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# SCIENCE APPLICATIONS INTERNATIONAL CORPORATION DATA MANAGEMENT TECHNICAL PROCEDURE

Title: Data Validation					
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Business Unit General M			Date:		
17 Jun	m 3/3/2009	C.B. Coward	3/3/2009		

# 1.0 PURPOSE

The purpose of this procedure is to define the process for validation of analytical sample results obtained from analyses of environmental samples collected for site characterization, assessment, determination of remedial actions, and risk assessment.

The primary goal of data validation is to provide an independent examination of the reported values and associated quality control. This will document that they are complete and accurately define the analytical context of the data set with respect to the project Data Quality Objectives (DQOs).

# 2.0 <u>SCOPE</u>

This procedure applies to all data generated as a result of analytical laboratory analyses of environmental samples for purposes of site characterization and environmental assessment activities conducted by Science Applications International Corporation (SAIC). This procedure is not applicable to in-situ field measurements, but may be applied to in-field analysis provided applicable documentation is available.

# 3.0 REFERENCES AND DEFINITIONS

# 3.1 <u>REFERENCES</u>

- 3.1.1 See the Common References at the front of the Data Management manual.
- 3.1.2 Science Applications International Corporation, Quality Assurance Administrative Procedure (SAIC QAAP) QAAP 15.1, Control of Nonconforming Items and Services.
- 3.1.3 Science Applications International Corporation, Quality Assurance Technical Procedure (SAIC QATP) TP-DM-300-2, Data Entry.
- 3.1.4 Science Applications International Corporation, Data Management Technical Procedure (SAIC DMTP) TP-DM-300-6, Data Package Receipt and Verification.

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- 3.1.5 Science Applications International Corporation, Quality Assurance Technical Procedure (SAIC QATP) TP-DM-300-9, Database Changes.
- 3.1.6 SAIC, Laboratory Data Validation Guidelines for Evaluating Radionuclide Analyses, Thomas L. Rucker and C. Martin Johnson Jr., SAIC document number 143.20020404.001, Revision 7, April 2002.
- 3.1.7 U.S. Environmental Protection Agency, Statement of Work for Organics Analysis Multi-Media, Multi-Concentration, Contract Laboratory Program, Document Number OLM01.0, and subsequent versions.
- 3.1.8 U.S. Environmental Protection Agency, Statement of Work for Inorganics Analysis Multi-Media, Multi-Concentration, Contract Laboratory Program, Document Number ILM01.0, and subsequent versions.
- 3.1.9 U. S. Environmental Protection Agency Contract Laboratory Program National Functional Guidelines for Organic Data Review, EPA-540/R-99/008, October 1999.
- 3.1.10 U. S. Environmental Protection Agency Contract Laboratory Program National Functional Guidelines for Low Concentrations Organic Data Review, EPA-540/R-00/006, June 2001.
- 3.1.11 U.S. Environmental Protection Agency Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540-R-004, October 2004.
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- 3.1.12 Region I, EPA New England, Data Validation Functional Guidelines for Evaluating Environmental Analyses, December 1996.
- 3.1.13 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983, PB84-128677.
- 3.1.14 U. S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, and all subsequent "Updates".
- 3.1.15 Science Applications International Corporation Quality Assurance Administrative Procedure (SAIC QAAP) QAAP 17.1, Records Management.

## 3.2 DEFINITIONS

- 3.2.1 <u>Data Validation</u> A systematic process for reviewing a body of data against a defined set of criteria to ensure that the data are adequate for their intended use. This review focuses on the technical aspects of the analytical process and quality control information. It should document that the analyses meet project specified QAPP and analytical SOW criteria.
- 3.2.2 <u>Electronic Data Deliverable (EDD)</u> Electronic representation of sample and analytical QC data as specified in the laboratory statement of work.
- 3.2.3 <u>Project</u> A finite, usually predetermined number of samples collected over a given time period for a particular site. A project consists of one or more Sample Delivery Groups.
- 3.2.4 <u>Sample Delivery Group (SDG)</u> A group of 20 or fewer samples received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. An SDG is defined by one of the following, whichever occurs first:
  - a) each 20 field samples;
  - b) each 14-day calendar period during which field samples are received, beginning with receipt of the first sample in the SDG; or
     c) as determined and defined by a specific project need.
  - c) as determined and defined by a specific project need.
- 3.2.5 <u>SAIC Environmental Information Management System (SEIMS)</u> A computerized repository of field and laboratory data arranged by project. If a given project has an identified alternate database, this should be substituted in this procedure where SEIMS is referenced.

# 4.0 **RESPONSIBILITIES**

4.1 See the Common Responsibilities at the front of the Data Management Manual.

## 4.2 PROJECT CHEMIST

The Project Chemist is responsible for:

- 4.2.1 preparing and disseminating appropriate guidance and project specific criteria for each verification and validation task;
- 4.2.2 ensuring that personnel are trained in and follow this procedure and all project specific requirements;
- 4.2.3 ensuring that data verification activities are conducted in accordance with this procedure and the defined project specific criteria;

- 4.2.4 monitoring project budget and schedule;
- 4.2.5 ensuring availability of necessary personnel, equipment, subcontractors, and services;
- 4.2.6 reviewing project analytical deliverables, verification checklists, and validation checklists for technical content, quality, and completeness; and
- 4.2.7 issuing "Requests for Missing or Incomplete Laboratory SDG Information" a full size form is provided immediately following this procedure or "Nonconformance Reports" as necessary.

# 4.3 DATA BASE ADMINISTRATOR (DBA)

The DBA is responsible for:

- 4.3.1 writing, testing, and maintaining all computer programs in support of the SEIMS database;
- 4.3.2 writing, testing, and maintaining computer programs for downloading laboratory EDDs into the appropriate SEIMS project database; and
- 4.3.3 ensuring electronic files are properly maintained and back-up files are completed.

# 4.4 DATA COORDINATOR

The Data Coordinator is responsible for:

- 4.4.1 date stamping and logging in all SDG data packages when received;
- 4.4.2 loading all project specific Sampling and Analysis Plan (SAP) information into SEIMS and downloading all laboratory EDDs into the established SEIMS project database, if required by the project;
- 4.4.3 ensuring that all data packages, electronic data, data verification checklists, and data validation checklists are maintained and complete;
- 4.4.4 establishing SAIC data review files by SDG for subsequent review by verification and validation staff;
- 4.4.5 ensuring that the laboratory EDD values are consistent with laboratory data deliverables;

- 4.4.6 ensuring original EDD files are stored properly;
- 4.4.7 ensuring data validation qualifiers and reason codes, (if validation is performed) are applied to each analytical result stored in the project database;
- 4.4.8 ensuring effective and efficient flow of project information; and
- 4.4.9 issuing "Requests for Missing or Incomplete Laboratory SDG Information" a full size form is provided immediately following this procedure or "Nonconformance Reports" as necessary.

# 4.5 DATA VALIDATORS

The Data Validators are responsible for:

- 4.5.1 ensuring that the appropriate guidance documents listed under references and outlined in the body of this procedure direct the data validation process;
- 4.5.2 ensuring that they are knowledgeable and informed of all project specific criteria and information necessary to complete the assigned validation task;
- 4.5.3 ensuring that appropriate checklists are used;
- 4.5.4 carefully reviewing the data packages;
- 4.5.5 completing the verification and validation checklists as identified in this procedure; and
- 4.5.6 issuing "Requests for Missing or Incomplete Laboratory SDG Information" a full size form is provided immediately following this procedure or "Nonconformance Reports" as necessary.

# 5.0 GENERAL

5.1 General direction is provided by the Environmental Protection Agency (EPA) under the Contract Laboratory Program (CLP) in the form of the National Functional Guidelines for Organic Data Review (EPA-540/R-99/008, October, 1999), the National Functional Guidelines for Low Concentration Organic Data Review (EPA-540/R-00/006, June 2001), and the National Functional Guidelines for Inorganic Data Review (EPA-540/R-004, October 2004). These guidelines provide specific criteria for determining data usability, however, they also allow for professional judgment. The requirements for LCS recoveries in this procedure have been modified for organic constituents based on professional judgment (See LCS section of organic data checklists).EPA Region I has provided the environmental

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community with a useful "Tiered Approach" to validation that allows a program or project to establish the level of intensity and depth of review applicable to their needs. Guidance to this approach appears in "Region I, EPA-New England Data Validation Functional Guidelines for Evaluating Environmental Analyses", July 1996, revised December 1996. This document and its appendices may prove useful during project data validation development. Interpretation of this guidance and its application to individual programs and projects needs to be made at the operational level and incorporated into the Sampling and Analysis Plan for a given investigation. Direction for radionuclide validation is provided by the Rucker and Johnson publication cited in Reference 3.1.6.

- 5.2 Specific requirements for analytical data validation are defined in the Quality Assurance Project Plan (QAPP) and/or the site Sampling and Analysis Plan (SAP) and/or the project specific data validation plan and are used to direct the systematic process to validate project data. Verification and validation must be consistent with the project data quality objectives, laboratory scope of work, and designated analytical methods. Data are validated against this set of accepted criteria to provide assurance that data are adequate for their intended use.
- 5.3 The validation of environmental data is the process by which data are evaluated in context to field and analytical QA/QC samples associated with the environmental samples. This process consists of data checking, auditing, verification, flagging, review, and certification. Validation is independent of the analytical laboratory data review. The project-specific Data Validators certify in writing that data have been validated and flagged in accordance with the defined process. Examples of the items evaluated during the validation process are:
  - iples of the items evaluated during the validation pro
    - technical holding times;
    - blanks (laboratory and field/trip/equipment);
    - duplicate samples (laboratory and field);
    - laboratory control samples;
    - matrix spike samples;
    - matrix spike duplicate samples;
    - surrogate / tracer recoveries;
    - calibration;
    - internal standards; and
    - external standards.
- 5.4 Data base entry of all data validation flags (Attachment I) and reason codes (Attachment II) that have been entered on the sample results forms is completed according to TP-DM-300-2 (Reference 3.1.3).

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## 6.0 PROCEDURE

## 6.1 DATA VALIDATION

## 6.1.1 STANDARD DATA VALIDATION GUIDELINES

- a) Data packages are validated in accordance with the QAPP, the site SAP, and Data Validation Plan.
- b) Standard data validation includes all aspects of data verification and implements an evaluation of laboratory quality control data and analytical procedures. This ensures the analytical process and instrumentation used to perform the analyses met all of the data quality requirements specified in the Data Quality Objectives (DQOs) and Sampling and Analysis Plan. Focus is given to laboratory /instrument performance criteria, sample preparation and matrix effects evaluation, and field quality control measures. Standard data validation involves evaluating the laboratory analytical data packages to confirm that:

## **Deliverable verification**

- the data packages are complete and contain all of the information specified in the Sampling and Analysis Plan [e.g., all samples and analyses requested, case narrative, summary data report, completed chain-of-custody form, analytical quality control data (blanks, matrix spikes, matrix spike duplicates, etc.), date and time when each analysis was performed],
- the laboratory ran the correct analytical methods specified in the Sampling and Analysis Plan,
- samples did not exceed the maximum analytical holding times specified in the Sampling and Analysis Plan,
- sample chain-of-custody was not broken from the time the sample was collected, analyzed, and the data reported, and
- the laboratory reported analytical results for each analytical method and each analyte required by the laboratory statement of work and the project Sampling and Analysis Plan.

## Laboratory/instrument performance criteria

- laboratory case narrative documentation is clear and accurate,
- analytical preparation procedures are acceptable and documented,

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- instrument operational and method calibration criteria have been achieved,
- laboratory calibration blank contamination is under control, and
- laboratory control standard criteria are being met.

# Sample preparation and matrix effects criteria

- laboratory method blank contamination is under control,
- sample surrogate compound recovery, tracer recovery, and internal standard criteria have been achieved,
- sample matrix spike recoveries meet minimum accuracy requirements specified in the DQOs and Sampling and Analysis Plan,
- sample matrix spike duplicate or duplicate comparisons meet minimum precision requirements specified in the DQOs and Sampling and Analysis Plan, and
- sample dilution review and re-analyses performance.

# Field quality control measures

- field source water blank, equipment rinsate blank, and sample trip blank contents have not impacted the project data results, and
- field duplicate comparisons meet minimum precision requirements specified in the DQOs and Sampling and Analysis Plan.
- c) Following application of TP-DM-300-6 (Reference 3.1.4) the Data Validator reviews the data package and data verification checklists. The appropriate work sheets (see checklist forms "Standard Validation Checklist", Attachment III, full size forms are provided immediately following this procedure), or QAPP, SAP, or Data Validation Plan specified checklists, available from the Data Coordinator, are used when validating data.
- d) All data presented on standardized reporting forms are validated against guideline criteria in all data packages.
- e) After completion of the work sheets, nonconforming items identified by the validation process are summarized and reported following TP-DM-300-9 (Reference 3.1.5) or QAAP 15.1 (Reference 3.1.2).
- f) Copies of the sample result forms are made and marked "DATA VALIDATION COPY".

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- g) Failures to meet specified criteria are documented on the work sheets for each analyte. The data for each sample/analyte are flagged accordingly on the data reporting forms marked "DATA VALIDATION COPY". This will involve professional judgement on the part of the data validator.
- h) For each data package, a two part data validation deliverable is generated consisting of:
  - data reporting forms marked "DATA VALIDATION COPY" with validation flags and reason codes; and
  - validation work sheet.

# 6.1.2 COMPREHENSIVE DATA VALIDATION GUIDELINES

Comprehensive data validation encompasses all Standard Data Validation information and adds an examination of the analytical raw data. This level of review requires all information generated by the laboratory to be presented as part of the data deliverable. This would include copies of all chromatograms, spectral printouts, quantification details, preparation logbooks, standard logbooks, calculation programs, etc., produced by the laboratory. In addition to the material reviewed during standard data validation, comprehensive data validation will include:

- a detailed examination of the raw data analyte identification,
- a check of calculations used to quantify analyte results, normally a minimum of 10% of the reported concentrations are checked by recalculation from original raw data information, and
- recalculated results are verified against final reported concentrations.

Following application of TP-DM-300-6 (Reference 3.1.4), the Data Validator reviews the data package and data verification checklists. The appropriate work sheets (see checklist forms "Comprehensive Validation Checklist", Attachment III, full size forms are provided immediately following this procedure), or QAPP, SAP, or Data Validation Plan specified checklists, available from the Data Coordinator, are used when validating data.

6.2 After completion of the validation, the Data Validator returns the validation package to the Data Coordinator. The Data Coordinator then sends the data validation package to the Project Chemist or another Data Validator for QA and technical review.

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6.3 After the QA and technical review is completed, the data validation flags and reason codes (when applicable) are entered into the database according to TP-DM-300-2 (Reference 3.1.3).

## 7.0 <u>RECORDS</u>

Documentation generated as a result of this procedure is submitted to the identified records system, in accordance with Section 17 of the Business Unit QAP.

## 8.0 ATTACHMENTS

- 8.1 Attachment I Data Qualifiers (validation qualifiers and laboratory qualifiers)
- 8.2 Attachment II Data Validation Reason Codes
- 8.3 Attachment III- Forms List

# ATTACHMENT I DATA QUALIFIERS

## Validation Data Qualifiers (Flags)

During the data validation process, all laboratory data are assigned appropriate data validation flags and reason codes. Validation flags are defined as follows:

- "U" Indicates the analyte was analyzed for, but not detected above the level of the associated value.
- "J" Indicates the analyte was positively identified, however, the associated numerical value is an estimated concentration of the analyte in the sample.
- "UJ" Indicates the analyte was analyzed for, but not detected, above the associated value, however, the reported value is an estimate and demonstrates a decreased knowledge of its accuracy or precision.
- "R" Indicates the analyte value reported is unusable. The integrity of the analyte's identification, accuracy, precision, or sensitivity have raised significant question as to the reality of the information presented.
- "=" Indicates the analyte has been validated, the analyte has been positively identified, and the associated concentration value is accurate.

## **Normal Laboratory Data Qualifiers**

During the laboratory production and internal review laboratory data may be assigned data qualifiers. These are reported as part of the laboratory data deliverable and will eventually be replaced by the more concise set of Validation Data Qualifiers. Normal laboratory data qualifiers are defined as follows:

Laboratory Qualifiers for Organic Analytical Data

- U Indicates that the compound was analyzed for but not detected. The sample quantitation limit (SQL) must be corrected for dilution. For a soil/sediment sample, the value must also be corrected for percent moisture.
- J Indicates an estimated value. This qualifier is used either when estimating a concentration for tentatively identified compounds (TICs) where a 1:1 response is assumed, or when the mass spectral data indicates the presence of a compound that meets the identification criteria but the result is less than the SQL but greater than zero.
- **N** Indicates presumptive evidence of a compound. This qualifier is used only for TICs, where the identification is based on a mass spectral library search.

- **P** Used for pesticide/PCB target analytes when there is greater than 25% difference for detected concentrations between the two GC columns.
- C Applies to pesticide results where the identification has been confirmed by gas chromatography/mass spectrometry (GC/MS). If GC/MS confirmation was attempted but was unsuccessful, this qualifier is not applied; instead a laboratorydefined qualifier is used.
- B Used when the compound is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and alerts the data user to take appropriate action. This qualifier is used for TICs as well as for positively identified target compounds.
- **E** Identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for that specific analysis.
- D Identifies all compounds identified in an analysis at a secondary dilution factor. This qualifier alerts data users that any discrepancies between the concentrations reported may be due to dilution of the sample or extract.
- **A** Indicates that a TIC was a suspected aldol-condensation product.
- X Indicates that other specific qualifiers were required to properly define the results. If used, the qualifier must be fully described and such description must be included in the Sample Data Summary Package and SDG narrative.

Laboratory Qualifiers for Inorganic Analytical Data

- B Indicates that the reported value was obtained from a reading that was less than the Contract Required Detection Limit (CRDL), but greater than or equal to the Instrument Detection Limit (IDL).
- **U** Indicates that the analyte was analyzed for but not detected.
- E Used when the reported value was estimated because of the presence of interference.
- **M** Indicates that the duplicate injection precision was not met.
- **N** Indicates that the spiked sample recovery was not within control limits.
- **S** Indicates that the reported value was determined by the method of standard additions (MSA).

- W Used when the post-digestion spike for furnace atomic absorption analysis was not within control limits (85 - 115%), while sample absorbance was less than 50% of spike absorbance.
- \* Indicates that the duplicate analysis was not within control limits.
- + Indicates that the correlation coefficient for the MSA was less than 0.995.

Laboratory Qualifiers for Radiochemical Analytical Data

- < The numerical value reported was less than the MDA.
- **N** The sample results were qualified to denote poor spike recovery.
- \*— The sample results were qualified to denote poor duplicate results.

ATTACHMENT II DATA VALIDATION REASON CODES

Organic, Inorganic, and Radiological Analytical Data

# Holding Times

- A01 Extraction holding times were exceeded.
- A02 Extraction holding times were grossly exceeded.
- A03 Analysis holding times were exceeded.
- A04 Analysis holding times were grossly exceeded.
- A05 Samples were not preserved properly.
- A06 Professional judgement was used to qualify the data.

# GC/MS Tuning

- B01 Mass calibration was in error, even after applying expanded criteria.
- B02 Mass calibration was not performed every 12 hours.
- B03 Mass calibration did not meet ion abundance criteria.
- B04 Professional judgement was used to qualify the data.

# Initial/Continuing Calibration - Organics

- C01 Initial calibration RRF was <0.05.
- C02 Initial calibration RSD was >30%.
- C03 Initial calibration sequence was not followed as required.
- C04 Continuing calibration RRF was <0.05.
- C05 Continuing calibration %D was not acceptable.
- C06 Continuing calibration was not performed at the required frequency.
- C07 Resolution criteria were not met.
- C08 RPD criteria were not met.
- C09 RSD criteria were not met.
- C10 Retention time of compounds was outside windows.
- C11 Compounds were not adequately resolved.
- C12 Breakdown of endrin or DDT was >20%.
- C13 Combined breakdown of endrin/DDT was >30%.
- C14 Professional judgement was used to qualify the data.

#### Initial/Continuing Calibration - Inorganics

- D01 ICV or CCV were not performed for every analyte.
- D02 ICV recovery was above the upper control limit.
- D03 ICV recovery was below the lower control limit.
- D04 CCV recovery was above the upper control limit.
- D05 CCV recovery was below the lower control limit.
- D06 Standard curve was not established with the minimum number of standards.
- D07 Instrument was not calibrated daily or each time the instrument was set up.
- D08 Correlation coefficient was <0.995.
- D09 Mid range cyanide standard was not distilled.
- D10 Professional judgement was used to qualify the data.

#### ICP and Furnace Requirements

- E01 Interference check sample recovery was outside the control limit.
- E02 Duplicate injections were outside the control limit.
- E03 Post digestion spike recovery was outside the control limit.
- E04 MSA was required but not performed.
- E05 MSA correlation coefficient was <0.995.
- E06 MSA spikes were not at the correct concentration.
- E07 Serial dilution criteria were not met.
- E08 Professional judgement was used to qualify the data.

## <u>Blanks</u>

- F01 Sample data were qualified as a result of the method blank.
- F02 Sample data were qualified as a result of the field blank.
- F03 Sample data were qualified as a result of the equipment rinsate.
- F04 Sample data were qualified as a result of the trip blank.
- F05 Gross contamination exists.
- F06 Concentration of the contaminant was detected at a level below the CRQL.
- F07 Concentration of the contaminant was detected at a level less than the action limit, but greater than the CRQL.
- F08 Concentration of the contaminant was detected at a level that exceeds the action level.
- F09 No laboratory blanks were analyzed.
- F10 Blank had a negative value >2x's the IDL.
- F11 Blanks were not analyzed at required frequency.
- F12 Professional judgement was used to qualify the data.
- F13 Reported blank net result is > than 1.65 sigma, radiochemistry.
- F14 Subtracted method blank exceeds 3 sigma of established blank value, radiochemistry.

#### Surrogate/Radiological Chemical Recovery

- G01 Surrogate/radiological chemical recovery was above the upper control limit.
- G02 Surrogate/radiological chemical recovery was below the lower control limit.
- G03 Surrogate recovery was <10%.
- G04 Surrogate recovery was zero.
- G05 Surrogate/radiological chemical recovery data was not present.
- G06 Professional judgement was used to qualify the data.
- G07 Radiological chemical recovery was <20%.
- G08 Radiological chemical recovery was >150%.
- G09 The 2 sigma uncertainty in the radiological sample specific chemical recovery was > 10%

#### Matrix Spike/Matrix Spike Duplicate (MS/MSD)

- H01 MS/MSD recovery was above the upper control limit.
- H02 MS/MSD recovery was below the lower control limit.
- H03 MS/MSD recovery was <10%.
- H04 MS/MSD pairs exceed the RPD limit.
- H05 No action was taken on MS/MSD results.
- H06 Professional judgement was used to qualify the data.
- H07 Radiological MS/MSD recovery was < 20%.
- H08 Radiological MS/MSD recovery was >160%.
- H09 Radiological MS/MSD samples were not analyzed at the required frequency.

## Matrix Spike

- I01 MS recovery was above the upper control limit.
- I02 MS recovery was below the lower control limit.
- I03 MS recovery was <30%.
- No action was taken on MS data.
- I05 Professional judgement was used to qualify the data.
- I06 MS samples were not analyzed at the required frequency.

#### Laboratory Duplicate

- J01 Duplicate RPD/radiological duplicate error ratio (DER) was outside the control limit.
- J02 Duplicate sample results were >5 x the CRDL.
- J03 Duplicate sample results were <5 x the CRDL.
- J04 Professional judgement was used to qualify the data.
- J05 Duplicate was not analyzed at the required frequency.
- J06 Radiological duplicate RPD and duplicate error ratio (DER) were outside acceptable limits.

#### Internal Area Summary

- K01 Area counts were outside the control limits.
- K02 Extremely low area counts or performance was exhibited by a major drop off.
- K03 IS retention time varied by more than 30 seconds.
- K04 Professional judgement was used to qualify the data.

#### Pesticide Cleanup Checks

- L01 10% recovery was obtained during either check.
- L02 Recoveries during either check were >120%.
- L03 GPC Cleanup recoveries were outside the control limits.
- L04 Florisil cartridge cleanup recoveries were outside the control limits.
- L05 Professional judgement was used to qualify the data.

#### **Target Compound Identification**

- M01 Incorrect identifications were made.
- M02 Qualitative criteria were not met.
- M03 Cross contamination occurred.
- M04 Confirmatory analysis was not performed.
- M05 No results were provided.
- M06 Analysis occurred outside 12 hr GC/MS window.
- M07 Professional judgement was used to qualify the data.
- M08 The %D between the two pesticide/PCB column checks was >25%.

## Compound Quantitation and Reported CRQLs

- N01 Quantitation limits were affected by large off-scale peaks.
- N02 MDLs reported by the laboratory exceeded corresponding CRQLs.
- N03 Professional judgement used to qualify the data.

#### **Tentatively Identified Compounds (TICs)**

- O01 Compound was suspected laboratory contaminant and was not detected in the blank.
- O02 TIC result was not above 10 x the level found in the blank.
- O03 Professional judgement was used to qualify analytical data.

## Laboratory Control Samples (LCSs)

- P01 LCS recovery was above upper control limit.
- P02 LCS recovery was below lower control limit.
- P03 LCS recovery was <50%.
- P04 No action was taken on the LCS data.
- P05 LCS was not analyzed at required frequency.

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- P06 Radiological LCS recovery was <50% for aqueous samples; <40% for solid samples.
- P07 Radiological LCS recovery was >150% for aqueous samples; >160% for solid samples.
- P08 Professional judgement was used to qualify the data.

#### Field Duplicate

- Q01 Field duplicate RPDs were >30% for waters and/or > 50% for soils.
- Q02 Radiological field duplicate error ratio (DER) was outside the control limit.
- Q03 Duplicate sample results were >5 x the CRDL.
- Q04 Duplicate sample results were <5 x the CRDL.

#### **Radiological Calibration**

- R01 Efficiency calibration criteria were not met.
- R02 Energy calibration criteria were not met.
- R03 Resolution calibration criteria were not met.
- R04 Background determination criteria were not met.
- R05 Quench curve criteria were not met.
- R06 Absorption curve criteria were not met.
- R07 Plateau curve criteria were not met.
- R08 Professional judgement was used to qualify the data.
- R09 Background quench curve criteria were not met.
- R10 Errors found in calculations.
- R11 Calibration required frequency not met.
- R12 Dark current criteria were not met.

## **Radiological Calibration Verification**

- S01 Efficiency verification criteria were not met.
- S02 Energy verification criteria were not met.
- S03 Resolution verification criteria were not met.
- S04 Background verification criteria were not met.
- S05 Cross-talk verification criteria were not met.
- S06 Professional judgement was used to qualify the data.
- S07 Calibration verification required frequency not met.

#### **Radionuclide Quantitation**

- T01 Detection limits were not met.
- T02 Analytical uncertainties were not met and/or not reported.
- T03 Inappropriate aliquot sizes were used.
- T04 Professional judgement was used to qualify the data.
- T05 Errors in calculation of reported result.
- T06 Errors in calculation of reported uncertainty.
- T07 Net negative result with absolute value greater than the reported uncertainty.

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- T08 Exceeded maximum mass/area on planchet for alpha/beta.
- T09 Quantification not possible due to interference.
- T10 Results do not compare with others related measurements on the same sample.
- T11 Reported result is less than 1.65 theta.
- T12 Analytical result is less than the associated MDA, but greater than the counting uncertainty.
- T13 Analytical result is less than both the associated counting uncertainty and the MDA.
- T14 Negative analytical result where absolute value exceeds 2x the associated MDA.

## System Performance

- V01 High background levels or a shift in the energy calibration were observed.
- V02 Extraneous peaks were observed.
- V03 Loss of resolution was observed.
- V04 Peak-tailing or peak splitting that may result in inaccurate quantitation were observed.
- V05 Professional judgement was used to qualify the data.
- V06 General degradation of system performance.

## **Radionuclide Identification**

- W01 Peak energy difference greater than 40 keV (alpha) or 2 keV (gamma).
- W02 Interference peak in region of interest.
- W03 Less than 50% total gamma abundance for tentatively identified radionuclides (TIRs).
- W04 Professional judgement was used to qualify the data.

# ATTACHMENT III Forms List

Immediately following this procedure are the full size forms for the following:

- Organic Data Review Checklist Standard Validation
- GC and LC Organic Data Review Checklist- Standard Validation
- Metals Data Review Checklist- Standard Validation
- Inorganic Data Review Checklist- Standard Validation
- Radiochemical Data Review Checklist- Standard Validation
- Organic Data Review Checklist Comprehensive Validation
- GC and LC Organic Data Review Checklist- Comprehensive Validation
- Metals Data Review Checklist- Comprehensive Validation
- Inorganic Data Review Checklist- Comprehensive Validation
- Radiochemical Data Review Checklist- Comprehensive Validation
- Requests for Missing or Incomplete Laboratory SDG Information

# SCIENCE APPLICATIONS INTERNATIONAL CORPORATION Organic Data Review Checklist - Standard Validation

Project:			Page 1 of 11
SDG No:		Analysis:	
Laboratory:		Method: Matrix:	
data have been su	ackage has been reviewed and Immarized. The general criteria Ination of the following:		trol/quality assurance performance ytical integrityof the data were
	Case Narrative Analytical Holding Times Sample Preservation Method Calibration Method and Project Blanks	Analytical Surrogate R Internal Standard Perform MS/MSD Recoveries a LCS Recoveries Re-analysis and Secon	ormance and Differences
Project Specific Q	A/QC or contract requirements	may take priority over val	idation criteria in this procedure.
Overall Remarks			
Definition of Qualif		inte d la cal	
	"U", not detected at the assoc "UJ", not detected and associa "J", associated value estimate "R", associated value unusabl "=", compound properly identit	ated value estimated d e or analyte identity unfo	unded
Reviewed by:			Date:
QA Reviewed by	/:		Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

# **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

## **III. Holding Times**

VOC - Waters - unpreserved: aromatic within 7 days, non-aromatic within 14 days of sample collection VOC - Waters - preserved: aromatic and non-aromatic within 14 days of sample collection VOC - Soils - preserve or analyze within 48 hours of sample collection; analyze within 14 days of preservation

SVOC, Pest., PCB - Waters - extract within 7 days of sample collection, analyze within 40 days of extraction SVOC, Pest., PCB - Soils - extract within 14 days of sample collection, analyze within 40 days of extraction

#### **Deviations:**

	VOC		SVOC			Pest/PCB		
Sample #	Date	Date	Date	Date	Date	Date	Date	Date
	Collected	Analyzed	Collected	Extracted	Analyzed	Collected	Extracted	Analyzed

#### Actions:

- 1. If holding times are exceeded, all results are qualified as estimated (J/UJ)
- 2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

## Page 4 of 11 IV. System Monitoring Compounds (SMC) Recoveries (VOC, SVOC, Pesticides, PCBs)

List SMC compounds with unacceptable recoveries:

#### **Deviations:**

	VOC			SVOC		SVOC			Pest	PCB	
Sample #			B/N Compounds			I Compo					
	TOL	BFB	DCE	NBZ	FBP	TPH	PHL	2FP	TBP	TCX	DCB
QC											
Limits											

#### Actions:

1. If any SMC recovery is <10%, qualify all positive results in associated fractions as estimated (J)

2. If any SMC recovery is <10%, qualify all nondetects in associated fractions as unusable (R)

3. If SMC recoveries fall between 10% and the lower recovery limit, qualify results as estimated (J/UJ)

4. If SMC recoveries fall above the upper recovery limit, qualify positive results as estimated (J)

5. Use professional judgement to qualify Pest/PCB results when SMC recoveries are >10%

6. Use professional judgement to qualify results when SMC recoveries have been diluted out of spec.

7. For SVOC, qualification of the data is required only when 2 or more SMC per fraction are not within control limits

8. Note: SMC formerly known as surrogates.

## V. Internal Standards Performance (VOC, SVOC)

VOC internal standard area counts within -50% to +100% of standard (Y/N) VOC internal standard retention times within  $\pm$  30 seconds of standard (Y/N)

SVOC internal standard area counts within -50% to +100% of standard (Y/N) SVOC internal standard retention times within + 30 seconds of standard (Y/N)

#### **Deviations:**

	IS	Area	Acceptable Range	RT	Std. RT
Sample #	Affected	Counts	Range		Value
ł			Ŭ		
				1	-
				1	
				1	1

#### Actions:

1. If area counts are outside limits, qualify positive results associated with that IS as estimated (J)

2. Non-detected compounds quantitated using an IS area count >100% should not be qualified

3. Non-detected compounds quantitated using an IS area count <50%, qualify as estimated (UJ)

4. If extremely low area counts are reported (<50% of the lower limit), qualify non-detects as unusable (R)

5. If an IS retention time varies more than 30 seconds, review the chromatographic profile for shifts and irregularities. Use professional judgement to qualify the data estimated (J/UJ) or unusable (R)

#### Remarks:

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# Page 6 of 11

## VI. Blanks

All blanks were reported per matrix per concentration level for each 12 hour period on each GC/ MS system used to analyze VOCs and SVOCs Yes No Review associated laboratory and project blank samples. List documented contamination below:

## Laboratory Method Blanks:

Date:	Lab ID #	Fraction	Compound	Conc. (ppb)
		·		
		·		
		· ·		
		· ·		
		· ·		
		·		
Associated F	Project Blanks (e.g.	, equipment rinsates	, trip blanks, etc.)	
Date	Lab ID #	Fraction	Compound	Conc. (ppb)
		. <u> </u>		
		· ·		
		·		
		· ·		
		·		
		· ·		
		·		
Remarks:				

## VI. Blanks (continued)

Calculate action levels based on 10X the highest blank concentration of "common laboratory solvents", VOCs (methylene chloride, acetone, toluene, 2-butanone, cyclohexane) or SVOCs (phthalates), and 5X the highest blank concentration for all other VOC, SVOC, Pesticides, and PCB compounds. Sample weights, volumes, and dilution factors must be taken into account when applying the 5X and 10X criteria. This allows the total amount of contaminant present to be considered.

#### **Deviations:**

	Maximum Conc.	Action Level (ppb)	Samples Affected
Compound	Detected, (ppb)		

#### Actions:

- 1. If compound results exceed the action levels, the data are not qualified
- 2. If compound results are below the required reporting level, report results as non-detect (U) at the reporting level
- 3. If the compound is detected above the reporting level, but below the action level, qualify as not-detected (U)
- 4. If gross contamination exists in blanks (i.e.,, saturated peaks by GC/ MS), all affected compounds in the associated samles should be qualifed as unusable (R) due to interference.
- 5. If blanks were not analyzed per matrix per concentration level for each 12 hour period on each GC/MS system

used to analyze VOCs and SVOCs use professional judgement to qualify data. Data may be rejected (R).

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# VII. Initial & Contining Calibration (VOC, SVOC)

GC/MS instrument performance checks (BFB / DFTPP) Acceptable Y or N All compounds must have and RRF > 0.01, %RSD < 30, and %D < 25

VOC - Date of initial calibration: VOC - Date(s) of continuing calibration: Was the 12 hour critieria met? Y or N

SVOC- Date of initial calibration: SVOC - Date(s) of continuing calibration: Was the 12 hour critieria met? Y or N

## **Deviations:**

Compound	Date	RRF	%RSD	%D	Samples Affected

\* % Difference =  $((RF_{CCV} - RF_{ICAL AVG})/RF_{ICAL AVG}) \times 100$ . In instances where the bias of the CCV impacts

validation qualifiers, review the RF values or amount reported to confirm that the % Difference or %

Drift are reported with the correct negative or positive value.

Actions:

- 1. If any compound has an initial or continuing RRF of < 0.01, qualify positive results as estimated (J)
- 2. If any compound has an intial or continuing RRF of < 0.01, qualify non-detects as unusable (R)
- 3. If any compound has a %RSD >30 or a %D >25, qualify positive results as estimated (J)
- 4. If any compound has a %RSD >40 or a %D >40, qualify non-detects as estimated (UJ)
- 5. If BFB or DFTPP mass assignment / ION abundance criteria are all associated data as unusable (R).
- 6. If samples were analyzed outside the 12 hour BFB or DFTPP performance check time period, qualify the affected sample data as estimated (J/UJ).
- 7. If separate calibration for water and soil were not performed, use professional judgement to evaluate the data. Data may be rejected (R).
- 8. If calibrations were not completed within the 12 hour criterion, qualify all associated data as estimated (J/UJ).

If the 12 hour criterion was grossly exceeded, reject all associated data (R).

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## VIII. Initial & Continuing Calibration (Pesticides, PCBs)

Linearity evaluation, are %RSD <20? (Y/N)

Is the RPD between calibration factors <25? (Y/N)

Are multicomponent calibration data provided for each analysis date? (Y/N)

Is the difference between columns check  $\leq$  25%D? (Y/N)

Are 4, 4'- DDT and endrin breakdown (PEM)  $\leq$  20% and combined breakdown  $\leq$  30% (Y/N)

#### **Deviations:**

Compound	%RSD	RPD	Samples Affected

\* % Difference = (( $RF_{CCV}$  -  $RF_{ICAL AVG}$ )/ $RF_{ICAL AVG}$ ) x 100. In instances where the bias of the CCV impacts

validation qualifiers, review the RF values or amount reported to confirm that the % Difference or %

Drift are reported with the correct negative or positive value.

## Actions:

- 1. If %RSD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 2. If RPD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 3. If %D criteria is not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 4. If breadkwon criteria are not met, positive 4, 4'-DDT and endrin should be qualified as estimated (J). And non-detects should be rejected (R).

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#### IX. Matrix Spike/Matrix Spike Duplicate Information

						_
General MS/MSD Criteria:		VOC	SVOC	Pest	PCB	
percent recovery (%R)	70-130	45-135	40-140	40-140		
relative percent difference	e (RPD)	<30	<50	<50	<50	
Project Sample(s) Spiked	:					
Deviations:						
	%R	%R	RPD	RPD		
Compound		Limits		Limits	Sa	mples Affected
•						

## Actions:

- 1. If the spike recovery is above the upper control limit (UCL), qualify all positive values in the unspiked sample as estimated (J) and non-detects as estimated (UJ).
- 2. If the spike recovery is below the lower control limit (LCL), qualify positive values as estimated (J). And non-detects as estimated (UJ).
- 3. If the spike recovery is <10%, qualify non-detect values as unusable (R)
- 4. If the RPD does not meet criteria, qualify positive values in the unspiked sample as estimated (J)
- 5. Use professional judgement to qualify additional samples in the analytical group based on MS/MSD results
- 6. Use professional judgement for qualification of data for unspiked compounds

# Page 11 of 11

#### X. Laboratory Control Sample Information

General LCS Criteria:	VOC	SVOC	Pest	PCB
percent recovery (%R)	80-120	60-120	50-130	50-130

Laboratory LCS Identifications:

**Deviations:** 

Compound	Date	%R	Samples Affected/Qualifiers Applied
oompound	Dato	7011	

## Actions:

Action should be based on both the number of compounds outside the criterion and the magnitude of the exceedance.

1. If the LCS recovery is below limits but > one- half the lower limit, qualify valves as estimated (J/UJ).

2. If the LCS recovery is < one-half the lower limit, qualify all data for that analyte as unusable (R).

3. If the LCS recovery is greater than the upper limit, qualify positive valves for that analyte as estimated (J).

4. If more than half the compounds in this LCS are not within recovery criteria, then qualify associated detected compounds as estimated (J).

5. Use professional judgement for qualification of data for compounds with no LCS information

	GC and LC Organic D		NAL CORPORATION st - Standard Validation RO, Methanol, etc.)
Project:			Page 1 of 9
SDG No:		Analysis: Method:	
Laboratory:		Matrix:	
data have been s	ackage has been reviewed and ummarized. The general criteria nination of the following:		rol/quality assurance performance ytical integrityof the data were
	Case Narrative Analytical Holding Times	Analytical Surrogate Ro MS/MSD Recoveries a	
	Sample Preservation Method Calibration Method and Project Blanks	LCS Recoveries Re-analysis and Secor	
Overall Remark	s:		
Definition of Qual	ifiers: "U", not detected at the assoc	sisted level	
	"UJ", not detected at the assoc "UJ", not detected and associ "J", associated value estimate "R", associated value unusab "=", compound properly identi	ated value estimated ed le or analyte identity unfou	unded
Reviewed by:			Date:
QA Reviewed b	y:		Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

# **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

#### **III. Holding Times**

VOC types - Waters - unpreserved: aromatic within 7 days, non-aromatic within 14 days of sample collection VOC types - Waters - preserved: aromatic and non-aromatic within 14 days of sample collection VOC types - Soils - preserve/analyze within 48 hours of sample collection; analyze within 14 days of preservation

SVOC types - Waters - extract within 7 days of sample collection, analyze within 40 days of extraction SVOC types - Soils - extract within 14 days of sample collection, analyze within 40 days of extraction

#### **Deviations:**

VOC types		SVOC types			Notes:	
Sample #	Date	Date	Date	Date	Date	
	Collected	Analyzed	Collected	Extracted	Analyzed	

#### Actions:

- 1. If holding times are exceeded, all results are qualified as estimated (J/UJ)
- 2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

#### Page 4 of 9

#### **IV. Initial & Continuing Calibration**

A blank and five standards should be analyzed, with one of the standards being within 2X the MDL Correlation coefficients must be  $\geq 0.995$ 

The RSD of the calibration factor or the relative response factor (RRF) must be  $\leq$  20% Continuing calibration %D must be within  $\pm$  15%

#### **Deviations:**

Compound	Correlation Coefficient	% RSD	%D	Samples Affected

\* % Difference =  $((RF_{CCV} - RF_{ICAL AVG})/RF_{ICAL AVG}) \times 100$ . In instances where the bias of the CCV impacts

validation qualifiers, review the RF values or amount reported to confirm that the % Difference or %

Drift are reported with the correct negative or positive value.

#### Actions:

- 1. If any compounds initial calibration linearity is <0.995, qualify the data as estimated (J/UJ)
- 2. If any compounds initial calibration linearity is <0.95, qualify the data as unusable (R)
- 3. If %RSD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 3. If %D criteria is not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)

## V. Surrogate Recoveries

List surrogate compounds with unacceptable recoveries:

#### **Deviations:**

Sample #	Surrogate ID	% R	QC Limits	Samples Affected
			LIIIIIIS	

#### Actions:

1. If any surrogate recovery is <10%, qualify all positive results in associated fractions as estimated (J)

2. If any surrogate recovery is <10%, qualify all nondetects in associated fractions as unusable (R)

3. If surrogate recoveries fall between 10% and the lower recovery limit, qualify results as estimated (J/UJ)

4. If surrogate recoveries fall above the upper recovery limit, qualify positive results as estimated (J)

6. Use professional judgement to qualify results when surrogate recoveries have been diluted out of spec.

# VI. Blanks

# Laboratory Method Blanks:

	Lab ID #	Fraction	Compound	Conc. (ppb)
		. <u> </u>		
·		. <u> </u>		
<u> </u>				
·				
<u> </u>				
Associated	Project Blanks (e.g.	, equipment rinsates	, trip blanks, etc.)	
Date	Lab ID #	Fraction	Compound	Conc. (ppb)
			Compound	
		. <u> </u>		
·				
·				
·				
·				
·				

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## VI. Blanks (continued)

Calculate action levels based on 5X the highest blank concentration of any given compound Sample weights, volumes, and dilution factors must be taken into account when applying the 5X criteria

#### **Deviations:**

	Maximum Conc.	Action Level (ppb)	Samples Affected
Compound	Detected, (ppb)		

## Actions:

1. If compound results exceed the action levels, the data are not qualified

2. If compound results are below the required reporting level, report results as non-detect (U) at the reporting level

3. If the compound is detected above the reporting level, but below the action level, qualify as not-detected (U)

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## VII. Matrix Spike/Matrix Spike Duplicate Information

General MS/MSD Criteria:

VOC	SVOC
types	types
70-130	45-135
<30	<50

percent recovery (%R) relative percent difference (RPD)

Project Sample(s) Spiked:

## **Deviations:**

Deviations.	0 ( D				
	%R	%R	RPD	RPD	
Compound		Limits		Limits	Samples Affected
	I	1		I	1

#### Actions:

1. If the spike recovery is outside limits, qualify all positive values in the unspiked sample as estimated (J)

2. If the spike recovery is <10%, qualify non-detect values as unusable (R)

3. If the RPD does not meet criteria, qualify positive values in the unspiked sample as estimated (J)

4. Use professional judgement to qualify additional samples in the analytical group based on MS/MSD results

5. Use professional judgement for qualification of data for unspiked compounds

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## VIII. Laboratory Control Sample Information

General LCS Criteria:

VOC	SVOC
types	types
80-120	60-120

Laboratory LCS Identifications:

percent recovery (%R)

#### **Deviations:**

Compound	Date	%R	Samples Affected/Qualifiers Applied
		L	

#### Actions:

1. If the LCS recovery is outside limits but >10%, qualify all positive values as esimated (J)

2. If the LCS recovery is outside limits but >10%, qualify non-detect values as estimated (UJ)

3. If the LCS recovery is <10%, qualify all data for that analyte as unusable (R)

4. Use professional judgement for qualification of data for compounds with no LCS information

# SCIENCE APPLICATIONS INTERNATIONAL CORPORATION Metals Data Review Checklist - Standard Validation

Project:	Page 1 of 13
SDG No:	Analysis:
Laboratory:	Method: Matrix:
	d the analytical quality control/quality assurance performance ia used to assess the analytical integrity of the data were
Case Narrative Analytical Holding Times Sample Preservation Method Calibration Method and Project Blanks LCS Recoveries Project specific QA/QC or contract requirements	MS/MSD Recoveries and Differences Duplicate Relative Percent Differences ICP Serial Dilution Furnace Atomic Absorption QC Re-analysis and Secondary Dilution Internal Standard Performance (if applicable) may take priority over validation criteria in this procedure.
Overall Remarks:	
Definition of Qualifiers: "U", not detected at the asso "UJ", not detected and assoc "J", associated value estimat "R", associated value unusal "=", compound properly iden	ciated value estimated ted ble or analyte identity unfounded
Reviewed by:	Date:
QA Reviewed by:	Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

# II. Re-analysis and Secondary Dilutions

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

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## **III. Holding Times**

Metals - Waters - preserved to pH<2, 180 days from sample collection Metals - Soils - 180 days from sample collection Mercury - Waters - preserved to pH<2, 28 days from sample collection Mercury - Soils - 28 days from sample collection

## **Deviations:**

		Metals				Mercury		
Sample #	Date	Date	Days	pН	Date	Date	Days	рΗ
		Analyzed	>HT	Check	Collected		>HT	Check
						-		

## Actions:

- 1. If preserved samples exceed holding time, qualify all associated results as estimated (J/UJ).
- 2. If unpreserved samples exceed holding time, qualify all associated results as unusable (R).
- 3. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)
- 4. If water samples are not acidified, use professional judgement. Minimally, qualify data as estimated (J) and non-detects unusable (R).
- 5. If soil samples exceed holding time, use professional judgement to qualify data.

## Page 4 of 13

## IV. Initial & Contining Calibration (ICP, GFAA, CVAA, etc.)

Initial calibration linearity criteria is  $r \ge 0.995$ ICV and CCV criteria are <u>+</u> 10% recovery, low level check standard allowed <u>+</u> 30% ICP inter-element check standard criteria <u>+</u> 20%

#### **Deviations:**

		Intial	ICV/		Samples Affected
Element	Date	Calib.	CCV	%R	

#### Actions:

- 1. If any elements initial claibration linearity is <0.995, qualify the data as estimated (J/UJ)
- 2. If any elements initial claibration linearity is <0.95, qualify the data as unusable (R)
- 3a. If any elements ICV or CCV recovery is <90%, qualify the data as estimated (J/UJ)
- 3b. If any elements ICV or CCV recovery is > 110%, qualify results > MDL as estimated (J), do not qualify non-detects
- 4a. If any elements ICV or CCV recovery is <75%, qualify the data as unusable (R)
- 4b. If any elements ICV or CCV recovery is > 125%, qualify positive results as estimated (J) or non-detects unusable (R)
- 4c. If any elements ICV or CCV recovery is > 160%, qualify positive results ≥ MDL us unusable (R). Do not qualify non-detects.
- 5a. If any elements CRI recovery is 50-69% (30-49% for Sb, Pb, Tl), qualify results ≥ MDL (but < 2 x CRQL) as estimated (J/UJ) and results > 2 x CRQL are not qualified.
- 5b.If any elements CRI recovery is < 50% (< 30% for Sb, Pb, Tl), qualify results > MDL (but < 2 x CRQL) as unusable (R) and results > 2 x CRQL as estimate (J).
- 5c. If any elements CRI recovery is > 130% but < 180 % (> 150% but < 200% for Sb, Pb, Tl) quality results > MDL (but < 2

x CRQL) as esimated (J) and non-detects and results > 2 x CRQL are not qualified.

5d. If CRI or (R) > 180% (> 200% for Sb, Pb, Ti), qualify results that are  $\geq$  MDL as unusable (R).

IV. Initial & Contining Calibration (ICP, GFAA, CVAA, etc.) (continued)	Page 5 of 13
Analytical Sequence and MS Tune	(Y/N)
<ol> <li>Were the appropriate number of ICP standards used?</li> <li>Were the appropriate number of AA standards used?</li> <li>Was calibration performed and documented at the beginning of each run?</li> <li>Were calibration check standards run at 10% frequency or every two hours?</li> <li>Were low level standard checks analyzed at approximately 2X the PQL?</li> <li>Was ICP-MS mass calibration within 0.1 AMU?</li> <li>Was ICP-MS % RSD of the absolute signals for all analytes &lt; 5%?</li> </ol>	

# **Deviations:**

Element	Deviation	Samples Affected

## Actions:

- 1. If instrument calibration is questionable, use professional judgement, qualify the data as estimated (J/UJ)
- 2. If instrument calibration documentation can not be obtained or is inadequate, qualify the data as unusable (R)
- 3. If mass calibration for ICP-MS was not within 0.1 AMU, qualify analyte results as estimated (J/UJ).
- 4. If % RSD for ICP-MS was > 5% for any analyte in the tuning solution, qualify associated resuts as estimated (J/UJ).

# V. Blanks (ICB, CCB, Method Blank, Equipment Rinsate Blank)

#### A. Blank Results

If the blank level is > CRQL for any analyte check that the analyte's concentration in a sample is > 10 x the blank value. The highest blank concentration of observed elements is the action level. Sample weights, volumes, and dilution factors must be taken into account when applying the action level. Blank results given in ug/L must be converted to mg/kg to compare them with soil sample results.

use the following equation:

ug/L x V/W x 1L/1000mL x 1000g/1kg x 1mg/1000ug = mg/kg

where:

V = volume of samples digest solution (usually 200 mL) W = weight of sample digested (usually 1 g)

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#### **Deviations:**

		Max. Conc.	Action	Samples Affected
Blank ID	Element	Detected	Level	
	1	<u> </u>		If additional space is required, use next page

#### Actions:

- For blank results 
   <u>></u> MDL but 
   <u>></u> CRQL, qualify sample results 
   <u>></u> MDL but 
   <u>></u> CRQL as CRQL U. Use
   professional judgement to qualify sample results exceeding the CRQL.
- 2a. If blank results are > CRQL: for sample values ≥ MDL but ≤ CRQL, qualify results as CRQL U; for sample values > CRQL but < 10 x the blank, qualify results as unusable (R) or estimated (J). No action is taken for sample results ≥ 10 x the blank values.</p>
- 2b. If ICB/ CCB results are > CRQL: for sample values > MDL but < CRQL, qualify results as CRQL U; for sample

values > CRQL but < blank results, qualify results as not detected (U) at the level of the blank or unusable (R).

Use proffessional judgement for sample results > blank results.

# Page 7 of 13

#### V. Blanks (continued)

The highest blank concentration of observed elements is the action level. Sample weights, volumes, and dilution factors must be taken into account. Blank results given in ug/L must be converted to mg/kg to compare them with soil sample results. use the following equation:

ug/L x V/W x 1L/1000mL x 1000g/1kg x 1mg/1000ug = mg/kg

where:

V = volume of samples digest solution (usually 200 mL) W = weight of sample digested (usually 1 g)

# **Deviations:** Samples Affected Max. Conc. Action Blank ID Element Detected Level

V. Blanks (continued)	Page 8 of 13
B. Frequency Requirements	(Y/N)
<ol> <li>Was a method (preparation) blank analyzed for each matrix?</li> <li>Was a method blank processed for every analytical batch (2)</li> <li>Was a calibration blank analyzed at 10% frequency or every</li> </ol>	0 samples)?
Deviations:	

Element	Deviation	Samples Affected

Remarks:

## C. Baseline Shift Evaluation

List the highest negative blank concentration for each analyte observed in laboratory or project blanks.

#### **Deviations:**

		Max. Neg.	Action	Samples Affected
Blank ID	Element	Conc.	Level	

\_\_\_\_\_

## Actions:

1. If the absolute value of the maximum negative blank result is > the CRQL, qualify positive results as estimated (J) and non-detects as estimated (UJ).

## VI. Laboratory Control Sample Evaluation

All LCS recovery criteria are set at 80-120%

An LCS must be analyzed for each matrix and for each digestion batch or set of twenty samples

## **Deviations:**

Element	Date	%R	Matrix	Samples Affected

# Actions:

1. If any element's LCS recovery is >120%, qualify positive results as (J).

2. If any element's LCS recovery is 50-79%, qualify positive results as (J) and non-detect results as (UJ).

3a. If any element's LCS recovery is <50%, qualify positive results as (J) and non-detect results as (R).

3b. If the LCS recovery is > 150%, qualify all results as unusable (R).

4. For soil LCS recovery > upper limit, qualify sample results > MDL as estimated (J).

5. For soil LCS recovery < lower limit, qualify results  $\geq$  MDL as estimated (J) and non-detects estimated (UJ).

6. Use professional judgement to qualify data if the LCS frequency criteria are not met.

## VII. Matrix Spike Evaluation

All MS recovery criteria are set at 75-125%

An MS must be analyzed for each matrix and for each digestion batch or set of twenty samples Verify that a field blank or PE sample was not used for spiked sample analysis.

Verify that a post-digestion spike was analyzed for those analytes where the pre-digestion spike recovery is outside control limits and the sample result is < 4 x the spike added.

#### Project Sample(s) Spiked:

#### **Deviations:**

	Spiked	Sample	Spike	%R	
	Spiked Sample			7013	
	Sample	Result	Amount		
Element	Result				Samples Affected

## Actions:

1. If the sample concentration exceeds the spiking level by a factor of 4X or more, do not qualify the data

2. If the spike recovery is >125%, qualify all positive values as (J).

- 3. If the spike recovery is between 30-74%, qualify positive values as (J) and non-detect values as estimated (UJ)
- 4. If the spike recovery is <30%, qualify positive values as (J) and non-detects are qualified unusable (R) if the post-digestion spike recovery is < 75% (or none was performed); non-detects are qualified as estimated (UJ) if the post-digestion spike recovery is ≥ 75%. There is no post-digestion spike performed for mercury.</p>
- 5. Qualify all samples of similar matrix to the spiked sample in the same manner
- 6. Use professional judgement to qualify data if the MS frequency criteria are not met.
- 7. Use professional judgement for qualification of data for unspiked elements

## Page 11 of 13

## VIII. Laboratory Duplicate Evaluation

Duplicate relative percent difference (RPD) for water is 20% (both results > 5 times CRDL) or < CRDL difference (if either result is < 5 times CRDL) and RPD for soil is 35% (if both results are > 5 times CRDL or < 2 times CRDL if either result is < 5 times CRDL.

When duplicate sample values are both less than the reporting level they are considered acceptable When duplicate sample values are within 5X the reporting level they are acceptable if their absolute difference is within 3X the reporting level

Verify that a field blank or PE samples was not used for duplicate analysis.

#### **Deviations:**

Element	Sample #	Duplicate #	RPD	Samples Affected

#### Actions:

1. If an element's RPD is >20% (water) / 35% (soil), qualify positive results as (J) and non-detect results as (UJ)

- 2. For low concentrations, if an element's duplicate absolute difference is > 3X the reporting level,
- qualify positive results as (J) and non-detect results as (UJ)

3. Use professional judgement to qualify data if the duplicate frequency criteria are not met.

Page 12 of 13

## IX. Inductively Coupled Plasma (ICP) Serial Dilution Analysis

Verify that a field blank or PE sample was not used for serial dulution. Serial dilution of positive results are performed when values exceed 50X the IDL Results from serial dilutions should agree within 10% of the original undiluted analysis

## **Deviations:**

Element	Sample #	Sample	Serial	%D	Action
		Result	Dilution		
		+			
		<u> </u>			
		1			
					l

#### Actions:

1. If the serial dilution %D is >10 and the analyte results are >50X the IDL, qualify all positive results as estimated (J) and non-detects as estimated (UJ).

		Page 13 of 13
X. Furnace Atomic	Absorption QC	5
A. Duplicate Precisio	on	(Y/N)
2. Were one point a	jections performed for all samples? nalytical spikes performed for all samples? ctions agree within <u>+</u> 20%?	
Deviations:		
Element	Deviation	Sample Affected
	<u> </u>	
A ations:		
1. If duplicate injection	n results are outside <u>+</u> 20%, qualify positive results as (J)	and non-detect results as (UJ)
Actions: 1. If duplicate injection Remarks: B. Post Digestion Sp		and non-detect results as (UJ)
<ol> <li>If duplicate injection</li> <li>Remarks:</li> <li>B. Post Digestion Sp</li> <li>Did post digestion</li> <li>If spike recoveries</li> </ol>		(Y/N) ia? zed by MSA?
<ol> <li>If duplicate injection</li> <li>Remarks:</li> <li>B. Post Digestion Sp</li> <li>Did post digestion</li> <li>If spike recoveries</li> </ol>	pike Recoveries n spike recoveries meet an 85-115% recovery criter s did not meet recovery criteria were samples analy	(Y/N) ia? zed by MSA?
<ol> <li>If duplicate injection</li> <li>Remarks:</li> <li>B. Post Digestion Sp</li> <li>Did post digestion</li> <li>If spike recoveries</li> <li>If MSA was used</li> </ol>	pike Recoveries n spike recoveries meet an 85-115% recovery criter s did not meet recovery criteria were samples analy	(Y/N) ia? zed by MSA?
<ol> <li>If duplicate injection</li> <li>Remarks:</li> <li>B. Post Digestion Sp</li> <li>Did post digestion</li> <li>If spike recoveries</li> <li>If MSA was used</li> <li>Deviations:</li> </ol>	pike Recoveries n spike recoveries meet an 85-115% recovery criter s did not meet recovery criteria were samples analy to analyze samples, was its' correlation coefficient	(Y/N) ia? zed by MSA? 
<ol> <li>If duplicate injection</li> <li>Remarks:</li> <li>B. Post Digestion Sp</li> <li>Did post digestion</li> <li>If spike recoveries</li> <li>If MSA was used</li> <li>Deviations:</li> </ol>	pike Recoveries n spike recoveries meet an 85-115% recovery criter s did not meet recovery criteria were samples analy to analyze samples, was its' correlation coefficient	(Y/N) ia? zed by MSA? 

## Actions:

- 1. If post digestion spike recoveries are >115%, qualify positive results as (J) and non-detect results as (U)
- 2. If post digestion spike recoveries are 11-84%, qualify positive results as (J) and non-detect results as (UJ)
- 3. If post digestion spike recoveries are <10%, qualify positive results as (R) and non-detect results as (R)
- 4. If MSA was used to quantitate values and the correlation coefficient was <0.995, qualify data as (J or UJ)
- 5. If MSA was used to quantitate values and the correlation coefficient was <0.95, qualify data as (R)

Project:		Inorganic Data I	ATIONS INTERNATION Review Checklist - Stan trate/Nitrite, Sulfate, Su	dard Validation
Laboratory:       Method: Matrix:         The above data package has been reviewed and the analytical quality control/quality assurance performance data have been summarized. The general criteria used to assess the analytical integrity of the data were based on an examination of the following:         Case Narrative       Method and Project Blanks         Analytical Holding Times       Method and Project Blanks         Sample Preservation       Duplicate Differences         LCS Recoveries       Re-analysis and Secondary Dilution         Overall Remarks:	Project:			Page 1 of 8
Laboratory:       Matrix:         The above data package has been reviewed and the analytical quality control/quality assurance performance data have been summarized. The general criteria used to assess the analytical integrity of the data were based on an examination of the following:         Case Narrative       Method and Project Blanks         Analytical Holding Times       Method and Project Blanks         Sample Preservation       Duplicate Differences         Method Calibration       LCS Recoveries         Re-analysis and Secondary Dilution         Overall Remarks:	SDG No:			
data have been summarized. The general criteria used to assess the analytical integrity of the data were based on an examination of the following: <ul> <li>Case Narrative Method and Project Blanks Analytical Holding Times Duplicate Differences Sample Preservation Duplicate Differences Re-analysis and Secondary Dilution</li> </ul> Overall Remarks:	Laboratory:			
Analytical Holding Times Sample Preservation Method Calibration       Matrix Spike Recoveries Duplicate Differences LCS Recoveries Re-analysis and Secondary Dilution         Overall Remarks:	data have been su	ummarized. The general criter		
Sample Preservation       Duplicate Differences         LCS Recoveries       Re-analysis and Secondary Dilution         Overall Remarks:			-	S
Pe-analysis and Secondary Dilution         Overall Remarks:		Sample Preservation	Duplicate Differences	
Definition of Qualifiers: "U", not detected at the associated level "U", not detected and associated value estimated "J", associated value estimated "J", associated value estimated "R", associated value estimated "=", compound properly identified and value positive Reviewed by: Date:				ry Dilution
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:	Overall Remarks	S:		
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"U", not detected at the associated level         "UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:       Date:				
"UJ", not detected and associated value estimated         "J", associated value estimated         "R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:	Definition of Quali			
"R", associated value unusable or analyte identity unfounded         "=", compound properly identified and value positive         Reviewed by:		"UJ", not detected and assoc	ciated value estimated	
Reviewed by: Date:		"R", associated value unusal	ole or analyte identity unfound	ed
		"=", compound properly iden	tified and value positive	
QA Reviewed by: Date:	Reviewed by:			Date:
	QA Reviewed by	/:		Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

# **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

## **III. Holding Times**

Sample should be preserved and analyzed according to the appropriate analytical method In general the following preservations and holding times for waters can be applied:

> Sulfate, 4 degress C, 28 days Sulfide, 4 degrees C, pH  $\geq$ 9 with zinc acetate/sodium hydroxide, 7 days Bromide/Chloride/Fluoride, no preservative required, 28 days Nitrate/Nitrite or Ammonia, 4 degrees C, pH  $\leq$  2 with sulfuric acid, 28 days Nitrate or Nitrite, 4 degrees C, 48 hours Alkalinity, 4 degrees C, 14 days TDS/TSS, 4degrees C, 7 days Phosphate (total), 4 degrees C, pH < 2 with sulfuric acid, 28 days Hexavalent Chromium, Cool 4 degress C, water- 24 hours, soil - 30 days

#### **Deviations:**

Sample #	Analyte	Date	Date	Date	Notes:
		Collected	Extracted	Analyzed	

#### Actions:

1. If holding times are exceeded, all results are qualified as estimated (J/UJ)

2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

3. If samples were not properly preserved, use professional judgement to qualify the data

## Page 4 of 8

#### **IV. Initial & Continuing Calibration**

A blank and at least three standards should be analyzed, with one of the standards being within 2X the MDL Correlation coefficients must be  $\geq$  0.995

Initial calibration check recoveries must be within 90-110%

Continuing calibration check recoveries must be within 85-115%

#### **Deviations:**

Compound	Correlation Coefficient	ICV/ CCV	%R	Samples Affected

#### Actions:

1. If any compounds initial calibration linearity is <0.995, qualify the data as estimated (J/UJ)

2. If any compounds initial calibration linearity is <0.95, qualify the data as unusable (R)

3. If ICV or CCV criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)

4. If ICV or CCV recoveries fall below 50%, qualify results as unusable (R)

# V. Blanks (Method Blanks and Project Blanks)

An analytical method blank must be analyzed with each batch of samples Calculate action levels based on 5X the highest blank concentration of any given analyte Sample weights, volumes, and dilution factors must be taken into account when applying the 5X criteria

#### **Deviations:**

	Maximum Conc.	Action Level (ppb)	Samples Affected
Analyte	Detected, (ppb)		

### Actions:

1. If analyte results exceed the action levels, the data are not qualified

2. If analyte results are below the required reporting level, report results as non-detect (U) at the reporting level

3. If the analyte is detected above the reporting level, but below the action level, qualify as not-detected (U)

## Page 6 of 8

## VI. Laboratory Control Sample Information

Each analyte's LCS % recovery must be within the control limits established by the laboratory In general LCS % recoveries should all be within 85-115%

#### **Deviations:**

Analyte	Date	%R	Samples Affected/Qualifiers Applied

#### Actions:

1. If the LCS recovery is outside limits but >10%, qualify all positive values as esimated (J)

- 2. If the LCS recovery is outside limits but >10%, qualify non-detect values as estimated (UJ)
- 3. If the LCS recovery is <10%, qualify all data for that analyte as unusable (R)
- 4. Use professional judgement for qualification of data for compounds with no LCS information

## Page 7 of 8

#### VII. Matrix Spike Information

Each analyte's Matrix Spike % recovery should be within the laboratory established control limits In general matrix spike % recoveries should all be within 75-125%

#### **Deviations:**

	%R	%R	
Analyte		Limits	Samples Affected

#### Actions:

1. If the spike recovery is outside limits, qualify all values in the unspiked sample as estimated (J/UJ)

- 2. If the spike recovery is <10%, qualify non-detect values as unusable (R)
- 3. Use professional judgement to qualify additional samples in the analytical group based on MS results
- 4. Use professional judgement for qualification of data for unspiked analytes

# Page 8 of 8

## VIII. Laboratory Duplicate Information

Each analyte's RPD should be within the laboratory established control limits In general RPDs should all be within 20%

#### **Deviations:**

	RPD	RPD	
Analyte		Limits	Samples Affected

#### Actions:

1. If the RPD is outside limits, qualify all values in the unspiked sample as estimated (J/UJ)

2. Use professional judgement to qualify additional samples in the analytical group based on RPD results

3. Use professional judgement for qualification of data when laboratory duplicates were not analyzed

# SCIENCE APPLICATIONS INTERNATIONAL CORPORATION Radiochemical Data Review Checklist - Standard Validation

Project:			Page 1 of 15
SDG No:		Analysis:	
Laboratory:		Method: Matrix:	
data have been sun	ckage has been reviewed and nmarized. The general criteria nation of the following:		trol/quality assurance performance ytical integrityof the data were
	Case Narrative Analytical Holding Times Sample Preservation Method Calibration Method and Project Blanks	Chemical and/or Trace Matrix Spike Results Duplicate Error Ratios LCS Recoveries Re-analysis and Secor	and RPDs
Overall Remarks:			
	ers: "U", not detected at the assoc "UJ", not detected and associa "J", associated value estimate "R", associated value unusabl "=", compound properly identif	ated value estimated d e or analyte identity unfo	unded
Reviewed by:			Date:
QA Reviewed by:			Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

# **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

# Page 3 of 15

## **III. Holding Times**

General analytical holding time for radionuclides is 6 months Water samples require preservation with nitric acid to pH <2, for dissolved radionuclide determination Radioactive iodine holding time is 7 days Consideration must always be given to the individual radionuclide half-life

#### **Deviations:**

Sample #	Radionuclide:	Date Collected	Date Analyzed	Action

## Actions:

1. If holding times are exceeded, all results are qualified as estimated (J/UJ)

2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

## Page 4 of 15

#### IV. Minimum Detectable Activities (MDAs)/ Reporting Levels

Verify MDAs with project requested reporting levels for all radionuclides Compare reported activities and uncertainties with reported MDAs

#### **Deviations:**

	Project Reporting	MDA	Samples Affected
Radionuclide	Level Goal	Achieved	

#### Actions:

1. Document all radionuclide determinations that do not meet project reporting level goals.

2. If the reported value with its uncertainty encompass the project reporting level goal, they are equivalent.

3. If the sample result is negative and its absolute value exceeds the MDA, qualify the result as estimated (UJ).

4. If the sample result is negative and its absolute value exceeds 2X the MDA, qualify the result ®.

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## V.A1. Calibration Alpha Spectroscopy

Initial efficiency calibration must be demonstrated for each detector. Initial energy calibration must be demonstrated for each detector. Resolution (FWHM) must be demonstrated for each detector. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.A2.Continuing Calibration Alpha Spectroscopy

Continuing calibration efficiency verification must be performed at least quarterly. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Continuing energy calibration must be demonstrated to be within 10% of the initial calibration. Continuing FWHM must be demonstrated to be within 10% of the initial FWHM. A long background count for each detector must be performed weekly or bi-weekly. Pulser counts and demonstration of FWHM for each detector must be demonstrated daily.

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	Detectors Affected		Samples Affected	

#### **Deviations:**

#### Actions:

1. If the initial calibration efficiencies, resolution, or standard information is not acceptable, qualify all affected results as estimated (J).

2. If the continuing calibration efficiency, energy, or FWHM are not acceptable,

qualify all affected results as estimated (J).

3. If background counts or pulser counts are not acceptable, qualify the affected data as estimated (J).

## V.B1. Calibration Gamma Spectroscopy

Initial efficiency calibration must be demonstrated on each detector for each geometry. Initial energy calibration must be demonstrated on each detector for each geometry. Resolution (FWHM) must be demonstrated for each detector for each geometry. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.B2.Continuing Calibration Gamma Spectroscopy

Continuing calibration efficiency verification must be performed for each detector at least quarterly. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Continuing energy calibration must be demonstrated to be within 10% of the initial calibration. Continuing FWHM must be demonstrated to be within 10% of the initial FWHM. A long background count for each detector must be performed monthly. Pulser counts and demonstration of FWHM for each detector must be demonstrated daily.

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	Detectors Affect		Samples Affected	

#### **Deviations:**

#### Actions:

1. If the initial calibration efficiency, energy, resolution, or standard information

is not acceptable, qualify all affected results as estimated (J).

2. If the continuing calibration efficiency, energy, or FWHM are not acceptable, qualify all affected results as estimated (J).

3. If background counts or pulser counts are not acceptable, qualify the affected data as estimated (J).

## Remarks:

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## Page 7 of 15

## V.C1. Calibration Liquid Scintillation Counters

Initial quench curves must be demonstrated for each radionuclide. Initial calibration must be demonstrated for each radionuclide. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.C2. Continuing Calibration Liquid Scintillation Counters

Continuing calibration efficiency verification must be performed afor each radionuclide. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Standards linear regression curve must be performed daily and documentation provided. Control charts for tritium and carbon-14 chi square and figure of merit values should be documented. A background count for each radionuclide window must be provided.

#### **Deviations:**

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	Detectors Affect	Range	Samples Affected	Value

#### Actions:

1. If the initial calibration quench curve or standard information is not acceptable,

- qualify all affected results as estimated (J).
- 2. If the continuing calibration efficiency or control charts are not acceptable, qualify all affected results as estimated (J).
- 3. If background counts are not acceptable, qualify the affected data as estimated (J).

## Page 8 of 15

## V.D1. Calibration Gas Proportional Counters

Initial efficiency calibration must be demonstrated for each detector. Absorption curve must be demonstrated for each detector. Plateau curve performance check must be demonstrated for each detector. Data used to determine alpha and beta cross-talk must be demonstrated. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.D2.Continuing Calibration Gas Proportional Counters

Continuing calibration efficiency verification must be performed at least quarterly. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Cross-talk value for each detector must be documented. Background count for each detector must be performed daily.

#### **Deviations:**

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	Detectors Affect	Range	Samples Affected	Value

#### Actions:

1. If the initial calibration absorption curve, plateau curve, % cross-talk, or standard information is not acceptable, qualify all affected results as estimated (J).

2. If the continuing calibration efficiency or percent cross-talk are not acceptable, qualify all affected results as estimated (J).

3. If background counts are not acceptable, qualify the affected data as estimated (J).

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## VI. Blanks

Review associated laboratory and project blank samples. List documented contamination below:

If the blank result is less than the associated uncertainty (error), no qualification will be warranted. If the blank result is greater than its associated uncertainty, but less than the MDA, then no

qualification will be warrented.

If the blank result is greater than the associated uncertainty and greater than the MDA, then qualification of sample results may be appropriate.

# Laboratory Method Blanks:

Date	Lab ID #	Radionulcide	Result and Error	MDA Result and Error
	Duciant Diamba (a. a.			
Associated	l Project Blanks (e.g.,	equipment rinsat	es, etc.)	
Date	Lab ID #	Radionuclide	Result and Error	MDA Result and Error
Remarks:				

## VI. Blanks (continued)

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Calculate action levels based on 10X the highest blank concentration.

#### **Deviations:**

	Max. Activity	Action Level	Samples Affected
Radionuclide	Detected		

#### Actions:

1. If the blank result falls outside criteria, qualify associated sample results that are less than 10X the blank value as estimated (J).

Example:	Blank Result	Uncert.	MDA or	Normalized absolute	<b>Qualification</b>	
				<u>difference</u>		
acceptable	0.3	0.45	0.5	>2.58	none	
acceptable	0.3	0.25	0.5	1.96 to 2.58	J	
outside criteria	0.3	0.25	0.2	<1.96	J	

2. If the absolute sample result is less than the MDA and the uncertainty is less than the result, qualify as non-detect (U).

3. If the absolute sample results is less than the MDA and the uncertainty is greater than the result, qualify as non-detect value uncertain (UJ).

4. If the sample result is greater than the MDA and the uncertainty is 50-100% of the result, qualify the data as estimated (J).

5. If the sample result is greater than the MDA and the uncertainty is greater than 100% of the result, qualify the data as rejected (R).

4. If the sample result is negative, and its absolute value exceeds 2X the MDA, qualify the data as rejected (R).

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### VII. Sample-Specific Carrier or Tracer Recovery

Sample-specific recoveries must be within limits as demonstrated by the applicable analytical procedures. Generally, recoveries of 30-110% are considered acceptable.

Documentation of traceable tracer solutions (NIST) and dilution documentation must be provided. Spot check sample-specific carrier or tracer recovery calculations.

#### **Deviations:**

			Action Taken
Radionuclide	Sample ID	%R	

#### Actions:

- 1. If recovery is between 30-110%, no qualification is necessary.
- 2. If recovery is between 10-30%, qualify the data as estimated (J).
- 3. If recovery is between 110-150%, qualify the data as estimated (J).
- 4. If recovery is less than 10%, qualify the data as rejected (R).
- 5. If recovery if greater than 150%, qualify the data as rejected (R).

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## VIII. Laboratory Control Sample Information

General LCS Criteria:	aqueous	solid
percent recovery (%R)	80-120	70-130

Laboratory LCS Identifications:

**Deviations:** 

Radionuclide	Date	%R	Samples Affected/Qualifiers Applied

#### Actions:

Aqueous	<u>&lt;50%</u>	<u>50-79%</u>	<u>121-150%</u>	<u>&gt;150%</u>
	R	J	J	R
Solid	<u>&lt;40%</u>	<u>40-69%</u>	<u>131-160%</u>	>160%
	R	J	J	R

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# IX. Matrix Spike Information

General MS Criteria:	Aqueous	Solid
percent recovery (%R)	50-120	40-130

Project Sample(s) Spiked:

**Deviations:** 

Radionuclide	Date	%R	Samples Affected/Qualifiers Applied

Aq	ueous	<u>&lt;20%</u> R	<u>20-49%</u> J	<u>121-160%</u> J	<u>&gt;160%</u> use professional judgement
	Solid	<u>&lt;10%</u> R	<u>10-39%</u> J		>160% use professional judgement
Remarks:					

## Page 14 of 15

## X. Duplicate Sample or Matrix Spike Duplicate Analysis

Identify the method utilized to evaluate duplicate analyses; duplicate error ration (DER), relative percent difference (RPD), or relative error ratio (RER). Duplicate actions should apply to all samples associated with the duplicate pair.

Duplicate Sample Identification:

## **Deviations:**

				Samples Affected
Radionuclide	DER	RPD	RER	

#### Actions:

1. If both sample and duplicate activities are within 2X the MDA comparison is acceptable.

- 2. If the DER is greater than 1.00, qualify the data as estimated (J).
- 3. If the RPD is greater than 50% qualify the data as estimated (J).
- 4. If one sample is <MDA and the other sample is >2X the MDA, qualify the data as estimated (J).

## XI. Overall Assessment of Data

It is appropriate for the data reviewer to make professional judgements and express concerns regarding the validity of the data, overall. This is particularly appropriate when there are several citeria outside the desired specifications. The additive nature of these factors may present data that needs to be further qualified beyond each individual qualification. The reviewer should summarize these concerns.

#### Actions:

1. Qualified data must be accompanied by all individual reason codes related to the qualification assigned.

2. If the sample result has been qualified for multiple reasons, the reviewer will use professional

judgement to determine if multiple estimations warrants rejection (R).

#### **Remarks:**

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# SCIENCE APPLICATIONS INTERNATIONAL CORPORATION Organic Data Review Checklist - Comprehensive Validation

Project:			Page 1 of 14
SDG No:		Analysis:	
Laboratory:		Method: Matrix:	
data have been su	ckage has been reviewed and t mmarized. The general criteria ination of the following:		trol/quality assurance performance ytical integrityof the data were
	Case Narrative Analytical Holding Times Sample Preservation Method Calibration Method and Project Blanks	Analytical Surrogate R Internal Standard Perfor MS/MSD Recoveries a LCS Recoveries Re-analysis and Secor	ormance and Differences
Project Sepcific QA	A/QC or contrqact requirements	may take priority over va	alidatin criteria in this procedure.
Overall Remarks	:		
Definition of Qualifi	iers: "U", not detected at the associa "UJ", not detected and associa "J", associated value estimated "R", associated value unusable "=", compound properly identifi	ted value estimated d e or analyte identity unfo	unded
Reviewed by:			Date:
QA Reviewed by	:		Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

## **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

## **III. Holding Times**

VOC - Waters - unpreserved: aromatic within 7 days, non-aromatic within 14 days of sample collection VOC - Waters - preserved: aromatic and non-aromatic within 14 days of sample collection VOC - Soils - preserve or analyze within 48 hours of sample collection; analyze within 14 days of preservation

SVOC, Pest., PCB - Waters - extract within 7 days of sample collection, analyze within 40 days of extraction SVOC, Pest., PCB - Soils - extract within 14 days of sample collection, analyze within 40 days of extraction

#### **Deviations:**

	VOC		SVOC			Pest/PCB		
Sample #			Dete		Data	<b>i</b>		
Sample #	Date	Date	Date	Date	Date	Date	Date	Date
	Collected	Analyzed	Collected	Extracted	Analyzed	Collected	Extracted	Analyzed
L								
					1			

#### Actions:

- 1. If holding times are exceeded, all results are qualified as estimated (J/UJ)
- 2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

## Page 4 of 14 IV. System Monitoring Compound (SMC) Recoveries (VOC, SVOC, Pesticides, PCBs)

Note: SMC formerly known as surrogates.

List SMC compounds with unacceptable recoveries:

## **Deviations:**

		VOC			SVOC			SVOC		Pest	PCB
Sample #	TO			B/N	Compou	unds	Acic		unds	TOY	DOD
	TOL	BFB	DCE	NBZ	FBP	TPH	PHL	2FP	TBP	ТСХ	DCB
	1										
QC	1										
Limits											

#### Actions:

1. If any SMC recovery is <10%, qualify all positive results in associated fractions as estimated (J)

2. If any SMC recovery is <10%, qualify all nondetects in associated fractions as unusable (R)

3. If SMC recoveries fall between 10% and the lower recovery limit, qualify results as estimated (J/UJ)

4. If SMC recoveries fall above the upper recovery limit, qualify positive results as estimated (J)

5. Use professional judgement to qualify Pest/PCB results when SMC recoveries are >10%

6. Use professional judgement to qualify results when SMC recoveries have been diluted out of spec.

7. For SVOC, qualification of the data is required only when 2 or more SMC per fraction are not within control limits.

## Page 5 of 14

## V. Internal Standards Performance (VOC, SVOC)

VOC internal standard area counts within -50% to +100% of standard (Y/N) VOC internal standard retention times within  $\pm$  30 seconds of standard (Y/N)

SVOC internal standard area counts within -50% to +100% of standard (Y/N) SVOC internal standard retention times within + 30 seconds of standard (Y/N)

#### **Deviations:**

	IS	Area	Acceptable	RT	Std. RT
Sample #	Affected	Counts	Range		Value

#### Actions:

1. If area counts are outside limits, qualify positive results associated with that IS as estimated (J)

2. Non-detected compounds quantitated using an IS area count >100% should not be qualified

3. Non-detected compounds quantitated using an IS area count <50%, qualify as estimated (UJ)

4. If extremely low area counts are reported (<50% of the lower limit), qualify non-detects as unusable (R)

5. If an IS retention time varies more than 30 seconds, review the chromatographic profile for shifts and irregularities. Use professional judgement to qualify the data estimated (J/UJ) or unusable (R)

Page 6 of 14

## VI. Blanks

All blanks were reported per matrix per concentration level for each 12 hour period on each GC/Ms system used to analyte VOC and SVOC. Yes / No

Review associated laboratory and project blank samples. List documented contamination below: Laboratory Method Blanks:

# Fraction Compound Date Lab ID # Conc. (ppb) Associated Project Blanks (e.g., equipment rinsates, trip blanks, etc.) Lab ID # Fraction Compound Conc. (ppb) Date **Remarks:**

## VI. Blanks (continued)

Calculate action levels based on 10X the highest blank concentration of "common laboratory solvents", VOCs (methylene chloride, acetone, toluene, 2-butanone cyclohexane) or SVOCs (phthalates), and 5X the highest blank concentration for all other VOC, SVOC, Pesticides, and PCB compounds. Sample weights, volumes, and dilution factors must be taken into account when applying the 5X and 10X criteria. This allows the total amount of contaminant present to be considered.

#### **Deviations:**

	Maximum Conc.	Action Level (ppb)	Samples Affected
Compound	Detected, (ppb)		

#### Actions:

- 1. If compound results exceed the action levels, the data are not qualified
- 2. If compound results are below the required reporting level, report results as non-detect (U) at the reporting level
- 3. If the compound is detected above the reporting level, but below the action level, qualify as not-detected (U)
- 4. If gross contamination exists in blanks (i.e., saturated peaks by GC/MS), all affected compounds in the associated samples should be qualified as unusable (R) due to interference.
- 5. If blanks were not analyzed per matrix per concentration level, for each 12 hour period on each GC/MS system used to analyze Vocs and SVOCs, use professional judgement to qualify data. Data may be rejected (R).

/II Initial & Contining	Calibration					Page 8 of 14
/II. Initial & Contining GC/MS instrument perforr Il compounds must have	mance checks	s (BFB or	DFTPP) acc		es 🗌	No 🗌
		, , ,	<u> </u>	<u>, , , , , , , , , , , , , , , , , , , </u>		
/OC - Date of initial cal /OC - Date(s) of contin Vas the 12 hour critieria	uing calibrat					
SVOC- Date of initial calib SVOC - Date(s) of conti		ation:				
Vas the 12 hour critieri	-					
Deviations:						
Compound	Date	RRF	%RSD	%D		Samples Affected
Data may be rejected (	n intial or cont %RSD >30 o %RSD >40 o signment/ ION ed outside the sestimated (J, r water and so R).	tinuing RF or a $\%$ D >2 abundance > 12 hour /UJ). bil were no	RF of < 0.01 25, qualify p 40, qualify n e critieria are i BFB or DFT ot performed hour criteric	, qualify no positive resu pon-detects n error, qual PP perform I, use profe	n-detects a ilts as estim as estimate lify all associ- nance chec essional jude all associate	s unusable (R) nated (J) ed (UJ) ated data as unusable (R).

VIII. Initial & Continuing Calibration (Pesticides, PCBs)	Page 9 of 14
Linearity evaluation, are %RSD <20?	Yes or No
Is the RPD between calibration factors <25? (Y/N)	Yes or No
Are multicomponent calibration data provided for each analysis date?	Yes or No
Is the difference between columns check $\leq$ 25%D?	Yes or No
Are 4, 4' - DDT and Endrin Breakdown (PEM) < 20%	Yes or No
And Combined breakdown < 30 (Y ? N)	Yes or No

## **Deviations:**

Compound	% RSD	RPD	Samples Affected

\* % Difference = (( $RF_{CCV}$  -  $RF_{ICAL AVG}$ )/ $RF_{ICAL AVG}$ ) x 100. In instances where the bias of the CCV impacts

validation qualifiers, review the RF values or amount reported to confirm that the % Difference or %

Drift are reported with the correct negative or positive value.

Actions:

- 1. If %RSD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 2. If RPD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 3. If %D criteria is not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 4. If breakdown criteria are not met, positive 4, 4'-DDT and Endrin should be qualified as estimated (J) and non- detects should be rejected (R).

## Page 10 of 14

## IX. Matrix Spike/Matrix Spike Duplicate Information

eneral MS/MSD Criteria:	VOC	SVOC	Pest	PCB
percent recovery (%R)	70-130	45-135	40-140	40-140
relative percent difference (RPD)	<30	<50	<50	<50

Project Sample(s) Spiked:

#### **Deviations:**

General

	%R	%R	RPD	RPD	
Compound		Limits		Limits	Samples Affected

## Actions:

- 1. If the spike recovery is above the upper control limit (UCL), qualify all positive values in the unspiked sample as estimated (J)
- 2. If the spike recovery is below the lower control limit (LCL), qualify positive valves as estimated (J). and non detects as estimated (UJ) in the unspiked sample.
- 3. If the spike recovery is <10%, qualify non-detect values as unusable (R)
- 4. If the RPD does not meet criteria, qualify positive values in the unspiked sample as estimated (J)
- 5. Use professional judgement to qualify additional samples in the analytical group based on MS/MSD results
- 6. Use professional judgement for qualification of data for unspiked compounds

## Page 11 of 14

#### X. Laboratory Control Sample Information

General LCS Criteria:	VOC	SVOC	Pest	PCB
percent recovery (%R)	80-120	60-120	50-130	50-130

Laboratory LCS Identifications:

**Deviations:** 

Compound	Date	%R	Samples Affected/Qualifiers Applied

#### Actions:

Action should be based on both the number of compounds outside the criterion and the magnitude of the exceedance.

- 1. If the LCS recovery is below limits but > one-half the lower limit, qualify values as estimated (J/UJ).
- 2. If the LCS recovery is < one-half the lower limit, qualify all non-detect values for the analyte as unusable (R). and all positive values for that analyte as estimated (J).
- 3. If the LCS recovery is greater than the upper limit, qualify positive values for that analyte as estimated (J).
- 4. If more than half the compounds in the LCS are not within recovery criteria, then qualify associated detected compounds as estimated (J).
- 5. Use professional judgement for qualification of data for compounds with no LCS information

Page 12 of 14 **XI. Identification Check** Are compound retention time (RT) windows confirmed and correct? Are individual mass spec. ion spectra confirmed and appropriate? **Deviations:** Compound RT Ion Spec Samples Affected

#### Actions:

- 1. Use professional judgement to qualify data if RT windows are exceeded.
- 2. Use professional judgement to qualify data if peak shape (i.e. tailing or splitting) is impacted.
- 3. Use professional judgement to qualify data if analyte ion spectra are compromised.

# Page 13 of 14

## XII. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Confirm appropriate instrument and manual peak integration. Confirm calculation of reported results for at least 10% of the data set.

## **Calculation Check:**

Analyte:	Method:	
Pamarka		
Remarks:		
Calculation Check:		
Analyte:	Method:	
	Method:	
Analyte: Remarks:	Method:	

# Page 14 of 14

## XII. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Confirm appropriate instrument and manual peak integration. Confirm calculation of reported results for at least 10% of the data set.

## **Calculation Check:**

Analyte:	Method:	
Remarks:		
Calculation Check:		
Calculation Check: Analyte:	Method:	
Analyte:	Method:	
Calculation Check:         Analyte:         Background         Remarks:	Method:	

	GC and LC Organic Data		NAL CORPORATION Comprehensive Validation RO, Methanol, etc.)
Project:			Page 1 of 12
SDG No:		Analysis: Method:	
Laboratory:		Matrix:	
data have been s	backage has been reviewed and ummarized. The general criteria mination of the following:		rol/quality assurance performance /tical integrityof the data were
	Case Narrative Analytical Holding Times	Analytical Surrogate Re	
	Sample Preservation Method Calibration Method and Project Blanks	LCS Recoveries Re-analysis and Secon	
Overall Remark	s:		
	10		
Definition of Qual	ifiers: "U", not detected at the assoc "UJ", not detected and associ "J", associated value estimate "R", associated value unusab "=", compound properly identi	ated value estimated ed le or analyte identity unfou	unded
Reviewed by:			Date:
QA Reviewed b	y:		Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

## **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

## **III. Holding Times**

VOC types - Waters - unpreserved: aromatic within 7 days, non-aromatic within 14 days of sample collection VOC types - Waters - preserved: aromatic and non-aromatic within 14 days of sample collection VOC types - Soils - preserve/analyze within 48 hours of sample collection; analyze within 14 days of preservation

SVOC types - Waters - extract within 7 days of sample collection, analyze within 40 days of extraction SVOC types - Soils - extract within 14 days of sample collection, analyze within 40 days of extraction

#### **Deviations:**

	VOC	types	S	VOC type	es	Notes:
Sample #	Date	Date	Date	Date	Date	
	Collected	Analyzed	Collected	Extracted	Analyzed	

#### Actions:

- 1. If holding times are exceeded, all results are qualified as estimated (J/UJ)
- 2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

## Page 4 of 12

## **IV. Initial & Continuing Calibration**

A blank and five standards should be analyzed, with one of the standards being within 2X the MDL Correlation coefficients must be  $\geq 0.995$ 

The RSD of the calibration factor or the relative response factor (RRF) must be  $\leq 20\%$ Continuing calibration %D must be within + 15%

#### **Deviations:**

Compound	Correlation Coefficient	% RSD	%D	Samples Affected

\* % Difference = ((RFCCV - RFICAL AVG)/RFICAL AVG) x 100. In instances where the bias of the CCV impacts validation qualifiers, review the RF values or amount reported to confirm that the % Difference or % Drift are reported with the correct negative or positive value.

#### Actions:

1. If any compounds initial calibration linearity is <0.995, qualify the data as estimated (J/UJ)

2. If any compounds initial calibration linearity is <0.95, qualify the data as unusable (R)

3. If %RSD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)

3. If %D criteria is not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)

## V. Surrogate Recoveries

List surrogate compounds with unacceptable recoveries:

#### **Deviations:**

Sample #	Surrogate ID	% R	QC Limits	Samples Affected
			Linito	

#### Actions:

1. If any surrogate recovery is <10%, qualify all positive results in associated fractions as estimated (J)

2. If any surrogate recovery is <10%, qualify all nondetects in associated fractions as unusable (R)

3. If surrogate recoveries fall between 10% and the lower recovery limit, qualify results as estimated (J/UJ)

4. If surrogate recoveries fall above the upper recovery limit, qualify positive results as estimated (J)

6. Use professional judgement to qualify results when surrogate recoveries have been diluted out of spec.

## VI. Blanks

Review associated laboratory and project blank samples. List documented contamination below:

# Laboratory Method Blanks:

Date	Lab ID #	Fraction	Compound	Conc. (ppb)
		<u> </u>		
ssociated	l Project Blanks (e.g.,	equipment rinsates	trip blanks, etc.)	
ate	Lab ID #	Fraction	Compound	Conc. (ppb)
		<u> </u>		
		<u> </u>		
		<u> </u>		
emarks:				
Remarks:				

## VI. Blanks (continued)

Calculate action levels based on 5X the highest blank concentration of any given compound Sample weights, volumes, and dilution factors must be taken into account when applying the 5X criteria

#### **Deviations:**

	Maximum Conc.	Action Level (ppb)	Samples Affected
Compound	Detected, (ppb)		

## Actions:

1. If compound results exceed the action levels, the data are not qualified

2. If compound results are below the required reporting level, report results as non-detect (U) at the reporting level

3. If the compound is detected above the reporting level, but below the action level, qualify as not-detected (U)

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#### VII. Matrix Spike/Matrix Spike Duplicate Information

General MS/MSD Criteria:

VOC	SVOC
types	types
70-130	45-135
<30	<50

relative percent difference (RPD)

Project Sample(s) Spiked:

percent recovery (%R)

## **Deviations:**

Deviations.		-		-	
	%R	%R	RPD	RPD	
Compound		Limits		Limits	Samples Affected
			1	J	1

#### Actions:

1. If the spike recovery is outside limits, qualify all positive values in the unspiked sample as estimated (J)

2. If the spike recovery is <10%, qualify non-detect values as unusable (R)

3. If the RPD does not meet criteria, qualify positive values in the unspiked sample as estimated (J)

4. Use professional judgement to qualify additional samples in the analytical group based on MS/MSD results

5. Use professional judgement for qualification of data for unspiked compounds

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## VIII. Laboratory Control Sample Information

General LCS Criteria:

VOC	SVOC
types	types
80-120	60-120

Laboratory LCS Identifications:

percent recovery (%R)

#### **Deviations:**

Compound	Date	%R	Samples Affected/Qualifiers Applied
·			

#### Actions:

1. If the LCS recovery is outside limits but >10%, qualify all positive values as esimated (J)

2. If the LCS recovery is outside limits but >10%, qualify non-detect values as estimated (UJ)

3. If the LCS recovery is <10%, qualify all data for that analyte as unusable (R)

4. Use professional judgement for qualification of data for compounds with no LCS information

#### Actions:

- 1. Use professional judgement to qualify data if RT windows are exceeded.
- 2. Use professional judgement to qualify data if peak shape (i.e. tailing or splitting) is impacted.
- 3. Use professional judgement to qualify data if analyte ion spectra are compromised.

## Page 11 of 12

## XII. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Confirm appropriate instrument and manual peak integration. Confirm calculation of reported results for at least 10% of the data set.

## **Calculation Check:**

Analyte:	Method:	
Remarks:		
Calculation Check:		
Calculation Check: Analyte:	Method:	
Analyte:	Method:	
Calculation Check:         Analyte:         Background         Remarks:	Method:	

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## XII. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Confirm appropriate instrument and manual peak integration. Confirm calculation of reported results for at least 10% of the data set.

## **Calculation Check:**

Analyte:	Method:	
Pamarka.		
Remarks:		
Calculation Check:		
Analyte:	Method:	
	Method:	
Analyte: Remarks:	Method:	

# SCIENCE APPLICATIONS INTERNATIONAL CORPORATION Metals Data Review Checklist - Comprehensive Validation

Project:		Page 1 o	of 16
SDG No:		Analysis: Method:	
Laboratory:		Matrix:	
data have been su		al quality control/quality assurance performances the analytical integrity of the data were	ce
	Analytical Holding TimesDuplicateSample PreservationICP SerialMethod CalibrationFurnace AMethod and Project BlanksRe-analysLCS RecoveriesInternal Second	Ntomic Absorption QC is and Secondary Dilution tandard Performance (if applicable)	
Project specific QA	/QC or contract requirements may take price	prity over validation criteria in this proceudre.	
Overall remarks	<u>.</u>		
Definition of Qualif			
	"U", not detected at the associated level "UJ", not detected and associated value es "J", associated value estimated "R", associated value unusable or analyte "=", compound properly identified and value	identity unfounded	
Reviewed by:		Date:	_
QA Reviewed by	:	Date:	-

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

## **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

Page 3 of 16

## **III. Holding Times**

Metals - Waters - preserved to pH<2, 180 days from sample collection Metals - Soils - 180 days from sample collection Mercury - Waters - preserved to pH<2, 28 days from sample collection Mercury - Soils - 28 days from sample collection

## **Deviations:**

		Metals				Mercury		
Sample #	Date Collected	Date Analyzed	Days >HT	pH Check	Date Collected	Date Analyzed	Days >HT	pH Check
	Concolod	Analyzeu	2111	Oneek	Concerca	Analyzeu	2111	Oneok

## Actions:

- 1. If preserved samples exceed holding time, qualify all associated results as estimated (J/UJ).
- 2. If unpreserved samples exceed holding time, qualify all associated results as unusable (R).
- 3. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)
- 4. If water samples are not acidified, use professional judgement. Minimally, qualify data as estimated (J) and non-detects unusable (R).
- 5. If soil samples exceed holding time, use professional judgement to qualify data.

## Page 4 of 16

## IV. Initial & Contining Calibration (ICP, GFAA, CVAA, etc.)

Initial calibration linearity criteria is  $r \ge 0.995$ ICV and CCV criteria are <u>+</u> 10% recovery, low level check standard allowed <u>+</u> 30% ICP inter-element check standard criteria <u>+</u> 20%

#### **Deviations:**

		Intial	ICV/		Samples Affected
Element	Date	Calib.	CCV	%R	

#### Actions:

- 1. If any elements initial claibration linearity is <0.995, qualify the data as estimated (J/UJ)
- 2. If any elements initial claibration linearity is <0.95, qualify the data as unusable (R)
- 3a. If any elements ICV or CCV recovery is <90%, qualify the data as estimated (J/UJ)
- 3b. If any elements ICV or CCV recovery is > 110%, qualify results  $\geq$  MDL as estimated (J).

Do not qualify non-detects.

- 4a. If any elements ICV or CCV recovery is <75%, qualify the data as unusable (R)
- 4b. If any elements ICV or CCV recovery is > 125% qualify positive results as estimated (J) or non-detects as unusable (R).
- 4c. If any element ICV or CCV recovery is ≥ 160%, qualify positive results ≥ MDL as unusable (R). Do not qualify non-detects.
- 5a. If any elements CRI recovery is 50 69% (30 49% for Sb, Pb, Tb), qualify results ≥ MDL (but < 2 times CRQL) as estimated (UJ) and results > 2 times CRQL are not qualified.
- 5b. If any elements CRI recovery is < 50% (<30% for Sb, Pb, Tl), qualify results ≥ MDL (but < 2 times CRQL) and non-detects unusable (R). Results > 2 times CRQL are estimated (J).
- 5c. If any elements CRI recovery is > 130% but < 180% (>150% but < 200% for Sb, Pb, Tl), qualify results
- $\geq$  MDL (but < 2 times CRQL) as esimated (J). And non-detects and results  $\geq$  the CRQL are not qualified.
- 5d. If CRI or(R) > 180% (> 200% for Sb, Pb, Ti), qualify results that are  $\geq$  MDL as unusable (R).

	IV. Initial & Contining C	alibration (ICP, GFAA, CVAA, etc.) (c	Page 5 of 16 ontinued)				
Analytical Sequence and MS Tune (Y/N							
1. Were the appropriate number of ICP standards used?							
	Element	Deviation	Samples Affected				

## Actions:

- 1. If instrument calibration is questionable, use professional judgement, qualify the data as estimated (J/UJ)
- 2. If instrument calibration documentation can not be obtained or is inadequate, qualify the data as unusable (R)
- 3. If mass calibration for ICP-MS was not within 0.1 AMU qualify analyte results as estimated (J/UJ)
- 4. If % RSD for ICP-MS was > 5% for any analyte in the tuning solution, qualify associated results as estimated (J/UJ).

## **Results:**

# V. Blanks (ICB, CCB, Method Blank, Equipment Rinsate Blank)

#### A. Blank Results

If the blank level is > CRQL for any analyte, check that the analyte's concentration in the samples is > 10 times the blank value. The highest blank concentration of observed elements is the action level. Sample weights, volumes, and dilution factors must be taken into account when applying the action level. Blank results given in ug/L must be converted to mg/kg to compare them with soil sample results.

use the following equation:

ug/L x V/W x 1L/1000mL x 1000g/1kg x 1mg/1000ug = mg/kg

where: V = volume of samples digest solution (usually 200 mL) W = weight of sample digested (usually 1 g)

#### **Deviations:**

Blank ID	Element	Max. Conc.		Samples Affected
		Detected	Level	

If additional space is required, use next page

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#### Actions:

- For blank results ≥ MDL but ≤ CRQL, qualify samples ≥ MDL but < CRQL as CRQL U. Use profession judgement to qualify sample results exceeding the CRQL.
- 2a. If blank results are > CRQL: For sample values > MDL but < CRQL, qualify results as CRQL U; for sample values > CRQL but < 10 times the blank qualify results as unusable (R) or estimated (J). No action is taken for sample results > 10 times the blank levels.

2b. If ICB/ CCB results are > CRQL; for sample values > MDL but < CRQL, qualify results as CRQL U; for

sample values > CRQL but < blank results, qualify results as not detected (U) at the level of the blank or

unusable (R). Use professional judgement for sample results > blank results.

# Page 7 of 16

## V. Blanks (continued)

The highest blank concentration of observed elements is the action level.

Sample weights, volumes, and dilution factors must be taken into account when applying the 5X criteria. Blank results given in ug/L must be converted to mg/kg to compare them with soil sample results. use the following equation:

ug/L x V/W x 1L/1000mL x 1000g/1kg x 1mg/1000ug = mg/kg

where:

V = volume of samples digest solution (usually 200 mL) W = weight of sample digested (usually 1 g)

# **Deviations:** Samples Affected Max. Conc. Action Blank ID Element Detected Level

V. Blanks (continued)	Page 8 of 16
B. Frequency Requirements	(Y/N)
<ol> <li>Was a method (preparation) blank analyzed for each matrix?</li> <li>Was a method blank processed for every analytical batch (20 sa</li> <li>Was a calibration blank analyzed at 10% frequency or every two</li> </ol>	

Element	Deviation	Samples Affected

Remarks:

## C. Baseline Shift Evaluation

List the highest negative blank concentration for each analyte observed in laboratory or project blanks.

#### **Deviations:**

		Max. Neg.	Action	Samples Affected
Blank ID	Element	Conc.	Level	

## Actions:

1. If the absolute value of the maximum negative blank result is > the CRQL, qualify positive results as estimated (J) and non-detects as estimated (UJ).

## VI. Laboratory Control Sample Evaluation

All LCS recovery criteria are set at 80-120%

An LCS must be analyzed for each matrix and for each digestion batch or set of twenty samples

#### **Deviations:**

Element	Date	%R	Matrix	Samples Affected

#### Actions:

- 1. If any element's LCS recovery is >120%, qualify positive results as (J).
- 2. If any element's LCS recovery is 50-79%, qualify positive results as (J) and non-detect results as (UJ)
- 3a. If any element's LCS recovery is <50%, qualify positive results as (J) and non-detect results as (R)
- 3b. If the LCS recovery is > 150%, qualify all results as unusable (R).
- 4. For soil LCS recovery > upper limit, qualify samples results > MDL as estimated (J).
- 5. For soil LCS recovery < lower limit, qualify results > MDL as esimated (J) and non-detected estimated (UJ).
- 6. Use professional judgement to qualify data if the LCS frequency criteria are not met.

## Page 10 of 16

## VII. Matrix Spike Evaluation

All MS recovery criteria are set at 75-125%

An MS must be analyzed for each matrix and for each digestion batch or set of twenty samples Verify that a field blank or PE sample was not used for spiked sample analysis

Verify that a post-digestion was analyzed for those anlytes where the pre-digestion spike recovery is outside control limits and the sample result is < 4 times the spike added.

Project Sample(s) Spiked:

#### **Deviations:**

	Spiked Sample	Sample Result	Spike Amount	%R			
Element	Result				Sar	mples Affec	ted

## Actions:

1. If the sample concentration exceeds the spiking level by a factor of 4X or more, do not qualify the data

2. If the spike recovery is >125%, qualify all positive values as (J).

3. If the spike recovery is between 30-74%, qualify positive values as (J) and non-detect values as estimated (UJ)

4. If the spike recovery is <30%, qualify positive values as (J) and non-detects are qualified unusable (R)

if the post-digestion spike recovery is < 75% (or none were performed); non-detects are qualified as estimated (UJ) If the post-digestion spike recovery is  $\geq$  75%. There is no post-digestion spike performed for mercury.

5. Qualify all samples of similar matrix to the spiked sample in the same manner

6. Use professional judgement to qualify data if the MS frequency criteria are not met.

7. Use professional judgement for qualification of data for unspiked elements

## Page 11 of 16

## VIII. Laboratory Duplicate Evaluation

Duplicate relative percent difference (RPD) for water is 20% (both results > 5 times CDRL) or < CRDL difference (if either result is < 5 times CRDL) and RPD for soil is 35% (if both results are > 5 times CRDL or < 2 times CRDL if either result is < 5 times CRDL).

When duplicate sample values are both less than the reporting level they are considered acceptable When dupicate sample values are within 5X the reporting level they are acceptable if their absolute difference is within 3X the reporting level

Verify that a field blank on PE sample was not used or duplicate analysis.

# **Deviations:**

Element	Sample #	Duplicate #	RPD	Samples Affected	

#### Actions:

- 1. If an element's RPD is >20% (water) / >35% (soil), qualify positive results as (J) and non-detect results as (UJ)
- 2. For low concentrations, if an element's duplicate absolute difference is > 3X the reporting level,

\_\_\_\_\_

- qualify positive results as (J) and non-detect results as (UJ)
- 3. Use professional judgement to qualify data if the duplicate frequency criteria are not met.

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## IX. Inductively Coupled Plasma (ICP) Serial Dilution Analysis

Verify that a field blank or PE sample was not used for serial dilution. Serial dilution of positive results are performed when values exceed 50X the IDL Results from serial dilutions should agree within 10% of the original undiluted analysis

#### **Deviations:**

Element	Sample #	Sample	Serial	%D	Action
		Result	Dilution		

# Actions:

1. If the serial dilution %D is >10 and the analyte results are >50X the IDL, qualify all positive results as estimated (J) and non-detects as estimated (UJ).

X. Furnace Atomic Abs	oration QC	Page 13 o	f 16
A. Duplicate Precision			(Y/N)
1. Were duplicate injection	ons performed for all samples? ical spikes performed for all samples? s agree within <u>+</u> 20%?		
Deviations:			
Element	Deviation	Sample Affe	cted
Remarks:	Its are outside <u>+</u> 20%, qualify positive result		
B. Post Digestion Spike F	Recoveries		(Y/N)
<ol> <li>If spike recoveries did</li> <li>If MSA was used to an</li> </ol>	ke recoveries meet an 85-115% recover not meet recovery criteria were sample nalyze samples, was its' correlation coef	s analyzed by MSA?	
Deviations: Element	Deviation	Sample Affe	ected

## Actions:

- 1. If post digestion spike recoveries are >115%, qualify positive results as (J) and non-detect results as (U)
- 2. If post digestion spike recoveries are 11-84%, qualify positive results as (J) and non-detect results as (UJ)
- 3. If post digestion spike recoveries are <10%, qualify positive results as (R) and non-detect results as (R)
- 4. If MSA was used to quantitate values and the correlation coefficient was <0.995, qualify data as (J or UJ)
- 5. If MSA was used to quantitate values and the correlation coefficient was <0.95, qualify data as (R)

## Page 14 of 16 XI. Inductively Coupled Plasma (ICP) Interference Check Sample Evaluation

Interference check samples should be analyzed at the beginning and end of each analysis run, or at a minimum of twice per 8 hour working shift.

Results for the ICS solution AB must fall within control limits of 20% for analytes included in the solution. Evaluate the ICS A solution raw data for results with an absolute value  $\geq$  MDL for analytes that are not present in the ICS A solution.

#### **Deviations:**

Element	Sample #	Sample	Interferent	Action
		Result	Result	

## Actions:

- 1. If the ICS AB %R for an analyte is > 120%, qualify sample results ≥ MDL as estimated (J) and non-detects should not be qualified.
- 2. If the ICS AB %R for an analytes is 50-79%, qualify sample results that are <u>></u> MDL as estimated (J) and non-detects as estimated (UJ).
- 3. If the ICS AB %R for an analyte is <50%, qualify all sample results that are > MDL and all non-detects as as unusable (R).
- 4. If results <u>></u> MDL are found for analytes not present in the ICS A solution, then in samples with comparable or higher levels of interferents and with analyte concentration that approximate those levels in the ICS A, sample results <u>></u> MDL should be qualified as estimated (J) and non-detects should not be qualified.
- 5. If negative results with absolute values > MDL are found for analytes not present in the ICS A solution, then in samples with comparable or higher levels of interferents, affected sample results > MDL should be qualified as estimated (J) and non-detects (UJ).

# XII. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Analyte identification should be confirmed in the original data output. Confirm calculation of reported results for at least 10% of the data set.

# **Calculation Check:**

Analyte:	Method:	
Remarks:		
Calculation Check: Analyte:	Method:	
Remarks <u>:</u>		
Remarks <u>:</u>		

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# XII. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Analyte identification should be confirmed in the original data output. Confirm calculation of reported results for at least 10% of the data set.

# **Calculation Check:**

Analyte:	Method:	
	I	
Remarks:		
Calculation Check:		
Analyte:	Method:	
Remarks <u>:</u>		
Remarks <u>:</u>		

Revision 2, 12/2008, TP-DM-300-7

	Inorganic Data Rev	ATIONS INTERNATION iew Checklist - Compr trate/Nitrite, Sulfate, S	
Project:			Page 1 of 10
SDG No:		Analysis:	
Laboratory:		Method: Matrix:	
data have been su	ackage has been reviewed and ummarized. The general criter nination of the following:		I/quality assurance performance cal integrityof the data were
	Case Narrative	Method and Project Blan	ks
	Analytical Holding Times Sample Preservation	Matrix Spike Recoveries Duplicate Differences	
	Method Calibration	LCS Recoveries	
		Re-analysis and Second	ary Dilution
Overall Remarks	6:		
Definition of Quali	fiers:		
	"U", not detected at the asso "UJ", not detected and assoc		
	"J", associated value estimat	ted	
	"R", associated value unusal "=", compound properly iden		ded
Reviewed by:			Date:
-			
QA Reviewed by	/:		Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

# **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

## **III. Holding Times**

Sample should be preserved and analyzed according to the appropriate analytical method In general the following preservations and holding times for waters can be applied:

> Sulfate, 4 degress C, 28 days Sulfide, 4 degrees C, pH  $\geq$ 9 with zinc acetate/sodium hydroxide, 7 days Bromide/Chloride/Fluoride, no preservative required, 28 days Nitrate/Nitrite or Ammonia, 4 degrees C, pH  $\leq$  2 with sulfuric acid, 28 days Nitrate or Nitrite, 4 degrees C, 48 days Alkalinity, 4 degrees C, 14 days TDS/TSS, 4degrees C, 7 days Phosphate (total), 4 degrees C, pH < 2 with sulfuric acid, 28 days Hexavalent Chromium, Cool 4 degress C, water- 24 hours, soil- 30 days

#### **Deviations:**

Sample #	Analyte	Date	Date	Date	Notes:
		Collected	Extracted	Analyzed	

#### Actions:

- 1. If holding times are exceeded, all results are qualified as estimated (J/UJ)
- 2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)
- 3. If samples were not properly preserved, use professional judgement to qualify the data

# Page 4 of 10

## **IV. Initial & Continuing Calibration**

A blank and at least three standards should be analyzed, with one of the standards being within 2X the MDL Correlation coefficients must be  $\geq$  0.995

Initial calibration check recoveries must be within 90-110%

Continuing calibration check recoveries must be within 85-115%

#### **Deviations:**

Compound	Correlation Coefficient	ICV/ CCV	%R	Samples Affected

#### Actions:

1. If any compounds initial calibration linearity is <0.995, qualify the data as estimated (J/UJ)

2. If any compounds initial calibration linearity is <0.95, qualify the data as unusable (R)

3. If ICV or CCV criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)

4. If ICV or CCV recoveries fall below 50%, qualify results as unusable (R)

# V. Blanks (Method Blanks and Project Blanks)

An analytical method blank must be analyzed with each batch of samples Calculate action levels based on 5X the highest blank concentration of any given analyte Sample weights, volumes, and dilution factors must be taken into account when applying the 5X criteria

#### **Deviations:**

	Maximum Conc.	Action Level (ppb)	Samples Affected
Analyte	Detected, (ppb)		

#### Actions:

1. If analyte results exceed the action levels, the data are not qualified

2. If analyte results are below the required reporting level, report results as non-detect (U) at the reporting level

3. If the analyte is detected above the reporting level, but below the action level, qualify as not-detected (U)

#### Remarks:

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## VI. Laboratory Control Sample Information

Each analyte's LCS % recovery must be within the control limits established by the laboratory In general LCS % recoveries should all be within 85-115%

#### **Deviations:**

Analyte	Date	%R	Samples Affected/Qualifiers Applied

#### Actions:

1. If the LCS recovery is outside limits but >10%, qualify all positive values as esimated (J)

- 2. If the LCS recovery is outside limits but >10%, qualify non-detect values as estimated (UJ)
- 3. If the LCS recovery is <10%, qualify all data for that analyte as unusable (R)
- 4. Use professional judgement for qualification of data for compounds with no LCS information

# Page 7 of 10

#### VII. Matrix Spike Information

Each analyte's Matrix Spike % recovery should be within the laboratory established control limits In general matrix spike % recoveries should all be within 75-125%

#### **Deviations:**

	%R	%R	
Analyte		Limits	Samples Affected

#### Actions:

1. If the spike recovery is outside limits, qualify all values in the unspiked sample as estimated (J/UJ)

- 2. If the spike recovery is <10%, qualify non-detect values as unusable (R)
- 3. Use professional judgement to qualify additional samples in the analytical group based on MS results
- 4. Use professional judgement for qualification of data for unspiked analytes

# Page 8 of 10

## VIII. Laboratory Duplicate Information

Each analyte's RPD should be within the laboratory established control limits In general RPDs should all be within 20%

#### **Deviations:**

	RPD	RPD	
Analyte		Limits	Samples Affected

#### Actions:

1. If the RPD is outside limits, qualify all values in the unspiked sample as estimated (J/UJ)

2. Use professional judgement to qualify additional samples in the analytical group based on RPD results

3. Use professional judgement for qualification of data when laboratory duplicates were not analyzed

# IX. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Analyte identification should be confirmed in the original data output. Confirm calculation of reported results for at least 10% of the data set.

# **Calculation Check:**

Analyte:	Method:	
Remarks:		
Calculation Check: Analyte:	Method:	
Remarks <u>:</u>		
Remarks <u>:</u>		

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# IX. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Analyte identification should be confirmed in the original data output. Confirm calculation of reported results for at least 10% of the data set.

# **Calculation Check:**

Analyte:	Method:	
	I	
Remarks:		
Calculation Check: Analyte:	Method:	
Remarks:		

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Project:			Page 1 of 21
SDG No:		Analysis:	
Laboratory:		Method: Matrix:	
data have been su	ackage has been reviewed and ummarized. The general criteria nination of the following:		trol/quality assurance performance lytical integrityof the data were
	Case Narrative	Chemical and/or Trace	er Recoveries
	Analytical Holding Times Sample Preservation	Matrix Spike Results Duplicate Error Ratios	and RPDs
	Method Calibration	LCS Recoveries	
	Method and Project Blanks	Re-analysis and Seco	ndary Dilution
Overall Remarks	5:		
Definition of Quali	fiers:		
	"U", not detected at the assoc		
	"UJ", not detected and associa "J", associated value estimate		
	"R", associated value unusabl	e or analyte identity unfo	unded
	II. II. A A A A A A A A A A A A A A A A	fied and value positive	
	"=", compound properly identi-		
Reviewed by:	"=", compound properly identi		Date:
·			Date:

## I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks:

# **II. Re-analysis and Secondary Dilutions**

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

# Page 3 of 21

# **III. Holding Times**

General analytical holding time for radionuclides is 6 months Water samples require preservation with nitric acid to pH <2, for dissolved radionuclide determination Radioactive iodine holding time is 7 days Consideration must always be given to the individual radionuclide half-life

### **Deviations:**

## Actions:

1. If holding times are exceeded, all results are qualified as estimated (J/UJ)

2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

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## IV. Minimum Detectable Activities (MDAs)/ Reporting Levels

Verify MDAs with project requested reporting levels for all radionuclides Compare reported activities and uncertainties with reported MDAs

#### **Deviations:**

	Project Reporting	MDA	Samples Affected
Radionuclide	Level Goal	Achieved	

#### Actions:

1. Document all radionuclide determinations that do not meet project reporting level goals.

2. If the reported value with its uncertainty encompass the project reporting level goal, they are equivalent.

3. If the sample result is negative and its absolute value exceeds the MDA, qualify the result as estimated (UJ).

4. If the sample result is negative and its absolute value exceeds 2X the MDA, qualify the result ®.

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## V.A1. Calibration Alpha Spectroscopy

Initial efficiency calibration must be demonstrated for each detector. Initial energy calibration must be demonstrated for each detector. Resolution (FWHM) must be demonstrated for each detector. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.A2.Continuing Calibration Alpha Spectroscopy

Continuing calibration efficiency verification must be performed at least quarterly. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Continuing energy calibration must be demonstrated to be within 10% of the initial calibration. Continuing FWHM must be demonstrated to be within 10% of the initial FWHM. A long background count for each detector must be performed weekly or bi-weekly. Pulser counts and demonstration of FWHM for each detector must be demonstrated daily.

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	<b>Detectors Affect</b>		Samples Affected	Value

### **Deviations:**

#### Actions:

1. If the initial calibration efficiencies, resolution, or standard information is not acceptable, qualify all affected results as estimated (J).

2. If the continuing calibration efficiency, energy, or FWHM are not acceptable,

qualify all affected results as estimated (J).

3. If background counts or pulser counts are not acceptable, qualify the affected data as estimated (J).

## V.B1. Calibration Gamma Spectroscopy

Initial efficiency calibration must be demonstrated on each detector for each geometry. Initial energy calibration must be demonstrated on each detector for each geometry. Resolution (FWHM) must be demonstrated for each detector for each geometry. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.B2.Continuing Calibration Gamma Spectroscopy

Continuing calibration efficiency verification must be performed for each detector at least quarterly. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Continuing energy calibration must be demonstrated to be within 10% of the initial calibration. Continuing FWHM must be demonstrated to be within 10% of the initial FWHM. A long background count for each detector must be performed monthly. Pulser counts and demonstration of FWHM for each detector must be demonstrated daily.

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	Detectors Affect		Samples Affected	

#### **Deviations:**

#### Actions:

1. If the initial calibration efficiency, energy, resolution, or standard information

is not acceptable, qualify all affected results as estimated (J).

2. If the continuing calibration efficiency, energy, or FWHM are not acceptable, qualify all affected results as estimated (J).

3. If background counts or pulser counts are not acceptable, qualify the affected data as estimated (J).

## Remarks:

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## V.C1. Calibration Liquid Scintillation Counters

Initial quench curves must be demonstrated for each radionuclide. Initial calibration must be demonstrated for each radionuclide. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.C2. Continuing Calibration Liquid Scintillation Counters

Continuing calibration efficiency verification must be performed afor each radionuclide. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Standards linear regression curve must be performed daily and documentation provided. Control charts for tritium and carbon-14 chi square and figure of merit values should be documented. A background count for each radionuclide window must be provided.

#### **Deviations:**

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	Detectors Affect	Range	Samples Affected	Value

#### Actions:

1. If the initial calibration quench curve or standard information is not acceptable,

- qualify all affected results as estimated (J).
- 2. If the continuing calibration efficiency or control charts are not acceptable, qualify all affected results as estimated (J).
- 3. If background counts are not acceptable, qualify the affected data as estimated (J).

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## V.D1. Calibration Gas Proportional Counters

Initial efficiency calibration must be demonstrated for each detector. Absorption curve must be demonstrated for each detector. Plateau curve performance check must be demonstrated for each detector. Data used to determine alpha and beta cross-talk must be demonstrated. Standards must be traceable and documentation must be provided. Standard preparation (dilutions, calculations, etc.) documentation must be provided.

## V.D2.Continuing Calibration Gas Proportional Counters

Continuing calibration efficiency verification must be performed at least quarterly. Continuing calibration efficiency must be demonstrated to be within 10% of the initial efficiency. Cross-talk value for each detector must be documented. Background count for each detector must be performed daily.

#### **Deviations:**

	IS	Area	Acceptable	RT	Std. RT
Deficiency	Affected	Detectors Affect	Range	Samples Affected	Value

#### Actions:

1. If the initial calibration absorption curve, plateau curve, % cross-talk, or standard information is not acceptable, qualify all affected results as estimated (J).

2. If the continuing calibration efficiency or percent cross-talk are not acceptable, qualify all affected results as estimated (J).

3. If background counts are not acceptable, qualify the affected data as estimated (J).

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## VI. Blanks

Review associated laboratory and project blank samples. List documented contamination below:

If the blank result is less than the associated uncertainty (error), no qualification will be warranted. If the blank result is greater than its associated uncertainty, but less than the MDA, then no

qualification will be warrented.

If the blank result is greater than the associated uncertainty and greater than the MDA, then qualification of sample results may be appropriate.

# Laboratory Method Blanks:

Date	Lab ID #	Radionulcide	Result and Error	MDA Result and Error
	Duciant Diamba (a. a.			
Associated	l Project Blanks (e.g.,	equipment rinsat	es, etc.)	
Date	Lab ID #	Radionuclide	Result and Error	MDA Result and Error
Remarks:				

# VI. Blanks (continued)

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Calculate action levels based on 10X the highest blank concentration.

#### **Deviations:**

	Max. Activity	Action Level	Samples Affected
Radionuclide	Detected		

#### Actions:

1. If the blank result falls outside criteria, qualify associated sample results that are less than 10X the blank value as estimated (J).

			·)·		
Example:	Blank Result	Uncert.	MDA or	Normalized absolute	<b>Qualification</b>
				<u>difference</u>	
acceptable	0.3	0.45	0.5	>2.58	none
acceptable	0.3	0.25	0.5	1.96 to 2.58	J
outside criteria	0.3	0.25	0.2	<1.96	J

2. If the absolute sample result is less than the MDA and the uncertainty is less than the result, qualify as non-detect (U).

3. If the absolute sample results is less than the MDA and the uncertainty is greater than the result, qualify as non-detect value uncertain (UJ).

4. If the sample result is greater than the MDA and the uncertainty is 50-100% of the result, qualify the data as estimated (J).

5. If the sample result is greater than the MDA and the uncertainty is greater than 100% of the result, qualify the data as rejected (R).

4. If the sample result is negative, and its absolute value exceeds 2X the MDA, qualify the data as rejected (R).

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## VII. Sample-Specific Carrier or Tracer Recovery

Sample-specific recoveries must be within limits as demonstrated by the applicable analytical procedures. Generally, recoveries of 30-110% are considered acceptable.

Documentation of traceable tracer solutions (NIST) and dilution documentation must be provided. Spot check sample-specific carrier or tracer recovery calculations.

#### **Deviations:**

			Action Taken
Radionuclide	Sample ID	%R	

#### Actions:

- 1. If recovery is between 30-110%, no qualification is necessary.
- 2. If recovery is between 10-30%, qualify the data as estimated (J).
- 3. If recovery is between 110-150%, qualify the data as estimated (J).
- 4. If recovery is less than 10%, qualify the data as rejected (R).
- 5. If recovery if greater than 150%, qualify the data as rejected (R).

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# VIII. Laboratory Control Sample Information

General LCS Criteria:	aqueous	solid
percent recovery (%R)	80-120	70-130

Laboratory LCS Identifications:

**Deviations:** 

Radionuclide	Date	%R	Samples Affected/Qualifiers Applied

#### Actions:

Aqueous	<u>&lt;50%</u> R	<u>50-79%</u> J	<u>121-150%</u> J	<u>&gt;150%</u> R	
Solid	<u>&lt;40%</u> R	<u>40-69%</u> J	<u>131-160%</u> J	>160% R	

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# IX. Matrix Spike Information

General MS Criteria:	Aqueous	Solid
percent recovery (%R)	50-120	40-130

Project Sample(s) Spiked:

**Deviations:** 

Radionuclide	Date	%R	Samples Affected/Qualifiers Applied

Aqueous	<u>&lt;20%</u> <u>20-49%</u> <u>121-160%</u> <u>&gt;160%</u> R J J use professional judgement
Solid	<u>&lt;10% 10-39% 131-160%</u> >160% R J J use professional judgement
Remarks:	

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## X. Duplicate Sample or Matrix Spike Duplicate Analysis

Identify the method utilized to evaluate duplicate analyses; duplicate error ration (DER), relative percent difference (RPD), or relative error ratio (RER). Duplicate actions should apply to all samples associated with the duplicate pair.

Duplicate Sample Identification:

#### **Deviations:**

				Samples Affected
Radionuclide	DER	RPD	RER	

#### Actions:

1. If both sample and duplicate activities are within 2X the MDA comparison is acceptable.

- 2. If the DER is greater than 1.00, qualify the data as estimated (J).
- 3. If the RPD is greater than 50% qualify the data as estimated (J).
- 4. If one sample is <MDA and the other sample is >2X the MDA, qualify the data as estimated (J).

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# XI. Chemical/Spectroscopic Separation Specificity (alpha spectroscopy)

Each alpha isotopic peak should be clear and free of interference from other energy peaks. Each isotopic energy peak should be evaluated for peak shape (i.e., tailing, splitting, etc.) The observed energy peak(s) for the radionuclide of interest must be confirmed as acceptable to theoretical.

# **Deviations:**

Radionuclide	Deficiency	Samples Affected

#### Actions:

1. If the energy of the radionuclide peak of interest is more than 100keV from the theoretical energy, qualify the results as rejected (R).

2. If the energy spectra contains any overlapping or interferent peaks that can not be resolved from the target peak, qualify the data as rejected (R).

3. If results have not been properly corrected for distinguishable interfering radionuclide peaks, qualify the data as rejected (R).

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# XII. Target Radionuclide Spectroscopic Identification (gamma spectroscopy)

Each sample target radionuclide energy must be within 2 keV of the observed standard peak energy. Multiple peak radionuclides must exhibit the appropriate peak energies and proportional status. At least 50% of the total gamma abundance must be accounted for by the quantified radionuclides. All peaks greater than 3X the background standard deviation must be identified and quantified. The observed energy peak(s) for radionuclides of interest must be confirmed as acceptable to theoretical. Radionuclide values must be consistent with related radionuclides (e.g., parent daughter relationships).

#### **Deviations:**

Radionuclide	Deficiency	Samples Affected

#### Actions:

1. For target radionuclides that are not detected, qualify the results as described in section VI.

2. For target radionuclides that are detected but fail to meet identification crtieria,

use professional judgement to qualify the data as estimated (J).

3. If the energy of the radionuclide peak of interest is more than 2 keV from the theoretical energy, use professional judgement to qualify the data.

4. If the energy spectra contains any overlapping or interferent peaks that can not be resolved from the target peak, qualify the data as rejected (R).

5. If results have not been properly corrected for distinguishable interfering radionuclide peaks, qualify the data as rejected (R).

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## XIII. Tentatively Identified Radionuclides (gamma spectroscopy)

Each sample tentatively identified radionuclide energy must be within 2 keV of the theoretical peak energy. Multiple peak radionuclides must exhibit the appropriate peak energies and proportional status. Tentatively identified radionuclide gamma spectra must match the radionuclide's library spectra. All peaks greater than 3X the background standard deviation must be identified and quantified. The observed energy peak(s) for radionuclides of interest must be confirmed as acceptable to theoretical. Judgments of this data should include: half-life consistencies; sample set consistencies; lab contamination. Radionuclide values must be consistent with related radionuclides (e.g., parent daughter relationships).

#### **Deviations:**

Radionuclide	Deficiency	Samples Affected

#### Actions:

1. Qualify all tentatively identified radionuclides as estimated (J).

2. If the energy of the tentatively identified radionuclide peak is more than 2 keV from the theoretical energy, use professional judgement to qualify the data.

3. If the reviewer judges anything regarding the identifcation of the tentatively identified radilnuclide as suspect, qualify the data as rejected (R).

# XIV. Evaluate System Performance (alpha spec, gamma spec, etc.)

Examples of system performance indicators:

Abrupt, discreet shifts in background or detector response. High background levels. Energy calibration shifts. Extraneous peaks. Loss of resolution. Peak tailing or splitting.

#### **Deviations:**

Radionuclide/Method	Deficiency	Samples Affected

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## Actions:

1. Based on the instrument performance indicators, the data reviewer must use professional judgement ot qualify the data.

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## XV. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Confirm appropriate instrument and manual peak integration. Confirm calculation of reported results for at least 10% of the data set.

# **Calculation Check:**

Radionuclide:	Method:	
Remarks:		
Calculation Check: Radionuclide:	Method:	
Remarks:		

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# XV. Analyte Quantitation Check

Original data information should fall within the established calibration range for the analytical run. Confirm appropriate instrument and manual peak integration. Confirm calculation of reported results for at least 10% of the data set.

# **Calculation Check:**

Radionuclide:	Method:	
Demostre		
Remarks:		
Calculation Check:		
Radionuclide:	Method:	
	I	
Remarks:		

## XVI. Overall Assessment of Data

It is appropriate for the data reviewer to make professional judgements and express concerns regarding the validity of the data, overall. This is particularly appropriate when there are several citeria outside the desired specifications. The additive nature of these factors may present data that needs to be further qualified beyond each individual qualification. The reviewer should summarize these concerns.

#### Actions:

1. Qualified data must be accompanied by all individual reason codes related to the qualification assigned.

2. If the sample result has been qualified for multiple reasons, the reviewer will use professional

judgement to determine if multiple estimations warrants rejection (R).

#### **Remarks:**

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SCIENCE APPLICATIONS INTERNATION Data Verification/Validation Request for Missing or Incomplete Laboratory	Review
Project:	
SDG No:	
Analyte Group:	
Sample Matrix:	
Requested Missing or Incomplete Information:	Date Requested:
Response:	Response Date: