data associated with the Case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the Case, or if the problem is an isolated occurrence not affecting the data.

B. Criteria

1. No contaminants should be detected in the blanks.

2. A preparation blank must be analyzed for each matrix, for every 20 samples digested, or for each batch digested, whichever is more frequent.

3. A calibration blank (CCB) must be analyzed after every ten samples or every 2 hours, whichever is more frequent.

C. Evaluation Procedure

1. Review the results reported on the Blank Summary (Form III) as well as the raw data (ICP printouts, strip charts, printer tapes, bench sheets, etc.) for all blanks and verify that the results were accurately reported.

2. Verify that the calibration blanks and prep blanks were analyzed at the proper frequency.

3. Report results >IDL for any trip blanks or equipment blanks taken with the Case.

D. Action

1. Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Any blank with a value below the negative IDL must be carefully evaluated to determine it's effect on the sample data. Blanks that are >2x the negative IDL indicate instrumental problems. The possibility of

false negatives exist , therefore the associated data must be qualified.

2. 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 a contaminant. The result must not be corrected by subtracting any blank value. Action levels should be calculated that are 5 times the maximum concentration of each contaminant detected in any blank. No positive sample results should be reported unless the concentration of the analyte in the sample exceeds 5 times the amount detected in any blank.

NOTE: The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil sample results reported on Form I will not be on the same basis (units, dilutions) as the calibration blank data reported on Form III or the aqueous field blank. Sample weights, volumes, and dilution factors must be taken into consideration when applying the 5x criteria. The reviewer may find it easier to work from the raw data when applying the 5x criteria to soil sample data/calibration blank data.

Sample results should be reported as follows:

a. If an analyte is detected in the blank but not in the sample no action is taken.

b. Positive results less than the action level shall be reported with a U.

c. Positive results greater than the action level shall be reported unqualified.

4. The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines. However, DPO approval for any deviation is required.

EXAMPLE 1:

Blank Result	7
IDL	5
Action Level	35
Sample Result	22
Qualified Sample Result	22 U

The sample result is > IDL but < the Action Level. Positive results less than 35 are qualified as non-detects.

EXAMPLE 2:

Blank Result	10
IDL	8
Action Level	50
Sample Result	70
Qualified Sample Result	70

The sample result is greater than the IDL and the Action Level so no qualifiers are necessary.

V. ICP INTEFERENCE CHECK SAMPLE

A. Objective

The ICP Interference Check Sample (ICS) Analysis is performed to verify the contract laboratory's interelement and background correction factors.

B. Criteria

1. An ICS must be run at the beginning and end of each sample analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent.

2. Results for the ICS solution AB analysis must fall within the control limits of [redacted] of the true value.

C. Evaluation Procedure

1. Verify the ICS was analyzed at the proper frequency.

2. Verify [redacted]

3. Recalculate from the raw data one or more recoveries using the following equation and verify that their calculated value agrees with the laboratory reported values on Form IV.

$$\text{ICS } \%R = \frac{\text{Found Solution AB}}{\text{True Solution AB}} \times 100$$

Where,

Found Solution AB = concentration (in ug/L) of each analyte measured in the analysis of solution AB.

True Solution AB = concentration (in ug/L) of each analyte in solution AB.

NOTE: The ICS solution may be diluted to bring the levels of the interferents (Ca, Mg, Fe, Al) within the linear range of the instrument. Dilutions may not be used for quantitation of any other elements.

4. Check ICS raw data for results with an absolute value >IDL for those analytes which are not present in the ICS A solution. Results greater than twice the absolute value of the IDL indicate either a positive or negative interference and must be qualified.

D. Action

1. If the ICS was not analyzed at the proper frequency the data may be affected. Use professional judgement to qualify the data.

2. For samples with concentrations of Al, Ca, Mg, and Fe which are 50% or more than their respective levels in the Interference Check Sample, the following actions are recommended:

   a. If the ICS recovery for an element is >120% and the reported sample results are non-detected, data are acceptable for use.

   b. If the ICS recovery for an element is >120% and the reported sample results are >IDL, qualify the affected data as estimated (J). Results may be biased high.

   c. If the ICS recovery for an element falls between 50 and 79% and reportable quantities of the analyte were detected, qualify the affected data as estimated (J). Results may be biased low.

   d. If an analyte is not detected in the sample, and the ICS recovery for that analyte falls within the range of 50-79%, the possiblity of false negatives may exist. Qualify the data for these samples as estimated (UJ).

   e. If ICS recovery results for an element are <50%, qualify the data as unusable (R).

3. If results >IDL are observed for elements which are not present in the EPA provided solution, the possibility of false positives exists. An evaluation

of the associated sample data for the affected elements should be made. Sample results > 2xIDL with levels of interferents that are 50% or more of the levels found in the ICS solution should be qualified as estimated (J). Certain circumstances may warrant rejection of the data (R) if it is impossible to determine whether the sample result is due entirely to interferences or not.

4. If negative results with absolute values >2xIDL are observed for elements which are not present in the EPA ICS solutions, the possibility of false negatives in the samples may exist. An evaluation of associated sample data should be made. For samples with levels of interferents that are 50% or more than the levels found in the ICS solution all results for the affected analytes which are reported as non-detected should be qualified as (UJ).

5. In general, the sample data can be accepted if the concentrations of Al, Ca, Mg, and Fe in the samples are found to be <50% of their respective concentrations in the ICS. However, if other elements are present in the sample at >10 mg/L, the reviewer should investigate the possibility of other inter- ference effects by using Table 2 given on page D-22 of the 7/87 SOW. These analyte concentration equiva-lents presented in the references should be considered only as estimated values, since the exact value of any analytical system is laboratory specific. Therefore, estimate the concentration produced by an interfering element. If the estimate is >2X CRDL and also greater than 10% of the reported concentration of the affected element, qualify the affected results as estimated (J).

The following are examples of estimating interferences and qualifying affected data.

EXAMPLE 1:

A positive result of 1050 ug/L was detected for Antimony in the ICS solution although there should not be any Antimony present in the solution. A review of sample concentrations indicate the levels of interferents in the samples are at least 50% of those found in the ICS solution (300,000 ug/L Aluminum and 150,000 ug/l Iron). Positive results for Antimony must be estimated (J) since they may be due to interferences from Iron or Aluminum.

Aluminum Result	300,000
Iron Result	150,000
Antimony Result	700
Qualified Antimony Result	700 J

EXAMPLE 2:

Samples contain 30,000 ug/L Vanadium. Table 2 on page D-22 of SOW 7/87 indicates the interelement interference factor for Vanadium on Beryllium is 0.05 mg/L Beryllium for every 100 mg/L of Vanadium. Estimating the concentration of Beryllium which is due to Vanadium yields a result of:

$$30{,}000 \text{ ug/L} \times \frac{0.05 \text{ mg/L}}{100 \text{ mg/L}} = 15 \text{ ug/L Beryllium.}$$

Vanadium Result	30,000
Reported Beryllium Result	70
Estimated Interference	15
Qualified Sample Result	20 J

The estimated concentration of Beryllium signal due to the interference of Vanadium is >2xCRDL and >10% of the reported concentration therefore the reported value is estimated (J).

VI. MATRIX SPIKE SAMPLE ANALYSIS

A. Objective

The matrix spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology.

B. Criteria

1. Samples identified as field blanks cannot be used for spiked sample analysis.

2. Spike recovery (%R) must be within the limits of 75-125%. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

3. If the matrix spike recovery does not meet criteria, a post-digestion spike is required and reported on Form VB for ICP, Flame, Mercury and Cyanide. (Post digestion spikes are also required for all furnace analyses but recoveries are reported on the raw data.) In some cases post digestion spike data may aid in

evaluating matrix interferences. This information must be included in the DPO report.

C. Evaluation Procedure

1. Review Form V and verify that results fall within the specified limits.

2. Check raw data and recalculate one or more %R using the following equation to verify that results were correctly reported on Form V.

$$\%R = \frac{(SSR-SR)}{SA} \times 100$$

Where,

SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Verify that the field blank was not used for spike analysis.

4. Verify that a matrix spike was prepared at the proper frequency.

5. Verify that a post-digestion spike was performed for all analytes with unacceptable pre-digestion spike recovery.

D. Action

1. If the spike recovery is >125% and the reported sample results are non-detected, the data are acceptable for use.

2. If the spike recovery is >125% or <75% and the reported sample levels are >IDL, qualify the data as estimated (J).

3. If the spike recovery falls within the range of 30-74% and the sample results are non-detected, qualify the data for these samples as estimated (UJ).

4. If spike recovery results fall <30% and the sample results are non-detected, qualify the data as unusable (R).

5. If the field blank was used for matrix spike analysis, all other QC data must be carefully checked and

professional judgement exercised when evaluating the data.

NOTE: Any action taken applies to all samples of the same matrix.

VII. DUPLICATE SAMPLE ANALYSIS

A. Objective

Duplicate analyses are indicators of the precision of the sample results.

B. Criteria

1. Samples identified as field blanks cannot be used for duplicate sample analysis.

2. A control limit of ± 20% (35% for soil) for the Relative Percent Difference (RPD) shall be used for sample values > 5 times the CRDL.

3. A control limit of ± CRDL (± 2xCRDL for soil) shall be used for sample values less than 5 times the CRDL, including the case when only one sample value is <5x CRDL or when one sample is above the IDL and one is non-detected.

4. A duplicate sample must be prepared and analyzed for every 20 samples, for every batch digested, or for every matrix, whichever is more frequent.

C. Evaluation Procedure

1. Review Form VI and verify that results fall within the control limits.

2. Check the raw data and recalculate one or more RPD using the following equation to verify that results have been correctly reported on Form VI.

$$RPD = \frac{S-D}{(S+D)/2} \times 100$$

Where,

S = Original Sample Value
D = Duplicate Sample Value

3. Verify that the field blank was not used for duplicate analysis.

4. Verify that duplicates were prepared at the required frequency.

D. Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify results > IDL for that analyte in all samples of the same matrix as estimated (J).

2. If the field blank was used for duplicate analysis, all other QC data must be carefully checked and professional judgement exercised when evaluating the data.

VIII. FIELD DUPLICATES

A. Objective

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than lab duplicates which measure only lab performance. It is also expected that soil duplicate results will have greater variance than watermatrices due to difficulties associated with collecting identical field samples.

B. Criteria

A control limit of ±30% (50% for soil) for the RPD shall be used for sample values >5xCRDL.

A control limit of ±2xCRDL (4xCRDL for soil) shall be used for sample values < 5xCRDL.

C. Evaluation Procedures

Samples which are field duplicates should be identified using EPA Sample Traffic Reports or sample field sheets. The reviewer should compare the results reported for each sample and calculate the Relative Percent Difference (RPD), if appropriate.

NOTE: The appropriate person should be contacted to determine whether field duplicates were taken during sampling.

D. Action

If field duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify results >IDL for that analyte in all samples of the same matrix as estimated (J).

IX. LABORATORY CONTROL SAMPLE ANALYSIS (LCS)

A. Objective

The laboratory control sample analysis is designed to serve as a monitor of the efficiency of the digestion procedure.

B. Criteria

1. All aqueous LCS results must fall within the control limits of [redacted]. According to SOW 7/87 Antimony and Silver are excluded from this criteria. For validation purposes the 20% limit will be applied to both Antimony and Silver.

2. All solid LCS results must fall within the control limits established by the EPA. The 80-120% criteria is not applied to the soil LCS.

3. An LCS must be prepared and analyzed for each matrix, for each batch of samples digested, or for every twenty samples, whichever is more frequent.

C. Evaluation Procedure

1. Review Form VII and verify that results fall within the control limits.

2. Check the raw data to verify reported results on Form VII. Recalculate one or more of the recoveries using the following equation.

$$\text{LCS } \%R = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Where,

LCS Found = concentration (in ug/L for aqueous, mg/kg for solid) of each analyte measured in the analysis of the LCS solution.

LCS True = concentration (in ug/L for aqueous, mg/kg for solid) of each analyte in the LCS source.

3. Verify that an LCS was prepared and analyzed at the proper frequency.

D. Action

1. Aqueous LCS

a. If the LCS recovery for any analyte (including Sb or Ag) falls within the range of 50-79% or >120%, qualify results >IDL as estimated (J).

b. If results are non-detected and the LCS recovery is greater than 120%, the data are acceptable for use.

c. If results are non-detected and the LCS recovery falls within the range of 50-79%, qualify the data for these samples as estimated (UJ).

d. If LCS recovery results fall <50%, qualify all data as unusable (R).

e. If an LCS was not analyzed at the proper frequency, professional judgement must be used to qualify the data. Matrix spike recovery may also indicate digestion efficiency. Provide explanations in the memo for all actions taken.

2. Solid LCS

a. If the solid LCS recovery for any analyte falls outside the EPA control limits, qualify all results >IDL as estimated (J).

b. If the LCS results are higher than the control limits and the samples are non-detected, no qualifiers are necessary.

c. If the LCS results are lower than the control limits, qualify all non-detected results as estimated (UJ). Results may be biased low.

3. If an LCS was not analyzed at the proper frequency, evaluate the affect on the sample data and qualify accordingly.

X. FURNACE ATOMIC ABSORPTION ANALYSIS

A. Objective

Duplicate injections and furnace post-digestion spikes establish the precision and accuracy of the individual analytical determinations.

B. Criteria

1. For sample concentrations >CRDL, duplicate injections must agree within [redacted] Relative Standard Deviation (RSD), or Coefficient of Variation (CV), otherwise the sample must be rerun once (two additional injections).

2. Spike recovery must be [redacted]

3. If post-digestion spike recovery is not within 85-115% and sample absorbance is > 50% of spike absorbance, the Method of Standard Addition is required. The sample must be spiked with standards at 50, 100 and 150% of the sample absorbance.

C. Evaluation Procedure

1. Check raw data to verify that duplicate injections were performed and agree within ± 20 % RSD (or CV) for sample concentrations >CRDL.

2. Review Furnace AA raw data to verify that the Furnace Atomic Absorption Scheme, as described in SOW, p.E-15, has been followed.

3. Verify the percent recoveries wer calcualted correctly.

4. Verify that all required MSA results are reported on Form VIII and check that the correlation coefficients and sample results are calculated correctly.

D. Action

1. If duplicate injections are outside the required criteria and the sample has not been rerun once as required, qualify positve results as estimated (J).

2. If the rerun sample results do not agree within ± 20% RSD or CV, qualify the positive results as estimated (J).

3. If sample absorbance is <50% of the post-digestion spike absorbance then:

   a. For sample results >IDL, if the furnace post-digestion spike recovery is not within 85-115%, qualify the sample results as estimated (J).

   b. For non-detected results, if the furnace post-digestion spike recovery is >10% but <85%, qualify the sample result as estimated (UJ).

   c. If post-digestion spike recovery <10%, qualify positive results and non-detected results as unusable (R).

4. If Method of Standard Addition (MSA) is required but has not been done, qualify the positive results as estimated (J).

5. If any of the samples run by MSA have not been spiked at the appropriate levels, qualify positive results as estimated (J).

6. If the MSA correlation coefficient is <0.995, qualify the positive results as estimated (J).

XI. ICP SERIAL DILUTION ANALYSIS

A. Objective

Serial dilution analysis determines whether significant physical or chemical interferences exist due to sample matrix.

B. Criteria

1. If the analyte concentration is sufficiently high (concentration in the original sample is minimally a factor of 50 above the IDL) the laboratory is required to report the results of a five fold dilution. Results that do not agree within 10% of the original results are flagged with "E" by the laboratory. For validation purposes, the criteria for action is 15%.

2. A serial dilution is required for each matrix analyzed.

3. If the sample used for the serial dilution had to be diluted for any elements in order to bring the result within the linear range of the instrument, an additional five fold dilution is required for evaluating matrix interferences for that particular element.

C. Evaluation Procedures

1. Verify that reported results for the serial dilution meet required criteria of ± 10% D.

2. Check the raw data and recalculate the %D using the following equation to verify that the dilution analysis results agree with initial sample results reported on Form IX.

$$\%D = \frac{I-S}{I} \times 100$$

Where,

I = Initial Sample Result

S = Serial Dilution Result (Instrument Reading X 5)

3. Check the raw data for evidence of negative interference, ie. results of the undiluted samples are significantly higher than the original sample.

D. Action

1. If the percent difference between results is >15 %, and the result of the diluted sample is greater than that of the undiluted sample qualify positive results (J) and non-detetced results as estimated (UJ). A supression due to sample matrix has resulted in the reported results being biased low.

2. If evidence of negative interference is found, use professional judgement to qualify the data. Positive results only need be estimated (J).

XII. DETECTION LIMITS

A. Objective

Instrument Detection Limits reported on Form XI are used for reporting the detection limit for all sample analyses. Any value less than the IDL is considered non-detected.

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B. Criteria

1. IDLs must be less than the CRDL for all elements.

2. ICP or other methods may be used that do not have IDLs that are less than the CRDLs if all sample results are greater than 5xIDL for that instrument.

3. IDLs must be multiplied by dilution factors and prep factors before being reported on Form I or the data summary tables.

C. Evaluation

1. Verify the instrument detection limits are present on Form XI for all elements and are less than the CRDLs.

2. Verify that results for all parameters are reported down to the IDL not CRDL on Form I.

3. Verify that any sample weights, volumes, and dilutions are taken into account when reporting the detection limit for all samples.

4. Verify that sample results are >5xIDL if ICP analysis results are used for As, Tl, Se, or Pb.

NOTE: When the laboratory provides both ICP and GFAA results for an analyte in a sample and the concentration is >ICP IDL, the results can assist in identifying quantitation problems.

D. Action

1. If the IDL is not less than the CRDL notify the DPO that the laboratory is not meeting contractual requirements. Use the laboratory's IDL as the detection limit on the data summary tables.

2. Change any results on the data summary tables for samples on Form I that are not reported down to the IDL or do not use proper dilution/prep factors.

3. Estimate or Reject any positive results or non-detected results for As, Tl, Se, or Pb that are analyzed by ICP but are not greater than 5xIDL.

XIII. SAMPLE RESULT VERIFICATION

A. Objective

The objective is to ensure that the reported quantitation results are accurate.

B. Criteria

Analyte quantitation must be calculated according to the appropriate SOW.

C. Evaluation Procedure

The raw data should be examined to verify the correct calculation of sample results reported by the laboratory. Digestion and distillation logs, instrument printouts, strip charts, etc. should be compared to the reported results on Form I.

1. Examine the raw data for anomalies (ie., baseline shifts, negative absorbances, omissions, legibility, etc.).

2. Verify that there are no transcription or reduction errors (eg., dilutions, percent solids, sample weights).

3. Verify that results fall within the linear range of the ICP (Form XIII) and within the calibrated range for the non-ICP parameters.

4. The following quidelines should be applied in determining the minimum level of data validation required to assure the acceptability of the data package.

   a. Choose at least two furnace AA parameters and check all related calculations and transcriptions.

   b. Choose at least two ICP parameters for complete validation. If any errors are identified, then evaluate an additional two parameters. If errors are still encountered, then all remaining ICP parameters must be evaluated.

   c. Data for Cyanide and Mercury must be validated 100 percent.

D. Action

1. If there are any discrepancies found, the laboratory must be contacted by the designated representative to resolve any discrepancies.

2. If a discrepancy remains unresolved, the reviewer may determine qualification of the data is warranted.

XIV. OVERALL ASSESSMENT OF DATA FOR A CASE

It is appropriate for the data reviewer to make professional judgements and express concerns and comments on the validity of the overall data for a Case. This is particularly appropriate when there are several QC criteria out of specification. The additive nature of QC factors out of specification is difficult to assess in an objective manner. However, the reviewer has a responsiblity to inform the user concerning data quality and data limitations in order to assist that user in avoiding inappropriate use of the data, while not precluding any consideration of the data at all. The data reviewer would be greatly assisted in this endeavor if the data quality objectives were provided.

GLOSSARY A

Data Qualifier Definitions

For the purposes of this document the following code letters and associated definitions are provided.

U - The material was analyzed for, but was not detected above the level of the associatied value. The associated value is either the sample quantitation limit or the sample detection limit.

J - The associated value is an estimated quantity.

R - The data are unusable. (Note: Analyte may or may not be present).

UJ - The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

GLOSSARY B

Additional Terms

AA	Atomic Absorption
Case	A finite, usually predetermined number of samples collected in a given time period for a particular site. A Case consists of one or more Sample Delivery Groups.
CCS	Contract Compliance Screening - process in which SMO inspects analytical data for contractual compliance and provides EMSL/LV, laboratories, and the Regions with their findings.
CLP	Contract Laboratory Program
CCB	Continuing Calibration Blank - a deionized water sample run every ten samples designed to detect any carryover contamination.
CCV	Continuing Calibration Verification - a standard run every ten samples designed to test instrument performance.
CRDL	Contract Required Detection Limit
CV	Coefficient of Variation
DPO	Deputy Project Officer
EMSL/LV	Environmental Monitoring System Laboratory/ Las Vegas
Equipment blank	Equipment blanks consist of water used to decontaminate sampling equipment as a sample check for cross-contamination from inadequate decontamination.
Holding Time	The time from sample collection to laboratory digestion, distillation, or analysis, whichever is appropriate.
ICP	Inductively Coupled Plasma
ICS	Interference Check Sample

Initial Calibration	The establishment of a calibration curve with the appropriate number of standards and concentration range. The calibra- tion curve plots absorbance or emission versus concentration of standards.
ICB	Initial Calibration Blank - first blank standard run to confirm the calibration curve.
ICV	Initial Calibration Verification - first standard run to confirm the calibration curve.
LCS	Laboratory Control Standard - A standard that has gone through digestion and is designed to measure digestion efficiency. The solid is suppplied by the EPA.
MS	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.
MD	Matrix Duplicate - a laboratory split sample to measure instrument precision.
MSA	Method of Standard Addition
NPO	National Program Office
PE Sample	Performance Evaluation Sample
PO	Project Officer
Preparation Blank	Laboratory blank water which has gone through digestion or distillation and analysis with each SDG.
QA	Quality Assurance - total program for assuring reliability of data.

QAC	Quality Assurance Coordinator
QC	Quality Control - routine application of procedures for controlling the monitoring process.
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
RSCC	Regional Sample Control Center
Serial Dilution	A sample run at a specific dilution to determine whether any signicant chemical or physical interferences exist due to sample matrix effects. (ICP only)
SDG	Sample Delivery Group - defined by one of the following, whichever occurs first: - Case of field samples - each twenty field samples in a Case - each 14 day calendar period which field samples in a Case are received, beginning with receipt of the first sample in the SDG.
SMO	Sample Management Office
SOP	Standard Operating Procedure
TCL	Target Compound List
Trip Blank	A trip blank consists of bottled water that accompanies sample bottles into the field and to the laboratory as a sample check for contamination along the trip.

APPENDIX A

Contract Compliance Screening Procedures for RAS Inorganic Data Packages

STANDARD OPERATING PROCEDURE
FOR
CONTRACT COMPLIANCE SCREENING (CCS)
OF
ROUTINE ANALYTICAL SERVICES ANALYSES
OF
INORGANICS DATA
UNDER SOW NO. 787

Introduction

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Appendix

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Introduction

The following is the Standard Operating Procedure (SOP) under which Contract Compliance Screening (CCS) screens routine inorganic data (RAS) under SOW 787.

CCS assesses the deliverables in terms of completeness and technical compliance. Discrepancies are reported under the relevant screening criteria as listed in the Table of Contents. The screening results are summarized on a summary form. The problems are described in detail in the comments section of Worksheets A and B; Worksheet A for ICP data, and Worksheet B for AA, mercury and cyanide data. Each problem is preceeded by the code for the relevant criterion. If there is more than one problem under a single code, sequential numbering is used to differentiate between problems (i.e., B_1, B_2, B_3... etc.) For each action, the affected samples are identified by placing the problem code on the bottom line of the comments section across from the corresponding sample.

The summary form consists of a matrix that lists the samples in the left margin, and the criteria in the top row. Each box in the matrix represents a sample and a criterion. An action code in a box means that there is a problem or an ambiguity in a sample for the related criterion. More than one action code may apply to a single box. The action codes are explained at the bottom of the summary page.

R = a requirement or deliverable is incomplete or defective and must be resubmitted.

S = a deliverable is missing and must be submitted.

N = a deliverable is technically noncompliant.

E = an ambiguity exists that requires an explanation in order that a decision can be made.

Comments listed with "No Action" on Worksheet A or B do not appear on the summary sheet, but need to be addressed in order to avoid future complications.

To ensure better understanding of terms used throughout this SOP, some of the terms are defined as follows:

- o A case may consist of one or more Sample Delivery Group(s).
- o A Sample Delivery Group (SDG) is defined by the following, whichever is most frequent:
  - each case of Field Samples received, OR
  - each 20 Field Samples within a case, OR
  - each 14 calendar day period during which Field Samples in a case are received.
- o All samples must initially be run undiluted. When an analyte concentration exceeds the calibrated or linear range, appropriate dilution and reanalysis of the prepared sample is required. The dilution should be minimal and should not dilute the sample below the instrument detection limit.
- o An analytical sample is defined as any cuvette in the auto sampler, excluding standardization, ICV, ICB, CCV and CCB. The ICS, CRI, Linear Range Standard, LCS, Analytical Spikes (post digest/post distillation spikes) and preparation blanks are all considered analytical samples.

A. Cover Page

1. Screening Procedure

a. Check to see that the Cover Page is present.

b. Ensure that all required information is filled in:

(1) Laboratory name, laboratory code, contract no., case #, SDG #, SAS # (where applicable), SOW #, and EPA sample numbers in alphanumeric order.

(2) All Sample #s in data package corresponding with Form I and Traffic Report.

(3) Ensure that the SDG number has been properly assigned.

(4) Lab managers signature and date.

(5) Statement concerning interelement and background corrections.

c. Check to see that the Cover Page is in EPA format.

2. Action

a. If the Cover Page is not present, it must be submitted. (S)

b. If the Cover Page is not properly completed, it must be resubmitted (R). IF the SDG number is incorrectly assigned the cover page as well as all affected forms and raw data must be resubmitted (R).

c. If the Cover Page format is different from that specified in Exhibit B, it must be resubmitted (R).

B. Form I - Data Sheets

1. Screening Procedure

a. Check to see that each sample in the data package has a Form I corresponding to the list on the Cover Page and the Traffic Reports.

b. Check to see that all required information is filled in:

1) EPA sample # and Laboratory Sample ID # must be the same as they appear on the Cover Page. Laboratory name, lab code, case #, SDG #, SAS #, and laboratory receipt date must be present.

2) Spot check the results versus the raw data.

3) Ensure that the correct number of significant figures and the proper rounding rules for reporting results on Form I have been followed 2 sig. fig. must be reported if the result < 10 and 3 sig. fig. if the result ≥ 10. For Hg report results as follows: < 0.2 ug/L, 0.2 U; between 0.2 and 10 ug/L, one decimal; above 10 ug/L, whole numbers.

4) Units must be UG/L for waters and MG/KG (dry wt) for solids.

5) Solid samples require a value for % solids (reported to one decimal place). Water samples must have "0" reported for % solids.

6) Ensure that soil results reported to the instrument detection limit are corrected for % solids.

7) Descriptions of coloration, clarity, texture and artifacts in sample are required before and after sample digestion/distillation. For water samples report color and clarity, for soil samples report color, texture and artifacts, as recommended in SOW 787, B-16.

c. Ensure that any required data flags resulting from requirements for Forms V - IX (N, M, *, S, +, W, and E) have been entered on Form I as appropriate.

d. Ensure that values are reported properly. Form I includes fields for three types of result qualifiers. These qualifiers must be completed as follows:

- C (concentration) qualifiers. Enter "B" if the reported value is less than CRDL but greater than IDL. If the analyte was analyzed for but not detected, a "U" must be entered. Samples must not be diluted below the detection limit of the instrument. The laboratory must use the lowest dilution factor necessary to bring each analyte within the valid linear range and report the highest valid value for each analyte.

- Q (Quality Control) qualifiers. Specified entries and their meanings are as follows:

  E - The reported value is estimated because of the presence of interference(s).

  M - Duplicate injection precision not met for GFAA.

  N - Spike sample recovery not within control limits.

  S - The reported value was determined by the method of standard additions (MSA).

  W - Post digestion spike for furnace AA analysis is out of control limits.

  * - Duplicate analysis not within control limits.

  + - Correlation coefficient for the MSA is less than 0.995.

o M (Method) qualifiers - Specified entries and their meanings are as follows:

P - ICP

A - Flame AA

F - Furnace AA

CV - Manual Cold Vapor AA

AV - Automated Cold Vapor AA

AS - Semi-Automated Spectrophotometric

C - Manual Spectrophotometric

T - Titrimetric

NR - If the analyte is not required to be analyzed

e. All dilutions not required by the SOW (and affecting IDL) must be noted on an analyte by analyte basis in the comment section.

f. Ensure that the results reported pertain to the analyses requested. If cyanide analysis is not requested Form I should state NR, under the method column.

g. Check to see that any analyte value reported by any method that has its IDL > CRDL is at least 5x IDL.

2. Action

a. A missing Form I must be submitted. (S)

b.,c.,d.,e. An incomplete Form I must be resubmitted. (R)

If transcription errors, improper flagging or incorrect units are found, Form I must be resubmitted (R).

If a sample is diluted below the IDL the sample must be reanalyzed using the appropriate dilution factor and the data submitted (S).

f. If a required analysis was not reported, results must be submitted (S) under CCS criterion R, as well as CCS criterion A, B, K.

g. If an analyte was determined by a method for which IDL > CRDL, samples with analyte results below 5x IDL should be marked noncompliant (N) under criterion L.

Table 1

Codes for Labelling Raw Data

Sample	XXXXXX
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Analytical Spike	XXXXXXA
Instrument Calibration Standards	STD1, STD2,...etc.
Initial Calibration Verifications	ICV1, ICV2,...etc.
Initial Calibration Blank	ICB
Continuing Calibration Verifications	CCV1, CCV2,...etc.
Continuing Calibration Blanks	CCB1, CCB2,...etc.
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
CRDL Standard for AA	CRA
CRDL Standard for ICP	CRI
Laboratory Control Samples:	
Aqueous (Water)	LCSW
Solid (Soil/Sediment)	LCSS
Preparation Blank	PB
Linear Range Analysis Standard	LRS
Sample Dilutions	3x - 3 fold - 1:2 - (one part sample and two parts dilutant)

CA. Form IIA - Initial and Continuing Calibration

1. Screening Procedure

a. Ensure that all required information on Form II has been completed: Laboratory name, lab code, Case #, SDG #, SAS #, Units (ug/L), Initial & Continuing Calibration sources and Analytical Methods used.

b. Check the Form IIA's versus the raw data to verify that results for all calibrations bracketing relevant samples in the case have been recorded.

c. Verify that 2-point (for ICP), 4-point plus blank (for Hg) and 3-point plus blank (for AA and cyanide) calibrations have been performed. If the instrument is not capable of performing the required calibration the standards must be run immediately after the instrument calibration sequence. The results must agree within ± 5% of the true value (exception: CRDL calibration standard).

d. Instruments must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

e. Spot check the calibration results versus the raw data and ensure proper rounding of results was followed.

f. Ensure that the %R calculations are correct and are within the control limits (90-110% for all metals, except 80-120% for Hg and 85-115% for CN). Ensure that the %R's are calculated from the results reported on Form IIA. Higher precision or unrounded values cannot be used to determine %R.

g. Verify that the proper calibration standards have been used, and that the same continuing calibration standard was used throughout the analysis run for a case of samples received. Verify that the reported true values for ICVs and CCVs are correct using EPA true values or values submitted by the laboratories.

h. Verify that the ICV has been run immediately after instrument calibration and is within the control limits (90-110%)

i. Verify that the 10% continuing calibration frequency requirement has been met. No more than ten analytical samples may be run between the ICV, first CCV or subsequent CCV's (this includes the ICS, LCS, CRI, Linear Range, analytical spikes and preparation blanks.) Ensure that every analytical sample is between two sets of ICV/ICB's or CCV/CCB's that have met all calibration requirements.

j. Verify that a CCV has been performed every 2 hours or at a 10% frequency whichever is more frequent.

k. Five full MSA's may be performed between consecutive CCV/CCB (whether single or duplicate injections were used). CCV/CCB values may also be determined by MSA if the instrument's mode cannot be changed while MSA is being performed.

l. Ensure that for all AA (except for Hg) and cyanide analyses one calibration standard is at CRDL.

2. Action

a. If any required information is missing from a Form II, the form must be resubmitted. (R)

b. If any calibration verification pertinent to the samples has not been reported, a Form II with the appropriate data must be submitted (S)

c. If the instrument calibration does not meet the required criteria (2-point (for ICP), 4 point plus blank (for Hg), 3-point plus blank (for AA and cyanide), or ±5% criteria) mark all related samples noncompliant (N).

d. If calibration raw data are not present, they must be submitted (S) under CCS criterion CA.

e,f. If the %R for the calibration result (rounded to the nearest whole number using EPA rounding rules) is outside the control limits, the instrument must be recalibrated, the previous ten samples re-analyzed for the affected analytes and the data submitted (S). If the %R calculations are incorrect, a corrected form must be resubmitted (R).

g. If improper calibration standards have been used, mark the associated samples as noncompliant (N). If the calibration sources have not been identified, Form II must be resubmitted (R) with the required information.

h. If the ICV is not run immediately after instrument calibration and/or is not within the control limits mark all affected samples noncompliant (N).

i,j,k. If the 10% continuing calibration frequency requirement has not been met, or if the 2 hour CCV has been violated, mark the preceding samples (to previous compliant CCV or ICV) as noncompliant (N).

l. If one of the calibration standards for all AA (except Hg) and cyanide analyses is not at CRDL, all samples will be considered noncompliant (N).

CB. Form IIB - CRDL Standard for AA and ICP

1. Screening Procedure

   a. Ensure that all required information on Form IIB has been completed.

   b. Verify that a standard at the CRDL has been analyzed for each analyte by AA (except Hg). Verify that a standard at 2x CRDL or 2x IDL, whichever is greater, has been analyzed for each analyte by ICP but not for Al, Ba, Ca, Fe, Mg, Na or K.

   c. Ensure that a CRDL standard has been performed at the beginning and end of each ICP run (but not before the ICV/ICB or after the final CCV/CCB at the end of the ICP run).

   d. Verify that the 8 hour CRDL requirement was satisfied for ICP analysis.

2. Action

   a. If any required information is missing from a Form IIB, the form must be resubmitted (R). A missing Form IIB must be submitted (S).

   b. If the true values of the CRDL standard are not at the appropriate levels the analysis for all samples must be marked noncompliant (N).

   c. If the CRDL standard for ICP has been run before the ICV/ICB or after the final CCV/CCB the analysis for all samples must be marked noncompliant (N).

   d. If the CRDL standard is not reanalyzed within 8 hours for ICP analysis, mark all samples in the run as noncompliant (N). If analysis times are not provided, the laboratory must submit them (S) under CCS criterion R.

D. Form III - Blanks

1. Screening Procedure

a. Ensure that all required information on Form III has been completed.

b. Ensure that all values for all required blanks were reported on Form III.

c. Spot check the results versus the raw data.

d. All blank results must be reported, positive, negative or zero.

e. The values for the preparation blank must be recorded in UG/L for water samples and MG/KG for soil samples. The values of ICB/CCB must be recorded in UG/L.

f. Ensure that the proper concentration flag "U" is applied for an absolute value below IDL, "B" for an absolute value greater than the IDL but below the CRDL.

g. Ensure that the blank values are within control limits.

h. Check the raw data to verify that a calibration blank has been analyzed immediately after each calibration verification, at the beginning and after the last analytical sample, every 2 hours, and at 10% frequency.

i. Ensure that a preparation blank has been analyzed for each batch of samples (up to 20). For Hg the preparation blank is the same as the calibration blank, in accordance with the mercury method. It must be labelled for both uses in the raw data.

2. Action

a. If Form III is incomplete, it must be resubmitted (R).

b. If required blanks are not documented, a Form III with the appropriate data must be submitted (S).

c. If the absolute value of the blank (ICB/CCB) results exceeds the CRDL for an element, mark the preceding samples (to the previous compliant ICB/CCB) as non-compliant (N). The instrument must be recalibrated and the samples re-analyzed for the affected analyte. If an element is determined on an instrument whose IDL exceeds the CRDL, mark the associated samples as noncompliant (N) if the calibration blank is greater than the IDL.

d. If the 2 hour or 10% frequency has not been met, mark the preceding samples (up to the last compliant ICB/CCB) as noncompliant (N).

e,g. If the absolute value for any analyte concentration in the prep. blank is above the CRDL, the lowest concentration of that analyte in the associated samples must be 10x the blank concentration. Otherwise, all samples must be redigested and reanalyzed for that analyte and the data submitted (S).

f. If results are improperly transcribed or the units are incorrect, Form III must be resubmitted (R).

h,i. If raw data are needed, code this as submit (S) under CCS criterion R.

E. Form IV - Interference Check Sample

1. Screening Procedure

a. Check to see that all information required on Form IV has been completed, including ICS source and ICP ID number.

b. Verify that an ICS analysis run has been performed at the beginning, (but not before the ICV and ICB), and end of each ICP run (but not after the final CCV/CCB).

c. Ensure that all ICS results are documented on Form IV. The analysis with the lowest dilution factor within the linear range for all elements must be reported.

d. If the ICS was Contractor prepared, verify that it contains all elements at the level specified in Table 2 (SOW 787 E-8) and that a mean and standard deviation are calculated for each analyte based upon at least 5 measurements.

e. Spot check the ICS results versus the raw data. Ensure that the %R calculations are correct and are within the control limits (80-120) except for Al, Ca, Fe, Mg. Recoveries must be calculated versus the "true" value if EPA solutions are used or a mean value when a contractor prepared solution is used.

f. Ensure that the %Rs are calculated from the values reported on Form IV. Higher precision or unrounded values are not permitted to be used to determine %R.

g. Verify that the 8 hour ICS requirement was satisfied.

2. Action

a. If any information on Form IV has not been completed, it must be resubmitted (R).

b. If the ICS is run before the ICV/ICB or after the final CCV/CCB mark all samples in the run as noncompliant (N). If ICS raw data have not been submitted for both the beginning and end of an ICP run, then the data must be submitted (S) under CCS criterion R.

c. If any required ICS value has not been documented on a Form IV, it must be submitted (S).

d. If Form IV lacks the true values for ICP elements, except for those not contained in an EPA solution, it must be resubmitted (R).

e. If any ICS recovery (except for "interferents" such as Al, Ca, Mg and Fe) is outside the control limit, mark all samples in that ICP run as noncompliant (N). Results for all elements must be within the linear range or a diluted value must be submitted (S) from the diluted ICS.

f. If the %Rs are calculated from values other than those reported on Form IV, the form must be resubmitted (R).

g. If the ICS is not reanalyzed within 8 hours, mark all samples in the run as noncompliant (N). If analysis times for ICP are not provided, the laboratory must submit (S) them under CCS criterion R.

FA. Form VA - Spiked Sample Analysis (Pre-Digestion/Pre-Distillation)

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1. Screening Procedure

a. Ensure that all information required on Form VA has been completed.

b. Verify that at least one spiked sample analysis has been performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium), or for each Sample Delivery Group (SDG), whichever is more frequent. If two methods (ICP, GFAA) are used for an element, spike samples must be run and reported for each method. The units for reporting spike sample results will be identical to those used for reporting sample results in Form I (i.e., UG/L for aqueous and MG/KG dry weight basis for solids).

Spike concentrations should conform to those in Table 3 (SOW 787 E-10). Results must be in consistent units; IDL is the minimum reportable level.

c. Ensure that %Rs are reported for every spike performed and spot check the results versus the raw data and %R calculations. In calculating %R, use sample result (SR)=0 for values < IDL. If the spike is performed on the sample that is used for the duplicate analysis ensure that the %Rs are calculated versus the results designated as the "original" sample. The duplicate results or the average of the duplicate and sample results cannot be used. Ensure that the %Rs are calculated from the values reported on Form VA. Higher precision or unrounded values cannot be used to determine %Rs. %R must be reported, whether it is negative, positive, or zero.

d. Under "control limit %R", "75-125" must be entered if the spike added (SA) value was greater than or equal to one-fourth of the sample result value. Results outside the control limits must be flagged with "N". This does not apply when SR (sample result) > 4 x SA (spike added).

e. If there is more than one spike (per matrix, level, and method) and only one requires the "N" flag, all samples of that matrix and level done by the same method, must be flagged.

f. An identified field blank cannot be used for matrix spike analysis. Except when it is the only sample of that concentration and matrix for that SDG.

g. Check the traffic reports to see if a specific sample is requested for the spike analysis.

2. Action

a. A Form VA missing any required information must be resubmitted (R).

b. If matrix spikes have not been performed at the required frequency, the data must be submitted (S). If required matrix spikes have not been documented on Form VA, they must be submitted (S). If spike concentrations do not conform to those in Table 3, the spike analyses (for affected elements) must be redone at the appropriate levels and submitted (S). If inconsistent units have been used, Form VA must be corrected and resubmitted (R).

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c. If the method of calculating the %R is incorrect, Form VA must be corrected and resubmitted (R). If the %Rs are calculated from values other than those reported on Form VA, the form must be resubmitted (R).

d,e. If a %R requires an "N" flag on Form VA and does not have it, Form VA must be resubmitted (R); Form I must also be resubmitted (R) as appropriate.

f. If an identified field blank has been used for the matrix spike analysis, a matrix spike analysis on a field sample must be submitted (S). (Except when the field blank is the only aqueous sample in a case of soil samples.)

g. If the sample designated to be used for the spike analysis has not been used then the proper sample must be spiked and the results submitted (S).

FB. Form VB - Spike Sample Analysis (Post-Digestion/Post-Distillation)

1. Screening Procedure

   a. Ensure that all required information on Form VB has been completed.

   b. For flame AA, ICP, Hg, and CN analyses, when the pre-digestion/pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, (i.e. an analyte that has been flagged "N"), a post-digestion/post-distillation spike must be performed for those elements that do not meet the specified criteria (except Ag). The unspiked aliquot of the sample must be spiked at 2x CRDL or 2x the indigenous level, whichever is greater. The units for reporting spike sample results will be UG/L.

   c. Spot check the results versus raw data and check %R calculations.

   d. Ensure that the proper reporting and calculation methodologies are followed.

2. Action

   a. If Form VB is missing any required information it must be resubmitted (R).

   b. If a post digestion/post distillation spike has not been done as required then the analysis must be performed and the data submitted (S). If the sample has been incorrectly spiked then the analysis must be redone at the appropriate spike level and the data submitted (S).

   c,d. If the results are improperly reported or calculated Form VB must be resubmitted (R).

G. Form VI - Duplicate Sample Analysis

1. Screening Procedure

a. Ensure that all information required on Form VI has been completed.

b. Verify that at least one duplicate sample analysis has been performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium), for each SDG, whichever is most frequent. If two methods (ICP, GFAA) are used for an element, duplicate samples must be run and reported for each method. Results must be in consistent units; IDL is the minimum reportable level. The sample and duplicate results cannot be averaged for reporting values on Form I. Duplicate analyses are required for % solids values and for all analyte results. Check for appropriate units (UG/L for water and MG/KG dry weight for soil).

c. Spot check the results versus raw data and the RPD calculations.

d. If both sample and duplicate values are $\geq$ 5x CRDL a control limit of $\pm$20% RPD is applied. If either the sample or duplicate results are below 5x CRDL a control limit of $\pm$CRDL is applied. The $\pm$CRDL value must be entered under "control limit" to applicable analytes. If both sample values are < IDL, the RPD is not to be calculated and "NC" must be entered under the method column. Values outside the control limits must be flagged with "*". If there is more than one duplicate sample (per matrix, level, and method), and only one is outside the control limits, all samples of that matrix and level, done by the same method, must be flagged.

e. The RPD values must be calculated from the values reported on Form VI. Higher precision or unrounded values cannot be used to determine the RPD.

f. Duplicate sample analysis may not be performed on an identified field blank, except when it is the only sample of that concentration and matrix for that SDG.

g. Check traffic reports to see if a specific sample is to be used for the duplicate analysis.

2. Action

a. If any required information is missing from Form VI, it must be resubmitted (R).

b. If duplicate analyses have not been performed at the required frequency, these analyses must be performed, and the data must be submitted (S).

If required duplicate analyses were performed, but were not documented on Form VI, these data must be submitted (S).

If inconsistent units were used, Form VI must be corrected and resubmitted (R).

c. If the RPD calculations are incorrect, Form VI must be resubmitted (R).

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d,e. If the incorrect control limits have been applied or the ±CRDL control limit is not entered when required, or if the flagging rules have been applied incorrectly Form VI must be corrected and resubmitted (R).

If the analytes have not been correctly flagged on Form I, the Form I's must be resubmitted (R) under criterion B.

f. If an identified field blank has been used for the duplicate analysis, a duplicate analysis on a field sample must be submitted (S).

g. If the sample designated to be used for the duplicate analysis has not been used then the proper sample must be used for the duplicate analysis and the results submitted (S).

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H. Form VII - Laboratory Control Sample

1. Screening Procedure

   a. Verify that all information required on Form VII has been completed. Ensure that the proper reporting methodology has been followed.

   b. Verify that at least one Form VII, with aqueous and/or solid laboratory control samples, has been submitted for each group of aqueous and/or solid samples in each SDG or each batch of samples digested. An aqueous LCS is not required for Hg and CN analysis. If an analyte is determined by more than one method a separate LCS must be analyzed by each method.

   c. Spot check results versus raw data and %R calculations.

   d. Ensure that the solid and aqueous LCS results are within specified control limits (80-120% for the aqueous LCS and within the specified concentration windows for the solid LCS). If either aqueous or solid LCS is out of control the analysis must be terminated, the problem corrected, and the samples associated with that LCS redigested and reanalyzed.

2. Action

   a. If any required information is missing from Form VII, it must be resubmitted (R).

   b. If the required number of LCS analyses has not been performed, these analyses must be performed and the data must be submitted (S). If a required LCS analysis has not been documented on a Form VII, it must be submitted (S).

   c,d. If any aqueous or solid LCS results are outside the control limits (80-120% for aqueous or concentration windows for solids) (except Ag and Sb in the aqueous LCS), mark all related samples as noncompliant (N).

I. Form VIII - Method of Standard Addition and Furnace Atomic Absorption (AA) QC Analysis

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1. Screening Procedure

a. Ensure that Form VIII has been completely and correctly filled in and results flagged as appropriate on Form I's.

b. Determine which elements have been analyzed by Furnace AA. Find the raw data and verify that the proper analytical sequence has been followed. Full MSA must be performed within the context of a regular analytical run, i.e., 3-point calibration plus blank between consecutive ICV/ICB and CCV/CCB with no more than five full MSA's. During full MSA, single injections for blanks and standards are permissible. If duplicate injections are used during MSA, their average value must be used to plot the MSA curve.

c. If sample concentrations are greater than the CRDL, ensure that the duplicate readings agree to within 20% relative standard deviation (RSD). If they do not agree within 20% RSD after one rerun, the result on Form I must be flagged with an "M".

d. Review the analytical spike recoveries and sample and spike absorbance (or concentration) to ensure that the MSA decision tree (Figure 1: SOW 787 E-15) has been followed. If the spiked sample requires dilution, the unspiked sample must be rerun at the same dilution factor. Analytical spikes are required on the LCS and prep blank. MSA is not to be performed on the LCS or preparation blank, regardless of spike recovery results. If MSA is performed twice on one sample as required by the MSA decision tree and both MSAs have a correlation coefficient < 0.995, ensure that the result reported on Form I for that analyte was from the MSA with the best correlation coefficient. An analytical spike is not required on the pre-digestion spike sample when the spike sample recovery is within the 75%-125% control limit or when SR > 4x SA.

A quick check for MSA.

%R for analytical spikes:

< 40% - Dilute (5 to 10x) sample and rerun once. If still < 40% the results must be flagged with an "E".

> 40%

(1) Sample absorbance or concentration is < 50% of the spike, the sample must be quantitated from the curve and report down to the IDL. If the spike recovery is less than 85% or greater than 115%, the results must be flagged with a "W".

(2) Sample absorbance or concentration is > 50% of the spike and the spike recovery is between 85% and 115%, the results must be quantitated from the curve and report down to the IDL.

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(3) Sample absorbance or concentration is > 50% of the spike and spike recovery is greater than 115% or less than 85%, the sample must be quantitated by MSA.

e. %R must be calculated using sample result, SR=0 for values < IDL. Values between IDL and CRDL must be used in calculating the analytical spike recovery.

If MSA's have been performed ensure that all MSA results have been reported correctly on Form VIII and in the raw data. Recalculate several linear regressions. Ensure that the curve is plotted with concentration added (x-axis) vs. absorbance (y-axis) and that the y intercept, slope, and correlation coefficient are reported on Form VIII or in the raw data as required. Also make sure all flags (S, +, W) are entered on Form I's as appropriate.

f. If the preparation blank analytical spike recovery is out of control (85-115%), the spiking solution must be verified by respiking and rerunning the preparation blank once. If the preparation blank analytical spike recovery is still out of control, the problem must be corrected and reanalysis of all analytical samples associated with that blank is required.

2. Action

a. If Form VIII is not present, it must be submitted (S). If the proper data flags (E, S, +, W and M) have not been written on Form I, then Form I must be resubmitted (R). An incomplete or incorrectly submitted Form VIII must be resubmitted (R).

b. If the proper analytical sequence has not been followed, mark the affected samples as noncompliant (N).

c. If the % RSD between a sample and its duplicate exceeds 20% and the sample has not been rerun, mark the sample as noncompliant (N).

d. If the proper decision tree process has not been followed for a sample, mark it as noncompliant (N). Examples are: (1) noncontract specified dilution of a sample before the first analysis, or (2) spiking at > 2 x CRDL, then diluting the spiked sample to bring the spike to 2 x CRDL or (3) rerunning a sample instead of diluting or performing MSA. If the spiked sample was not run at the same dilution factor as the unspiked sample, it is non-compliant (N).

e. If the %R is improperly calculated, the calculation must be corrected and the data must be resubmitted (R). If MSA is required, the analyses must be performed and the data submitted (S). If the slope, y-intercept and correlation coefficient are not present in the raw data they must be submitted (S).

f. If the preparation blank analytical spike recovery is out of control and it was rerun but remained out of control mark all affected samples as noncompliant (N).

J. Serial Dilution (Form IX - ICP Only)

1. Screening Procedure

a. Verify that Form IX has been submitted for each required serial dilution and that all information required on Form IX has been completed.

b. Ensure that at least one serial dilution has been performed on each group of samples of a similar matrix type (i.e., water, soil) and concentration (i.e., low, medium), for each SDG, whichever is most frequent. If no samples in the case contain analytes at least ten times (10x) the IDL in the original (undiluted) analysis, then serial dilution is not required (NR). The initial sample value must be entered on Form IX and (NR) entered under the method column if the original sample is below 10x IDL.

c. Check to see that a 5-fold dilution that does not agree within 10% of the initial value has been flagged "E" for that analyte on Form IX and all associated Form Is. If there is more than one serial dilution per SDG, but at least one is not within the 10% limit for a specific analyte, flag all samples of the same matrix and concentration with "E" flags for that analyte.

d. Ensure that identified field blanks have not been used for serial dilution analysis.

e. A 5x dilution is required (one part of sample mixed with four parts distilled water).

f. Spot check the results on Form IX against the raw data and recalculate the % Difference for a few elements.

g. Ensure that the proper concentration flags have been reported on Form IX.

2. Action

a. If Form IX is not present, it must be submitted (S). An incomplete Form IX must be resubmitted (R).

b. If no, or too few serial dilution analyses have been performed (provided that analyte concentrations are sufficiently high), these data must be submitted (S).

c. If "E" flags are required, but are not entered on Form IX, the form must be resubmitted (R). The affected Form Is must also be resubmitted (R) under CCS criterion B.

d. If an identified field blank has been used for serial dilution analysis, an analysis on a field sample must be submitted (S).

e. If a dilution factor other than 5x is used, the analysis is noncompliant (N).

f. If the results on Form IX do not correspond to the raw data, or the % Difference for any element is incorrect, the form must be resubmitted (R).

g. If Form IX is improperly completed it must be resubmitted (R).

KA,KB. **Holding Times - Form X**

1. Screening Procedure

Cyanide (CN) - 14 days

Mercury (Hg) - 26 days

Other metals - 6 months

a. Ensure that Form X has been submitted and completed properly.

b. For CCS purposes, the holding time is defined as the number of days from the date of sample receipt at the laboratory to the date of sample preparation (digestion or distillation). Compare these two dates for each analyte and sample to see if holding times have been exceeded. Holding time = prep date - laboratory receipt date.

c. Ensure that the holding time has been properly calculated. The dates on Form X must correspond to the dates on the distillation (or digestion) log and the Traffic Reports.

2. Action

a. If Form X is not present, it must be submitted (S).

b. If information to determine holding times is not contained in the data package, it must be submitted (S). If the holding time for an analyte in a sample has been exceeded, mark that sample as noncompliant (N) and report the number of days in excess on the CCS summary sheet.

c. If the holding time calculations on Form X are incorrect, Form X must be resubmitted (R).

L. Form XI - Instrument Detection Limits

1. Screening Procedure

   a. Verify that at least one Form XI has been submitted.

   b. An IDL must be reported for each wavelength used in the sample analysis (no IDL is required for CN). Verify that the IDL are not greater than the CRDL for each analyte. If an analyte has been determined by an instrument whose IDL exceeds the CRDL for that element, check to see that the concentration in the sample exceeds the CRDL by at least a factor of 5.

   c. Verify that the IDL's reported are for the current quarter, (i.e. no more than 3 months old). If multiple instruments are used for the analysis of an analyte within an SDG, the highest IDL for the instrument must be used for reporting concentration values for that SDG.

   d. Verify that all information is recorded on Form XI including Instrument I.D.

2. Action

   a. If a required Form XI is missing it must be submitted (S).

   b. If an IDL is not reported for an analyte it must be submitted (S). If the IDL for an analyte exceeds the CRDL, mark all samples noncompliant (N) and notify the laboratories Project Officer (P.O.). (Except when the sample result is greater than 5x's the IDL).

   c. If the case is submitted using out of date IDL's, the new IDL's must be submitted (S) and all affected Forms I-IX resubmitted (R). If the analyte has been determined by more than one instrument and the highest IDL is not reported then all affected Forms I-IX must be resubmitted (R).

   d. An incomplete or ambiguous Form XI (unclear as to which IDL corresponds to which instrument) must be resubmitted (R).

M. Forms XII A & B Interelement Correction Factors

1. Screening Procedure

   a. Verify that all required information is recorded on Forms XII A & B.

   b. Verify that a current Form XII has been submitted with every case package. Ensure that correction factors for Al, Ca, Fe and Mg have been determined. Also ensure that the date of the correction factors do not exceed the ICP analysis date or precede the analysis date by more than 12 months.

2. Action

   a. An incomplete form must be resubmitted (R).

   b. If interelement correction factors have not been determined for Al, Ca, Fe and Mg mark all samples noncompliant (N). If a Form XII is missing, it must be submitted (S), under criterion M.

N. **Form XIII - ICP Linear Ranges**

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1. Screening Procedure

   a. Ensure that all required information is recorded on Form XIII.

   b. Verify that a current Form XIII has been submitted for each ICP instrument used in determining analyte results. Ensure that the date of ICP analysis does not exceed the Linear Range determination by more than 3 months.

   c. Make sure that all the results obtained from each ICP instrument for each analyte and wavelength fall below its established linear range for that instrument. For AA, CN and Hg the linear range of the instrument is the value of the highest standard used to calibrate that instrument.

2. Action

   a. If Form XIII is improperly completed or incomplete it must be resubmitted (R).

   b. A missing Form XIII must be submitted (S). If the linear range(s) are out of date then the current linear ranges must be submitted (S).

   c. If any value obtained from an instrument falls above its established linear range of that instrument the sample should be marked as noncompliant (N).

000101

R. Raw Data

1. Screening Procedure

a. Verify that legible raw data (ICP, Flame AA, Hg, CN, Digestion and Distillation Logs, and % solids) including instrument readouts and indication of pH < 2 or > 12, as applicable, have been submitted to support all sample analyses and QC operations reported in the case.

b. Ensure that the raw data are properly labelled using EPA Sample numbers and codes and conform strictly to the contract requirements (See Table 1: SOW 787 B-8).

c. Ensure that the raw data are in the correct order (ICP, Flame AA, Furnace AA, Hg, CN, digestion and Distillation Logs, and % solids).

d. Verify that proper background correction and multiple injection/exposure requirements have been fulfilled. The Cover Page statement on background correction applies only to ICP.

e. Ensure that the time and date of each analysis has been given and the run sequence is clear and follows the contract requirements.

f. Ensure that no QA/QC samples are improperly run. If the first run of a QA/QC sample is outside of its specified control limits, the analysis must be terminated, the problem(s) corrected, the instrument recalibrated, and all affected samples reanalyzed.

g. Ensure that any crossouts made to the raw data are initialed and dated by lab personnel.

2. Action

a. If the raw data for any sample or QC operation are missing they must be submitted (S). If the data are illegible they must be resubmitted (R).

b. If the raw data are not properly labelled or do not strictly conform to the contract requirements, the data msut be resubmitted (R).

c. If the raw data are not in the correct order, note it on the worksheet as a "No Action" item. If the problem persists, report it to the laboratory's Project Officer.

d. If background corrections have not been applied to Furnace AA, ICP, or Flame AA measurements below 350 nm, mark all samples as noncompliant (N). If duplicate injections/exposures for Furnace ICP or AA (except MSA) have not been performed, mark the affected samples as noncompliant (N).

e. If time and dates of each analysis are not given in the raw data, the data must be resubmitted (R). If the raw data is not clear (i.e. too many crossouts, inconsistency between actual raw data and summary sheets, ambiguous explanations, etc...) the data must be resubmitted (R).

f. If QA/QC samples are rerun becuase the first analysis was outside of specified control limits for that QA/QC sample the analytical samples associated with that particular QA/QC sample must be marked noncompliant (N) under the QA/QC criterion it pertains to.

g. If crossouts in the raw data are not initialed and dated, the raw data must be resubmitted (R) with the appropriate initials and dates.

T. Sample Traffic Reports

1. Screening Procedure

   a. Verify that all samples listed on the Cover Page, and having a Form I, also have a Traffic Report copy in the data package.

   b. Ensure that the Traffic Report photocopy is legible as to case number, sample number, sample type, required analysis, receipt date and signature. The Traffic Reports shall be arranged in increasing EPA sample number order, considering both alpha and numeric designations.

   c. Check to see that all analyses requested on the Traffic Report have been carried out. (Note: If "Filtered" is entered on the Traffic report a "dissolved" metal analysis is not automatically requested. The specific analysis requested must be under the proper column in section "C RAS ANALYSIS" of the traffic report).

   d. Check to see if any of the samples are labeled as a field blank or a rinsate.

2. Action

   a. If a Traffic Report photocopy is missing, it must be submitted (S). If there is a Traffic Report photocopy but no Form I or raw data, the problem should be coded as explain (E).

   b. If the Traffic Report photocopy is illegible, it must be resubmitted (R).

   c. If requested analyses have not been performed, the data for that analysis and all required forms must be submitted (S).

   d. If an identified field blank or rinsate has been used for the duplicate, spike or serial dilution analysis (except if the sample is the only sample of that concentration and matrix in that SDG) the analysis must be submitted (S) on an appropriate sample, under the proper criterion.

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CONTRACT COMPLIANCE SCREENING SUMMARY FOR INORGANICS

CASE: SAMPLES: DATE MAILED:

LAB NAME: CONTRACT: SCREENER: DATE SCREENED:

SDG NUMBER: REGION: DATE RECEIVED:

SAMPLE NO.	A COVER PAGE	B DATA SHEET	CA CALIBRATION ICV/CCV	CB CRDL	D BLANK	E ICS	FA SPIKES PRE.	FB POST	G DUP.	H LCS	I MS@	J SER. DIL.	KA HOLDING HG	KB TIME CN	L IDL	M CORR. FACTR	N LIN. RANGE	R RAW DATA	T TR	DI-SK

PROBLEM CODE: E = EXPLAIN, R = RESUBMIT, S = SUBMIT, N = NONCOMPLIANT

000406

Appendix A

CONTRACT COMPLIANCE SCREENING FOR INORGANICS

WORKSHEET A - ICP

PAGE:

CASE:

CONTRACT:

SAMPLES:

SDG_NO.:

DATE SCREENED:

LAB NAME:

REGION:

SCREENED BY:

SAMPLE NO.	C/M	SAMP WT.	DATE RECD.	DATE ANAL.	QC ANALYSIS TYPE	QC ANALYSIS TIME	PROBLEMS

000102

2601

CONTRACT COMPLIANCE SCREENING FOR INORGANICS WORKSHEET B - AA AND CN PAGE:

CASE: CONTRACT: SAMPLES: SDG_NO.: DATE SCREENED:

LAB NAME: REGION: SCREENED BY:

SAMPLE	DATE															PROBLEMS

Appendix C

INORGANIC TARGET ANALYTE LIST (TAL)

Analyte	Contract Required Detection Limit (1,2) (ug/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

Appendix D

TABLE 2. INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/L)	Interferents	(mg/L)
Ag	1.0	Al	500
Ba	0.5	Ca	500
Be	0.5	Fe	200
Cd	1.0	Mg	500
Co	0.5		
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

Appendix E

TABLE 3. SPIKING LEVELS[1] FOR SPIKE SAMPLE ANALYSIS

	For ICP/AA (ug/L)		For Furnace AA (ug/L)		Other[2] (ug/L)
Element	Water	Soil	Water	Soil	
Aluminum	2,000	*			
Antimony	500	500	100	100	
Arsenic	2,000	2,000	40	40	
Barium	2,000	2,000			
Beryllium	50	50			
Cadmium	50	50	5	5	
Calcium	*	*			
Chromium	200	200			
Cobalt	500	500			
Copper	250	250			
Iron	1,000	*			
Lead	500	500	20	20	
Magnesium	*	*			
Manganese	500	500			
Mercury					1
Nickel	500	500			
Potassium	*	*			
Selenium	2,000	2,000	10	10	
Silver	50	50			
Sodium	*	*			
Thallium	2,000	2,000	50	50	
Vanadium	500	500			
Zinc	500	500			
Cyanide					100

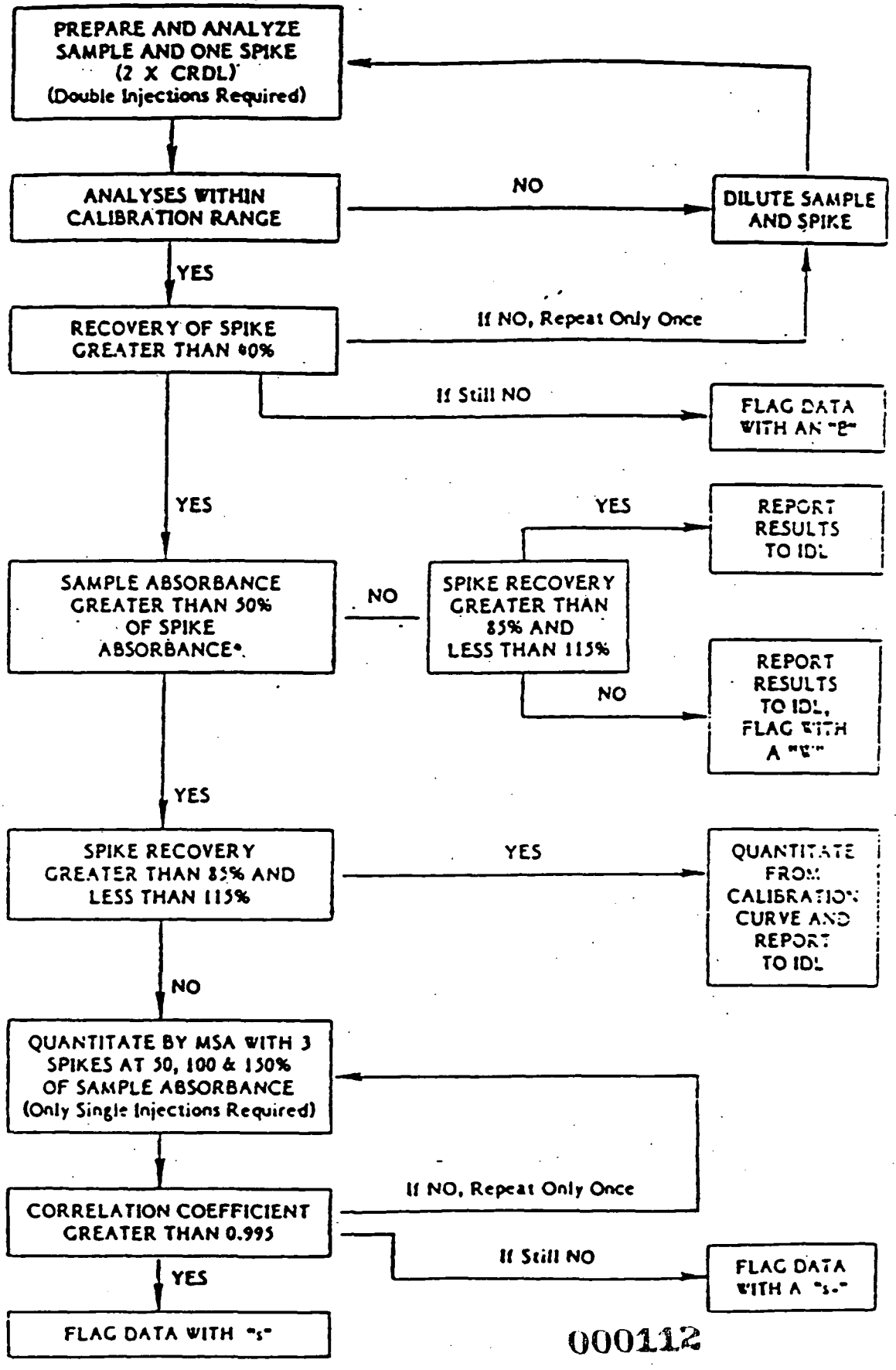
NOTE: Elements without spike levels and not designated with an asterisk, must be spiked at appropriate levels.

[1]The levels shown indicate concentrations in the final digestate of the spiked sample (200 mL final volume).

[2]Spiking level reported is for both water and soil/sediment matrices.

*No spike required.

Figure 1.
FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME



000112

*Spike absorbance defined as (absorbance of spike sample) minus (absorbance of the sample).

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SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER SHEET

Lab Name: _____ Contract No.: 68-01-_____

Lab Code: _____ Case No.: _____ SAS No.: _____-___

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ Sample Receipt Date: _____
(Lowest EPA Sample Number in first shipment of samples received under SDG) (MM/DD/YY)

Last Sample in SDG: _____ Sample Receipt Date: _____
(Highest EPA Sample Number in last shipment of samples received under SDG) (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

000113

_____ _____
Sample Custodian Date

SUGGESTED INSTRUMENT RUN SEQUENCES (SOW787)

The following suggested run sequences are based upon a 20 sample SDG (Sample Delivery Group). The example Sample IDs and respective concentrations are listed below. Also note the abbreviations key supplied for raw data.

Sample ID	Sample Type
MZZ001	ILW
MZZ002	ILW
MZZ003	ILW
MZZ004	ILW
MZZ005	ILW
MZZ006	IMW
MZZ007	IMW
MZZ008	IMW
MZZ009	IMW
MZZ010	IMW
MZZ011	ILS
MZZ012	ILS
MZZ013	ILS
MZZ014	ILS
MZZ015	ILS
MZZ016	IMS
MZZ017	IMS
MZZ018	IMS
MZZ019	IMS
MZZ020	IMS

Abbreviations Key

ILW = Inorganic Low Water

IMW = Inorganic Medium Water

ILS = Inorganic Low Soil

IMS = Inorganic Medium Soil

XXXXXX = Sample

XXXXXXD = Duplicate

XXXXXXS = Matrix Spike (Predigest./Predistill)

XXXXXXL = Serial Dilution

XXXXXXA = Analytical Spike (Postdigest./Postdistill)

ICP (Duplicate Exposures Required)

1) Blank	16) CCV1[a]	31) MZZ008	46) MZZ016D
2) Std1	17) CCB1[a]	32) MZZ009	47) MZZ016S
3) Std2	18) MZZ001D	33) MZZ010	48) MZZ017
4) ICV[a]	19) MZZ001S	34) MZZ010L	49) MZZ018
5) ICB[a]	20) MZZ002	35) MZZ011	50) MZZ019
6) ICSA[b]	21) MZZ003	36) MZZ011D	51) MZZ0020
7) ICSAB[b]	22) MZZ004	37) MZZ011S	52) CCV4[a]
8) CRII[c]	23) MZZ005	38) MZZ012	53) CCB4[a]
9) PB1 (ILW)	24) MZZ005L	39) MZZ013	54) MZZ020L
10) PB2 (IMW)	25) MZZ006	40) CCV3[a]	55) ICSA[b]
11) PB3 (ILS)	26) MZZ006D	41) CCB3[a]	56) ICSAB[b]
12) PB4 (IMS)	27) MZZ006S	42) MZZ014	57) CRIF[c]
13) LCSW	28) CCV2[a]	43) MZZ015	58) CCV5[a]
14) LCSS	29) CCB2[a]	44) MZZ015L	59) CCB5[a]
15) MZZ001	30) MZZ007	45) MZZ016	

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a An ICV/CCV and ICB/CCB must be performed at 10% frequency or every 2 hours, whichever is more frequent.

b The initial and final analyses of the ICSA and ICSAB solutions must be performed within 8 hours of each other.

c The initial and final analyses of the CRI standards must be performed within 8 hours of each other.

FURNACE AA (Duplicate Injection Required)

1)	Blank	27)	MZZ002	53)	CCV4	79)	MZZ017
2)	Std1	28)	MZZ002A	54)	CCB4	80)	MZZ017A
3)	Std2	29)	CCV2	55)	MZZ011	81)	MZZ0018
4)	Std3 (CRA)	30)	CCB2	56)	MZZ011A	82)	MZZ018A
5)	ICV	31)	MZZ003	57)	MZZ011D	83)	MZZ019
6)	ICB	32)	MZZ003A	58)	MZZ011DA	84)	MZZ019A
7)	PB1 (ILW)	33)	MZZ004	59)	MZZ011S	85)	MZZ020
8)	PB1A (ILW)	34)	MZZ004A	60)	MZZ011SA	86)	MZZ020A
9)	PB2 (IMW)	35)	MZZ005	61)	MZZ012	87)	MZZ001+0*
10)	PB2A (IMW)	36)	MZZ005A	62)	MZZ012A	88)	MZZ001+10*
11)	PB3 (ILS)	37)	MZZ006	63)	MZZ013	89)	MZZ001+20*
12)	PB3A (ILS)	38)	MZZ006A	64)	MZZ013A	90)	MZZ001+30*
13)	PB4 (IMS)	39)	MZZ006D	65)	CCV5	91)	CCV7
14)	PB4A (IMS)	40)	MZZ006DA	66)	CCB5	92)	CCB7
15)	LCSW	41)	CCV3	67)	MZZ014		
16)	LCSWA	42)	CCB3	68)	MZZ014A		
17)	CCV1	43)	MZZ006S	69)	MZZ015		
18)	CCB1	44)	MZZ006SA	70)	MZZ015A		
19)	LCSS	45)	MZZ007	71)	MZZ016		
20)	LCSSA	46)	MZZ007A	72)	MZZ016A		
21)	MZZ001	47)	MZZ008	73)	MZZ016D		
22)	MZZ001A	48)	MZZ008A	74)	MZZ016DA		
23)	MZZ001D	49)	MZZ009	75)	MZZ016S		
24)	MZZ001DA	50)	MZZ009A	76)	MZZ016SA		
25)	MZZ001S	51)	MZZ010	77)	CCV6		
26)	MZZ001SA	52)	MZZ010A	78)	CCB6		

*MSA single injection

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FLAME (Single Exposure Required)

1) Blank
2) Std1
3) Std2
4) Std3 (CRA)
5) ICV
6) ICB
7) PB1 (ILW)
8) PB2 (ILW)
9) PB3 (ILS)
10) PB4 (IMS)
11) LCSW
12) LCSS
13) MZZ001
14) MZZ001D
15) MZZ001S
16) MZZ002
17) CCV1
18) CCB1
19) MZZ003
20) MZZ004
21) MZZ005
22) MZZ006
23) MZZ006D
24) MZZ006S
25) MZZ007
26) MZZ008
27) MZZ009
28) MZZ010
29) CCV2
30) CCB2
31) MZZ011
32) MZZ011D
33) MZZ011S
34) MZZ012
35) MZZ013
36) MZZ014
37) MZZ015
38) MZZ016
39) MZZ016D
40) MZZ016S
41) CCV3
42) CCB3
43) MZZ017
44) MZZ018
45) MZZ019
46) MZZ020
47) CCV4
48) CCB4

MERCURY (Single Exposure Required)

1) Blank
2) Std1
3) Std2
4) Std3
5) Std4
6) ICV
7) ICB
8) PB1 (ILW)
9) PB2 (IMW)
10) PB3 (IMS)
11) PB4 (IMS)
12) LCSS (ILS, IMS)
13) MZZ001
14) MZZ001D
15) MZZ001S
16) MZZ002
17) MZZ003
18) CCV1
19) CCB1
20) MZZ004
21) MZZ005
22) MZZ006
23) MZZ006D
24) MZZ006S
25) MZZ007
26) MZZ008
27) MZZ009
28) MZZ010
29) MZZ011
30) CCV2
31) CCB2
32) MZZ011D
33) MZZ011S
34) MZZ012
35) MZZ013
36) MZZ014
37) MZZ015
38) MZZ016
39) MZZ016D
40) MZZ016S
41) MZZ017
42) CCV3
43) CCB3
44) MZZ018
45) MZZ019
46) MZZ020
47) CCV4
48) CCB4

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CONTRACT COMPLIANCE SCREENING

7601

INORGANIC DEFECT STATEMENTS LISTING

Prepared for

Analytical Operations Branch

Hazardous Site Evaluation Division

US EPA Office of Emergency and Remedial Response

by

Viar & Company

under

Contract No. 68-01-7235

Draft Version January 1989

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000120

The error statements and SOW references described in this document are used for all notification of defects identified during CCS screening. Each statement is structured as follows:

Square brackets [ ] enclose the list of possible items that may be associated with part of a defect statement. E.g., [slope/y-intercept/correlation coefficient/x-intercept (concentration)] [is/are] missing. Only applicable items should be included for the report statement.

Parentheses ( ) enclose the page reference to the Statement of Work on which notification of noncompliance is based.

{ } enclose the element with which the defect is associated.

For example, statement R14:

{ } MSA [data/slope/y-intercept] [is/are] missing. (E14-17).

could be reported as:

Pb MSA slope is missing. (E14-17).

The following pages list the error statements to be used for each type of defect, organized by the CCS criterion under which they should be reported. Note that the compliance of sample-associated blanks, spikes, and duplicates is partially assessed on the basis of their own associated QC criteria.

1. [Contract No./Lab Code/Case No./SAS No./SDG No.] is/are missing or incorrect on Form _____. (B12).
2. Lab code incorrect on Form _____. (B12).
3. [Case No./SAS No.] is inconsistent on Forms _____. (B13).
4. EPA format is not followed for Form _____. (A4).
5. Form suffix not correctly incremented for records corresponding to Form _____. (H5).

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Criterion A. Cover Page.

1. SOW No. is missing or incorrect. (B14).
2. EPA Sample No. is [missing/incorrect/not in alphanumeric order]. (B14). 7601
3. [Question one/Question two] is not answered (B14). **B**
4. Question Three is [answered incorrectly/not left blank] (B14). **B**
5. Lab manager's signature is not present. (B14).
6. Spike suffix (S) is [missing/associated with an incorrect sample]. (B14).
7. Duplicate suffix (D) is [missing/associated with an incorrect sample]. (B14).
8. Cover page is missing. (B14).

Criterion B. Form 1.

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1. EPA Sample No. is [missing/incorrect]. (B15).
2. Matrix is [missing/incorrect]. (B15).
3. Lab Sample ID. is inconsistent with the cover page. (B14).
4. Level is [missing/incorrect]. (B12).
5. Date received is [missing/inconsistent with the traffic report]. (B15).
6. % Solids is [missing/inconsistent with the raw data]. (B15).
7. Concentration units are [missing/inconsistent with the Sample Matrix]. (B15).
8. [Color/clarity [before/after]/texture/artifacts] are [improperly entered/missing]. (B16).
9. ( ) Concentration is [missing/inconsistent with the raw data]. (B15).
10. ( ) Concentration flag in Form I column C is [missing/inconsistent with the raw data]. (B15).
11. ( ) Form I "Q" qualifier is [missing/incorrect]. (B15, C1, D48, D69).
12. ( ) Form I "M" qualifier is [missing/incorrect]. (B15).
13. ( ) Reported concentration is not to the required significant figures. (B15).
14. ( ) Concentration is not corrected for % solids. (B5).
15. Form I is missing. (B5).

1. Initial calibration source is missing. (B16).
2. Continuing calibration source is missing. (B16,17).
3. ( ) Initial calibration true value is [missing/inconsistent with the source value]. (B17).
4. ( ) Initial calibration found value is [missing/inconsistent with the source value]. (B17).
5. Initial calibration %R is [missing/incorrect/not reported to one decimal place]. (B17).
6. ( ) Continuing calibration true value is [missing/inconsistent with the source value]. (B17).
7. ( ) Continuing calibration found value is [missing/incorrect/not reported to two decimal places]. (B17).
8. ( ) Continuing calibration %R is [missing/incorrect/not reported to one decimal place]. (B18).
9. ( ) M column method is [missing/incorrect]. (B18).
10. ( ) [Initial calibration/continuing calibration] %R is outside the specified control limits. (E5).
11. The same continuing calibration standard was not used throughout for ( ). (E5).
12. Form 2A is missing. (B5).

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1. AA CRDL standard source is missing. (B18).

2. ICP CRDL standard source is missing. (B18).

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3. ( ) CRDL standard source for (AA/ICP) is [missing/not at the CRDL/not run at two times the IDL when IDL is greater than the CRDL/not run at two times the CRDL when the CRDL is greater than the IDL]. (B18,E6).

4. ( ) CRDL standard (CRI) analyzed in the middle of the run is [not reported/not reported in the Final Found section of the Form]. (B19).

5. ( ) [CRI/CRA] is missing when more than one wavelength is used. (B19).

6. ( ) CRDL standard for AA true value is not reported to one decimal place. (B18).

7. ( ) CRDL standard for AA found value is not reported to two decimal places. (B18).

8. ( ) CRDL standard for ICP [initial found/final found] is not reported to two decimal places. (B19).

9. ( ) %R for [AA/ICP] is [missing/incorrect/not reported to one decimal place. (B18,19).

10. Form 2B is missing. (B5).

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1. Preparation blank matrix is [missing/reported incorrectly]. (B19).
2. Preparation concentration units are [missing/inconsistent with the matrix]. (B19).
3. ( ) [initial calibration blank/aqueous preparation blank/continuing calibration blank] is [missing/inconsistent with the raw data/not in ug/L/not reported to one decimal place]. (B20).
4. ( ) absolute value of [initial/continuing] calibration blank exceeds the [IDL/CRDL]. (E7/C1).
5. ( ) column C qualifier is [missing/incorrect]. (B20).
6. ( ) M qualifier is [missing/incorrect]. (B20).
7. ( ) Sample value is below 10 times CRDL and is reported incorrectly because the preparation blank value is below the negative CRDL. (E7).
8. ( ) Sample value is less than 10 times the preparation blank value and the preparation blank value exceeds the CRDL. (E7).
9. Form 3 is missing. (B5).

1. ICP ID No. is missing. (B20).
2. ( ) true values for [solution A/solution AB] are [missing/incorrect/not reported to the nearest whole number]. (B21).
3. ICS source ID (or Lot number) is missing. (B21).
4. ( ) [Initial/final found] [Sol/A/Sol. AB] values are [missing/inconsistent with the raw data/not reported to the nearest whole number for solution A/not reported to one decimal place for solution AB]. (B21).
5. ( ) [Negative/zero] found value for [solution A/solution AB] is [missing/incorrect/inconsistent with the raw data]. (B21).
6. ( ) Found values for [solution A/solution AB] are missing for the wavelength used. (B21).
7. ( ) %R is [missing/incorrect/not reported to one decimal place] for [initial found value/final found value]. (B21).
8. Form 4 is missing. (B6).

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1. Form 5A is [missing/not performed for specified sample]. (B6/E9).
2. [Matrix/level] is [missing/inconsistent with the traffic report]. (B12/B22). 7601
3. Concentration is [missing/inconsistent with the matrix]. (B22).
4. ( ) control limits are [missing/incorrect/not left empty when the sample is greater than four times the spike added]. (B22). **B**
5. ( ) spiked sample result is [missing/inconsistent with the raw data/not in the appropriate units]. (B22).
6. ( ) [spiked sample result/sample result] C qualifier is [missing/incorrect]. (B22).
7. ( ) sample result is [missing/inconsistent with the raw data/inconsistent with Form 1]. (B22).
8. ( ) spike added is [missing/incorrect]. (B22).
9. ( ) %R is [missing/incorrect/not reported to one decimal place]. (B22).
10. ( ) Q qualifier is [missing/incorrect when the sample is four times greater than the spike added]. (B22/E9).
11. ( ) M qualifier is [missing/incorrect]. (B23).
12. ( ) [spiked sample result/sample result/spike added] is missing for the alternate method used. (E9).
13. EPA Sample No. is missing suffix. (B13). **B**

1. Form 5B is missing. (B6). **B**
2. [Matrix/Level] is [missing/inconsistent with the traffic report]. (B22/B23). **B**
3. ( ) [control limit/%R/Q field] is reported when it should be blank. (B23). **B**
4. ( ) Sample result is [missing/inconsistent with Form 1/not required/not reported to two decimal places/not in ug/L]. (B23). **B**
5. ( ) spiked sample result is [missing/inconsistent with the raw data/not reported to two decimal places/not reported in ug/L]. (B23). **B**
6. ( ) spike added is [missing except for Ag/incorrect/not reported in ug/L/not reported to one decimal place/not added at the greater of two times CRDL or twice the sample value]. (B23/E9). **B**
7. ( ) %R is [missing/incorrect/not calculated to one decimal place/not reported when negative/not reported when zero]. (B23). **B**
8. ( ) Q qualifier is not left blank. (B23). **B**
9. ( ) M qualifier is [missing/incorrect]. (B24). **B**
10. EPA Sample No. is missing an A suffix. (B8). **B**

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1. Form is [missing/not submitted for specified sample]. (B6/E11).
2. [Matrix/Level] is [missing /inconsistent with the traffic report]. (B24).
3. % solids for [sample/duplicate] is missing. (B24).
4. Concentration units are [missing /inconsistent with the matrix]. (B24).
5. EPA Sample No is missing the D suffix. (B13). **B**
6. ( ) control limit is [missing incorrect/not corrected for original sample weight/not corrected for % solids/not left empty when sample or duplicate is greater than five times CRDL]. (B24,27,E11). **B**
7. ( ) sample value is [missing/inconsistent with Form 1/inconsistent with the raw data]. (B24).
8. ( ) Duplicate value is [missing/inconsistent with the raw data]. (B24).
9. [C,M] qualifier is [missing/incorrect]. (B24,25).
10. ( ) Q qualifier is [missing/incorrect]. (B24,25).
11. ( ) RPD is [missing/incorrect/not reported to one decimal place]. (B24,25).

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2. [Solid/Aqueous] LCS Source ID (or Lot number) is missing. (B25).

3. ( ) [Aqueous/Solid] true value is [missing/incorrect]. (B25). 7601

4. ( ) Aqueous found value is [missing/inconsistent with the raw data/not reported to two decimal places]. (B25,26/E12).

5. ( ) Solid found value is [missing/outside the control limits/inconsistent with the raw data/not reported to one decimal place]. (B25,26/E12).

6. ( ) Aqueous %R is [missing/incorrect/not reported to one decimal place/outside control limits]. (B25,26).

7. ( ) Solid %R is [missing/incorrect/not reported to one decimal place]. (B25,26). **B**

8. ( ) C qualifier is [missing/incorrect]. (B25).

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1. Form 8 is missing. (B6).
2. [EPA Sample No./Analyte/dilution factor] is [missing/not in alphanumeric order/incorrect/inconsistent with the raw data]. (B26).
3. Added [concentration/absorbance] is [missing/inconsistent with the raw data]. (B26,27).
4. Final concentration is [missing/inconsistent with the matrix/inconsistent with the raw data] for EPA Sample No. (B27).
5. MSA was incorrectly done on the LCS. (E14).
6. Final concentration is [missing/not reported to one decimal/not reported as an absolute value/is not reported as less than the IDL] for EPA Sample No. (B27).
7. R value is [missing/not consistent with the raw data] for EPA Sample No. (B27).
8. MSA was not reported as required. (E14-17) for EPA No.
9. Q qualifier is [missing/incorrect]. (E14-17).

1. Form 9 is missing. (B6).
2. [Matrix/Level] is [missing /inconsistent with the traffic report]. (B28).
3. ( ) Initial Sample result is [missing/inconsistent with Form 1]. (B28).
4. ( ) serial dilution result is [missing/inconsistent with the raw data/not multiplied by 5]. (B28).
5. ( ) % difference is [missing/incorrect]. (B28).
6. ( ) [Initial Sample/Serial dilution] result C qualifier is [missing/incorrect]. (B28).
7. ( ) Q qualifier is [missing/incorrect]. (B29).
8. ( ) M qualifier is [missing/incorrect]. (B29). **B**

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1. [Mercury/Cyanide] holding time [cannot be determined/exceeds SOW specification]. (D4).

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1. Form 10 is missing. (B6).
2. Date is missing. (B30).
3. [ICP/Flame/Furnace] ID Number is missing. (B30).
4. ( ) Wavelength is [missing/not reported to two decimal places]. (B30).
5. ( ) Background code is [missing/incorrect]. (B30).
6. ( ) IDL is [missing/not reported to one decimal place]. (B30).
7. ( ) M qualifier is [missing/incorrect]. (B30).
8. Instrument (_____) IDLs were determined more than three months before the date of analysis. (E14).
9. Sample values are invalid because the IDL exceeds the CRDL and reported values are less than 5x IDL. (A2).

1. Form 11 is [missing/not reported as required]. (B6).
2. Date is missing. (B30).
3. ICP ID No. is missing. (B31).
4. ( ) Wavelength is [missing/not reported to two decimal places]. (B31).
5. [Al/Ca/Fe/Mg] correction factor is [missing/not reported when [zero/negative]. (B31).
6. Instrument (_____) correction factor(s) [was/were] determined more than one year from date of analysis. (E16).

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1. Form 12 is missing. (B32).
2. ICP ID No. is missing. (B32).
3. Date is [missing/incorrect]. (B32).
4. ( ) Integration time [missing/not in seconds/greater than five spaces]. (B32).
5. ( ) Concentration is [missing/not in ug/L]. (B32).
6. ( ) M qualifier is [reported improperly/missing (NR)]. (B32).
7. Instrument (_____) linear range(s) [was/were] determined more than three months before the date of analysis. (E16).
8. ( ) Concentration reported on Form 1 was obtained from a value exceeding the [linear/calibration] range. (A3, B34).

1. Form 13 is missing. (B35).
2. More than 32 analyses were reported on one Form 13.
3. Method of analysis is [missing/not an acceptable code]. (B35).
4. [EPA sample no./duplicate/spike/LCS/prep. blank] is [missing/not in ascending alpha numeric order/formatted incorrectly]. (B35).
5. Repreparation of [EPA sample no./duplicate/spike/LCS/prep. blank] was [missing/not in the order of increasing preparation date/formatted incorrectly]. (B35).
6. Preparation date is [formatted incorrectly/incorrectly/missing/incorrect with respect to the method used]. (B35).
7. Under "weight", the wet weight is [missing/not reported to two decimal places/not in grams/not the wet weight/incorrect with regards to the method fo analysis] for soil samples. (B35).
8. Weight is not blank for water samples. (B35)
9. The final volume is [missing/not in ml/not to the nearest whole number]. (B35).

1. Form 14 is missing. (B36).

2. The following [field sample/QC sample (i.e. the calibration standards, ICVs, CCVs, ICBs, CCBs, CRAs, CRIs, ICSs, LRSs, LCSs, PBs, duplicate, serial dilutions, pre-digestion spikes, post digestion spikes, analytical spikes) MSA addition], associated with the SDG, [was/were] not reported for the following [Analytes/Method] on Form 14. (B36).

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3. More than 32 analysis were reported on one Form 14. (B36).

4. Instrument ID number is [missing/greater than 12 spaces/not uniquely identifying the instrument used to produce the data]. (B36).

5. Method code is [missing/greater than two characters]. (B36).

6. ["Start Date"/"End Date"]is [missing/formatted incorrectly/not when the analysis was [done/ended]. (B36).

7. Sample No. is not [formatted properly/in temporal order]. (B36).

8. Dilution factor is [missing/not to two decimal places/incorrectly including the inherent preparation dilution]. (B37).

9. Time is [not in the correct format/missing]. (B7) (B37).

10. Percent recovery for the furnace analytical spike(s) [is/are] [missing/incorrect]. (B37).

11. Data missing for ( ). (B37).

12. Instrument calibration is not [performed/performed with proper number of standards] for ( ). (E3).

13. Instrument calibration was not performed within 24 hours of analysis for ( ). (E3).

14. The two hour calibration time limit for is violated for ( ). (E5).

15. The 10% calibration frequency is violated for ( ). (E5, E6, E14, E15).

16. [ICS/CRI] [was/were] not analyzed twice within the 8 hour working shift for ( ). (E6, E8).

17. The MSA tree was not followed correctly for ( ). (E14).

18. [ICS/CRI/ICV] was not performed after calibration for ( ). (E6).

19. [ICB/CCB] was not performed after [ICV/CCV] for ( ). (E6).

20. [ICS/CRI/CCV] was not performed at the end of the run for ( ). (E6).

21. Preparation blank analytical spike recovery is out of SOW specified range for ( ). (E17).

22. Sample was not run initially undiluted for ( ). (A3).

23. Serial dilution was not performed at 5x dilution for ( ). (E12).

Criterion R. Raw Data

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1. [ICP/Furnace/Flame/Hg/CN] raw data are missing. (B7).
2. SDG package is not numbered sequentially. (B4).
3. ( ) Raw data is [illegible/deleted but not signed/unclear]. (B7).
4. SDG package not submitted with single-sided pages. (B3).
5. SDG package forms are not in increasing EPA sample alphanumeric order. (B5).
6. ( ) Instrument readouts as strip charts/tapes/or hard copies are necessary. (B6).
7. ( ) Raw data are submitted out of order. (B6).
8. ( ) Raw data are not labelled with [EPA sample number appropriate codes]. (B7).
9. ( ) Data for [calibration [standards/source/solution preparation date/blanks]/sample [volume/weight/duplicate/spikes/serial dilution/analytical spike/%R/CV/]/instrument used/background correction used/] are missing. (B7).
10. ( ) MSA [data/slope/y-intercept] [is/are] missing. (E14-17).
11. ( ) Integration times are missing for AA analyses. (B7).
12. Digestion log(s) [is missing[sample/preparation blank/LCS/date/volume/weight used]. (B9).
13. Aqueous pH is [less than 2/greater than 12/missing]. (B9).
14. ( ) Value is not reported uncorrected in the raw data. (A3).
15. Duplicate exposures are missing for ICP. (D1).
16. ( ) Furnace element did not have double injections (except MSA). (E14).
17. [ICP/Furnace/Flame/HG/CN] standard curve is incomplete. (E4).
18. [ICP/Furnace/Flame/HG/CN] standard at the CRDL is missing for ( ). (E4).
19. EPA specified [ICP/Furnace/Flame/Hg/CN] method was not used for ( ). (A2).
20. Furnace spike was added incorrectly for ( ). (E10).
21. Percent solids data are [missing/incorrect]. (B15, E11).
22. The MSA tree was not followed correctly for ( ). (E14).
23. Post digest spiking volume exceeded 10% of the sample volume. (E16).
24. Standardization solution concentrations are missing. (B7).
25. Instrument was not calibrated [daily/when it was set-up]. (E3).

2. SDG Group No. is [missing/improperly assigned] on traffic report. (B4).
3. SDG final sample is missing on traffic report. (B4).
4. [Lab manager's signature/receipt date/sample condition on receipt] is missing on the traffic report. (B4).
5. [Matrix spike/duplicate/serial dilution] was run on a field blank. (E9,11,12).

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APPENDIX C

Completed Data Validation Reports

FIGURE 6
DQO SUMMARY FORM

1. SITE

NAME: DQO DEMONSTRATION
LOCATION:
NUMBER:

EPA REGION: 1
PHASE: RI 1 RI 2 RI 3 ERA FS RD RA (CIRCLE ONE)

2. MEDIA (CIRCLE ONE): SOIL | GW | SW/SED | AIR | BIO | OTHER

3. USE (CIRCLE ALL THAT APPLY): SITE CHARAC. (RI&S) | RISK ASSESS. | EVAL. ALTS. | ENGG DESIGN | PRP DETER. | MONITORING REMEDIAL ACTION | OTHER

4. OBJECTIVE Existing wells will be sampled to determine if contaminants are present in residential wells and to obtain inforamtion on the levels of contaminants in on-site observation wells

5. SITE INFORMATION

AREA: 200ft X 200ft depression DEPTH TO GROUND WATER: 15 feet
GROUND WATER USE: Drinking water
SOIL TYPES: Glacial till-depth 0-30ft; shale 30->100ft
SENSITIVE RECEPTORS:

6. DATA TYPES (CIRCLE APPROPRIATE DATA TYPES)

A. ANALYTICAL DATA

pH	PESTICIDES	TOX
CONDUCTIVITY	PCB	TOC
VOA	METALS	BTX
ABN	CYANIDE	COD
TCLP		

B. PHYSICAL DATA

PERMEABILITY	HYDRAULIC HEAD
POROSITY	PENETRATION TEST
GRAIN SIZE	HARDNESS
BULK DENSITY	

7. SAMPLING METHOD (CIRCLE METHOD(S) TO BE USED)

ENVIRONMENTAL | BIASED | GRAB | NON-INTRUSIVE | PHASED
SOURCE | GRID | COMPOSITE | INTRUSIVE

8. ANALYTICAL LEVELS (INDICATE LEVEL(S) AND EQUIPMENT & METHODS)

LEVEL 1 FIELD SCREENING - EQUIPMENT:
LEVEL 2 FIELD ANALYSIS - EQUIPMENT:
LEVEL 3 NON-CLP LABORATORY - METHODS:
LEVEL 4 CLP/RAS - METHODS: Total Metals Analysis
LEVEL 5 NON STANDARD:

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR: Well OW1 & OW2
CRITICAL (LIST): 3 Private well samples (A1, A2 & A3)
PROCEDURES: Private wells - sample at Tap. OW1 & OW2 Bailer

10. QUALITY CONTROL SAMPLES (CONFIRM OR SET STANDARD)

A. FIELD
COLLOCATED - 5% OR: 4 Duplicates
REPLICATE - 5% OR:
FIELD BLANK - 5% OR: 1
TRIP BLANK - 1 PER DAY OR:

B. LABORATORY
REAGENT BLANK - 1 PER ANALYSIS BATCH OR:
REPLICATE - 1 PER ANALYSIS BATCH OR:
MATRIX SPIKE - 1 PER ANALYSIS BATCH OR:
OTHER:

11. BUDGET REQUIREMENTS

BUDGET: $6,000 SCHEDULE: 2 days for sampling, 5 days chemist
STAFF: 2 person field crew, chemist for data validation & interpretation

CONTRACTOR: PRIME CONTRACTOR:
SITE MANAGER: DATE:

FOR DETAILS SEE SAMPLING & ANALYSIS PLAN

CDM SF DQO 1.002

000144

November 16, 1988

Dennis Gagne
Regional Sample Control Custodian
U.S. Environmental Protection Agency
90 Canal Street
Boston, Massachusetts 02114

Re: TID No. 01-8809-15
Case 9164, SDG235
XYZ Laboratories
ABC Site, Lexington, MA
Metals: 10/aqueous

Dear Mr. Gagne:

A validation was performed on the inorganic analytical data from 10 aqueous samples collected by LMO at the ABC site. The data were evaluated based on the following parameters:

- * ● data completeness
- * ● holding times
- ● calibration verification
- ● laboratory and field blank analyses
- ● ICP interference check sample results
- ● matrix spike recoveries
- * ● laboratory and field duplicates
- ● laboratory control sample results
- ● furnace atomic absorption results
- * ● serial dilution results
- * ● detection limit results
- * ● sample results

● All criteria were met for this parameter.

<u>Table I summarizes the validation recommendations which were based on the following information</u>:

Calibration Verification

The 2xCRDL standard for Cadmium had a recovery of 65%. Results near the CRDL may be biased low. Estimate (J) positive results less than 3xCRDL and non-detected results (UJ).

Blanks

Element	Maximum Conc./Units	Action Level
Antimony	53.4 ug/l	267 ug/l
Copper	12.1 ug/l	60.5 ug/l
Zinc	6.0 ug/l	30 ug/l

Value > IDL and < Action Level = Report value U
Value > IDL and > Action Level = Report value unqualified

ICP Interference Check Sample

Positive results were observed for Antimony in ICS A solution although there is no Antimony present in the solution. The positive results may be due to an interference from Iron. Significant levels of Iron are present in the samples. It is recommended to estimate all positive results for Antimony.

Matrix Spike Recoveries

TR#s	Analyte	Percent Recovery
MAE236	Silver	72
MAE236	Lead	60

Positive and non-detected results shall be estimated for both Silver and Lead.

Laboratory Duplicates

Laboratory duplicates did not meet required criteria for Copper. Estimate (J) positive results only.

Post-digestion Spikes

The following samples analyzed by graphite furnace had low post digestion spike recoveries:

November 16, 1988

Element	Samples Affected
Selenium	MAE237, MAE247
Lead	MAE246
Thallium	All except MAE240 and MAE247

All affected samples are non-detected and shall be estimated (UJ).

Serial Dilution Analysis

Serial dilution results did not meet required criteria for Calcium. Estimate (J) all positive results.

No other problems were encountered with this case.

Very truly yours,

ROY F. WESTON, INC.

Karol-Anne O'Leary
Data Reviewer

Joseph D. Mastone
Team Manager
ESAT Region I

KAO/kao
Enlosures
cc. D. Szaro

ABC Site Name
Case 9164
Table I. Recommendation Summary

Aluminum		Magnesium	
Antimony	A^1	Manganese	
Arsenic		Mercury	
Barium		Nickel	
Berylium		Potassium	
Cadmium	J^1	Selenium	
Calcium	J^5	Silver	J^2
Chromium		Sodium	
Cobalt		Thallium	J^4
Copper	A^1, J3	Vanadium	
Iron		Zinc	A^1
Lead	J^2	Cyanide	

If the field is left blank the qualifier is A - Accept all data.

A^1 - Accept data, raise the sample detection limit(s) due to blank contamination.

J^1 - Estimate (UJ) non-detetced results due to poor linearity near the CRDL. Detection limit is biased low.

J^2 - Estimate (J) positive values and detection limits (UJ) due to low matrix spike recoveries. These estimated results would be minimum values.

J^3 - Estimate (J) all positive values due to poor duplicate precision.

J^4 - Estimate (UJ) non-detected results due to low matrix spike recoveries for Se (MAE237 and MAE247), and Tl (all except MAE240 and MAE247).

J^5 - Estimate (J) positive results due to a supression observed through the serial dilution analysis. Estimated results represent minimum values.

SITE: A' 'ITE
CASE: 9. SDG: MAE235
LABORATORY: XYZ LABORATORIES

INORC AQUEOUS ANALYSIS
ug/L

SAMPLE NUMBER:			MAE235	MAE236	MAE237	MAE238	MAE239	MAE240	MAE245	MAE246	MAE247	MAE258	
SAMPLE LOCATION:			SW-1	SW-2	SW-3	SW-4	SW-5	SW-5	SW-6	SW-7	SW-8	SW-9	
LABORATORY NUMBER:													
INORGANIC ELEMENTS		INSTRUMENT DETECTION LIMITS (ug/l, ppb)											CONTRACT DETECTION LIMITS (ug/l,ppb)
ALUMINUM	P	25	60.5 J	--	307	--	--	--	--	--	--	25 J	200
ANTIMONY	P	37	80 U	--	--	91 U	--	87 U	--	--	--	--	60
ARSENIC	F	3.0	--	--	--	--	--	--	--	--	--	--	10
BARIUM	P	2.0	18.8	8.0	10.8	18.7	19.6	--	23.6	178	--	22.4	200
BERYLLIUM	P	2.0	--	--	--	--	--	--	--	--	--	--	5
CADMIUM	P	5.0	UJ	UJ	UJ	UJ	UJ	UJ	UJ	UJ	UJ	UJ	5
CALCIUM	P	81	33400 J	35200 J	4140 J	17000 J	16900 J	104 J	29400 J	23000 J	--	37300 J	5000
CHROMIUM	P	4.0	--	--	--	--	--	--	--	--	--	--	10
COBALT	P	4.30	--	--	--	--	--	--	--	--	--	--	50
COPPER	P	10	24.2 U	33.1 U	103 J	--	--	--	--	--	--	63 J	25
IRON	P	14	102000	49.2	21.7 J	91000	21.6 J	120000	875	1490	--	626	100
LEAD	P	3.0	5.2 J	4.1 J	16.3 J	UJ	UJ	4.3 J	UJ	UJ	UJ	19 J	5
MAGNESIUM	P	81	1980	1870	752	2460	2430	--	2950	2940	--	2980	5000
MANGANESE	P	6.0	22.0	8.4 J	38.2	13.0	12.4	--	76.4	2920	--	20.7	15
MERCURY	CV	0.1	--	--	--	--	--	--	--	--	--	--	0.2
NICKEL	P	10	--	--	--	--	--	--	--	--	--	--	40
POTASSIUM	P	95	1810	3550	785	1750	1710	--	5030	4560	--	3700	5000
SELENIUM	F	2.0	--	--	UJ	--	--	--	--	--	UJ	--	5
SILVER	P	4.0	UJ	UJ	UJ	UJ	UJ	UJ	UJ	UJ	UJ	UJ	10
SODIUM	P	1360	18900	10700	7500	52600	51800	--	35700	88200	--	21700	5000
THALLIUM	F	2.0	UJ	UJ	UJ	UJ	UJ	--	UJ	UJ	--	UJ	10
VANADIUM	P	4.0	--	--	--	--	--	--	--	--	--	--	50
ZINC	P	3.0	39.6	19.9 U	44.4	7.8 U	8.4 U	--	23.9 U	23.3 U	--	74.0	20

ANALYTICAL METHOD
F - FURNACE
P - ICP/FLAME AA
CV - COLD VAPOR

NOTE:
J - QUANTITATION IS APPROXIMATE DUE TO LIMITATIONS IDENTIFIED IN THE QUALITY CONTROL REVIEW (DATA REVIEW).
R - VALUE IS REJECTED
-- VALUE IS NON-DETECTED

REGION I
Data Review Worksheets

Site Name ABC Site
Reference Number 01-8809-15

REGION I REVIEW OF INORGANIC CONTRACT LABORATORY DATA PACKAGE

The hardcopied (laboratory name) XYZ Laboratories data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

Case No.	9164	SAS No.		Sampling Date(s)	03/14/88-03/17/88
SDG. No.	MAE235	Matrix	Aqueous	Shipping Date(s)	03/18/88
No. of Samples	10			Date Rec'd by Lab	03/19/88

Traffic Report Nos: MAE235 - MAE240, MAE245 - MAE247, MAE258

Trip Blank No.:
Equipment Blank No.:
Field Dup Nos:

SOW No. 7/87 requires that specific analytical work be done and that associated reports be provided by the laboratory to the Regions, EMSL-LV, and SMO. The general criteria used to determine the performance were based on an examination of:

- -Data Completeness
- -Holding Times
- -Calibrations
- -Blanks
- -ICP Interference Check Results
- -Matrix Spike Recoveries
- -Laboratory Duplicates
- -Field Duplicates
- -Lab Control Sample Results
- -Furnace AA Results
- -ICP Serial Dilution Results
- -Detection Limit Results
- -Sample Quantitation

Overall Comments: The data package was complete and the copies were of good quality.

Definitions and Qualifiers:

A - Acceptable data.
J - Approximate data due to quality control criteria.
R - Reject data due to quality control criteria.
U - Analyte not detected.

Reviewer: Karol-Anne O'Leary Date: 11/15/88

II. HOLDING TIMES

Complete table for all samples and circle the fractions which are not within criteria.

SAMPLE ID	DATE SAMPLED	HG DATE ANALYSIS	CYANIDE DATE ANALYSIS	OTHERS DATE ANALYSIS	pH	ACTION
MAE235	03/14/88	13 03/27/88	NA	04/17/88	< 2	
MAE236	03/14/88	13 03/27/88	NA	04/17/88	< 2	
MAE237	03/14/88	13 03/27/88	NA	04/17/88	< 2	
MAE238	03/14/88	13 03/27/88	NA	04/17/88	< 2	
MAE239	03/14/88	13 03/27/88	NA	04/17/88	< 2	
MAE240	03/15/88	12 03/27/88	NA	04/17/88	< 2	
MAE245	03/17/88	10 03/27/88	NA	04/17/88	< 2	
MAE246	03/17/88	10 03/27/88	NA	04/17/88	< 2	
MAE247	03/18/88	9 03/27/88	NA	04/17/88	< 2	
MAE258	03/16/88	11 03/27/88	NA	04/17/88	< 2	

METALS - 180 DAYS FROM SAMPLE COLLECTION
MERCURY - 28 DAYS FROM SAMPLE COLLECTION
CYANIDE - 14 DAYS FROM SAMPLE COLLECTION

ACTION:

1. If holding times are exceeded all positive results are estimated (J) and non-detects are estimated (UJ).

2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable (R).

III A. INSTRUMENT CALIBRATION (Section 1)

1. Recovery Criteria

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

DATE	ICV/CCV#	ANALYTE	%R	ACTION	SAMPLES AFFECTED

ACTIONS:

If any analyte does not meet the %R criteria follow the actions stated below:

For Positive Results:

	Accept	Estimate (J)	Reject (R)
Metals	90-110%R	75-89%R, 111-125%R	<75%R, >125%R
Mercury	80-120%R	65-79%R, 121-135%R	<65%R, >135%R
Cyanide	85-115%R	70-84%R, 116-130%R	<70%R, >130%R

For Non-detected Results:

	Accept	Estimate (UJ)	Reject (R)
Metals	90-125%R	75-89%R	<75%R, >125%R
Mercury	80-135%R	65-79%R	<65%R, >135%R
Cyanide	85-130%R	70-84%R	<70%R, >130%R

All criteria were met.

III B. INSTRUMENT CALIBRATION (Section 2)

2. Analytical Sequence

A. Did the laboratory use the proper number of standards for calibration as described in the SOW? Yes or No

B. Were calibrations performed at the beginning of each analysis? Yes or No

C. Were calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent or every two hours during analysis, whichever is more frequent? Yes or No

D. Were the correlation coefficients for the calibration curves for AA, Hg, and CN $\geq$ 0.995? Yes or No

E. Was a standard at 2xCRDL analyzed for all ICP analyses? Yes or No

If No,

The data may be affected. Use professional judgement to determine severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

The CRDL standard for Cadmium had a recovery of 65%. Estimate all values for Cadmium < 3 X CRDL (15ug/L).

REGION I
Data Review Worksheet

IV A. BLANK ANALYSIS RESULTS (Sections 1-3)

List the blank contamination in Sections 1 & 2 below. A separate worksheet should be used for soil and water blanks.

1. Laboratory Blanks

MATRIX: Aqueous

DATE	ICB/CCB#	PREP BL	ANALYTE	CONC./UNITS
4/13/88	ICB		Sb	53.4
4/13/88		Aqueous	Cu	12.1
4/13/88		Aqueous	Zn	6.0

2. Equipment/Trip Blanks

DATE	EQUIP BL#	ANALYTE	CONC./UNITS

3. Frequency Requirements

A. Was a preparation blank analyzed for each matrix, for every 20 samples and for each digestion batch? (Yes) or No

B. Was a calibration blank run every 10 samples or every 2 hours whichever is more frequent? (Yes) or No

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below, and list the samples affected.

-
-

IV B. BLANK ANALYSIS RESULTS (Section 4)

. Blank Actions

The Action Levels for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a U.

2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

MATRIX: Aqueous

ELEMENT	MAX. CONC./ UNITS	AL/ UNITS
Sb	53.4	267 ug/L
Cu	12.1	605 ug/L
Zn	6.0	30 ug/L

MATRIX: _____

ELEMENT	MAX. CONC./ UNITS	AL/ UNITS

NOTE: Blanks analyzed during a soil case must be converted to mg/kg in order to compare them with the sample results.

$$\text{Conc. in ug/L} \times \frac{\text{Volume diluted to (200ml)}}{\text{Weight digested (1gram)}} \times \frac{1\text{L}}{1000\text{ml}} \times \frac{1000\text{gm}}{1\text{kg}} \times \frac{1\text{mg}}{1000\text{ug}} = \text{mg/kg}$$

Multiplying this result by 5 to arrive at the action level gives a final result in mg/kg which can then be compared to sample results.

V A. ICP INTERFERENCE CHECK SAMPLE (Sections 1 & 2)

1. Recovery Criteria

List any elements in the ICS AB solution which did not meet the criteria for %R.

DATE	ELEMENT	%R	ACTION	SAMPLES AFFECTED

ACTIONS:

If an element does not meet the %R criteria , follow the actions stated below:

	PERCENT RECOVERY		
	<50%	50-79%	>120%
Positive Sample Results	R	J	J
Non-detected Sample Results	R	UJ	A

2. Frequency Requirements

Were Interference QC samples run at the beginning and end of each sample analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent? Yes or No

If no,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

All acceptable.

V B. ICP INTERFERENCE CHECK SAMPLE (Section 3)

Report the concentration of any elements detected in the ICS A solution > 2xIDL that should not be present.

ELEMENT	CONC. DETECTED IN THE ICS	CONC. OF INTERFERENTS IN THE ICS AL	CA	FE	MG
Sb	50 ug/L	506000	525000	188000	521000

Estimate the concentration produced by the interfering element in all affected samples. See guidelines for examples. List the samples affected by interferences below:

SAMPLE AFFECTED	ELEMENT AFFECTED	SAMPLE CONC. (ug/L)	SAMPLE INTERFERENT CONC. AL	CA	FE	MG	ESTIMATED INTERF. (ug/L)
MAE235	Sb	80			102000		27
MAE238	Sb	91			91800		24
MAE240	Sb	87			120000		32

ACTIONS:

1. In general, the sample data can be accepted without qualification if the sample concentrations of Al, Ca, Fe, and Mg are less than 50% of their respective levels in the ICS solution.

2. Estimate (J) positive results for affected elements for samples with levels of interferents 50% or more of that in the ICS solution.

3. Reject (R) positive results if the reported concentration is due entirely to the interfering element.

4. Estimate (UJ) non-detected results for which false negatives are suspect.

Give explanations for any actions taken below:
Estimate (J) positive results for antimony.

REGION I
Data Review Worksheets

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VI. MATRIX SPIKE

..« # MAE236 MATRIX: Aqueous

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

S - amount of spike added
SSR - spikes sample result
SR - sample result

Analyte	SSR	SR	S	%R	Action
Silver	36	4.0 u	50	72	J(+), UJ ND
Lead	16	4.1	20	60	J(+), UJ ND

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS:

1. If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.

2. If any analyte does not meet the %R criteria follow the actions stated below:

	PERCENT RECOVERY		
	<30%	30%-74%	>125%
Positive Sample Results	J	J	J
Non-detected Results	R	UJ	A

3. Frequency Criteria

A. Was a matrix spike prepared at the required frequency? (Yes) or No

B. Was a post digestion spike analyzed for elements that did not meet required criteria for matrix spike recovery? (Yes) or No

Separate worksheet should be used for each matrix spike pair.

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VII. LABORATORY DUPLICATES

List the concentrations of any analyte not meeting the criteria for duplicate precision. For soil duplicates, calculate the CRDL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate precision by circling either the RPD or CRDL for each element.

MATRIX: Aqueous

Element	CRDL water ug/L	CRDL soil mg/kg	Sample # MAE236	Duplicate# MAE236	RPD	Action
Aluminum	200		60.5	54.7		
Antimony	60		37 u	37 u	NC	
Arsenic	10		3.0u	3.0u	NC	
Barium	200		18.8	18.3		
Beryllium	5		2.0u	2.0u	NC	
Cadmium	5		5.0u	5.0u	NC	
Calcium	5000		33400	33500	0.3	
Chromium	10		4.0u	4.0u	NC	
Cobalt	50		4.3u	4.3u	NC	
Copper	25		24.2	62.9		J(+)
Iron	100		142	146	2.8	
Lead	5		5.2	3.7		
Magnesium	5000		1980	2000		
Manganese	15		22.2	23.2		
Mercury	0.2		0.2u	0.2u	NC	
Nickel	40		10 u	10 u	NC	
Potassium	5000		1810	1740		
Selenium	5		1.0u	1.0	NC	
Silver	10		4.0u	4.0u	NC	
Sodium	5000		18900	18500		
Thallium	10		2.0u	2.0u	NC	
Vanadium	50		4.0u	4.0u	NC	
Zinc	20		59.6	41.3		
Cyanide	10					

Laboratory Duplicate Actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >20% for waters and >35% for soils.

2. If sample results are less than 5x the CRDL, estimate (J) positive results and (UJ) nondetected results for elements whose absolute difference is >CRDL, (2xCRDL for soils). If both samples are nondetected, the RPD is not calculated (NC).

VIII. FIELD DUPLICATES - *not applicable*

List the concentrations of all analytes in the field duplicate pair. For soil duplicates, calculate the CRDL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evalute the precision by circling either the RPD or CRDL for each element.

MATRIX:_____

Element	CRDL water ug/L	CRDL soil mg/kg	Sample #	Duplicate#	RPD	Action
Aluminum	200					
Antimony	60					
Arsenic	10					
Barium	200					
Beryllium	5					
Cadmium	5					
Calcium	5000					
Chromium	10					
Cobalt	50					
Copper	25					
Iron	100					
Lead	5					
Magnesium	5000					
Manganese	15					
Mercury	0.2					
Nickel	40					
Potassium	5000					
Selenium	5					
Silver	10					
Sodium	5000					
Thallium	10					
Vanadium	50					
Zinc	20					
Cyanide	10					

Field Duplicate Actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >30% for waters and >50% for soils.

2. If sample results are less than 5x the CRDL, estimate (J) positive results and (UJ) nondetected results for elements whose absolute difference is >2xCRDL, (4xCRDL for soils). If both samples are non-detected, the RPD is not calculated (NC).

IX. LABORATORY CONTROL SAMPLE - *all acceptable*

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. Aqueous LCS

List any LCS recoveries not within the 80-120% criteria and the samples affected.

DATE	ELEMENT	%R	ACTION	SAMPLES AFFECTED

2. Solid LCS

List any analytes that were not within the control windows set by the EPA for the solid LCS sample. The 80-120% criteria is not used to evaluate solid LCS results.

ELEMENT	LCS CONC.	CONTROL WINDOWS	ACTION	SAMPLES AFFECTED

ACTIONS:

AQUEOUS LCS	Percent Recovery <50%	51-79%	>120%
Positive Results	R	J	J
Non-detected Results	R	UJ	A

SOLID LCS	<EPA Control Windows	>EPA Control Windows
Positive Results	J	J
Non-detected Results	UJ	A

3. Frequency Criteria

A. Was an LCS analyzed for every matrix, every digestion batch, and every 20 samples? Yes or No

REGION I
Data Review Worksheets

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X A. FURNACE ATOMIC ABSORPTION ANALYSIS

1. Duplicate Precision

--✓-- Duplicate injections and one-point analytical spikes were performed for all samples: duplicate injections agreed within ± 20%.

-------- Duplicate injections and/or spikes were not performed for the following samples/elements: _____

-------- Duplicate injections did not agree within ± 20% for samples/elements: _____

2. Post Digestion Spike Recoveries

-------- Spike recoveries met the 85-115% recovery criteria for all samples.

--✓-- Spike recoveries did not meet the 85-115% criteria but did not require MSA for the following samples/elements:
Se - MAE237, MAE247 Pb MAE246
Tl - all samples except MAE240, MAE247

--✓-- MSA was used to quantitate analytical results when contractually required.
✓ Correlation coefficients ≥0.995, accept results.
___ Correlation coefficients <0.995 for sample numbers/elements: _____

-------- Method of Standard Addition (MSA) was not performed as required for samples/elements: _____

ACTIONS:

1. Estimate (J) positive results if duplicate injections are outside ± 20 % RSD or CV.

2. If the sample absorbance is <50% of post digestion spike absorbance the following actions should be applied:

	PERCENT RECOVERY		
	<10%	11%-84%	>115%
Positive Sample Results	J or R	J	J
Non-detected Results	R	UJ	A

3. Estimate (J) sample results if MSA was required and not performed.

4. Estimate (J) sample results if correlation coefficient was <0.995.

✓ 000162

XI. INDUCTIVELY COUPLED PLASMA (ICP) SERIAL DILUTION ANALYSIS

_____ Serial Dilutions were performed for each matrix and results of the diluted sample analysis agreed within ten percent of the original undiluted analysis.

_____ Serial Dilutions were not performed for the following: _____

✓ Serial Dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the IDL before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

MATRIX: Aqueous

ELEMENT	IDL	50xIDL	SAMPLE RESULT	SERIAL DILUTION	%D	ACTION
Aluminum	25	1250	60.5	125 u	NR	
Barium	2.0	100	18.8	10 u	NR	
Beryllium						
Cadmium						
Calcium	81	4050	33400	41000	22.8	J(+)
Chromium						
Cobalt						
Copper	10	500	24.2		NR	
Iron	14	700	102000	97000	4.9	
Lead						
Magnesium	81	4050	1980			
Manganese	6	300	22		NR	
Nickel						
Potassium	95	4750	1810		NR	
Silver						
Sodium	1360	68000	18900		NR	
Vanadium						
Zinc	3.0	150	39.6		NR	

Actions apply to all samples of the same matrix.

ACTIONS:

1. Estimate (J) positive results if %D >15.

XII. DETECTION LIMIT RESULTS

Instrument Detection Limits

✓ Instrument Detection Limit results were present and found to be less than the Contract Required Detection Limits.

_____ IDLs were not included in the data package on Form XI.

_____ IDLs were present, but the criteria was not met for the following elements:_____

2. Reporting Requirements

_____ Were sample results on Form I reported down to the IDL not the CRDL for all analytes? (Yes) or No

_____ Were sample results that were analyzed by ICP for Se, Tl, As, or Pb at least 5x IDL. (Yes) or No

_____ Were sample weights, volumes, and dilutions taken into account when reporting detection limits on Form I. (Yes) or No

If No,

The reported results may be inaccurate. Make the necessary changes c the data summary tables and request that the laboratory resubmit the corrected data.

XIII. SAMPLE QUANTITATION

✓ Sample results fall within the linear range for ICP and within the calibrated range for all other parameters.

_____ Sample results were beyond the linear range/ calibration range of the instrument for the following samples/elements:

_____

In the space below, please show a minimum of one sample calculation per method:

ICP - *Magnesium - Result from instrument - 1.984 mg/L*

$$984 \frac{mg}{L} \times \frac{1000\ ug}{1\ mg} = 1980\ ug/L$$

FURNACE - *Instrument printout in ug/L. Prep factor = 1*

MERCURY - *Instrument printout in ug/L. Prep factor = 1*

CYANIDE

For soil samples, the following equation may be necessary to convert raw data values (usually reported in ug/L) to actual sample concentrations (mg/kg):

The lab is required to use 1 gram sample (wet weight) to 200 ml.

Wet weight concentration =

$$\text{digest conc. in } \frac{ug}{L} \times \frac{200ml}{1\ gm} \times \frac{1L}{1000\ ml} \times \frac{1000gm}{1kg} \times \frac{1mg}{1000ug} = \frac{mg}{kg}$$

In addition the sample results are converted to dry weight using the percent solids calculations:

$$\frac{\text{Wet weight conc.}}{\%\text{solids}} \times 100 = \text{final concentration, dry weight (mg/kg)}$$

APPENDIX D

Figures

FIGURE 1

Region I Data Validation

Roles and Responsibilities

FIGURE 1

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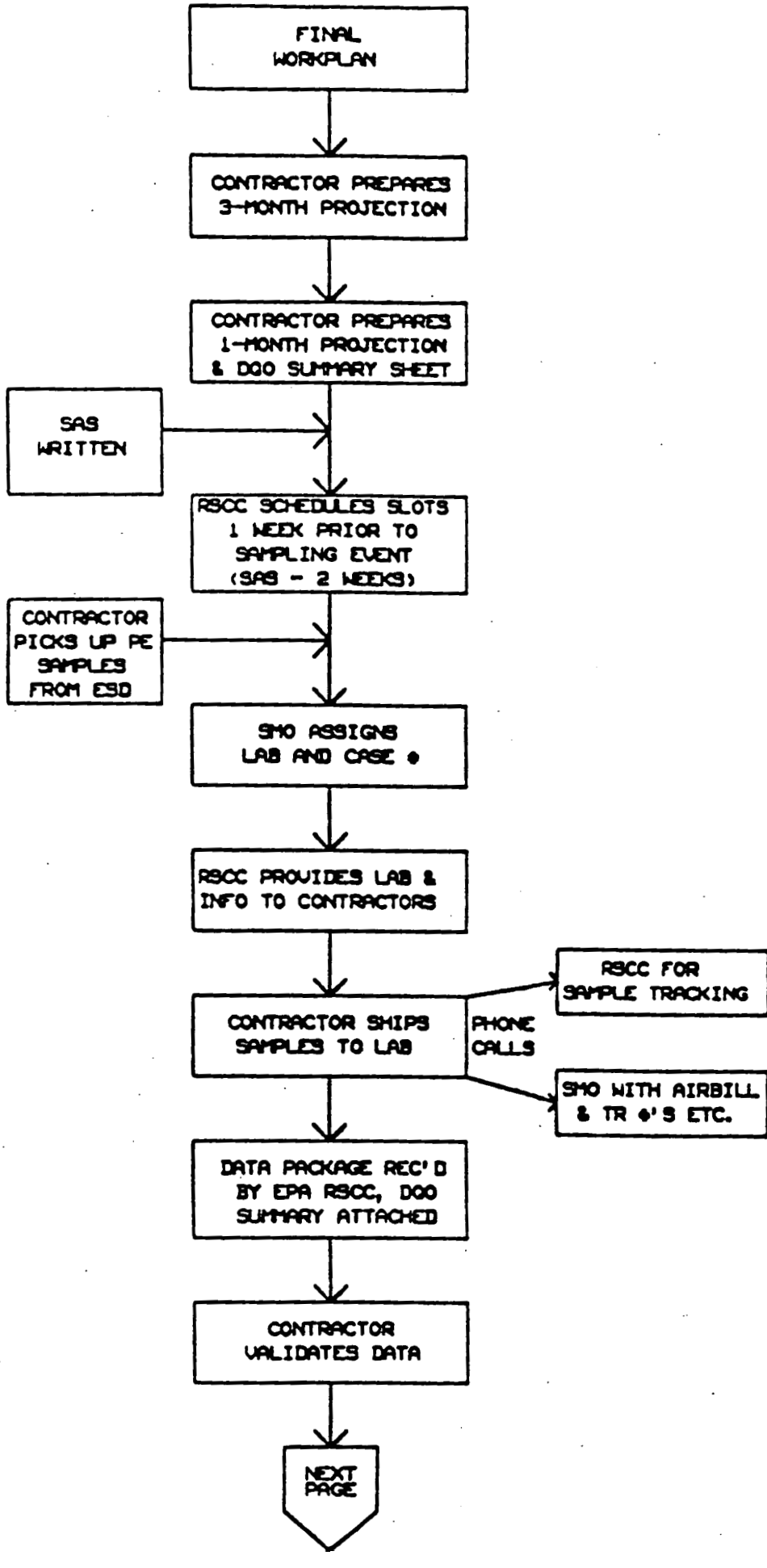
DATA VALIDATION - ROLES AND RESPONSIBILITIES



000168

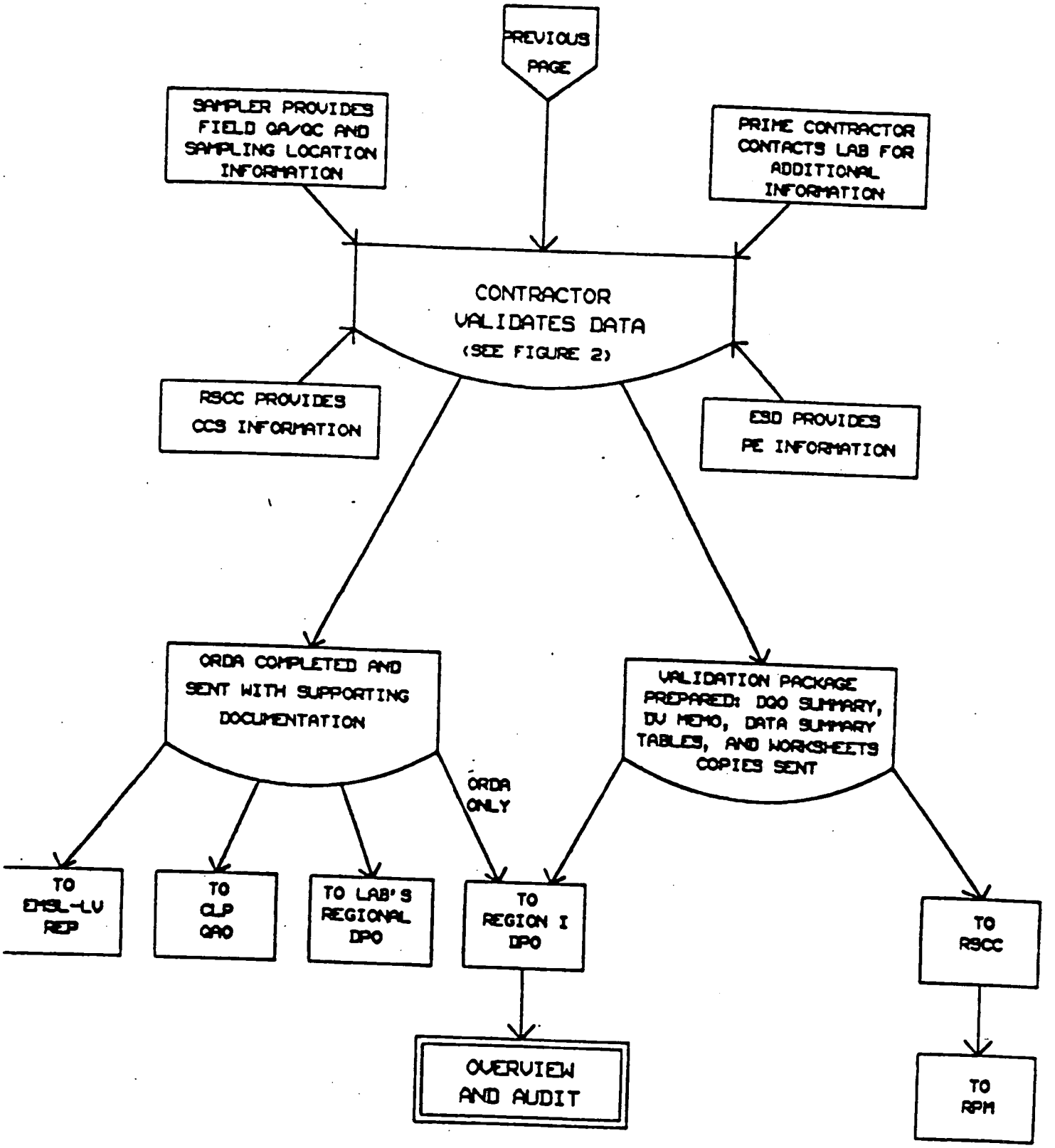
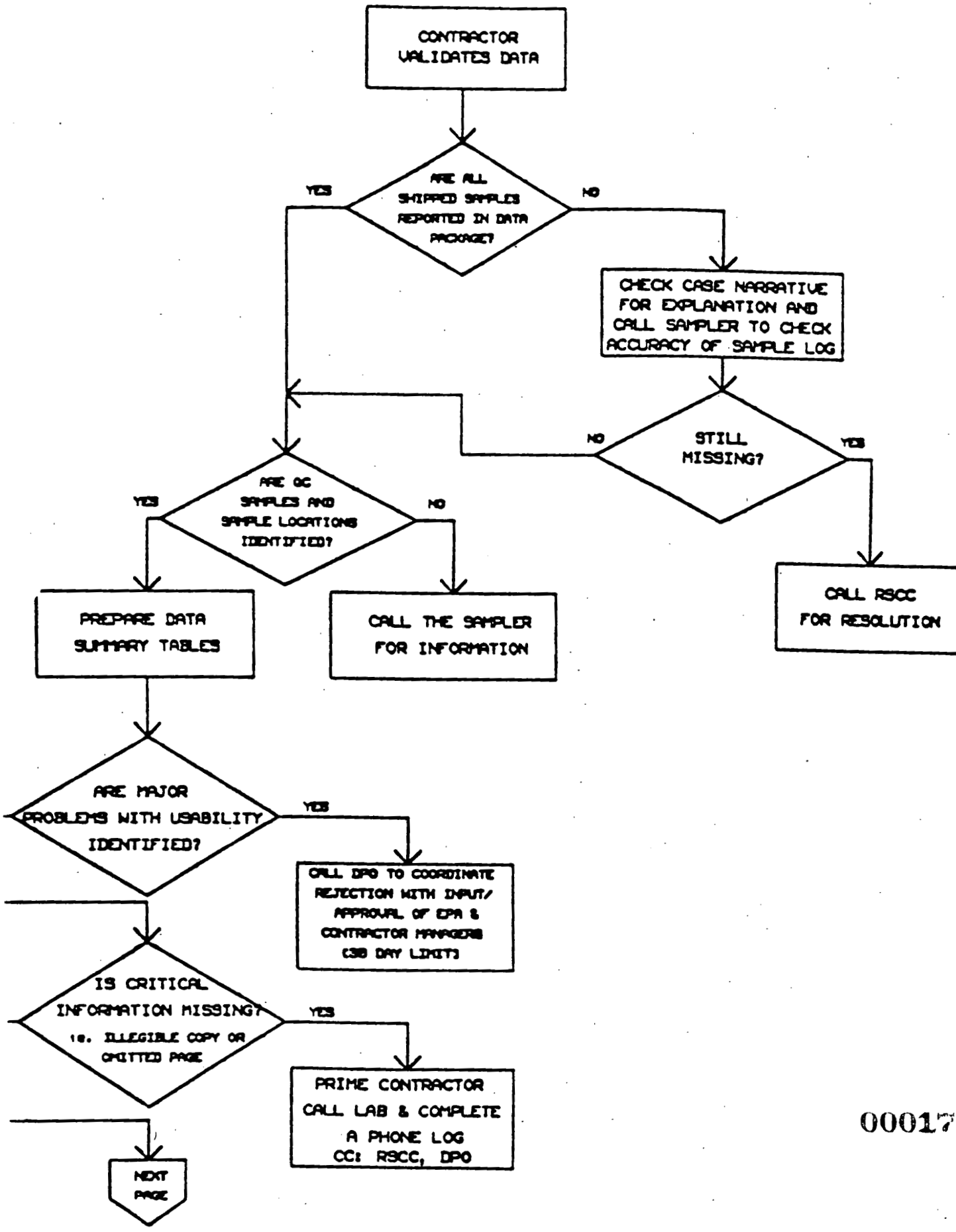

PREVIOUS PAGE
SAMPLER PROVIDES FIELD QA/QC AND SAMPLING LOCATION INFORMATION
PRIME CONTRACTOR CONTACTS LAB FOR ADDITIONAL INFORMATION
CONTRACTOR VALIDATES DATA (SEE FIGURE 2)
RSCC PROVIDES CCS INFORMATION
ESD PROVIDES PE INFORMATION
ORDA COMPLETED AND SENT WITH SUPPORTING DOCUMENTATION
VALIDATION PACKAGE PREPARED: DQO SUMMARY, DV MEMO, DATA SUMMARY TABLES, AND WORKSHEETS COPIES SENT
ORDA ONLY
TO EMSL-LV REP
TO CLP QAO
TO LAB'S REGIONAL DPO
TO REGION I DPO
TO RSCC
OVERVIEW AND AUDIT
TO RPM

FIGURE 2

Overview of the Data Review Process

FIGURE 2

OVERVIEW OF THE DATA REVIEW PROCESS



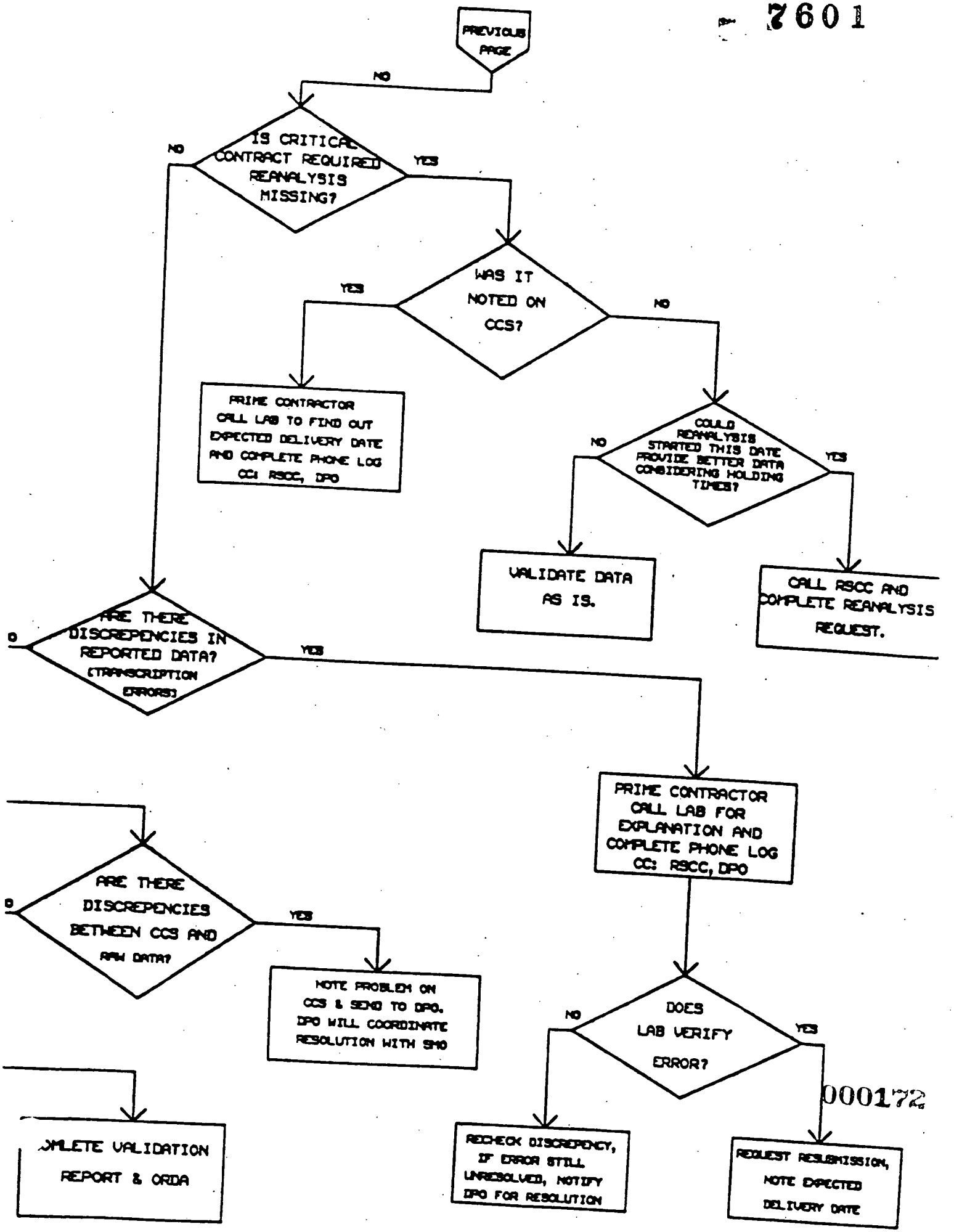

PREVIOUS PAGE
NO
IS CRITICAL CONTRACT REQUIRED REANALYSIS MISSING?
NO
YES
WAS IT NOTED ON CCS?
YES
NO
PRIME CONTRACTOR CALL LAB TO FIND OUT EXPECTED DELIVERY DATE AND COMPLETE PHONE LOG CC: RSCC, DPO
COULD REANALYSIS STARTED THIS DATE PROVIDE BETTER DATA CONSIDERING HOLDING TIMES?
NO
YES
VALIDATE DATA AS IS.
CALL RSCC AND COMPLETE REANALYSIS REQUEST.
ARE THERE DISCREPENCIES IN REPORTED DATA? (TRANSCRIPTION ERRORS)
YES
PRIME CONTRACTOR CALL LAB FOR EXPLANATION AND COMPLETE PHONE LOG CC: RSCC, DPO
ARE THERE DISCREPENCIES BETWEEN CCS AND RAW DATA?
YES
NOTE PROBLEM ON CCS & SEND TO DPO. DPO WILL COORDINATE RESOLUTION WITH SMO
DOES LAB VERIFY ERROR?
NO
YES
RECHECK DISCREPENCY, IF ERROR STILL UNRESOLVED, NOTIFY DPO FOR RESOLUTION
REQUEST RESUBMISSION, NOTE EXPECTED DELIVERY DATE
OMLETE VALIDATION REPORT & ORDA

FIGURE 3

Inorganic Traffic Report

FIG : 3A

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report
(For CLP Use Only)

Case Number

SAS No. (If applicable)

1. Type of Activity (Check one)
   - ENF
   - ER
   - ESI
   - NPLD
   - O&M
   - PA
   - RA
   - RD
   - RIFS
   - SI
   - ST
   - STPA
   - STSI
   - Other *(Specify)*

2. Region Number

Sampling Co.

Sampler (Name)

3. Ship To:

4. Date Shipped

Airbill Number

Carrier

Non-Superfund Program

Site Name

City, State

Site Spill ID

Double volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

5. Sample Description *(Enter in Column A)*
   1. Surface Water
   2. Ground Water
   3. Leachate
   4. Rinsate
   5. Soil/Sediment
   6. Oil (SAS)
   7. Waste (SAS)
   8. Other (SAS) *(Specify)*

CLP Sample Number *(From labels)*	(A) Sample Description *(From box 9)*	(B) Concentration L=low M=med H=high	(C) RAS Analysis		(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding Organic Sample Number
			Total Metals	Cyanide				

EPA Form 9150-1 (8-88) Replaces EPA Form 2075-8, which may be used.

Green - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

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EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report
(For CLP Use Only)

Case Number: 10101

5. (If applicable)

1. Type of Activity (Check one): ENF, ER, ESI, NPLD, O&M, PA, RA, RD, RIFS, SI, ST, STPA, STSI, Other (Specify)

Non-Superfund Program

Site Name: Drum Site

City, State: Green City, OR

Site Spill ID: 05

2. Region Number: X

Sampling Co.: Acme Co.

Sampler (Name): Joan Sampler

3. Ship To: Analytical Lab, 100 Center Ave, Anytown, CA 94568, Attn A. Metal

4. Date Shipped: 11-4-88

Airbill Number: 0987654321

Carrier: Fed Ex

Double volume required for matrix spike/duplicate aqueous sample.

Ship medium and high concentration samples in paint cans.

See reverse for additional instructions.

6. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

CLP Sample Number (From labels)	(A) Sample Description (From box 6)	(B) Concentration L=low M=med H=high	(C) RAS Analysis: Total Metals	(C) RAS Analysis: Cyanide	(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding Organic Sample Number
MJZ 900	1	L	X	X		LOC-1	11-4/0700	JA 321
MJZ 901	1	L	X	X		LOC-2	11-4/0730	JA 322
MJZ 902	1	L	X	X		LOC-3	11-4/0800	JA 323
MJZ 903	1	L	X	X		LOC-4	11-4/0830	JA 324
MJZ 904	1	L	X	X		LOC-5	11-4/0900	JA 325
MJZ 905	1	L	X	X		LOC-6	11-4/0930	JA 326
MJZ 906	1	L	X	X		LOC-7	11-4/0945	JA 327
MJZ 907	1	L	X	X		LOC-8	11-4/1000	JA 328
MJZ 908	1	L	X	X		LOC-9	11-4/1030	JA 329
MJZ 909	1	L	X	X		LOC-10	11-4/1100	JA 330
MJZ 910	1	L	X	X		LOC-11	11-4/1130	JA 331
MJZ 911	1	L	X	X		LOC-12	11-4/1200	JA 332
MJZ 912	1	L	X	X		LOC-13	11-4/1215	JA 333
MJZ 913	1	L	X	X		LOC-14	11-4/1245	JA 334
MJZ 914	1	L	X	X		LOC-15	11-4/1300	JA 335
MJZ 915	1	L	X	X		LOC-16	11-4/1330	JA 336
MJZ 916	1	L	X	X		LOC-17	11-4/1400	JA 337
MJZ 917	1	L	X	X		LOC-18	11-4/1430	JA 338
MJZ 918	1	L	X	X		LOC-19	11-4/1500	JA 339
MJZ 920	1	L	X	X	MS/dup	LOC-20	11-4/1530	JA 340

EPA Form 9110-1 (9-88) Replaces EPA Form 3075-6, which may be used. Green - SMO Copy Pink - Region Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

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FIGURE 4

Chain of Custody Form

PROJ. NO. | PROJECT NAME

Boston, Massachusetts 02203

SAMPLERS: *(Signature)*

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CON-TAINERS							REMARKS

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field

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1-2705

FIGURE 5

Contract Laboratory Program

Telephone Record Log

In Reference to Case No(s):

FIGURE 5

Contract Laboratory Program
REGIONAL/LABORATORY COMMUNICATION SYSTEM
Telephone Record Log

Date of Call: _____

Laboratory Name: _____
Lab Contact: _____

Region: _____
Regional Contact: _____

Call Initiated By: ____ Laboratory ____ Region

In reference to data for the following sample number(s):

_____
_____
_____

Summary of Questions/Issues Discussed:

_____
_____
_____
_____
_____
_____
_____

Summary of Resolution:

_____
_____
_____
_____
_____
_____
_____
_____

_____ _____
Signature Date

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy

FIGURE 6

DQO Summary Form

FIGURE 6
DQO SUMMARY FORM

1. SITE

NAME _____
LOCATION _____
NUMBER _____

EPA REGION ____
PHASE _____
RI 1 RI 2 RI 3 ERA FS RD RA
(CIRCLE ONE)

2. MEDIA *(CIRCLE ONE)*	SOIL	GW	SW/SED	AIR	BIO	OTHER ____

3. USE *(CIRCLE ALL THAT APPLY)*	SITE CHARAC. (H&S)	RISK ASSESS.	EVAL. ALTS.	ENG'G DESIGN	PRP DETER.	MONITORING REMEDIAL ACTION	OTHER ____

4. OBJECTIVE _____

5. SITE INFORMATION

AREA _____ DEPTH TO GROUND WATER _____
GROUND WATER USE _____
SOIL TYPES _____
SENSITIVE RECEPTORS _____

6. DATA TYPES *(CIRCLE APPROPRIATE DATA TYPES)*

A. ANALYTICAL DATA

pH	PESTICIDES	TOX
CONDUCTIVITY	PCB	TOC
VOA	METALS	BTX
ABN	CYANIDE	COD
TCLP	____	____

B. PHYSICAL DATA

PERMEABILITY	HYDRAULIC HEAD
POROSITY	PENETRATION TEST
GRAIN SIZE	HARDNESS
BULK DENSITY	____
____	____

7. SAMPLING METHOD *(CIRCLE METHOD(S) TO BE USED)*

ENVIRONMENTAL	BIASED	GRAB	NON- INTRUSIVE	PHASED
SOURCE	GRID	COMPOSITE	INTRUSIVE	____

8. ANALYTICAL LEVELS *(INDICATE LEVEL(S) AND EQUIPMENT & METHODS)*

LEVEL 1 FIELD SCREENING - EQUIPMENT _____
LEVEL 2 FIELD ANALYSIS - EQUIPMENT _____
LEVEL 3 NON-CLP LABORATORY - METHODS _____
LEVEL 4 CLP/RAS - METHODS _____
LEVEL 5 NON STANDARD _____

9. SAMPLING PROCEDURES

BACKGROUND - 2 PER EVENT OR _____
CRITICAL (LIST) _____
PROCEDURES _____

10. QUALITY CONTROL SAMPLES *(CONFIRM OR SET STANDARD)*

A. FIELD
COLLOCATED - 5% OR ____
REPLICATE - 5% OR ____
FIELD BLANK - 5% OR ____
TRIP BLANK - 1 PER DAY OR ____

B. LABORATORY
REAGENT BLANK - 1 PER ANALYSIS BATCH OR ____
REPLICATE - 1 PER ANALYSIS BATCH OR ____
MATRIX SPIKE - 1 PER ANALYSIS BATCH OR ____
OTHER _____

11. BUDGET REQUIREMENTS

BUDGET _____ SCHEDULE _____
STAFF _____

CONTRACTOR _____ PRIME CONTRACTOR _____
SITE MANAGER _____ DATE _____

FOR DETAILS SEE SAMPLING & ANALYSIS PLAN

COM SF DQO 1.002

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FIGURE 7

Data Summary Tables

SITE:
CASE: SDG:
LABORATORY:

INORGANIC AQUEOUS ANALYSIS
ug/L

SAMPLE NUMBER:
SAMPLE LOCATION:
LABORATORY NUMBER:

INORGANIC ELEMENTS	INSTRUMENT DETECTION LIMITS (ug/l, ppb)	CONTRACT DETECTION LIMITS (ug/l,ppb)
ALUMINUM	P	200
ANTIMONY	P	60
ARSENIC	F	10
BARIUM	P	200
BERYLLIUM	P	5
CADMIUM	P	5
CALCIUM	P	5000
CHROMIUM	P	10
COBALT	P	50
COPPER	P	25
IRON	P	100
LEAD	P	5
MAGNESIUM	P	5000
MANGANESE	P	15
MERCURY	CV	0.2
NICKEL	P	40
POTASSIUM	P	5000
SELENIUM	F	5
SILVER	P	10
SODIUM	P	5000
THALLIUM	F	10
VANADIUM	P	50
ZINC	P	20
CYANIDE	C	10

ANALYTICAL METHOD
F - FURNACE
P - ICP/FLAME AA
CV - COLD VAPOR
C - COLORIMETRIC

NOTE: J - QUANTITATION IS APPROXIMATE DUE TO LIMITATIONS IDENTIFIED IN THE QUALITY CONTROL REVIEW (DATA REVIEW).
R - VALUE IS REJECTED.
-- VALUE IS NON-DETECTED

FIGURE 7

FIGURE 7

SITE:
CASE: SDG:
LABORATORY:

SAMPLE NUMBER:
SAMPLE LOCATION:
LABORATORY NUMBER:

INORGANIC SOIL ANALYSIS
mg/kg

To calculate the sample detection limits, divide the IDL by % solids.

INORGANIC ELEMENTS		INSTRUMENT DETECTION LIMITS (mg/kg, ppm)	CONTRACT DETECTION LIMITS (mg/kg, ppm)
ALUMINUM	P		40
ANTIMONY	P		12
ARSENIC	F		2
BARIUM	P		40
BERYLLIUM	P		1
CADMIUM	P		1
CALCIUM	P		1000
CHROMIUM	P		2
COBALT	P		10
COPPER	P		5
IRON	P		20
LEAD	P		1
MAGNESIUM	P		1000
MANGANESE	P		3
MERCURY	CV		0.1
NICKEL	P		8
POTASSIUM	P		1000
SELENIUM	F		1
SILVER	P		2
SODIUM	P		1000
THALLIUM	F		2
VANADIUM	P		10
ZINC	P		4
CYANIDE	C		
% SOLIDS			

ANALYTICAL METHOD
F - FURNACE
P - ICP/FLAME AA
CV - COLD VAPOR
C - COLORIMETRIC

NOTE:
J - QUANTITATION IS APPROXIMATE DUE TO LIMITATIONS IDENTIFIED IN THE QUALITY CONTROL REVIEW (DATA REVIEW).
R - VALUE IS REJECTED.
-- VALUE IS NON-DETECTED

FIGURE 8

Standard Worksheets

REGION I
Data Review Worksheets

Site Name_____
Reference Number_____

REGION I REVIEW OF INORGANIC CONTRACT LABORATORY DATA PACKAGE

The hardcopied (laboratory name)_____ data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data review included:

Case No. _____ SAS No._____ Sampling Date(s) _____
SDG. No. _____ Matrix _____ Shipping Date(s) _____
No. of Samples _____ Date Rec'd by Lab _____

Traffic Report Nos: _____
_____
Trip Blank No.: _____
Equipment Blank No.:_____
Field Dup Nos: _____

SOW No. _____ requires that specific analytical work be done and that associated reports be provided by the laboratory to the Regions, EMSL-LV, and SMO. The general criteria used to determine the performance were based on an examination of:

- -Data Completeness
- -Holding Times
- -Calibrations
- -Blanks
- -ICP Interference Check Results
- -Matrix Spike Recoveries
- -Laboratory Duplicates
- -Field Duplicates
- -Lab Control Sample Results
- -Furnace AA Results
- -ICP Serial Dilution Results
- -Detection Limit Results
- -Sample Quantitation

Overall Comments:_____
_____
_____
_____.

Definitions and Qualifiers:

A - Acceptable data.
J - Approximate data due to quality control criteria.
R - Reject data due to quality control criteria.
U - Analyte not detected.

Reviewer: _____ Date: _____

REGION I
Data Review Worksheets

7601

I. DATA COMPLETENESS

MISSING INFORMATION	DATE LAB CONTACTED	DATE REC'D

II. HOLDING TIMES Complete table for all samples and circle the analysis date for samples not within criteria.

SAMPLE ID	DATE SAMPLED	HG DATE ANALYSIS	CYANIDE DATE ANALYSIS	OTHERS DATE ANALYSIS	pH	ACTION

METALS - 180 DAYS FROM SAMPLE COLLECTION
MERCURY - 28 DAYS FROM SAMPLE COLLECTION
CYANIDE - 14 DAYS FROM SAMPLE COLLECTION

ACTION:

1. If holding times are exceeded all positive results are estimated (J) and non-detects are estimated (UJ).

2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable (R).

III A. INSTRUMENT CALIBRATION (Section 1)

Recovery Criteria

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

DATE	ICV/CCV#	ANALYTE	%R	ACTION	SAMPLES AFFECTED

ACTIONS:

If any analyte does not meet the %R criteria follow the actions stated below:

For Positive Results:

	Accept	Estimate (J)	Reject (R)
Metals	90-110%R	75-89%R, 111-125%R	<75%R, >125%R
Mercury	80-120%R	65-79%R, 121-135%R	<65%R, >135%R
Cyanide	85-115%R	70-84%R, 116-130%R	<70%R, >130%R

For Non-detected Results:

	Accept	Estimate (UJ)	Reject (R)
Metals	90-125%R	75-89%R	<75%R, >125%R
Mercury	80-135%R	65-79%R	<65%R, >135%R
Cyanide	85-130%R	70-84%R	<70%R, >130%R

III B. INSTRUMENT CALIBRATION (Section 2)

2. Analytical Sequence

A. Did the laboratory use the proper number of standards for calibration as described in the SOW? Yes or No

B. Were calibrations performed at the beginning of each analysis? Yes or No

C. Were calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent or every two hours during analysis, whichever is more frequent? Yes or No

D. Were the correlation coefficients for the calibration curves for AA, Hg, and CN $\geq$ 0.995? Yes or No

E. Was a standard at 2xCRDL analyzed for all ICP analyses? Yes or No

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

_____

_____

_____

_____

IV A. BLANK ANALYSIS RESULTS (Sections 1-3)

List the blank contamination in Sections 1 & 2 below. A separate worksheet should be used for soil and water blanks.

1. Laboratory Blanks MATRIX:_____

DATE	ICB/CCB#	PREP BL	ANALYTE	CONC./UNITS

2. Equipment/Trip Blanks

DATE	EQUIP BL#	ANALYTE	CONC./UNITS

3. Frequency Requirements

A. Was a preparation blank analyzed for each matrix, for every 20 samples and for each digestion batch? Yes or No

B. Was a calibration blank run every 10 samples or every 2 hours whichever is more frequent? Yes or No

If No,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below, and list the samples affected.

_____

_____

IV B. BLANK ANALYSIS RESULTS (Section 4)

4. Blank Actions

The Action Levels for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a U.

2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

MATRIX:_____ MATRIX:_____

ELEMENT	MAX. CONC./ UNITS	AL/ UNITS	ELEMENT	MAX. CONC./ UNITS	AL/ UNITS

NOTE: Blanks analyzed during a soil case must be converted to mg/kg in order to compare them with the sample results.

$$\text{Conc. in ug/L} \times \frac{\text{Volume diluted to (200ml)}}{\text{Weight digested (1gram)}} \times \frac{1\text{L}}{1000\text{ml}} \times \frac{1000\text{gm}}{1\text{kg}} \times \frac{1\text{mg}}{1000\text{ug}} = \text{mg/kg}$$

Multiplying this result by 5 to arrive at the action level gives a final result in mg/kg which can then be compared to sample results.

V A. ICP INTERFERENCE CHECK SAMPLE (Sections 1 & 2)

Recovery Criteria

List any elements in the ICS AB solution which did not meet the criteria for %R.

DATE	ELEMENT	%R	ACTION	SAMPLES AFFECTED

ACTIONS:

If an element does not meet the %R criteria , follow the actions stated low:

	PERCENT RECOVERY		
	<50%	50-79%	>120%
Positive Sample Results	R	J	J
Non-detected Sample Results	R	UJ	A

2. Frequency Requirements

Were Interference QC samples run at the beginning and end of each sample analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent? Yes or No

If no,

The data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

_____

_____

_____

V B. ICP INTERFERENCE CHECK SAMPLE (Section 3)

Report the concentration of any elements detected in the ICS A solution > 2xIDL that should not be present.

ELEMENT	CONC. DETECTED IN THE ICS	CONC. OF INTERFERENTS IN THE ICS			
		AL	CA	FE	MG
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____
_____	_____	_____	_____	_____	_____

Estimate the concentration produced by the interfering element in all affected samples. See guidelines for examples. List the samples affected by interferences below:

SAMPLE AFFECTED	ELEMENT AFFECTED	SAMPLE CONC. (ug/L)	SAMPLE INTERFERENT CONC.				ESTIMATED INTERF. (ug/L)
			AL	CA	FE	MG	
_____	_____	_____	___	___	___	___	_____
_____	_____	_____	___	___	___	___	_____
_____	_____	_____	___	___	___	___	_____
_____	_____	_____	___	___	___	___	_____

ACTIONS:

1. In general, the sample data can be accepted without qualification if the sample concentrations of Al, Ca, Fe, and Mg are less than 50% of their respective levels in the ICS solution.

2. Estimate (J) positive results for affected elements for samples with levels of interferents 50% or more of that in the ICS solution.

3. Reject (R) positive results if the reported concentration is due entirely to the interfering element.

4. Estimate (UJ) non-detected results for which false negatives are suspect.

Give explanations for any actions taken below:

_____

_____

_____

VI. MATRIX SPIKE

TR # _____ MATRIX: _____

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

S - amount of spike added
SSR - spikes sample result
SR - sample result

Analyte	SSR	SR	S	%R	Action

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS:

1. If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.

2. If any analyte does not meet the %R criteria follow the actions stated below:

	PERCENT RECOVERY		
	<30%	30%-74%	>125%
Positive Sample Results	J	J	J
Non-detected Results	R	UJ	A

2. Frequency Criteria

A. Was a matrix spike prepared at the required frequency? Yes or No

B. Was a post digestion spike analyzed for elements that did not meet required criteria for matrix spike recovery? Yes or No

1 separate worksheet should be used for each matrix spike pair.

VII. LABORATORY DUPLICATES

List the concentrations of any analyte not meeting the criteria for duplicate precision. For soil duplicates, calculate the CRDL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evaluate precision by circling either the RPD or CRDL for each element.

MATRIX:_____

Element	CRDL water ug/L	CRDL soil mg/kg	Sample # _____	Duplicate# _____	RPD	Action
Aluminum	200					
Antimony	60					
Arsenic	10					
Barium	200					
Beryllium	5					
Cadmium	5					
Calcium	5000					
Chromium	10					
Cobalt	50					
Copper	25					
Iron	100					
Lead	5					
Magnesium	5000					
Manganese	15					
Mercury	0.2					
Nickel	40					
Potassium	5000					
Selenium	5					
Silver	10					
Sodium	5000					
Thallium	10					
Vanadium	50					
Zinc	20					
Cyanide	10					

Laboratory Duplicate Actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >20% for waters and >35% for soils.

2. If sample results are less than 5x the CRDL, estimate (J) positive results for elements whose absolute difference is >CRDL, (2xCRDL for soils). If both samples are non-detected, the RPD is not calculated (NC).

VIII. FIELD DUPLICATES

List the concentrations of all analytes in the field duplicate pair. For soil duplicates, calculate the CRDL in mg/kg using the sample weight, volume and percent solids data for the sample. Indicate what criteria was used to evalute the precision by circling either the RPD or CRDL for each element.

MATRIX:_____

Element	CRDL water ug/L	CRDL soil mg/kg	Sample #	Duplicate#	RPD	Action
Aluminum	200					
Antimony	60					
Arsenic	10					
Barium	200					
Beryllium	5					
Cadmium	5					
Calcium	5000					
Chromium	10					
Cobalt	50					
Copper	25					
Iron	100					
Lead	5					
Magnesium	5000					
Manganese	15					
Mercury	0.2					
Nickel	40					
Potassium	5000					
Selenium	5					
Silver	10					
Sodium	5000					
Thallium	10					
Vanadium	50					
Zinc	20					
Cyanide	10					

Field Duplicate Actions should be applied to all other samples of the same matrix type.

ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >30% for waters and >50% for soils.

2. If sample results are less than 5x the CRDL, estimate (J) positive results and (UJ) nondetected results for elements whose absolute difference is >2xCRDL, (4xCRDL for soils). If both samples are non-detected, the RPD is not calculated (NC).

IX. LABORATORY CONTROL SAMPLE

Aqueous LCS

List any LCS recoveries not within the 80-120% criteria and the samples affected.

DATE	ELEMENT	%R	ACTION	SAMPLES AFFECTED

2. Solid LCS

List any analytes that were not within the control windows set by the EPA for the solid LCS sample. The 80-120% criteria is not used to evaluate solid LCS results.

ELEMENT	LCS CONC.	CONTROL WINDOWS	ACTION	SAMPLES AFFECTED

ACTIONS:

AQUEOUS LCS	Percent Recovery <50%	51-79%	>120%
Positive Results	R	J	J
Non-detected Results	R	UJ	A

SOLID LCS	<EPA Control Windows	>EPA Control Windows
Positive Results	J	J
Non-detected Results	UJ	A

3. Frequency Criteria

A. Was an LCS analyzed for every matrix, every digestion batch, and every 20 samples? Yes or No

X A. FURNACE ATOMIC ABSORPTION ANALYSIS

Duplicate Precision

-------- Duplicate injections and one-point analytical spikes were performed for all samples: duplicate injections agreed within ± 20%.

-------- Duplicate injections and/or spikes were not performed for the following samples/elements:_____

_____

-------- Duplicate injections did not agree within ± 20% for samples/elements:_____

_____

2. Post Digestion Spike Recoveries

-------- Spike recoveries met the 85-115% recovery criteria for all samples.

-------- Spike recoveries did not meet the 85-115% criteria but did not require MSA for the following samples/elements:

_____

_____

-------- MSA was used to quantitate analytical results when contractually required.

_____ Correlation coefficients ≥0.995, accept results.

_____ Correlation coefficients <0.995 for sample numbers/elements:_____

-------- Method of Standard Addition (MSA) was not performed as required for samples/elements:_____

ACTIONS:

1. Estimate (J) positive results if duplicate injections are outside ± 20 % RSD or CV.

2. If the sample absorbance is <50% of post digestion spike absorbance the following actions should be applied:

	PERCENT RECOVERY		
	<10%	11%-84%	>115%
Positive Sample Results	J or R	J	J
Non-detected Results	R	UJ	A

3. Estimate (J) sample results if MSA was required and not performed.

4. Estimate (J) sample results if correlation coefficient was <0.995.

XI. INDUCTIVELY COUPLED PLASMA (ICP) SERIAL DILUTION ANALYSIS

_____ Serial Dilutions were performed for each matrix and results of the diluted sample analysis agreed within ten percent of the original undiluted analysis.

_____ Serial Dilutions were not performed for the following:

_____

_____ Serial Dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the IDL before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

MATRIX:_____

ELEMENT	IDL	50xIDL	SAMPLE RESULT	SERIAL DILUTION	%D	ACTION
Aluminum						
Barium						
ryllium						
admium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Silver						
Sodium						
Vanadium						
Zinc						

Actions apply to all samples of the same matrix.

ACTIONS:

1. Estimate (J) positive results if %D >15.

XII. DETECTION LIMIT RESULTS

Instrument Detection Limits

_____ Instrument Detection Limit results were present and found to be less than the Contract Required Detection Limits.

_____ IDLs were not included in the data package on Form XI.

_____ IDLs were present, but the criteria was not met for the following elements:_____

2. Reporting Requirements

_____ Were sample results on Form I reported down to the IDL not the CRDL for all analytes? Yes or No

_____ Were sample results that were analyzed by ICP for Se, Tl, As, or Pb at least 5x IDL. Yes or No

_____ Were sample weights, volumes, and dilutions taken into account when reporting detection limits on Form I. Yes or No

If No,

The reported results may be inaccurate. Make the necessary changes on the data summary tables and request that the laboratory resubmit the corrected data.

XIII. SAMPLE QUANTITATION

_____ Sample results fall within the linear range for ICP and within the calibrated range for all other parameters.

_____ Sample results were beyond the linear range/ calibration range of the instrument for the following samples/elements:

_____

In the space below, please show a minimum of one sample calculation per method:

ICP

FURNACE

MERCURY

CYANIDE

For soil samples, the following equation may be necessary to convert raw data values (usually reported in ug/L) to actual sample concentrations (mg/kg):

The lab is required to use 1 gram sample (wet weight) to 200 ml.

Wet weight concentration =

$$\text{digest conc. in } \frac{ug}{L} \times \frac{200ml}{1\ gm} \times \frac{1L}{1000\ ml} \times \frac{1000gm}{1kg} \times \frac{1mg}{1000ug} = \frac{mg}{kg}$$

In addition the sample results are converted to dry weight using the percent solids calculations:

$$\frac{\text{Wet weight conc.}}{\%\text{solids}} \times 100 = \text{final concentration, dry weight (mg/kg)}$$

FIGURE 9

Inorganic Regional Data Assessment Form

FIGURE 9

Region 7601

INORGANIC REGIONAL DATA ASSESSMENT

CASE NO. _____ SITE _____

LABORATORY _____ NO. OF SAMPLES/ MATRIX _____

SDG# _____ REVIEWER (IF NOT ESD) _____

SOW# _____ REVIEWER'S NAME _____

DPO: ACTION _____ FYI _____ COMPLETION DATE _____

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES				
2.	CALIBRATIONS				
3.	BLANKS				
4.	ICS				
5.	LCS				
6.	DUPLICATE ANALYSIS				
7.	MATRIX SPIKE				
8.	MSA				
9.	SERIAL DILUTION				
10.	SAMPLE VERIFICATION				
11.	OTHER QC				
12.	OVERALL ASSESSMENT				

O = Data had no problems/or qualified due to minor problems.
M = Data qualified due to major problems.
Z = Data unacceptable.
X = Problems, but do not affect data.

ACTION ITEMS: _____

AREAS OF CONCERN: _____

NOTABLE PERFORMANCE: _____

APPENDIX E

Completed Inorganic Regional

Data Assessment Form (IRDA) and

Guidance for Completing the ORDA

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Guidelines for Evaluating Validation Memos and Completing the Inorganic Regional Data Assessment Form

Introduction

This document is designed to offer guidance in evaluating validation memos and completing the Inorganic Regional Data Assessment (IRDA) Form.

The document is designed to define and clarify the areas evaluated and the resulting actions for the Inorganic Regional Data Assessment Form. The procedure must be performed by a qualified data reviewer, since technical expertise is utilized in completing the form. An IRDA Form must be completed for each data package reviewed and validated for Region I.

I. HOLDING TIMES

A. Definition or Explanation

The holding time of the sample can be defined as the time from sample collection to the time of analysis or sample preparation, as appropriate.

B. Actions

If all the sample holding time criteria were met, the category will be qualified with "O", data had no problems/or qualified due to minor problems.

If the sample holding times were exceeded and all the positive results and detection limits are estimated, the Inorganic Regional Data Assessment Form (IRDA) will be qualified with "O", data had no problems/or qualified due to minor problems.

If the holding times were grossly exceeded and all the positive results are estimated and non-detected results are rejected, the IRDA form will be qualified with "M", data qualified due to major problems.

If the holding times were grossly exceeded and all positive and non-detected results are rejected, the IRDA form will be qualified with "Z", data unacceptable.

II. CALIBRATIONS

A. Definition or Explanation

Calibrations refer to the instrument's initial and continuing calibrations and demonstrate the instrument's ability to produce acceptable quantitative data.

B. Actions

If all the calibration criteria are within specifications, and met the frequency requirements the IRDA form will be qualified with "O", data had no problems/or qualified due to minor problems.

If the %R for the ICV or CCV is between 75-89 or 111-125, (or the respective ranges for CN and Hg) and the positive results have been estimated, the IRDA form will be qualified with "M", data qualified due to major problems.

If the %R is outside the 75-125% limits, (or the respective ranges for CN and Hg) and the positive results and non-detected results are rejected, the IRDA will be qualified with "Z", data unacceptable.

If other problems related to calibrations exist that do

not affect the data (eg. standards were not run at the proper frequency) , the IRDA will be qualified with "X", problems but do not affect data.

III. BLANKS

A. Definition or Explanation

The blanks consist of preparation blanks, calibration blanks, and equipment or trip blanks. The blanks are reviewed to determine the existence and magnitude of contamination problems. The problems associated with one blank apply to all the associated samples. Blanks are reported per matrix, and for each digestion batch.

B. Actions

If no contamination is present, and the proper number of blanks were analyzed, the IRDA form will require "O", data had no problems/or qualified due to minor problems.

If low levels of contaminants are present, and the detection limit is raised, the IRDA form will be qualified with "X", problems, but do not affect the data.

If contaminants were found in the laboratory blanks at levels greater than the CRDL, the associated samples must be reanalyzed or redigested depending on the source of contamination. If the samples are not reanalyzed, the IRDA qualifier will be "Z", data unacceptable.

If high levels of contaminants are detected in the equipment blanks, the IRDA shall be qualified with "M", data qualified due to major problems.

If blanks were not analyzed at the proper frequency but do not affect the data, the IRDA will be qualified with "X", problems but do not affect the data.

IV. INTERFERENCE CHECK SAMPLE (ICS)

A. Definition or Explanation

The ICP Interference Check Sample is analyzed at the beginning and end of each sample analysis run to verify the contract laboratory's interelement and background correction factors.

B. Actions

If all the recoveries for the ICS meet criteria and an ICS was analyzed at the proper frequency, the IRDA qualifier will be "O", data had no problems/or qualified due to minor problems.

If the ICS recovery does not meet criteria but the levels of interferents in the samples are not 50% or more of those found in the ICS solution, the qualifier will be "X", problems, but do not affect the data.

If ICS recoveries are 50-79% or >120%, and levels of interferents are 50% or more of that found in the ICS solution, the IRDA will be qualified with "O", data had no problems or qualified due to minor problems.

If results >2x IDL are detected for elements not present in the EPA solution and the samples have levels ofinterferents that are 50% or more of those found in the ICS solution, the IRDA qualifier will be "M", data qualified due to major problems.

If sample results are rejected due to major interferences, the IRDA form will be qualified with "Z", data unacceptable.

If the ICS recovery falls <50 %, the IRDA qualifier will be "Z", data unacceptable.

If the ICS was not analyzed at the proper frequency but the data are not affected, qualify the IRDA with "X", problems, but do not affect the data.

V. LABORATORY CONTROL SAMPLE

A. Definition or Explanation

The laboratory control sample analysis is designed to serve as a monitor of the efficiency of the digestion procedure. Reported results may be biased either high or low for elements with LCS recoveries outside required criteria.

B. Actions

If the aqueous LCS recovery falls within the range of 50-150%, or the solid LCS falls within the control windows and LCSs were analyzed at the required frequency, the IRDA qualifier will be "O", data had no problems or qualified due to minor problems.

If the aqueous LCS recovery >150%, or soil LCS results

are > control windows and results are >IDL, the IRDA qualifier will be "M", data qualified due to major problems.

If the aqueous LCS recovery >150%, or soil LCS results are > control windows and results are non-detected, the IRDA qualifier will be "X", problems but do not affect the data.

If the aqueous LCS recovery is <50% or the solid LCS falls below the control windows the qualifier should be "Z", data unacceptable.

If an LCS was not analyzed at the proper frequency but the data are not affected, the qualifier shall be "X", problems, but do not affect data.

VI. DUPLICATE ANALYSIS

A. Description or Explanation

Duplicate analyses are indicators of the precision of the sample results. Laboratory duplicates give an indication of the precison of the laboratory analysis. Field duplicates are indicators of field precision as well as laboratory precision. Laboratory duplicates are required per matrix and per digestion batch. Qualifiers apply to all samples of a similar matrix.

B. Actions

If samples have an RPD for lab or field duplicates that <50% for waters, <75 % for soils and were analyzed at the required frequency, the IRDA qualifier will be "O", data had no problems/or qualified due to minor problems.

For samples that are <5xCRDL , if the difference between the results is ± 2xCRDL for laboratory duplicates or ± 4xCRDL for field duplicates and were analyzed at the required frequency, the IRDA qualifier will be "O", data had no problems or qualified due to minor problems.

If the RPD is >50% for waters and 75 % for soils, for either laboratory or field duplicates for samples that are >5xCRDL, the qualifier should be "M", data qualified due to major problems.

If the absolute difference between samples is > ±2xCRDL for laboratory duplicates and >± 4xCRDL for field duplicates for samples less than 5xCRDL, the qualifier

should be "M", data qualified due to major problems.

If duplicates were not analyzed at the proper frequency but the data are not affected, qualify the IRDA with "X", problems, but do not affect the data.

VII. MATRIX SPIKE

A. Definition or Explanation

The matrix spike sample analysis is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. A matrix spike is required for each matrix analyzed and qualifiers apply to all samples of similar matrix.

B. If all the matrix spike recoveries are within the specified criteria, and were anlayzed at the required frequency, the IRDA form will be qualified with "O", data had no problems/or qualified due to minor problems.

If the matrix spike recovery is >125% and the reported results are non-detected, the qualifier should be "X", problems, but do not affect the data.

If the matrix spike recovery is between 50-150%, and the results are >IDL, the qualifier shall be "O", data had no problems/or qualified due to minor problems.

If the matrix spike recovery is >150% and positive results are estimated, qualify the IRDA with "M", data qualified due to major problems.

If the matrix spike recovery is between 30% and 50%, for non-detected results or <50% for results >IDL, the qualifier shall be "M", data qualified due to major problems.

If the spike recovery results fall below 30% and the sample results are non-detected, the IRDA form should be qualified with "Z", data unacceptable.

If a matrix spike was not analyzed at the proper frequency, or a post-digestion spike was not analyzed as required, but the data are not affected, qualify the IRDA with "X", problems, but do not affect the data.

VIII. METHOD OF STANDARD ADDITION (MSA)

A. Description or Explanation

The Method of Standard Addition (MSA) is required when the furnace post digestion spike recovery is not within 85-115%, and the sample absorbance is >50% of spike absorbance. This section of the IRDA is used to qualify problems with post-digestion spike recovery as well as MSAs.

B. Actions

If duplicate injections were performed as required and the %RSD or CV <40, qualify the IRDA with "O", data had no problems/or qualified due to minor problems.

If the %RSD or CV >40, qualify the IRDA with "M", data qualified due to major problems.

If post digestion spike recoveries were within 75-125% and were analyzed for every sample, the IRDA will be qualified with "O", data had no problems/or qualified due to minor problems.

If post digestion spike recoveries were >125% but samples were non-detected, qualify the IRDA with "X", problems but do not affect the data.

If post digestion spike recoveries 11-75%, (or >125% for positive results) the IRDA qualifer will be "M", data qualified due to major problems.

If post digestion spike recoveries were <10%, qualify the IRDA with "Z", data unacceptable.

If MSAs were performed as required and the correlation coefficient is >0.995, the IRDA form will be qualified with "O", data had no problems/or qualified due to minor problems.

If the correlation coefficient is <0.995 for both MSA analyses, the form shall be flagged with "M", data qualified due to major problems.

IX. SERIAL DILUTION

A. Description or Explanation

Serial dilution analysis determines whether significant physical or chemical interferences exist due to sample

matrix.

B. **Actions**

If the %D is <30%, the IRDA shall be flagged with "O", data had no problems/or qualified due to minor problems.

If the %D is >30%, the IRDA shall be flagged with "M", data qualified due to major problems.

If a serial dilution was not performed as required, the IRDA form shall be flagged with "X", problems, but do not affect the data.

X. SAMPLE RESULT VERIFICATION

A. **Description or Explanation**

The objective is to ensure that the reported quantitation results are accurate.

B. **Actions**

If transcription, calculation, linear range, or raw instrument data discrepancies are found and are resolved after contacting the laboratory, the IRDA form shall be qualified with "O", data had no problems/or qualified due to minor problems.

If transcription, calculation, linear range, or raw instrument data discrepancies cannot be resolved, qualify the form with "M", data qualified due to major problems.

XI. OTHER QC

A. **Description or Explanation**

Other problems with quality control parameters pertaining to the Case may be qualified using professional judgement. (ie. detection limits, post digestion spikes, etc.)

XII. OVERALL ASSESSMENT

A. **Description or Explanation**

The data reviewer makes professional judgements and expresses concerns and comments on the validity of the overall data package for a Case. The additive nature of

QC parameters out of specification is difficult to
in an objective manner. The data quality objectives
aid in making the decision of the overall validity o
data.

B. **Actions**

If there are no significant problems associated with th
Case, the IRDA will be qualified with "O", data had no
problems/or qualified due to minor problems.

If there were small problems with the data package or contractual violations but the data could be utilized, qualify the form with "X", problems, but do not affect the data.

If there was a significant problem which interfered with use of the data, the category will be qualified, "Z", data unacceptable.