

**US Army Corps  
of Engineers**  
Louisville District

**LOUISVILLE RADIOLOGICAL GUIDELINE**

**(LRG)**

**[DATA ANALYSIS AND VALIDATION GUIDELINES]  
VERSION 4**

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LOUISVILLE DISTRICT

September 2004

This document is broken into four categories and found in the chapters following.

## **LOUISVILLE RADIOLOGICAL GUIDELINE (LRG):**

- **RADIOLOGICAL MEASUREMENT CRITERIA**
- **RADIOLOGICAL INTERFERENCES**
- **DATA REPORTING**
- **DATA VALIDATION RADIOLOGICAL REPORT**

## **ACKNOWLEDGEMENT**

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**This document was built upon information obtained during visits at Severn Trent Laboratory (STL) ST. Louis, Missouri; General Engineering Laboratory (GEL), Charleston, South Carolina; and Eberline, Oak Ridge, Tennessee.**

**The author acknowledges the valuable contribution to the preparation of this document by Brigid Brooks from MWH Americas, Inc**

**Also, the author acknowledges the valuable comments received during the revision of the LRG Versions from the following:**

**STL – ST. Louis**

**General Engineering Laboratory, Charleston, South Carolina**

**Eberline Laboratory, Oak Ridge, Tennessee**

**USACE  
Louisville District**

**Environmental Standards , Inc.**

# FORWARD

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## LOUISVILLE RADIOLOGICAL GUIDELINE

This document is intended to summarize the requirements for evaluating environmental radiological data quality and to help ensure proper preparation of the appropriate documentation for Radiological projects.

There are no restrictions on the distribution or reproduction of this document. This is a living document and it is the responsibility of the User to maintain the most current version. Revisions and additions to this document will be posted on the United States Army Corps of Engineers (USACE) Louisville District web page: <http://www.lrl.usace.army.mil/ed/article.asp?id=197>. Users are encouraged to frequently visit the web page to obtain current updates and make suggestions for further improvement of this document.

It is the intent of Louisville District to ensure that all laboratories and contractors who are involved in any environmental radiological process (with Louisville District) are familiar with and follow the guidelines contained herein.

If you have any suggestions, revisions, additions, or need clarification of any part of this document, please contact the author, Subject Matter Expert – Chemistr (502) 315-6324.

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Author  
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## **REVISIONS AND ADDENDA**

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Revisions to this LRG will be noted below and incorporated into this document as warranted. All revisions and addenda will be reviewed by the USACE Louisville District Senior Chemist prior to incorporation.

<b>VERSION NO.</b>	<b>DATE OF INCORPORATION</b>	<b>BRIEF DESCRIPTION OF REVISION</b>	<b>USACE SENIOR CHEMIST INITIALS</b>
1	1-7-04	Incorporation of comments	SAM
2	3-10-04	Incorporation of comments	SAM
3	3-23-04	Incorporation of comments	SAM
4	September 2004	Incorporation of comments	SAM

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## **ACRONYMS AND SYMBOLS**

%R	Percent Recovery
$\alpha$	Alpha
$\gamma$	Gamma
°C	Degrees Celsius
$\mu$ Ci	microcurie
$\beta$	Beta
Bq	Becquerel
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
Ci	curie
CLP	Contract Laboratory Program
DVRR	Data Validation Radiological Report
FWHM	Full Width – Half Maximum
g	grams
GEL	General Engineering Laboratory
HNO <sub>3</sub>	Nitric Acid
HTRW-CX	Hazardous, Toxic and Radioactive Waste Center of Expertise
ICQ	Instrument Quality Control
L	Liter
LCG	Louisville Chemistry Guidelines
LCS/LCSD	Laboratory Control Sample/Laboratory Control Sample Duplicate
LRG	Louisville Radiological Guidelines
mCi	millicurie
MDA	Minimum Detectable Amount
MS/MSD	Matrix Spike/Matrix Spike Duplicate

## **ACRONYMS CONTINUED**

MSA	Method of Standard Additions
MWH	MWH Americas Inc.
ND	Normalized Difference
pCi	picocurie
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
QO	Quality Objective
RER	Relative Error Ratio
RL	Reporting Limit
RL	Reporting Limit
ROI	Region of Interest
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
$\sigma$	Sigma
STL	Severn Trent Laboratories
TPU	Total Propagated Uncertainty
US	United States
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency

# 1.0 INTRODUCTION

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This document summarizes the environmental data assessment for radiological analyses as required for Louisville District projects. Primary and Quality Assurance (QA) laboratories must be validated by Corps of Engineers Hazardous, Toxic and Radiological Waste Center of Expertise (HTRW-CX) branch in order to conduct sample analysis. Data Review steps have been defined in relation to laboratories, contractors and independent 3<sup>rd</sup> party Data Validators. These steps are summarized on the Flow Chart presented in Figure 1 in Appendix B.

The Louisville Radiological Guideline (LRG) is written to describe the process of data review by the laboratory, data verification and/or validation by the contractor or an independent validator, comparison of QA and Quality Control (QC) data (primary) and the final report describing the acceptance/rejection of the data, as applicable. In order to avoid the situation of rejecting all the data, it is expected that all parties involved in Louisville District projects implement the LRG. It is available for whomever in USACE that wishes to utilize this document. This document has been divided into four sections as described below.

**Section I, Radiological Measurement Criteria:** acceptance criteria for analyses with flagging criteria have been summarized in tables. All parties involved in a project for the Louisville District should be aware of these Quality Objectives (QOs). It is very important that these tables be given to the laboratory analyst, so he/she becomes aware of Validators' technical judgment on the data that he/she produces.

**Section II, Radiological Interferences:** a summary of interferences associated with the preparation, extraction and analysis of samples for radiological isotopes.

**Section III, Data Reporting:** a summary of the hard copy deliverable is presented. It is expected that laboratories include detailed information in the case narrative.

**Section IV, Data Validation Radiological Report (DVRR):** this chapter covers the reporting of data validation findings and provides a tool to be used to compare the results from the primary laboratory with those analyzed by the QA laboratory.

All laboratory personnel who participate in US Army Corps of Engineers projects must sign the attached ETHICS AND INTEGRITY AGREEMENT in Appendix A.

## 2.0 Do's AND Don'ts CHECKLIST

This checklist is to be utilized by the laboratory, contractors and/or 3<sup>rd</sup> party validators as applicable. This checklist is in addition to the laboratories own checklist. It is provided to point out important items for the laboratory to take into account.

Project Name:

<b>Table 2-1 CHECKLIST</b>	
<b>Do's and Don't Items</b>	<b>Completed Y/N/NA</b>
Have all laboratory personnel who participate in US Army Corps of Engineers projects sign the attached ETHICS AND INTEGRITY AGREEMENT.	
Report percentage solid for soil and sediment samples on the data sheet.	
Report the following dates of samples on their corresponding analytical data sheet: Date Collected, Date Extracted, and Date Analyzed.	
Report tracer/carrier recoveries and their QC limits on their corresponding analytical sheet.	
Dilute samples extracts/digestate to the calibration range when initial concentration levels are outside the upper limit of the calibration curve. Report results of the diluted and undiluted analyses in the data package.	
Include the following information for all MS/MSD and LCS conducted: theoretical concentration of solutes spiked to sample matrices, concentration of the analytes present in the matrices before spiking, concentrations of determined solutes (recovered) after spiking, % recovery, and RPDs.	
Control charts and/or QC limits for the following: Tracer/Carrier Recoveries, Blank Spike Recoveries, and/or LCS for the period of sample analyses, shall be available on request.	
Include Chain of Custody and Cooler Receipt Forms in the data packages.	
Include the Quality Assurance Officer/Manager signature and Laboratory Manager signature in each data package and report.	
Data Validator must initial and date pages of data results that have been validated	
When a tracer/carrier is utilized, an MS/MSD must be conducted. When a tracer or carrier is not utilized, only an MS is required along with a sample duplicate.	
If soil samples were not ground and split in the field, the primary laboratory will grind and then split off (10%) of the ground samples to be sent to the QA laboratory.	
Validators must not alter the reported results by crossing out data and changing. If qualifying data, only place qualifier. If result change in necessary, make note on laboratory form and contact laboratory for corrected form. Further investigate additional samples.	
Validator can request laboratory limits and statistics for review as applicable.	

### 3.0 LRG vs. LCG – DIFFERENCES IN PROTOCOL

Several differences can be noted between the Louisville Radiological Guideline (LRG) and the Louisville Chemistry Guidelines (LCG). Among these differences are the following:

LRG	LCG
<ul style="list-style-type: none"><li>• <b>MS/MSDs</b> are conducted on radiological samples because carriers and/or tracers are added to the samples. Carriers and tracers are utilized similar to that of surrogates in organic analysis.</li></ul>	<ul style="list-style-type: none"><li>• <b>MS ONLY</b> is conducted on metals samples. MSDs are NOT required.</li></ul>
<ul style="list-style-type: none"><li>• Validators receive <b>Primary and QA Data</b> for review.<ul style="list-style-type: none"><li>• As shown in Figure 1, Appendix B, the validator receives both sets of data to ensure separation techniques can be compared. Error can increase in the separation process.</li><li>• The validator utilizes the QA data to support what is being looked at from the primary laboratory.</li></ul></li></ul>	<ul style="list-style-type: none"><li>• Validators receive only the <b>Primary Data</b> for review/validation.</li><li>• Validators may receive QA data for CQAR preparation.</li></ul>
<ul style="list-style-type: none"><li>• Validators prepare one report, the DVRR as described in Section IV of this document.</li></ul>	<ul style="list-style-type: none"><li>• Validators prepare a Data Validation Report (DVR) for the <b>Primary Data</b>.</li><li>• Validators may prepare CQAR for the <b>QA Data</b>. (a separate report from the DVR)</li></ul>

## **4.0 RADIOLOGICAL DATA ASSESSMENT GUIDELINE**

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The purpose of this document is to summarize the requirements set forth by US Army Corps of Engineers (USACE), Louisville District for evaluating data quality and for providing the appropriate Analytical Reports for radiological analyses. The goal of USACE-Louisville District is to obtain analytical data of definitive quality that meet all project specific requirements.

### **4.1 Laboratory Validation**

The Louisville District requires environmental laboratories be validated by the USACE-HTRWCX, Omaha, Nebraska. The Louisville District and/or contractor's firms are the laboratory contract holders. The District Chemist and/or Project Chemist initiates the validation process for both the Louisville District and contractor's firms. There are two types of environmental laboratories for HTRW -projects:

#### **4.1.1 Primary Laboratory**

Primary samples constitute 100% of the samples collected and may include field duplicates (normally 10%), to be analyzed by a certified laboratory. This laboratory is called a Primary or QC laboratory. Primary Sample Analysis Data is usually called QC data. QC data should not be confused with the laboratory QC analyses performed during analysis such as method blanks, duplicates, and spiked samples. See Figure 1 Flow Chart, Appendix B. All soil samples will be ground either by the primary laboratory or if possible by the field samplers. Water samples will be split in the field.

#### **4.1.2 QA Laboratory**

Samples that are split off primary or duplicate samples (normally 10% of primary) are called QA samples, and are to be analyzed by another certified laboratory. This laboratory is called a QA laboratory. See Figure 1 Flow Chart, Appendix B.

Approval of primary and QA laboratories must be obtained from the Louisville District Chemist.

### **4.2 Radiological Analysis**

The analytical testing of environmental samples for radiological isotopes is conducted utilizing one or more of the following techniques: Gamma Spectroscopy, Alpha Spectroscopy, Liquid Scintillation Counting, Gas Flow Proportional Counting System, Laser Induced Kinetic Phosphorescence and Lucas Cell Counting. In order to assure that the analytical data are accurate and legally defensible, the Louisville District prepared this document as a guideline for contractors, environmental laboratories, and data validation contractors. Its purpose is for the laboratories to generate data for the intended usage on the first attempt.

The laboratory should review this document and be aware of the Quality Assurance Project Plan (QAPP) or Sampling and Analysis Plan (SAP) (project specific), EM 200-1-3 (available on <http://www.lrl.usace.army.mil/ed/article.asp?id=197> ).

The Radiological Analysis Criteria, Section I, provides summaries of Quality Objectives (QO) for the Louisville District.

### **4.3 Data Assessment Process**

Review of the radiological analytical data may be conducted incrementally on Sample Analytical Groups instead of the entire analytical data at the end of the project. Size and frequency of the Sample Analytical Groups will be determined at project initiation. Analytical results for samples that are sent to primary and/or QA laboratory will be reviewed when their data packages are ready for release to the client (contractor or Louisville District). See Figure 1, Appendix B.

#### **4.3.1 Laboratory Data Review (Steps 1 & 1A)**

Primary- and QA-Laboratories review their data before releasing data packages/reports to the contractor or to Louisville District. The review process is the same for both primary and QA laboratories. Accordingly, STEP-1 is designated for review by the primary laboratory and STEP-1A is designated for review by the QA laboratory.

##### **4.3.1.1 Process**

The review may be variable as a function of the laboratory Quality Assurance Plan, and the type of work that is being performed. At a minimum, review should include routine quality control (QC) data check, analytical results check and may also include the checking of Specific Reporting Requirements. The data review requirement should be determined prior to the start of the analysis, i.e., during project scope set up and negotiations.

##### **4.3.1.2 Product**

Analytical Reports must contain the analytical results with laboratory QC data. The reports will have the following items:

**Cover Page:** the cover page must contain the Project Name, and a statement indicating the authenticity of the data. Laboratory Director, Quality Assurance Manager/Officer and/or Project Manager must enter their signatures with dates on this page.

**Case Narrative:** a case narrative describing any non-conformances with methodology and/or the Tables of Quality Objectives (attached).

**Analytical Data Packages:** The laboratories will provide comprehensive data packages that will contain the raw instrument printouts, with a copy of the analytical data sheet (including all pertinent quality control data and forms) attached to the Sample Delivery Groups (SDGs). The SDG is a unique number given by the laboratory associated with the samples received and the final data package.

### **4.3.2 Data Validation Radiological Report (Step-2)**

An independent entity or laboratory contract holder for the primary laboratory (contractor) performs this process to produce the Data Validation Radiological Report (DVRR). See a memo by Office of Council in Appendix C for definition of the independent 3<sup>rd</sup> party.

#### **4.3.2.1 Process**

This process must evaluate the Completeness, Consistency, and Compliance of a data package against the Quality Assurance Project Plan and the applicable analytical methods and the LRG. This process requires a Comprehensive Data Package. This verification/validation process will include, but is not limited to the following (instrument dependent): Calibration Criteria, tracer and carrier recoveries, blanks, results of energy and efficiency checks, results of quality checks or pulser checks, results of Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSDs), and/or Matrix Spike/Matrix Spike Duplicate (MS/MSD), as applicable, and results of duplicates. The reviewer performs verification of 100% and validation of 10% of the primary samples. During the validation process, the validator must first determine if overall data quality problems exist, or if data quality problems are specific to a given matrix/method. If the rejected data are deemed to be part of an overall quality problem, the Validator randomly chooses 10% of the data package to evaluate and so on until a level of confidence is reached to accept or reject the data (up to 100%). If the rejected data are deemed systematic to a particular matrix/method, the Validator chooses 10% from that specific matrix/method to evaluate. Full data validation consists of validating the data using these Guidelines and recalculating the positive hits above the QL.

This process also provides a complete assessment of the quality of the data by examining primary samples, duplicates at 10% of the primary samples, and their split samples (QA) via comparison of the QA sample results to the duplicate and/or primary sample results. Examination of the primary (confirmation) sample data, and their 10% split samples (QA) provides the data user with a degree of the acceptance and usability of the Radiological Data Quality. The finding should be summarized in a report format.

#### **4.3.2.2 Product**

The Data Validation Radiological Report is a document that is prepared by an independent entity or contractor that is not involved directly in the analysis of the samples. Any nonconformance with the QAPP will be relayed to the laboratories for corrective actions. Corrective actions will be implemented in order to avoid such deficiency in the subsequent phases of analysis. This approach allows in real-time, determination of the lab analytical performance, allows immediate determination of data integrity, and data usability.

Validators choose randomly the 10% of the raw data for validation. Full data validation consists of validating the data using the Guidelines in this LRG and verifying/recalculating reported values. Validator will qualify the data as outlined in the LRG. If serious problems, based on percent (%) usability as defined in the QAPP, are encountered during the validation process, validation should be conducted on the next 10% of the raw data, and so on till a level of

confidence is reached to accept or reject the data. The Validation Guidelines, and a Data Validation Checklist, are presented in Section IV Attachment A.

## 5.0 PRECISION DISCUSSION

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Analytical data are generated for matrix spike/matrix spike duplicate (MS/MSD) to determine precision and accuracy of the analytical method and sample preparation. For radiological analyses, MS/MSDs are not utilized in Gamma Spectroscopy. For the other instruments, where carriers and tracers are utilized, (Section 6.0) an MS/MSD is required. For other instruments where a carrier or tracer is NOT utilized matrix duplicate must be utilized in place of an MSD. Qualification based on relative percent differences (RPDs) of duplicate analyses is presented in Table 5-1.

When reviewing MS/MSD, or MD data three criteria are utilized in determining the acceptability of the precision of analysis.

The first criteria is relative percent difference which is calculated with the following equation:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where:

For duplicate analysis:

$C_1$  = Measured total activity off the first detection or the first sample aliquot

$C_2$  = Measured total activity off the second detection or the duplicate sample aliquot

For MS/MSD analyses:

$C_1$  = Spike sample result minus sample result

$C_2$  = Spike sample result minus sample result

The second criteria for determining the acceptability of the precision of the analysis is through the use of the Relative Error Ratio (RER). The RER is the ratio between the difference in measured activity to the summation of potential errors.

$$RER = \frac{|C_1 - C_2|}{\sum \text{counting uncertainties}} = Z_{REP} = \frac{|X_1 - X_2|}{\sqrt{u_c^2(x_1) + u_c^2(x_2)}}$$

Where for RER:

For duplicate analysis:

$C_1$  = Measured total activity off the first detection or the first sample aliquot

$C_2$  = Measured total concentration off the second detection or the duplicate sample aliquot

$\Sigma$ uncertainties = See Appendix G for definition

For MS/MSD analyses:

$C_1$  = Spike sample result minus sample result

$C_2$  = Spike sample result minus sample result

$\Sigma$ uncertainties = See Appendix G for definition

For  $Z_{REP}$ :

$X_1$  and  $X_2$  denotes two measure activity concentrations

$u_c(X_1)$  and  $u_c(X_2)$  denotes the respective measure activity concentrations uncertainty

The third criteria is Normalized Difference (ND) which takes into account the difference in sample results and the relative uncertainty for samples with low activity.

$$ND = \frac{|X_1 - X_2|}{TPU}$$

Where:

$X_1$  – Sample result

$X_2$  – Sample result

TPU – sample uncertainty @ 1 sigma (68.3%) level

The fourth criteria is the Total Propagated Uncertainty (TPU) which takes into account the total uncertainty of the sample analysis. See Appendix G for the TPU equation. The difference between TPU for the sample and its duplicate should not be greater than 10%. If the TPU is > 10% then the data is rejected and qualified “R”. One or more of the following criteria should be met (Table 5-1).

**Table 5-1**  
Precision Comparison

ND Activity <5X QL	RPD $\Delta/\text{mean} * 100 =$ Activity >5X QL	RER $\frac{ C_1 - C_2 }{\sum \text{uncertainties}}$	TPU Relative uncertainty	Flagging Criteria
$\geq 2.6$	High (>25)	Low < 2 $\sigma$	<10%	J
$\geq 2.6$	High (>25)	Low < 2 $\sigma$	>10%	R
$\geq 2.6$	High (>25)	High > 2 $\sigma$	>10%	R
$\leq 2.6$	Low (<25)	Low < 2 $\sigma$	<10%	None
$\leq 2.6$	Low (<25)	High > 2 $\sigma$	<10%	J/R
$\leq 2.6$	Low (<25)	Low < 2 $\sigma$	>10%	R
$\leq 2.6$	Low (<25)	High > 2 $\sigma$	>10%	R

Furthermore, with Gamma Spectroscopy, uncertainty can be increased due to the Compton effect. (See Appendix D)

## 6.0 TRACERS AND CARRIERS

Tracers and carriers are utilized in the analysis of some radioactive isotopes depending on the instrument that is being utilized. Carriers are typically nonradioactive elements with similar chemical characteristics as the analyte being analyzed. Tracers are radionuclides that chemically mimic and do not interfere with the target radio analyte through the chemical preparation and instrument analysis. Carriers and tracers are added to samples to determine the overall chemical yield for the analytical preparation steps. Table 6-1 provides an **EXAMPLE** of some radionuclides and whether a tracer or carrier is utilized in the analysis. The tracer or carrier utilized can vary from laboratory to laboratory as well as instrument to instrument.

**Table 6-1**  
Tracers and Carriers

<b>Isotope</b>	<b>Instrument</b>	<b>Carrier or Tracer</b>	<b>Possible Carrier or Tracer Utilized</b>
Americium-241	Alpha Spectroscopy	Tracer	Americium-243
Curium-244	Alpha Spectroscopy	Tracer	Americium-243
Curium-245/246	Alpha Spectroscopy	Tracer	Americium-243
Neptunium-237	Alpha Spectroscopy	Tracer	Neptunium-239
Plutonium-238	Alpha Spectroscopy	Tracer	Plutonium-242
Plutonium-239/240	Alpha Spectroscopy	Tracer	Plutonium-242
Thorium-228	Alpha Spectroscopy	Tracer	Thorium-229
Thorium-230	Alpha Spectroscopy	Tracer	Thorium-229
Thorium-232	Alpha Spectroscopy	Tracer	Thorium-229
Uranium-234	Alpha Spectroscopy	Tracer	Uranium-232
Uranium-235/236	Alpha Spectroscopy	Tracer	Uranium-232
Uranium-238	Alpha Spectroscopy	Tracer	Uranium-232
Nickel-59	Liquid Scintillation	Carrier	Nickel
Nickel-63	Liquid Scintillation	Carrier	Nickel
Plutonium-241	Liquid Scintillation	Tracer	Plutonium-242
Polonium-210	Alpha Spectroscopy	Tracer	Polonium-208
Technetium-99	Liquid Scintillation	Tracer	Technetium-95m
Lead-210	Gas Flow Prop Counting	Carrier	Lead
Iron-55	Liquid Scintillation	Carrier	Elemental Iron
Radium-226	Gas Flow Prop. Counting	Tracer	Barium-133
Radium-228	Gas Flow Prop. Counting	Tracer/Carrier	Barium-133 and Yttrium
Radium(Total)	Gas Flow Prop. Counting	Tracer	Barium-133
Strontium-89	Gas Flow Prop. Counting	Carrier	Strontium or Barium
Strontium-90	Gas Flow Prop. Counting	Carrier	Strontium or Barium
Strontium(89 and 90l)	Gas Flow Prop. Counting	Carrier	Strontium or Barium

## 7.0 DILUTIONS

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Dilutions performed during the analytical process that raise the project reporting limits may render all results unusable. Dilutions, on the other hand, may be necessary, due to the level of activity in the sample or matrix interferences. Also, dilutions may be required to maintain method compliance. It is important that the laboratories balance as best as possible the project requirements vs. other factors to use the most appropriate dilution.

In cases where high contamination is suspected prior to analysis, dilutions are acceptable provided that the high concentration is confirmed. However, if the dilution shows the target compounds as not detected or detected at the low end of the calibration range, the sample must be re-analyzed at a lesser dilution.

In cases where high contamination is not known until after the first analysis, dilution would be necessary for target compounds that have exceeded the calibration range. The laboratory should dilute as little as possible so that the target analyte of the highest concentration is above the low end of the calibration range.

## 8.0 DATA QUALIFIERS

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Data qualifier flags are used in an effort to best describe the quality of each piece of data to the data user. These flags are letter codes appended to the numerical data (or in some instances used alone). A series of standard remarks is used to give a more detailed explanation of the data. The following data qualifiers along with the United States Environmental Protection Agency (USEPA) definitions will be used during the data validation process. The validator may also utilize the QA sample results in the qualification of the primary data. The use/application of these qualifiers is further discussed in the Quality Objective Tables in Section 1.

- U -** The radionuclide was analyzed for but not detected. The value preceding the U is the sample-specific Minimum Detectable Amount (MDA).
- J -** The identification of the analyte is acceptable, but the quality assurance criteria indicate that the quantitative values may be outside the normal expected range of accuracy (i.e. the quantitative value is considered estimated).
- R -** Data are considered to be rejected and shall not be used. This flag denotes the failure of quality control criteria such that it cannot be determined if the analyte is present or absent from the sample. Re-sampling and analysis are necessary to confirm or deny the presence of the analyte.
- UJ -** This flag is a combination of the U and J qualifiers, which indicates that the analyte is not present. The reported value is considered to be an estimated sample-specific MDA.
- B -** The B flag is to be used when the analyte is found in the associated blank as well as the in the sample. Caution: If using CLP software, override the CLP “B” designation. However, blanks should not have any contamination unless associated with the instrument blank.

## 9.0 REFERENCES

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Nuclear Regulatory Commission (NRC). August 2001. *Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)*.

Nuclear Regulatory Commission (NRC), August 2000. *Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM) Revision 1*.

**SECTION I**  
**RADIOLOGICAL MEASUREMENT**  
**CRITERIA**

This Section describes Quality Objectives for the six techniques utilized in determining radiological isotopic activity in samples. The six methods/techniques include: Gamma Spectroscopy, Alpha Spectroscopy, Liquid Scintillation Counting, a Gas Flow Proportional Counting System, Laser Induced Kinetic Phosphorescence and a Lucas Cell Counting.

The validator may request the laboratory limits and/or statistics for review.

**Table 1**  
**Summary of Quality Objectives for Measurement by Gamma Spectroscopy ( $\gamma$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	Energy Calibrations is established utilizing minimum of six calibration points evenly distributed across the energy range (0 – 2MeV)	Annually or when the calibration control check indicated an unacceptable change in the energy calibration parameters or major repairs to the instrumentation have been conducted	$< \pm 2\sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$ OR Energy difference should be $\leq 0.5$ keV for all points  For first calibration, compare to manufacturers specifications	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved $\leq 0.5$ keV no action $\geq 0.5$ keV – take corrective action, instrument is not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags $\leq 0.5$ keV – No flag  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data $\geq 0.5$ keV – Reject all data Qualify data with “R”
Full Width at the Half Maximum (FWHM) Calibration	Energy Calibration Check for peak shape monitoring	Annually or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters  Defines shape of peak and how sharp it is	$< \pm 2\sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$  OR  FWHM $\leq 3.0$ keV at 1332 KeV or reference manufactures specifications  FWHM difference $< 0.5$ keV for selected peaks – one low end (ie: $^{241}\text{Am}$ ), one middle (ie: $^{60}\text{Co}$ ) and one high (ie: $^{137}\text{Cs}$ )	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved  If $\geq 3 \sigma$ rerun once to determine statistical significance. If found to be acceptable, no corrective action is necessary. If fails, perform one or more of the following: 1) Check expiration date of standard 2) Check source positioning 3) Check instrument setup (ie: cables, connectors) Rerun daily calibration If rerun fails do not use instrument	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”  OR  If $\geq$ FWHM Limits then reject all data.  Qualify data with “R”

**Table 1**  
**Summary of Quality Objectives for Measurement by Gamma Spectroscopy ( $\gamma$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Efficiency Calibration and/or Geometry Calibration Check	Calibration source. Efficiency for each peak and geometry for each matrix used in samples on each day of use	Annually or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters  Geometry check daily.	$< \pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$ Difference within $2\sigma$ and/or $3\sigma$ of manufacturers specifications or historical laboratory information  Geometry check should be within + or - 10% of the known value	$\bar{x}$ and $2\sigma$ – no action  between $2\sigma$ and $3\sigma$ – investigate, notate warning  $\geq 3\sigma$ - take corrective action, instrument not usable until resolved  Investigate and re-calibrate/re-analyze samples.	$\bar{x}$ and $2\sigma$ - No flags (90%-110)  between $2\sigma$ and $3\sigma$ - J(60%-90%) or (110% - 140%)  $\geq 3\sigma$ - Reject all data $\leq 60\%$ or $\geq 140\%$  Qualify data with “R”  Reject all data in which a geometry calibration check has not been conducted.
Blanks	Instrument and geometry specific blanks used to assess method contamination  Water – utilize distilled or deionized water (radon free)  Soil/other – utilize empty counting container  Filter – utilize physically and chemically identical filter media	One blank per matrix and geometry per batch (or every 20 samples)	No target analyte may be present above Reporting limit	If analyte found in instrument blank is non-detect in samples  If analyte detected in associated samples  If analyte concentration in instrument blank exceeded 5% of concentration in one or more samples, re-analyze all affected samples	No effect  Compare concentrations  If concentration in instrument blank exceeds 5% of the concentration found in one or more samples, report with qualification (B/UB).

**Table 1**  
**Summary of Quality Objectives for Measurement by Gamma Spectroscopy ( $\gamma$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Background subtraction spectrum	Long count with empty chamber used to accumulate data to determine cpm. Count must be longer than sample count or as appropriate when stabilized.	Minimum of bi-weekly, when the background quality control check indicates an unacceptable change in the daily background parameters, or when counting chamber changes have been made (ie: cleaning, replacement etc..)	$< \pm 2\sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, Conduct an empty chamber count; instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags between $2\sigma$ and $3\sigma$ - J $\geq 3\sigma$ - Reject all data Qualify data with “R”
Energy, resolution and efficiency checks		Daily when the instrument is utilized or before and after each analytical batch	$< \pm 2\sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$  When compared to previous efficiencies %D < 10%	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags between $2\sigma$ and $3\sigma$ - J $\geq 3\sigma$ - Reject all data Qualify data with “R”
Detector background check	Counting conducted for a standard count time	Daily when the instrument is utilized or as appropriate	$< \pm 2\sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags between $2\sigma$ and $3\sigma$ - J $\geq 3\sigma$ - Reject all data Qualify data with “R”

**Table 1**  
**Summary of Quality Objectives for Measurement by Gamma Spectroscopy ( $\gamma$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Counting Duplicate or matrix duplicate		Per sample batch	<p><math>RPD \leq 25\%</math> for high activity When result is <math>&lt; 5 \times</math> the QL, then use Normalized Difference (ND) <math>\leq 2.6</math> or RER <math>&lt; 1.0</math></p> <p>If one sample below QL and the other at least <math>5 \times</math> above the QL</p> <p>If both sample results are below the QL – not applicable</p>	<p>Re – Analyze</p> <p>Lab should determine discrepancy and initiate corrective action</p>	No action is taken based on this only, use professional judgement
LCS	Interference-free matrix containing target radionuclide	One per matrix per batch (or every 20 samples)	75-125%	Correct problem and repeat	When recovery is $> UL$ flag positives with J. For recovery $< LL$ , flag J/UJ. For a recovery $< 30\%$ , flag J/R.
QA sample results (split off primary sample)	Comparison of primary laboratory sample result with QA laboratory sample result	10% of total primary samples	<p><math>RPD \leq 25\%</math> for high activity When result is <math>&lt; 5 \times</math> the QL, then use Normalized Difference (ND) <math>\leq 2.6</math> or RER <math>&lt; 1.0</math></p>	<p>Investigate primary and QA data For other qualification</p> <p>If data has not been qualified due To other criteria, re-analyze</p>	<p>Report data that has not Been qualified due to Other QA/QC criteria</p> <p>Qualify data utilizing Professional judgement. (J/R)</p>

**Table 2**  
**Summary of Quality Objectives for Measurement by Alpha Spectroscopy ( $\alpha$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Energy Calibration Check	Performed using at least 3 isotopes within the energy range of 3 to 6 MeV	Monthly or when QC Check or Pulser is unacceptable	Slope $\leq 15$ keV/channel  Final peak energy positions of observed isotopes within $\pm 40$ keV.	Rerun	If outside acceptance criteria, reject all data ( R ).
Pulser Check	Performed to verify the proper operation of the detectors	Daily	FWHM $\leq 25$ keV  Shift of peak $< 40$ keV	Rerun to determine statistical significance of errant parameter  Check electronics Check interface	If $\geq$ FWHM Limits then reject all data.  Qualify data with "R"
Efficiency Calibration Check	Calibration source. Average efficiency for three (3) peaks	Annually or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters	$< \pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$ Difference within $2\sigma$ and/or $3\sigma$ of manufacturers specifications or historical laboratory information	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags between $2\sigma$ and $3\sigma$ - J $\geq 3\sigma$ - Reject all data  Qualify data with "R"
Tracer	Internal tracer utilized for isotope specific analysis	With every sample – added prior to sample preparation with the exception of grinding, when it is added after, but prior to dissolution of sample	30-110% Recovery or as determined by the client within this window.  FWHM of tracer peak $< 100$ keV  Tracer peak energy $\pm 50$ keV for all samples  Normalize Results	Investigate: Potential high recovery of actual isotope in sample  If recovery outside criteria – reanalysis is required beginning with the preparation.	If % recovery outside acceptance criteria, reject (R) results  Check for yield correction, if not contact laboratory to correct in calculations

**Table 2**  
**Summary of Quality Objectives for Measurement by Alpha Spectroscopy ( $\alpha$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Full Width at the Half Maximum (FWHM) Calibration	Energy Calibration Check for peak shape monitoring	Annually or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters  Defines shape of peak.	$< \pm 2 \sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$  and/or peak energy $\leq 50$ keV	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved  If $\geq 3 \sigma$ rerun once to determine statistical significance. If found to be acceptable, no corrective action is necessary. If fails, perform one or more of the following: 4) Check expiration date of standard 5) Check source positioning 6) Check instrument setup (ie: cables, connectors) Rerun daily calibration If rerun fails do not use instrument until resolved  If tracer FWHM fails than reanalysis is required	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”
Background	Long count with empty chamber used to accumulate data to determine cpm. Count must be $\geq$ than sample count	Weekly, when change in the daily background parameters, or when counting chamber changes have been made (ie: cleaning, replacement etc..)	$< \pm 2\sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved and conduct an empty chamber count; instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”

**Table 2**  
**Summary of Quality Objectives for Measurement by Alpha Spectroscopy ( $\alpha$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Blank	Blank to assess method / instrument contamination	One blank per matrix per batch (or every 20 samples)	No target analyte may be present above Reporting limit	<p>If analyte found in blank is non-detect in samples</p> <p>If analyte detected in associated samples</p> <p>If analyte concentration in blank exceeded 5% of concentration in one or more samples, re-analyze all affected samples</p>	<p>No effect</p> <p>Compare concentrations</p> <p>If concentration in blank exceeds 5% of the concentration found in one or more samples, report with qualification (B/UB).</p>
LCS	Interference-free matrix containing target radionuclide	One LCS per matrix per batch (or every 20 samples)	75-125%	Correct problem and repeat	When recovery is $\geq$ UL flag positives with J. For recovery <LL, flag J/UJ. For a recovery <30%, flag J/R.
MS/MSD	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	75-125%	Re-analyze or Recount	No action is taken based on MS recovery alone, use professional judgment.
Counting Duplicate or matrix duplicate		Per sample batch	<p>RPD <math>\leq</math> 25 for high activity When result is <math>&lt;5</math> X the QL, then use Normalized Difference (ND) <math>\leq</math> 2.6 or RER &lt; 1.0</p> <p>If one sample below QL and the other at least 5X above the QL</p> <p>If both sample results are below the QL – not applicable</p>	<p>Re – Analyze</p> <p>Lab should determine discrepancy and initiate corrective action</p>	No action is taken based on this only, use professional judgement

**Table 2**  
**Summary of Quality Objectives for Measurement by Alpha Spectroscopy ( $\alpha$ )**

<b>Quality Control Element</b>	<b>Description of Element</b>	<b>Frequency of Implementation</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>	<b>Flagging Criteria for Validator</b>
QA sample results	Comparison of primary laboratory sample result with QA laboratory sample result	10% of total primary samples	RPD $\leq$ 25 for high activity When result is $< 5 \times$ the QL, then use Normalized Difference (ND) $\leq$ 2.6 or RER $<$ 1.0	Investigate primary and QA data for other qualification  If data has not been qualified due to other criteria, re-analyze	Report data that has not been qualified due to other QA/QC criteria  Qualify data utilizing professional judgement. (J/R)

**Table 3**  
**Summary of Quality Objectives for Measurement by Liquid Scintillation Counting System ( $\beta$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	Series of $^{14}\text{C}$ and $^3\text{H}$ (or appropriate isotopes) and background (concentration must be at least 40,000 dpm)	3 vials each of series conducted annually	$< \pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$  For first calibration, compare to manufacturers specifications	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”
Efficiency Check	$^{14}\text{C}$ and $^3\text{H}$ (or appropriate isotopes)	At least every 24 hours or at closing of sample or analytical batch	$< \pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”
Background Check	$^{14}\text{C}$ and $^3\text{H}$ (or appropriate isotopes)	At least every 24 hours or at closing of sample or analytical batch	$< \pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$	$\bar{x}$ and $2\sigma$ – no action between $2\sigma$ and $3\sigma$ – investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”
Tracer/Carrier (as applicable)	Internal tracer utilized for isotope specific analysis	As applicable to the isotope being analyzed for.	30-110% Recovery Normalize Results	Investigate: Potential high recovery of actual isotope in sample  If recovery outside criteria – reanalysis is required beginning with the preparation.	Check for yield correction, if not contact laboratory to correct in calculations

**Table 3**  
**Summary of Quality Objectives for Measurement by Liquid Scintillation Counting System ( $\beta$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Quench Curve	Quench standards should contain at least 40,000 dpm of $^{14}\text{C}$ and/or $^3\text{H}$ (or appropriate isotope)	Annually or when a measured change in instrument or when process or instrument repair or modification is made (ie: different cocktail utilized, process modified with different acid, photomultiplier change, cleaning, replacement etc..)	$\pm 10\%$ of the quench of the efficiency standard (of a traceable standard)	Reanalyze beginning with the vial preparation	Reject all samples in which a quench curve outside of acceptance criteria is utilized.
Blank	Blank to assess method / instrument contamination	One blank per matrix per batch (or every 20 samples)	No target analyte may be present above Reporting limit	<p>If analyte found in blank is non-detect in samples</p> <p>If analyte detected in associated samples</p> <p>If analyte concentration in blank exceeded 5% of concentration in one or more samples, re-analyze all affected samples</p>	<p>No effect</p> <p>Compare concentrations</p> <p>If concentration in blank exceeds 5% of the concentration found in one or more samples, report with qualification (B/UB).</p>
LCS	Interference-free matrix containing target radionuclide	One LCS per matrix per batch (or every 20 samples)	75-125%	Correct problem and repeat	When recovery is $> \text{UL}$ flag positives with J. For recovery $< \text{LL}$ , flag J/UJ. For a recovery $< 30\%$ , flag J/R.

**Table 3**  
**Summary of Quality Objectives for Measurement by Liquid Scintillation Counting System ( $\beta$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
MS/MSD	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	75-125%	Re-analyze or reprep	No action is taken based on MS recovery alone, use professional judgment.
Counting Duplicate or matrix duplicate		Per sample batch	<p><math>RPD \leq 25</math> for high activity When result is <math>&lt; 5 \times</math> the QL, then use Normalized Difference (ND) <math>\leq 2.6</math> or <math>RER &lt; 1.0</math></p> <p>If one sample below QL and the other at least <math>5X</math> above the QL</p> <p>If both sample results are below the QL – not applicable</p>	<p>Re – Analyze</p> <p>Lab should determine discrepancy and initiate corrective action</p>	No action is taken based on this only, use professional judgement
QA sample results	Comparison of primary laboratory sample result with QA laboratory sample result	10% of total primary samples	<p><math>RPD \leq 25</math> for high activity When result is <math>&lt; 5 \times</math> the QL, then use Normalized Difference (ND) <math>\leq 2.6</math> or <math>RER &lt; 1.0</math></p>	<p>Investigate primary and QA data for other qualification</p> <p>If data has not been qualified due to other criteria, re-analyze</p>	<p>Report data that has not been qualified due to other QA/QC criteria</p> <p>Qualify data utilizing professional judgement. (J/R)</p>

**Table 4**  
**Summary of Quality Objectives for Measurement by Low Background Gas Flow Proportional Counting System**  
**( $\alpha$  and  $\beta$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Instrument Quality Control (IQC)	Counting results of Radioactive Check Source is compared to statistical average data		< $\pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$  For first calibration, compare to manufacturers specifications	$\bar{x}$ and $2\sigma$ - no action between $2\sigma$ and $3\sigma$ - investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with "R"
Radioactive Check Source Efficiency	Total Beta Radiation of a 1000 dpm Sr-90 Or Sr-90 or Cs-137 >10000 counts	Daily	< $\pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$  For first calibration, compare to manufacturers specifications	$\bar{x}$ and $2\sigma$ - no action between $2\sigma$ and $3\sigma$ - investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with "R"
Radioactive Check Source Efficiency	Total Alpha Radiation of a 1000 dpm Am-241, Th-230 or appropriate source	Daily	< $\pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$  For first calibration, compare to manufacturers specifications	$\bar{x}$ and $2\sigma$ - no action between $2\sigma$ and $3\sigma$ - investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with "R"
Background	Background for Alpha and Beta - measure for 30 minutes	Daily when instrument is utilized	< $\pm 2\sigma$ or within $\pm 2\sigma$ or $\pm 3\sigma$	$\bar{x}$ and $2\sigma$ - no action between $2\sigma$ and $3\sigma$ - investigate, notate warning $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags  between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with "R"

**Table 4**  
**Summary of Quality Objectives for Measurement by Low Background Gas Flow Proportional Counting System**  
**( $\alpha$  and  $\beta$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Self-absorption Curve	Required for both alpha and beta counting.	Annually – same matrix and geometry as samples – with a minimum of 7 points distributed throughout the mass range and at least 10,000 counts conducted	$\leq 1\%$ or $r^2 \geq 0.90$ or $\leq 10\%$ of independent source	No action if $\leq 1\%$ Reanalyze all associated samples when $> 1\%$  $r^2 \leq 0.90$ create a new curve  No action if $< 10\%$ Reanalyze all associated samples when $> 10\%$	$\leq 1\%$ - No flags $> 1\%$ R  No valid curve – reject data – “R” flag  $\leq 10\%$ no flag $> 10\%$ Reject (R)
Cross Talk Curves	Gross alpha and beta overlap. Potential contamination of beta emitters in alpha region. Potential alpha emitters in Beta region	Annually – same matrix and geometry as samples – with a minimum of 7 points distributed throughout the mass range and at least 10,000 counts conducted	$< 1\%$ or compare to manufacture specifications	No action if $\leq 1\%$ Reanalyze all associated samples when $> 1\%$	$\leq 1\%$ - No flags $> 1\%$ R
Tracer or Carrier (as applicable)	Internal tracer utilized for isotope specific analysis	With every sample	30-110% Recovery Normalize Results	Investigate: Potential high recovery of actual isotope in sample  If recovery outside criteria – reanalysis is required beginning with the preparation.	Check for yield correction, if not contact laboratory to correct in calculations

**Table 4**  
**Summary of Quality Objectives for Measurement by Low Background Gas Flow Proportional Counting System**  
**( $\alpha$  and  $\beta$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Blank	Blank to assess method / instrument contamination	One blank per matrix per batch (or every 20 samples)	No target analyte may be present above Reporting limit	If analyte found in blank is non-detect in samples If analyte detected in associated samples  If analyte concentration in blank exceeded 5% of concentration in one or more samples, re-analyze all affected samples	No effect  Compare concentrations If concentration in blank exceeds 5% of the concentration found in one or more samples, report with qualification (B/UB).
LCS	Interference-free matrix containing target radionuclide	One LCS per matrix per batch (or every 20 samples)	75-125%	Correct problem and repeat	When recovery is >UL flag positives with J. For recovery <LL, flag J/UJ. For a recovery <30%, flag J/R.
MS/MSD as applicable	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	75-125%	Re-analyze or Reprep	No action is taken based on MS recovery alone, use professional judgment.
Counting Duplicate or matrix duplicate		Per sample batch	RPD $\leq$ 25 for high activity When result is $<5$ X the QL, then use Normalized Difference (ND) $\leq$ 2.6 or RER $<$ 1.0  If one sample below QL and the other at least 5X above the QL  If both sample results are below the QL – not applicable	Re – Analyze  Lab should determine discrepancy and initiate corrective action	No action is taken based on this only, use professional judgement

**Table 4**  
**Summary of Quality Objectives for Measurement by Low Background Gas Flow Proportional Counting System**  
**( $\alpha$  and  $\beta$ )**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
QA sample results	Comparison of primary laboratory sample result with QA laboratory sample result	10% of total primary samples	RPD $\leq$ 25 for high activity When result is $< 5 \times$ the QL, then use Normalized Difference (ND) $\leq$ 2.6 or RER $<$ 1.0	Investigate primary and QA data for other qualification  If data has not been qualified due to other criteria, re-analyze	Report data that has not been qualified due to other QA/QC criteria  Qualify data utilizing professional judgement. (J/R)

**Table 5**  
**Summary of Quality Objectives for Measurement by Laser Induced Kinetic Phosphorescence (Uranium)**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	Series of 3 standards per range that encompasses the concentration range of the samples being analyzed.  Low range – approx. 1.0 µg/L to 10 µg/L  High Range – approx. 10 µg/L to 1000 µg/L	Daily	$R^2 > 0.990$  And  150<Lifetime<350 us (microseconds)  % Discrepancy < 10%	Rerun  If continues to fail, instrument is unusable.	Qualify with R when less than 3 standards are used  If r2 >0.990 or lifetime is not met conduct post-spike sample analysis
Continuing Calibration Verification	A standard within each range  Low range – 1 µg/L to 10 µg/L  High range – 500 µg/l	Every 10 samples	± 10% (90-110%)	Correct problem and rerun.	For each target analyte, when D>10% with a negative bias –J/UJ When D>10% with a positive bias –J positives only. For <60% Reject (R)
Continuing Calibration Blank	Reagent Blank to assess method contamination	Every 10 samples	No target analyte may be present above Reporting limit	If analyte found in blank is non-detect in samples  If analyte detected in associated samples  If analyte concentration in blank exceeded 10% of concentration in one or more samples, re-analyze all affected samples	No effect  Compare concentrations  If concentration in blank less than 10% of the concentration found in one or more samples, report with qualification (B/UB).

**Table 5**  
**Summary of Quality Objectives for Measurement by Laser Induced Kinetic Phosphorescence (Uranium)**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Method of Standard Additions (MSA)	KPA measurements of total uranium on filters are made by MSA	When total uranium on cellulose ester filters is required	Results must agree within 10%	Rerun	If agreement > 10 % R
Calibration Checks	During sample analysis. Analysis is conducted on two reference solutions with one being within the calibration range, and must be a NON-calibration curve solution	Daily for both high and low range	± 10%	Correct problem and rerun.	For each target analyte, when D>10% with a negative bias -J/R. when D>10% with a positive bias -J positives only.
Blank	Blank to assess method / instrument contamination	One blank per matrix per batch (or every 20 samples)	No target analyte may be present above Reporting limit	If analyte found in blank is non-detect in samples If analyte detected in associated samples  If analyte concentration in blank exceeded 5% of concentration in one or more samples, re-analyze all affected samples	No effect  Compare concentrations If concentration in blank exceeds 5% of the concentration found in one or more samples, report with qualification (B/UB).
LCS	Interference-free matrix containing target radionuclide	One LCS per matrix per batch (or every 20 samples)	75-125%	Correct problem and repeat	When recovery is >UL flag positives with J. For recovery <LL, flag J/UJ. For a recovery <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	75-125%	Reanalyze or Reprep	No action is taken based on MS recovery alone, use professional judgment.

**Table 5**  
**Summary of Quality Objectives for Measurement by Laser Induced Kinetic Phosphorescence (Uranium)**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Matrix duplicate		Per sample batch	<p>RPD <math>\leq</math> 25 for high activity When result is <math>&lt;5</math> X the QL, then use Normalized Difference (ND) <math>\leq</math> 2.6 or RER <math>&lt;</math> 1.0</p> <p>If one sample below QL and the other at least 5X above the QL</p> <p>If both sample results are below the QL – not applicable</p>	Re – Analyze	No action is taken based on this only, use professional judgement
QA sample results	Comparison of primary laboratory sample result with QA laboratory sample result	10% of total primary samples	<p>RPD <math>\leq</math> 25 for high activity When result is <math>&lt;5</math> X the QL, then use Normalized Difference (ND) <math>\leq</math> 2.6 or RER <math>&lt;</math> 1.0</p>	<p>Investigate primary and QA data for other qualification</p> <p>If data has not been qualified due to other criteria, re-analyze</p>	<p>Report data that has not been qualified due to other QA/QC criteria</p> <p>Qualify data utilizing professional judgement. (J/R)</p>

**Table 6**  
**Summary of Quality Objectives for Measurement by Lucas Cell Counting (Radon)**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
Initial Calibration	Series of known Standards (at least three)	Annually or when daily check indicates a condition that warrants recalibration	Standard deviation < 10% of the average cell constant	Rerun Calibration	>10% standard deviation J/R as appropriate.
Daily Background Check	Radon Counting cell utilized to determine gross counts per minute	Daily performance of two checks: 1)photomultiplier without a cell in it 2)photomultiplier with a Lucas Cell in place.	< $\pm 2 \sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$ or $\leq 0.3$ cpm	$\bar{x}$ and $2\sigma$ – no action  between $2\sigma$ and $3\sigma$ – investigate, notate warning  $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”
Instrument Performance Check	Record gross count of a known level	Daily	< $\pm 2 \sigma$ or within $\pm 2 \sigma$ or $\pm 3 \sigma$	$\bar{x}$ and $2\sigma$ – no action  between $2\sigma$ and $3\sigma$ – investigate, notate warning  $\geq 3\sigma$ - take corrective action, instrument not usable until resolved	$\bar{x}$ and $2\sigma$ - No flags between $2\sigma$ and $3\sigma$ - J  $\geq 3\sigma$ - Reject all data  Qualify data with “R”
Blank	Blank to assess method / instrument contamination	One blank per matrix per batch (or every 20 samples)	No target analyte may be present above Reporting limit	If analyte found in blank is non-detect in samples If analyte detected in associated samples  If analyte concentration in blank exceeded 5% of concentration in one or more samples, re-analyze all affected samples	No effect  Compare concentrations If concentration in blank exceeds 5% of the concentration found in one or more samples, report with qualification (B/UB).

**Table 6**  
**Summary of Quality Objectives for Measurement by Lucas Cell Counting (Radon)**

Quality Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria	Corrective Action	Flagging Criteria for Validator
LCS	Interference-free matrix containing target radionuclide	One LCS per matrix per batch (or every 20 samples)	75-125%	Correct problem and repeat	When recovery is >UL flag positives with J. For recovery <LL, flag J/UJ. For a recovery <30%, flag J/R.
MS	Sample matrix spiked with all target analytes prior to digestion	1 per sample batch	75-125%	Reanalyze or Reprep	No action is taken based on MS recovery alone, use professional judgment.
Matrix duplicate		Per sample batch	RPD $\leq$ 25 for high activity When result is <5 X the QL, then use Normalized Difference (ND) $\leq$ 2.6 or RER < 1.0  If one sample below QL and the other at least 5X above the QL  If both sample results are below the QL – not applicable	Re – Analyze	No action is taken based on this only, use professional judgement
QA sample results	Comparison of primary laboratory sample result with QA laboratory sample result	10% of total primary samples	RPD $\leq$ 25for high activity When result is <5 X the QL, then use Normalized Difference (ND) $\leq$ 2.6 or RER < 1.0	Investigate primary and QA data for other qualification  If data has not been qualified due to other criteria, re-analyze	Report data that has not been qualified due to other QA/QC criteria  Qualify data utilizing professional judgement. (J/R)

## **SECTION II**

# **RADIOLOGICAL INTERFERENCES**

This Section provides a summary of interferences associated with the preparation, extraction and analysis of samples for radiological isotopes.

## 1.0 Gamma Spectroscopy

Gamma spectrometry has many potential interferences which are usually in the form of radionuclides with unresolved photon emissions.

### Common Radionuclide Interferences in Gamma Spec.

<b>Radionuclide</b>	<b>Interferences</b>
Ac-228	Am-241, Cs-135, Zn-65, Zr-95, Mn-54, Cr-51, Bi-212, Eu-155, Nb-94, Be-7, K-40, Th-231, Cd-109, Bi-207, Ra-223, Ce-141
Ag-110m	Nb-95, Rh-106
Am-241	Pm-147 (from summation)
Bi-212	Pm-149
Bi-214	Cd-109, I-126, Pb-211, Sn-11, Nb-95, Nb-94, La-140
Co-57	Pm-147
Co-60	Pa-233 (from double escape peak)
Cs-134	Be-7, Sb-124
Cs-137	Te-125m
Eu-152	Pb-210, Bi-210, Pm-147, Co-57, Co-58
Eu-154	Zr-95, Pm-147, Co-57, Be-7, Na-22, Tl-208
Eu-155	Bi-210, Pb-210, Sn-126, Am-241, Cd-109, Np-237
Np-237	Sn-126
Pa-231	Ce-139, Ba-133
Pa-233	Pm-147
Pa-234	Hg-203, Gd-153, Ce-144, Sb-125
Pa-234m	Nb-95
Pb-212	Cd-109, Pm-146, Nb-95m, Np-237, Ra-224, Sn-126, Np-239
Pb-214	Np-237, Cd-109, Ce-143, Ra-224, Ac-227, Bi-211, Sn-126
Ra-223	Sn-117m
Ra-226	Cd-109
Rn-219	Cm-247
Sb-125	Cs-134, Cf-251, Cr-51
Sn-126	Cd-109, U-235, Np-237
Th-227	Nb-95m
Th-228	Ce-144
Th-230	Tm-171
Th-231	Am-241, Gd-153
Th-232	Am-241
Tl-208	Hg-203
U-234	Co-57, Pm-147
U-235	Tm-125m, Cd-109, Eu-155, Y-90, Ce-141, Ce-139, Mo-99, Co-57, Th-229, Pm-147, Tc-99m

## **2.0 Alpha Spectroscopy**

Alpha spectrometry has many potential interferences which are usually in the form of radionuclides with unresolved alpha emissions. The unresolved alpha emissions are sometimes due to detector quality, counting chamber pressure, distance between the source and the detector or source plate quality. Other potential interferences are often due to high alpha activity rates or attenuation of the alpha emissions, which results in poorly resolved peaks.

## **3.0 Liquid Scintillation Counting**

Uranium daughters such as  $^{234m}\text{Protactinium}$  and  $^{234}\text{Thorium}$  can lead to false positive results for  $^{99}\text{Technetium}$ . The  $^{242}\text{Plutonium}$  tracer may lead to positive biased results for  $^{241}\text{Plutonium}$  and must be compensated for. Uranium may also interfere with the determination of  $^{55}\text{Iron}$  and  $^{147}\text{Promethium}$ .

Quenching may occur for several reasons among these are impurities in the scintillation solution.

Also the analysis may be affected if the scintillators are exposed to light and are not dark-adapted.

Other interferences may also be encountered that can only be identified through a thorough review of spectral data.

## **4.0 Gas Flow Proportional Counting System**

Interferences associated with the Gas Flow Proportional Counting System may include a detector contaminated with radioactive material, therefore resulting in a high background reading and interference with the measurement of a sample.

The radioactivity of the sample is not separated from the solids of the sample; therefore the solids concentration is a limiting factor in the sensitivity of the method for a sample.

Radionuclides that are volatile under sample preparation conditions of this method can't be measured. Other radionuclides may also be lost during the sample evaporation and drying.

Moisture absorbed by the sample residue, if uncorrected, can lead to low-biased results due to the increase in self-absorption.

Heterogeneity of the sample residue in the counting planchet interferes with the accuracy and precision of the method.

Cross talk may also cause interference.

### **5.0 Laser Induced Kinetic Phosphorescence**

High levels of chlorides and organic materials can suppress the phosphorescence and lead to low biased results.

### **6.0 Lucas Cell Counting**

No known interferences for Radon measurements.

# **SECTION III DATA REPORTING**

# Data Reporting

Radiological data packages submitted by laboratories should contain sufficient data in order to allow the data reviewer to assess the accuracy, precision, representativeness, comparability, and sensitivity. The analytical data packages must be presented in a well-organized manner, with pages sequentially numbered and include at a minimum the following:

1. Cover Page
2. Table of contents
3. Case narrative
4. Data Reports (computer generated)
5. Results Reports (instrument generated or spreadsheet)
6. Monthly instrument calibration data package (as applicable)
7. Calibration Standards
8. Run Log
9. Raw Data
10. Preparation Data Sheets
11. Instrument Printouts
12. QC/QA Summary
13. Corrective Actions Documentation
14. Chain of Custody/Cooler Receipt Forms

## 1. Cover Page:

Name of the Laboratory, and full address  
Project Name & Site location  
Date Report Issued  
Contract Number  
Signature/Date of Laboratory Director, Quality Assurance Officer, and/or Project Manager  
Statement indicating the authenticity of the data

## 2. Table of Content:

An index or table of content should be included to allow easy retrieval of information for sample analyses.

## 3. Case Narrative:

A detailed description of any problems with analysis should be discussed. Corrective actions and resolution should be addresses in the case narrative. Sample preparation and/or analysis out of the holding time should be noted. Samples that were received and were not analyzed should be indicated. Within the case narrative, a discussion must be given as to whether sample results were normalized.

## 4. Result Reports:

The following information is needed:  
Project Name  
Field Sample ID as indicated on the Chain of Custody  
Laboratory sample ID that correspond to the field sample ID  
Method Numbers for sample preparation

Method Number for sample analysis  
Sample date collected  
Sample date prepared  
Sample date analyzed  
Dilution Factor (as applicable)  
Sample weight for soil/sediment  
Sample volume for water samples  
Matrix type  
Percent solid  
Tracer/Carrier Recoveries (as applicable)  
Instrument Printouts

**5. QC/QA Summary:**

This summary should include results of method blanks, LCS, MS/MSDs (as applicable), laboratory duplicates and recovery of the carriers/tracers (as applicable per method) spikes for the QC/QA samples. All levels of spikes, accuracy (% recovery) and precision (RPD) must be provided. Also, the acceptance criteria for the recoveries and for the RPD must be documented in the data packages.

**6. Corrective Actions:**

All corrective actions that were initiated during the project and their resolution must be included in the data packages.

**7. Chain-of-Custody/Cooler Receipt Forms:**

Original chain of custody and the cooler receipt forms, that are applicable to the analyzed samples, must be included in the data packages.

**Section IV**  
**Data Validation Radiological Reports**  
**(DVRR)**

# DATA VALIDATION REPORTS

Purpose: The purpose of the Data Validation Radiological Report (DVRR) is to provide the data user with a timely review of data quality, both the verification and validation. This is achieved through the review and evaluation of QA samples, and through an examination of the corresponding project sample data by the contractor.

Report Contents: The DVRR should contain the items listed below, although not necessarily in the format nor in the order presented. The format should present the information in an organized fashion, which the reader can easily comprehend. The information below assumes that QA samples were collected and analyzed as part of the project QA effort.

**Project Information:** This Section should contain any pertinent reference information to aid the reader in assessing the relevance of this report.

**Executive Summary:** A summary description of the QA/QC effort expended on this data should be presented. The identities of the laboratories performing the various project tests should be cited.

The report content is mostly left up to the author, keeping in mind that the intent is to convey the overall results of the QA effort. Any major findings should be summarized here. State the possible effects upon the project sample data based upon: 1) a review of QA sample inspection results; 2) a comparison of QA sample data with project sample data; 3) a comparison of QC sample data with project sample data; 4) a review of primary and QC data; and 5) a review of field QC data. State when a data review revealed no potential effects upon the project data, based on percent (%) usability. Also state when a complete data review could not be performed, i.e., "A complete data review could not be performed because there were no QC or QA samples collected.

# STANDARD OPERATING PROCEDURE: DATA VALIDATION REPORT

This report serves as the Standard Operating Procedure (SOP) for executing data validation. Attachment A contains the data validation checklists.

## **Attachment A:**

The checklists for each method are provided in Attachment B as follows:

**Gamma Spectroscopy (Gamma –  $\gamma$ )**

**Alpha Spectroscopy (Alpha –  $\alpha$ )**

**Liquid Scintillation Counting (Beta –  $\beta$ )**

**Gas Flow Proportional Counting System (Alpha –  $\alpha$  and Beta –  $\beta$ )**

**Laser Induced Kinetic Phosphorescence (Uranium)**

**Lucas Cell Counting (Radon)**

The reviewer/validator will submit a Validation Report that summarizes the findings to inform the data user on the limitation of the data, discussing areas of concerns, and issues that might affect the quality of the data. The validator will review raw data submitted by the laboratory, QC results, Chain of Custody forms, Cooler Receipt forms, and laboratory narratives. The validator may use the attached Checklists, or modification of them in documenting the findings, however, the validator must use the guideline presented in the attached checklists, and the QO tables included in this document under Radiological Analysis Criteria, (Section I). Laboratories might have QC limits that are more stringent than the criteria included in the QO tables, however, the validator must validate the data based on the QO tables, and not on the laboratory QC limits. So, data should not be considered estimated "J" if they are out of the laboratory but within the established QC limits that are presented in the QO tables, and the validator would override the laboratory's qualifier "J" and consider the data unqualified.

When a deviation from a procedure is observed, the Validator must contact the laboratory to obtain additional information in order to reach a decision. The report will contain an overall assessment, and specifics of any relevant issues.

## **The proposed contents of Data Validation Reports is as follows:**

Cover

Title Page: With signature blocks for US Army Corps principal member and senior reviewers

Table of Contents

Glossary

Acronyms

Background

Description of Prior Activities

Description of Prior Data

Description of Work Performed

Approach/Methodology

Data validation

Data Completeness

Date Usability Summary of Qualifications

Impact on data Quality

Data Review of both Primary and QA Laboratory Data

Description of Data Completeness Review

Data Summary Tables

Summary of Laboratory Completeness

Summary of Field Quality Control Sample Completeness

Deficiencies in Data and any Flagging Codes

Source of Deficiencies

Impact on Quality of Data

Data Review Comments

Analysis Specific Comments

Discussion of comparison of Primary and QA data

Finding on Overall Quality of Data (%Usability) (see Appendix F for definition)

Attachments:

1. Chain of Custody and Cooler Receipt Forms
2. Analytical Results with qualifiers for compounds/analyte based on reviewer's findings

**ATTACHMENT A  
DATA VALIDATION GUIDELINES AND  
CHECKLISTS**

# DATA VALIDATION GUIDELINES

## INTRODUCTION

This Section presents checklists on the radiological laboratory data evaluation to be used by Louisville District Chemists and/or contractors. This review process will be conducted using calibration, QC results, chain-of-custody forms, and laboratory narratives. When a deviation from the procedure is observed, the laboratory will be requested to provide additional information to enable the reviewer to reach a decision. The data validation report will address each of the following sections as applicable.

- 1.0 Gamma Spectroscopy (Gamma –  $\gamma$ )
- 2.0 Alpha Spectroscopy (Alpha –  $\alpha$ )
- 3.0 Liquid Scintillation Counting (Beta –  $\beta$ )
- 4.0 Gas Flow Proportional Counting System (Alpha –  $\alpha$  and Beta –  $\beta$ )
- 5.0 Laser Induced Kinetic Phosphorescence (Uranium)
- 6.0 Lucas Cell Counting (Radon)

## VALIDATION PROCEDURE

The Validator will review the following requirements as applicable per method:

- Holding times
- Background checks
- Tracer or carriers
- Matrix spike/Matrix Spike Duplicate (as applicable)
- Calibration
  - Initial calibration
  - Energy Calibration (as applicable)
  - FWHM (as applicable)
  - Efficiency Calibrations (as applicable)
  - Geometry Calibrations (as applicable)
  - QC or pulser Checks
  - Quench Curves (as applicable)
- Blanks (as applicable)
  - Method blank
  - Field blank
  - Instrument blank
- Laboratory Control Sample
- Duplicate sample results
- Preparation and/or run logs
  -
- Laboratory duplicate
  - Field duplicate
- Quality Assurance Sample Results (from QA Laboratory)
- Laboratory data assessment (Narrative report)

# **DATA VALIDATION CHECK LIST**

## GAMMA SPECTROSCOPY- $\gamma$

Project Name: \_\_\_\_\_  
 Laboratory: \_\_\_\_\_  
 Batch Number(s): \_\_\_\_\_  
 Sample Delivery Group (SDG): \_\_\_\_\_

	<u>Yes</u>	<u>No</u>	<u>NA</u>
1. Holding Time: (a) Were samples preserved?	[ ]	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]	[ ]
2. Initial Calibration: (a) Were energy calibrations evenly distributed across the Energy range (0-2MeV)?	[ ]	[ ]	[ ]
(b) Was the energy difference $\leq 0.5\text{KeV}$ for all points?	[ ]	[ ]	[ ]
OR (c) Initial calibration between $\bar{x}$ and $2\sigma$ ?	[ ]	[ ]	[ ]
(d) Initial calibration between $2\sigma$ and $3\sigma$ ? - investigate and notate warning	[ ]	[ ]	[ ]
(e) Initial calibration $\geq 3\sigma$ ? - take corrective action – instrument not usable	[ ]	[ ]	[ ]
3. FWHM – check for peak shape monitoring (a) Was a FWHM calibration conducted?	[ ]	[ ]	[ ]
(b) FWHM $\leq 3.0\text{keV}$ at 1332 keV or within manufacturers specifications?	[ ]	[ ]	[ ]
(c) FWHM difference $< 0.5\text{ keV}$ for selected peaks? (Am-241,Co-60 and Cs-137)	[ ]	[ ]	[ ]
OR (d) FWHM $\bar{x}$ and $2\sigma$ ? - no action	[ ]	[ ]	[ ]
(e) FWHM between $2\sigma$ and $3\sigma$ ? - Investigate and notate warning	[ ]	[ ]	[ ]
(f) FWHM $> 3\sigma$ ? - take corrective action – instrument not usable	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
4. Efficiency Calibration and/or Geometry Calibration Check			
(a) Was an efficiency/geometry calibration conducted? (annually or as necessary)	[ ]	[ ]	[ ]
(b) Was an energy resolution and efficiency check conducted daily?	[ ]	[ ]	[ ]
Was it within acceptable limits?	[ ]	[ ]	[ ]
(c) Efficiency calibration $\bar{x}$ and $2\sigma$ ? - no action	[ ]	[ ]	[ ]
(d) Efficiency calibration between $2\sigma$ and $3\sigma$ ? - Investigate and notate warning	[ ]	[ ]	[ ]
(e) Efficiency calibration $\geq 3\sigma$ ? - take corrective action – instrument not usable	[ ]	[ ]	[ ]
(f) Geometry Check within + or – 10% of known value?	[ ]	[ ]	[ ]
5. Background			
(a) Was a background subtraction spectrum conducted bi-weekly or when necessary? Was it within acceptable limits?	[ ]	[ ]	[ ]
(b) Was the detector background conducted Daily when the instrument was utilize? Was it within acceptable limits?	[ ]	[ ]	[ ]
6. Sample Quality Control:			
(a) Blanks: Was a blank conducted one per matrix and geometry per batch? - Were target analytes $\leq$ RL?	[ ]	[ ]	[ ]
(b) LCS: Were the percent recoveries for LCS within the limits? (75-125%)	[ ]	[ ]	[ ]
(c) Counting duplicate or matrix duplicate: Was the RPD within acceptable criterion? ( $\leq 25\%$ for high activity or $ND \leq 2.6$ for results $< 5X$ the QL) or $RER < 1.0$	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
7. QA Sample Results			
(a) Were samples sent to a QA laboratory?	[ ]	[ ]	[ ]
(b) Was the RPD within acceptable criterion? ( $\leq 25\%$ for high activity or $ND \leq 2.6$ for results $< 5X$ the QL) or $RER < 1.0$ ?	[ ]	[ ]	[ ]
8. Comments (attach additional sheets if necessary):			
Validated/Reviewed by:			
Signature:		Date:	
Name:			

## ALPHA SPECTROSCOPY - $\alpha$

Project Name: \_\_\_\_\_  
 Laboratory: \_\_\_\_\_  
 Batch Number(s): \_\_\_\_\_  
 Sample Delivery Group (SDG): \_\_\_\_\_

	<u>Yes</u>	<u>No</u>	<u>NA</u>
1. Holding Time:			
(a) Were samples preserved?	[ ]	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]	[ ]
2. Energy Calibration:			
(a) Was an energy calibration conducted?	[ ]	[ ]	[ ]
Utilizing 3 isotopes within the energy range of 3 to 6 MeV?	[ ]	[ ]	[ ]
(b) Was the slope $\leq 15$ keV / channel?	[ ]	[ ]	[ ]
(c) Was the final peak energy position of the observed isotopes within $\pm 40$ keV?	[ ]	[ ]	[ ]
3. Pulser Check:			
(a) Was a pulser check conducted daily?	[ ]	[ ]	[ ]
(b) Was the FWHM $\leq 25$ keV and the peak shift $< 40$ keV?	[ ]	[ ]	[ ]
4. Efficiency Calibration:			
(a) Was an efficiency calibration conducted? (annually)	[ ]	[ ]	[ ]
(b) Efficiency calibration $\bar{x}$ and $2\sigma$ ? - no action	[ ]	[ ]	[ ]
(c) Efficiency calibration between $2\sigma$ and $3\sigma$ ? - investigate and notate warning	[ ]	[ ]	[ ]
(d) Efficiency calibration $\geq 3\sigma$ ? take corrective action – instrument not usable	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
5. Tracer: (a) Was a tracer utilized and within acceptable recovery? (30-110%)  (b) Was the FWHM of tracer peak <100 keV?  (c) Was the tracer peak energy $\pm 50$ keV for all samples  (d) Were the sample results normalized?	[ ]	[ ]	[ ]
6. FWHM – check for peak shape monitoring (a) Was a FWHM Calibration conducted?  (b) FWHM of tracer peak < 100keV and/or the peak energy within $\pm 50$ keV? OR (c) FWHM $\bar{x}$ and $2\sigma$ ? - no action  (d) FWHM between $2\sigma$ and $3\sigma$ ? - investigate and notate warning  (e) FWHM $\geq 3\sigma$ ? - take corrective action – instrument not usable	[ ]	[ ]	[ ]
7. Background (a) Was a background subtraction spectrum conducted bi- weekly or when necessary?  (b) Was it within acceptable limits?	[ ]	[ ]	[ ]
8. Sample Quality Control: (a) Blanks: Was a blank conducted one per matrix per batch? - Were target analytes $\leq$ RL?  (b) LCS: Were the percent recoveries for LCS within the limits? (75-125%)  (c) MS/MSD: Were the percent recoveries within the limits? (75-125%)  (d) Counting duplicate or matrix duplicate: Was the RPD within acceptable criterion? ( $\leq 25\%$ for high activity or ND $\leq 2.6$ for results < 5X the QL) or RER < 1.0?	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
9. QA Sample Results:			
(a) Were samples sent to a QA laboratory?	[ ]	[ ]	[ ]
(b) Was the RPD within acceptable criterion? ( $\leq 25\%$ for high activity or $ND \leq 2.6$ for results $< 5X$ the QL) or $RER < 1.0$ ?	[ ]	[ ]	[ ]
10. Comments (attach additional sheets if necessary):			
Validated/Reviewed by:			
Signature:		Date:	
Name:			

## LIQUID SCINTILLATION COUNTING - $\beta$

Project Name: \_\_\_\_\_  
 Laboratory: \_\_\_\_\_  
 Batch Number(s): \_\_\_\_\_  
 Sample Delivery Group (SDG): \_\_\_\_\_

	<u>Yes</u>	<u>No</u>	<u>NA</u>
1. Holding Time:			
(a) Were samples preserved?	[ ]	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]	[ ]
2. Initial Calibration:			
(a) Was a Calibration conducted from a series of C-14, H-3 (or appropriate isotopes) and background standards?	[ ]	[ ]	[ ]
(b) Was the concentrations > 40,000 dpm?	[ ]	[ ]	[ ]
(c) Initial calibration between $\bar{x}$ and $2\sigma$ ? - no action	[ ]	[ ]	[ ]
(d) Initial calibration between $2\sigma$ and $3\sigma$ ? - investigate and notate warning	[ ]	[ ]	[ ]
(e) Initial calibration $\geq 3\sigma$ ? - take corrective action – instrument not usable	[ ]	[ ]	[ ]
3. Efficiency Calibration:			
(a) Was an efficiency calibration conducted every 24 hours or at the closing of the sample or analytical batch?	[ ]	[ ]	[ ]
(b) Efficiency calibration $\bar{x}$ and $2\sigma$ ? - no action	[ ]	[ ]	[ ]
(c) Efficiency calibration between $2\sigma$ and $3\sigma$ ? - investigate and notate warning	[ ]	[ ]	[ ]
(d) Efficiency calibration $\geq 3\sigma$ ? - take corrective action – Instrument not usable	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
<p>4. Background Check:</p> <p>(a) Was a background check conducted every 24 hours or at the closing of the sample or analytical batch?</p> <p>(b) Efficiency calibration <math>\bar{x}</math> and <math>2\sigma</math>? - no action</p> <p>(c) Efficiency calibration between <math>2\sigma</math> and <math>3\sigma</math>? - investigate and notate warning</p> <p>(d) Efficiency calibration <math>\geq 3\sigma</math>? - take corrective action – instrument not usable</p>	[ ]	[ ]	[ ]
<p>5. Tracer/Carrier:</p> <p>(a) Was a tracer or carrier utilized and within acceptable recovery? (30-110%)</p> <p>(b) Were the sample results normalized?</p>	[ ]	[ ]	[ ]
<p>6. Quench Curve:</p> <p>(a) Was a quench curve conducted at least annually?</p> <p>(b) Was the quench standard at least 40,000 dpm of C-14 and/or H-3?</p> <p>(c) Was the quench curve within <math>\pm 10\%</math> of the efficiency standards?</p>	[ ]	[ ]	[ ]
<p>7. Sample Quality Control:</p> <p>(a) Blanks: Was a blank conducted one per matrix per batch? - Were target analytes <math>\leq</math> RL?</p> <p>(b) <u>LCS</u>: Were the percent recoveries for LCS within the limits? (75-125%)</p> <p>(c) <u>MS</u>: Were the percent recoveries for MS within the QC limits? (75-125%)</p> <p>(d) <u>Counting duplicate or matrix duplicate</u>: Was the RPD within acceptable criterion? (<math>\leq 25\%</math> for high activity or <math>ND \leq 2.6</math> for results <math>&lt; 5X</math> the QL) or RER <math>&lt; 1.0</math></p>	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
8. QA Sample Results:			
(a) Were samples sent to a QA laboratory?	[ ]	[ ]	[ ]
(b) Was the RPD within acceptable criterion? ( $\leq 25\%$ for high activity or $ND \leq 2.6$ for results $< 5X$ the QL) or $RER < 1.0$ ?	[ ]	[ ]	[ ]
9. Comments (attach additional sheets if necessary):			
Validated/Reviewed by:			
Signature:		Date:	
Name:			

# GAS FLOW PROPORTIONAL COUNTING SYSTEM - $\alpha$ and $\beta$

Project Name: \_\_\_\_\_  
 Laboratory: \_\_\_\_\_  
 Batch Number(s): \_\_\_\_\_  
 Sample Delivery Group (SDG): \_\_\_\_\_

	<u>Yes</u>	<u>No</u>	<u>NA</u>
1. Holding Time:			
(a) Were samples preserved?	[ ]	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]	[ ]
2. Instrument Quality Control:			
(a) Was a counting source checked and compared to statistical average data?	[ ]	[ ]	[ ]
(b) Initial calibration between $\bar{x}$ and $2\sigma$ ? - no action	[ ]	[ ]	[ ]
(c) Initial calibration between $2\sigma$ and $3\sigma$ ? - investigate and notate warning	[ ]	[ ]	[ ]
(d) Initial calibration $\geq 3\sigma$ ? - take corrective action – instrument not usable	[ ]	[ ]	[ ]
3. Efficiency Calibration:			
(a) Was an efficiency calibration conducted daily for both alpha and beta applicable?	[ ]	[ ]	[ ]
(b) Efficiency calibration $\bar{x}$ and $2\sigma$ ? - no action	[ ]	[ ]	[ ]
(c) Efficiency calibration between $2\sigma$ and $3\sigma$ ? - investigate and notate warning	[ ]	[ ]	[ ]
(d) Efficiency calibration $\geq 3\sigma$ ? - take corrective action – instrument not usable	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
4. Background			
(a) Was a background check conducted for both alpha and beta?	[ ]	[ ]	[ ]
Was it within acceptable limits?	[ ]	[ ]	[ ]
5. Self-Absorbtion Curve			
(a) Was a self-absorbtion curve performed daily for both alpha and beta?	[ ]	[ ]	[ ]
- For the same matrix and geometry as the samples?			
- With a minimum of 7 points distributed throughout the mass range?			
- For at least 10,000 counts?			
Was it within acceptable limits? ( $\leq 1\%$ or $r^2 \geq 0.90$ or $\leq 10\%$ of an independent source)	[ ]	[ ]	[ ]
6. Cross Talk Curve			
(a) Was a cross talk curve performed daily for both gross alpha and beta?	[ ]	[ ]	[ ]
- For the same matrix and geometry as the samples?			
- Within a minimum of 7 points distributed throughout the mass range?			
- For at least 10,000 counts?			
(b) Was it within acceptable limits? ( $\leq 1\%$ )	[ ]	[ ]	[ ]
7. Tracer/Carrier (as applicable)			
(a) Was a tracer or carrier utilized and within acceptable recovery? (30-110%)	[ ]	[ ]	[ ]
(b) Were the sample results normalized?	[ ]	[ ]	[ ]
8. Sample Quality Control:			
(a) Blanks: Was a blank conducted one per matrix per batch?	[ ]	[ ]	[ ]
- Were target analytes $\leq$ RL?			
(b) LCS: Were the percent recoveries for LCS within the limits? (75-125%)	[ ]	[ ]	[ ]
(c) MS/MSD as applicable: Were the percent recoveries for MS within the QC limits? (75-125%)	[ ]	[ ]	[ ]
(d) Counting duplicate or matrix duplicate: Was the RPD within acceptable criterion? ( $\leq 25\%$ for high activity or $ND \leq 2.6$ for results $< 5X$ the QL) or $RER < 1.0$	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
9. QA Sample Results			
(a) Were samples sent to a QA laboratory?	[ ]	[ ]	[ ]
(b) Was the RPD within acceptable criterion? ( $\leq 25\%$ for high activity or $ND \leq 2.6$ for results $< 5X$ the QL) or $RER < 1.0$ ?	[ ]	[ ]	[ ]
10. Comments (attach additional sheets if necessary):			
Validated/Reviewed by:			
Signature:	Date:		
Name:			

# LASER INDUCED KINETIC PHOSPHORESCENCE Uranium

Project Name: \_\_\_\_\_

Laboratory: \_\_\_\_\_

Batch Number(s): \_\_\_\_\_

Sample Delivery Group (SDG): \_\_\_\_\_

	<u>Yes</u>	<u>No</u>	<u>NA</u>
1. Holding Time: (a) Were samples preserved?	[ ]	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]	[ ]
2. Initial Calibration: (a) Was a series of 3 standards per range utilized ?	[ ]	[ ]	[ ]
(b) Was $R^2 > 0.990$ ?	[ ]	[ ]	[ ]
(c) Was $150 < \text{Lifetime} < 350$ microseconds (us) ?	[ ]	[ ]	[ ]
(d) Was % Discrepancy $< 10$ ?	[ ]	[ ]	[ ]
3. Continuing Calibration Verification (CCV): (a) Was a CCV conducted every 10 samples ?	[ ]	[ ]	[ ]
(b) Was the CCV within $\pm 10\%$ (90-110%)?	[ ]	[ ]	[ ]
4. Continuing Calibration Blank (CCB): (a) Were analytes in the blank $\leq \text{RL}$ ?	[ ]	[ ]	[ ]
(b) Was a CCB analyzed every 10 samples ?	[ ]	[ ]	[ ]
5. Method of Standard Additions (MSA) (a) Was the MSA performed?	[ ]	[ ]	[ ]
(b) Did results agree within 10%?	[ ]	[ ]	[ ]
6. Calibration Checks (a) Was a calibration check performed and at the required frequency?	[ ]	[ ]	[ ]
(b) Did the results agree within 10%?	[ ]	[ ]	[ ]

	<u>Yes</u>	<u>No</u>	<u>NA</u>
7. Sample Quality Control:			
(a) Blanks: Was a blank conducted one per matrix per batch? - Were target analytes $\leq$ RL?	[ ]	[ ]	[ ]
(b) LCS: Were the percent recoveries for LCS within the limits? (75-125%)	[ ]	[ ]	[ ]
(c) MS: Were the percent recoveries for MS within the QC limits? (75-125%)	[ ]	[ ]	[ ]
(d) Matrix Duplicate: Was the RPD within acceptable criterion? Was the RPD within acceptable criterion? ( $\leq$ 25% for high activity or ND $\leq$ 2.6 for results $<$ 5X the QL) or RER $<$ 1.0	[ ]	[ ]	[ ]
8. QA Sample Results			
(a) Were samples sent to a QA laboratory?	[ ]	[ ]	[ ]
(b) Was the RPD within acceptable criterion? Was the RPD within acceptable criterion? ( $\leq$ 25% for high activity or ND $\leq$ 2.6 for results $<$ 5X the QL) or RER $<$ 1.0	[ ]	[ ]	[ ]
9. Comments (attach additional sheets if necessary):			
Validated/Reviewed by:			
Signature:		Date:	
Name:			

# LUCAS CELL COUNTING

## Radon

Project Name: \_\_\_\_\_  
 Laboratory: \_\_\_\_\_  
 Batch Number(s): \_\_\_\_\_  
 Sample Delivery Group (SDG): \_\_\_\_\_

	<u>Yes</u>	<u>No</u>	<u>NA</u>
1. Holding Time: (a) Were samples preserved?	[ ]	[ ]	[ ]
(b) Were samples analyzed within holding time?	[ ]	[ ]	[ ]
2. Initial Calibration: (a) Was a series of known standards used?	[ ]	[ ]	[ ]
(b) Was the standard deviation < 10% of the average cell constant	[ ]	[ ]	[ ]
3. Daily Background Check (a) Was a daily background check performed?	[ ]	[ ]	[ ]
(b) $\bar{x}$ and $2\sigma$ ? - No action	[ ]	[ ]	[ ]
(c) Between $2\sigma$ and $3\sigma$ ? - Investigate and notate warning	[ ]	[ ]	[ ]
(d) $\geq 3\sigma$ ? Take corrective action – instrument not usable	[ ]	[ ]	[ ]
Or			
$\leq 0.3$ cpm			
4. Instrument Performance Check (a) Was a daily Instrument performance check performed?	[ ]	[ ]	[ ]
(b) $\bar{x}$ and $2\sigma$ ? - No action	[ ]	[ ]	[ ]
(c) Between $2\sigma$ and $3\sigma$ ? - Investigate and notate warning	[ ]	[ ]	[ ]
(d) $\geq 3\sigma$ ? - Take corrective action – instrument not usable	[ ]	[ ]	[ ]



# **Appendix A**

## **Ethics and Data Integrity Agreement**

## ETHICS AND DATA INTEGRITY AGREEMENT

\_\_\_\_\_  
*(Laboratory/Company)*

I. I, \_\_\_\_\_ *(Name)*, state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at \_\_\_\_\_ *(Laboratory/Company)*.

II. I agree that in the performance of my duties at \_\_\_\_\_ *(Laboratory/Company)*:

- a. I shall not intentionally report data values that are not the actual values obtained;
- b. I shall not intentionally report the dates and times of data analysis that are not the actual dates and times of data analyses; and
- b. I shall not intentionally represent another individual's work as my own.

III. I agree to inform \_\_\_\_\_ *(Laboratory/Company)* of any accidental reporting of non-authentic data by myself in a timely manner.

III. I agree to inform \_\_\_\_\_ *(Laboratory/Company)* of any accidental or intentional reporting of non-authentic data by other employees.

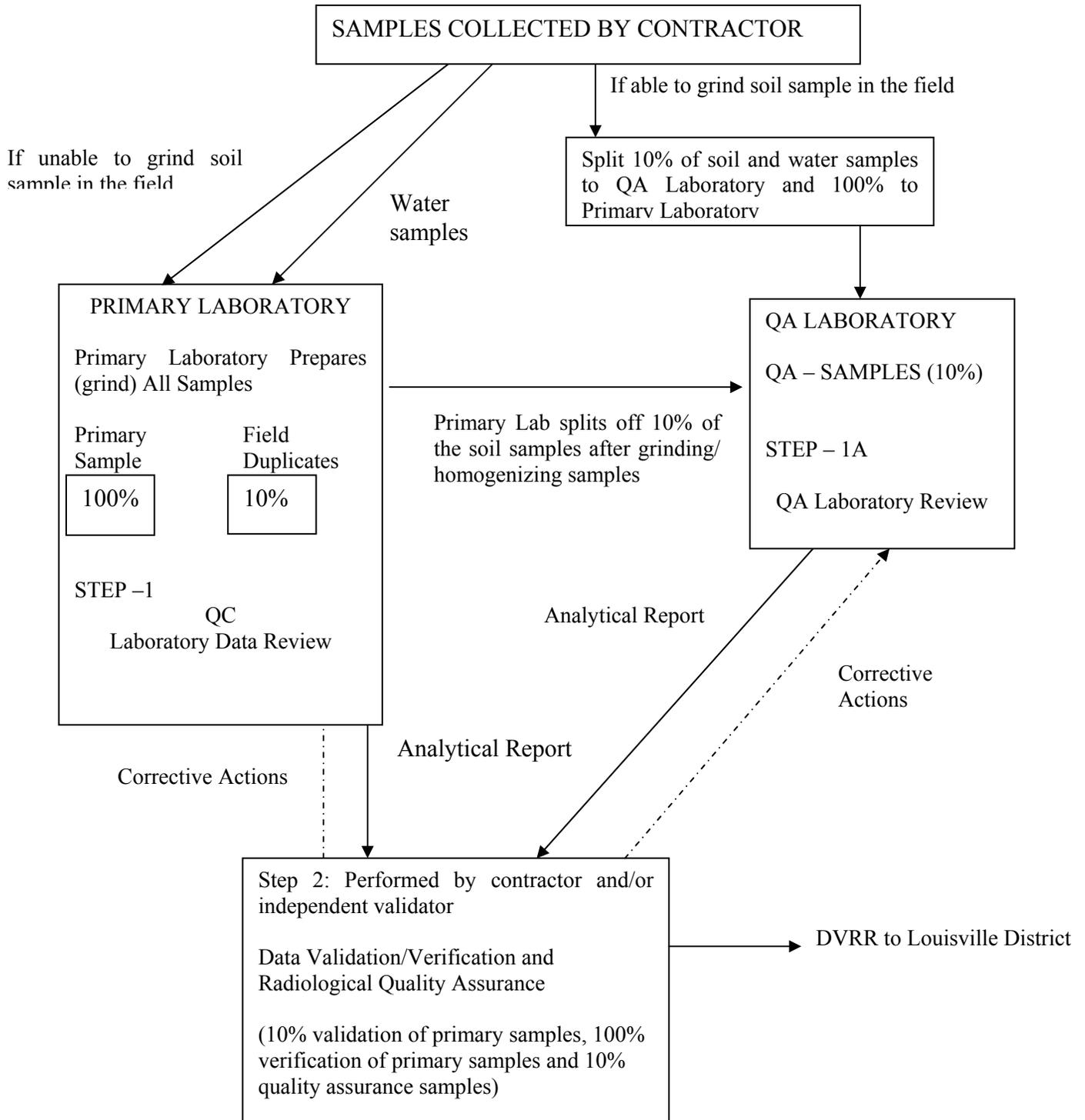
\_\_\_\_\_  
**(Signature)**

\_\_\_\_\_  
**(Date)**

## **Appendix B**

### **Flow Chart – Samples Collected by Contractor**

**FIGURE 1**  
**FLOW CHART**



**Appendix C**

**Independent 3<sup>rd</sup> Party Validation of  
Laboratory Sample Data Results  
Memorandum**



**DEPARTMENT OF THE ARMY**  
U. S. ARMY ENGINEER DISTRICT, LOUISVILLE  
CORPS OF ENGINEERS  
P. O. BOX 59  
LOUISVILLE, KENTUCKY 40201-0059

31 October 2000

MEMORANDUM FOR: CELRL-ED-E-B

MEMORANDUM FROM: CELRL-OC

SUBJECT: Independent 3<sup>rd</sup> Party Validation of Laboratory Sample Data Results

1. The issue that has been presented concerns how to satisfy the requirement for independent third party validation of laboratory sample data results. This issue is independent of the quality control/quality assurance processes that have established criteria in place.
2. The main issue that is involved is conflict of interest either real or perceived. Both should be avoided at all times for several reasons. There are ethical issues and standards of professionalism, there are technical issues in requiring valid information upon which decisions can be based and there are legal issues in fulfilling our responsibilities under CERCLA or other environmental laws. If this process can be successfully challenged at any level it could require the process to be repeated costing time and money and depending on when this occurs in the process the cost could be significant.
3. Therefore, in the procurement process care should be exercised as to the relation between the sampling laboratory and the Data Validator. The party contracting with the Data Validator should have no vested interest in the outcome of the process. Therefore, the sampling laboratory should not subcontract out the data validation. Beyond that, one has to look at the relationships and make some common sense determinations. For example the Corps may have separate actions for sampling laboratory and a Data Validator. Depending on the circumstances, the TERC, as a cost reimbursable contractor with no vested interest in the outcome, may be able hire both the sampling laboratory and the Data Validator. In fixed price contracts one should exercise extreme care. A fixed price AE contractor who subcontracts separately with a sampling laboratory and a Data Validator can be financially impacted by the negative results of the Data Validator.
4. This concept extends not only to the contractual relations but also to the technical relationships. The sampling laboratory should not be communicating directly with the Data Validator concerning the process or results. The determination of the validation process and which sampling results are to be used in the validation process should be independent of the sampling laboratory.

5. Those involved in the process may be aware of additional circumstances where potential areas of conflict of interest arise in this area. The general guidance provided above is just that – general. Individuals need to recognize the potential conflicts and use caution and common sense. This office is available to assist in evaluation and resolution of any concerns in this area.

Kevin M. Finley

Assistant District Counsel

# **Appendix D**

## **The Compton Effect**

The Compton effect is a phenomenon in which higher energy photons may lose only a portion of their energy to the atomic electron, which is again ejected from its atom. This electron goes on to create ionization as before. The remaining energy is taken up by another reduced energy photon, which is then scattered, in a new direction. The new photon will either be absorbed by a photoelectric effect or if the energy is still high, will continue with further Compton scattering. The Compton effect occurs in all materials and mostly in those with photons of medium energy ranging from approximately 0.5 to 3.5 MeV.

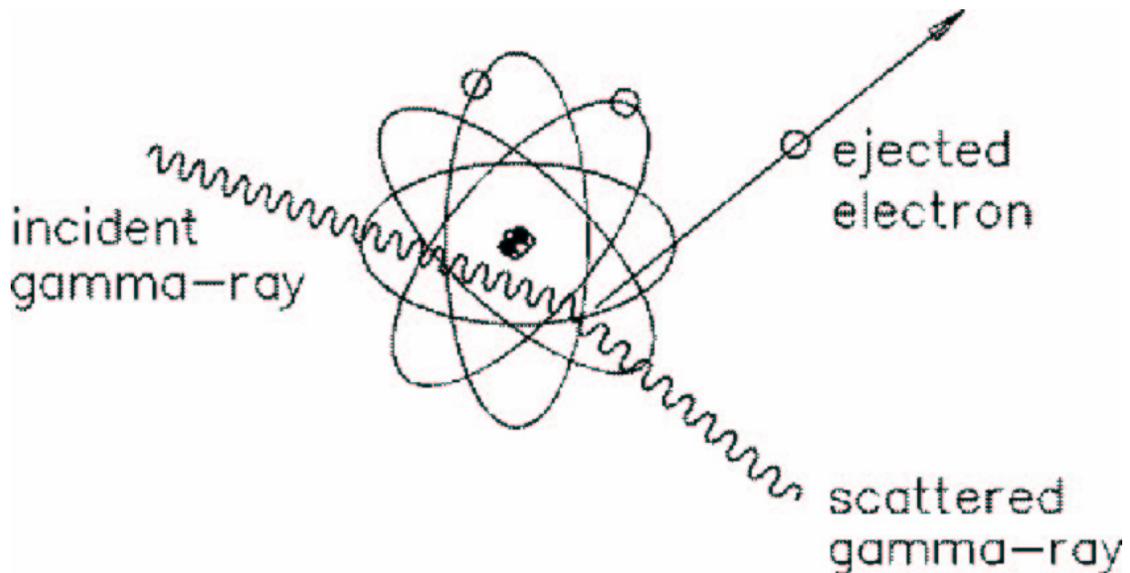


Figure 2: Compton Scattering<sup>1</sup>

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<sup>1</sup> Obtained from [www.truimf.ca/safety/rpt/rpt\\_2/node19.html](http://www.truimf.ca/safety/rpt/rpt_2/node19.html)

# **Appendix E**

## **Sample Container, Preservation, and Holding Times**

## Sample Containers, Preservation, and Hold Times

Parameter	Matrix	Volume/ Container	Preservation	Holding Time
<b>Radiochemistry</b>				
Americium	Soil, vegetation, and air filters	8 ounces / P,G	None	6 months
Americium	Water	1 L/ P,G	HNO <sub>3</sub> to pH<2	6 months
Carbon – 14	Soil	4 ounces / P,G	4°C	6 months
Carbon-14	Water	1 L P,G	4°C	6 months
Curium	Soil, vegetation and air filters	8 ounces /P	None	6 months
Curium	Water	8 ounces /P	HNO <sub>3</sub> to pH<2	6 months
Gamma radionuclides	Soil	8 ounces / P,G	None	6 months
Gamma radionuclides	Water	1 L / P,G	HNO <sub>3</sub> to pH<2	6 months
Gross Alpha	Water	500 mL/P,G	HNO <sub>3</sub> (pH<2)	6 months
Gross Alpha and Beta	Soil	4 ounces / P	None	6 months
Gross Alpha/Beta	Water	200 mL/P,G	HNO <sub>3</sub> (pH<2)	6 months
Iodine – 131	Water	P	None	8 days
Iodine -129	Soil	8 ounces/P	None	6 months
Iodine -129	Water	P	None	6 months
Iron-55	Soil	8 ounces/P	None	6 months
Iron-55	Water	P	HNO <sub>3</sub> to pH<2	6 months
Lead 210	Soil	4 ounces / P,G	None	6 months
Lead 210	Water	1 L / P,G	HNO <sub>3</sub> (pH<2)	6 months
Neptunium-237	Soil, vegetation and air filters	4 ounces / P	None	6 months
Neptunium –237	Water	P, 1L	HNO <sub>3</sub> to pH<2	6 months
Nickel-59	Soil	4 ounces / P	None	6 months
Nickel-59	Water and soil	1L/8 ounces/P	None	6 months
Nickel-63	Soil	4 ounces / P	None	6 months
Nickel-63	Water and soil	1L/8 ounces/P	None	6 months
Phosphorus–32	Soil	4 ounces / P	None	6 months
Phosphorus-32	Water	P, 1L	HNO <sub>3</sub> to pH<2	6 months
Plutonium	Soil, vegetation, and air filters	8 ounces/P ,G	None	6 months
Plutonium	Water	1 L / P,G	HNO <sub>3</sub> to pH<2	6 months
Polonium 210	Soil	4 ounces / P,G	None	6 months
Polonium 210	Water	1 L / P,G	HNO <sub>3</sub> (pH<2)	6 months
Promethium-147	Soil	4 ounces / P	None	6 months
Promethium-147	Water	P, 1L	HNO <sub>3</sub> to pH<2	6 months
Ra 226/228	Soil	8 ounces/P,G	None	6 months
Ra 226/228	Water	1 L / P,G	HNO <sub>3</sub> (pH<2)	6 months
Radium-223	Water	P	None	6 months
Radium-224	Water	P	None	6 months
Radon-222	Soil	8 ounces/P	4°C	6 months
Radon-222	Water	40 ml volatile bottle	4°C, Zero headspace	7 days

Parameter	Matrix	Volume/ Container	Preservation	Holding Time
Sr 89/90	Soil	8 ounces/P,G	None	6 months
Sr 89/90	Water	1 L / P,G	HNO <sub>3</sub> (pH<2)	6 months
Technetium 99	Soil	8 ounces/P,G	None	6 months
Technetium 99	Water	1 L / P,G	HNO <sub>3</sub> (pH<2)	6 months
Thorium	Soil, vegetation, and air filters	8 ounces/P,G	None	6 months
Thorium	Water	1 L / P,G	HNO <sub>3</sub> to pH<2	6 months
Total Alpha Radium	Soil	8 ounces/P	None	6 months
Total Alpha Radium	Water	P	HNO <sub>3</sub> to pH<2	6 months
Total Uranium	Soil	4 ounces/P,G	None	6 months
Total Uranium	Water	5 mL /P,G	HNO <sub>3</sub> (pH<2)	6 months
Tritium	Soil	8 ounces / G	None	6 months
Tritium	Water	120 mL / G	None	6 months
Uranium	Soil, vegetation, and air filters	8 ounces/P,G	None	6 months
Uranium	Water	1 L / P,G	HNO <sub>3</sub> to pH<2	6 months

NOTES

1. P = Polyethylene (preferred when acceptable)
2. G = Borosilicate glass with Teflon lined cap
3. L = Liter
4. g = gram

# **Appendix F**

## **Glossary of Terms**

## GLOSSARY<sup>2</sup>

**Actinide Series:** Series of elements beginning with actinium, element number 89 and continuing through lawrencium, element number 103.

**Alpha Decay:** The spontaneous emission of an alpha particle during radioactive decay of a nucleus. An alpha particle is a strong ionizing particle from the nucleus having a mass and charge equal to that of a positively charged helium ion.

**Analyte:** The particular Radionuclide to be determined in a sample of interest.

**Background:** Ambient signal response recorded by measurement instruments that is independent of radioactivity contributed by the radionuclides being measured in the sample.

**Batch:** A batch of samples is 20 samples or less including a blank, laboratory control sample, a duplicate and a matrix spike as appropriate. A batch of samples is processed throughout the entire analytical process together.

**Beta Decay:** The emission of a beta particle during radioactive decay of a nucleus. A beta particle is a charged particle emitted from the nucleus, having a mass and charge equal in magnitude to that of an electron (negatively charged).

**Bias:** The deviation of a single measured value of a random variable from a corresponding expected value.

**Carriers:** Carriers are typically nonradioactive (ie: natural strontium, barium, yttrium) elements with similar chemical characteristics as the analyte being analyzed. Carriers are added to samples to determine the overall chemical yield for the analytical preparation steps.

**Check Source:** A radioactive source used to confirm the satisfactory operation of the instrument.

**Cocktail:** The solution in which samples are placed for measurement in a liquid scintillation Counting. Solvents and scintillators are major components of scintillation cocktails.

**Counting Efficiency:** The ratio of the net count rate of a radionuclide standard source to its corresponding known activity.

**Counting Efficiency Factor:** The fraction of actual disintegrations in the sample, which are counted by the detector as a function of residue weight.

---

<sup>2</sup> Terms obtained from various sources including laboratory SOPs and written methodologies.

## GLOSSARY<sup>2</sup> CONTINUED

**CPM:** Counts per minute (emission rate measured by the instrument).

**Crosstalk:** the detection of alpha events in the beta channel or the detections of beta events in the alpha channel during simultaneous counting.

**Curies:** The traditional unit of measure used to express the amount of radioactivity in material. The SI unit for activity is the bequerel.

1 curie (Ci) =  $2.22 \times 10^{12}$  disintegrations/minute

1 millicurie(mCi) =  $2.22 \times 10^9$  disintegrations/minute

1 microcurie (uCi) =  $2.22 \times 10^6$  disintegrations/minute

1 picocurie (pCi) = 2.22 disintegrations/minute

1 becquerel (Bq) = 1 disintegration/second

**Daughter:** A nuclide formed by radioactive decay of a parent radionuclide.

**Dead Time:** the time while the analog to digital converter is processing a pulse and is unable to process another pulse.

**DPM:** Disintegrations per minute (true emission rate of a source).

**Duplicate Sample:** A second aliquot of a sample that serves as a Batch QC sample, demonstrating analytical method precision and sample homogeneity.

**Efficiency:** A measure of the fraction of actual disintegrations in the sample, which are counted by a detector.

**Energy Calibration:** The correlation of the multichannel analyzer (MCA) channel number to decay energy, obtained from the location of peaks from known radioactive standards.

**Field Blank:** A sample prepared in the field by transferring ASTM Type II Water to a clean sample container. The field blank is used to indicate the presence of contamination due to sample collection and handling.

**Full Width Half Maximum (FWHM):** the full width of a gamma-ray peak distribution measured at half the maximum peak height, measure above the continuum (background).

**Gamma Radiation:** Electromagnetic radiation of nuclear origin usually accompanying another form of radioactive decay.

---

<sup>2</sup> Terms obtained from various sources including laboratory SOPs and written methodologies.

## GLOSSARY<sup>2</sup> CONTINUED

**Geometry:** a standard sample or source counting configuration (ie: 20 ml vial, marinelli beaker, jar and others) and its relationship to the detector.

**H Number/Quench Indicator:** A parameter which indicates the level of quench in a sample using the energy distribution pattern of Compton electrons induced in the sample by an external Cs source.

**HALF-LIFE ( $T_{1/2}$ ):** The time required for 50 percent of a radioactive isotope to decay.

**Ionizing Radiation:** Any electromagnetic or particulate radiation capable of producing ions directly or indirectly in its passage through matter.

**Key Peak:** a spectral peak used for identification or quantification of an isotope.

**Laboratory Control Sample (LCS):** The LCS is a laboratory generated sample prepared by adding known quantities of analyte(s) to an appropriate matrix which contains no analyte activity and is carried throughout the entire analysis procedure with the samples. The laboratory control sample is a quality indicator and provides information about the relative bias of the analysis. It is used to assess the overall process for any inherent biases or trends.

**Lifetime:** the measurement of the length of phosphorescence in a sample or standard

**Linear Regression Coefficient or  $R^2$ :** the measurement of the linearity of decay. A low  $R^2$  indicates poor phosphorescence or quench.

**Matrix Duplicate:** Duplicate aliquot of a sample processed and analyzed independently, however under the same laboratory conditions.

**Matrix Spike:** A matrix spike is an aliquot of a sample to which known quantities of analyte(s) have been added. It is carried through the entire analytical procedure with the sample in order to evaluate the appropriateness of the method for the matrix by measuring recovery of the added analyte(s).

**Minimum Detectable Activity (MDA):** The minimum detectable activity is the smallest amount (activity or mass) of an analyte in a sample that will be detected.

---

<sup>2</sup> Terms obtained from various sources including laboratory SOPs and written methodologies

## GLOSSARY<sup>2</sup> CONTINUED

$$\text{Normalized Difference (ND)} = \text{ND} = \frac{|X_1 - X_2|}{\text{TPU}}$$

Where:

$X_1$  – Sample result

$X_2$  – Sample result

TPU – sample uncertainty @ 1 sigma (68.3%) level

**Nuclide:** An atomic species characterized by the constitution of its nucleus, specifically by the number of protons and neutrons.

**Percent Usability:** the percent of data that is not rejected due to QA/QC issues

$$\% \text{ usability} = \frac{(\text{unqualified sample data} + \text{estimated sample data})}{\text{total data points measured}}$$

**Proportional Counter:** a gas filled radiation counter tube operated in the range of high voltage in which the total charge collected for each ionizing event is proportional to the number of ion pairs formed in the tube by the initial event.

**Quality Assurance (QA):** an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

**Quality Control (QC):** the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

**Quench:** Something that interferes with either the production or the detection of a light pulse in or from a scintillation sample.

**Radioactive Decay:** The process by which a spontaneous change in nuclear state takes place. This process is accompanied by the emission of energy and subatomic particles.

**Radiation Yield:** The amount of radiation of the type being measured that is produced per each disintegration, which occurs.

**Region of Interest (ROI):** In radiochemical analysis, the Multichannel Analyzer region defining the isotope of interest displayed in terms of energy or channels.

**Relative Bias:** The quotient of the bias divided by the expected value.

---

<sup>2</sup> Terms obtained from various sources including laboratory SOPs and written methodologies

## GLOSSARY<sup>2</sup> CONTINUED

**Relative Error Ratio:** The ration between the difference in measured activity to the summation of potential errors.

**Scintillator:** A transparent substance that emits visible or near-ultraviolet light when traversed by an ionizing particle. This substance absorbs decay energy transferred from the solvent and emits light energy (photons) approximately proportional in intensity to the decay energy.

**Self-Absorption:** absorption of radioactive emissions by the solids contained on the counting planchet, thereby preventing the emission from reaching the detector.

**Simultaneous Counting:** the measurement of both gross alpha and gross beta activity at the same time.

**Spike:** In radiochemical analysis, an accurately measure amount of tracer quantitatively introduced or transferred into a sample aliquot.

**Total Propagated Uncertainty (TPU):** The TPU is an estimated number that can be calculated by taking into account the effect of random and systematic uncertainties

**Tracer:** A radionuclide that chemically mimics and does not interfere with the target radioanalyte through the chemical preparation and instrument analysis.

**Tracer Chemical Recovery:** The percent yield of the recovered tracer radioisotope after the sample/tracer aliquot has undergone preparation and instrument analysis.

---

<sup>2</sup> Terms obtained from various sources including laboratory SOPs and written methodologies

# **Appendix G**

## **CALCULATIONS**

**Calculations may vary from laboratory to laboratory, but the same principles apply. These are example calculations.**

**Efficiency:**

$$Eff = \frac{CPM_o}{DPM_c}$$

Where:

Eff – Efficiency

CPMo – observed counts per minute of the standard

DPMc – decay corrected disintegration rate of the certified calibration  
Standard

**Minimum Detectable Amount (MDA) Calculation:**

$$MDA = \left( \frac{X + C\sqrt{B * T_C}}{2.22 * V * Eff * A * T_C * D} \right)$$

Where: X – Statistical Factor (95% Confidence Level) = 2.71  
C – Confidence statistical factor (95% Confidence Level) = 4.65  
B – Total background counts for sample count time  
2.22– Dpm to Pico Curies conversion factor  
V - Sample volume or weight (typically in liters or grams)  
Eff – Counting Efficiency  
A – Abundance  
T<sub>s</sub> – Sample Count Time  
D – Decay or ingrowth correction as necessary

**Activity:**

$$\text{Activity (pCi/unit)} = \left( \frac{Cn}{2.22 * V * Eff * A * D} \right) * \exp$$

$$\text{Exp} = - \left( - \frac{\ln 2}{t_{1/2}} * T \right)$$

Where: Cn – Sample Count rate in cpm (NET) = CPM – BKG CPM  
2.22– Dpm to Pico Curies conversion factor  
V - Sample volume or weight  
Eff – Counting Efficiency  
A – Abundance  
D – Decay or ingrowth correction as necessary  
T – Sample Decay time (Difference between sample date/time and count date/time)  
t<sub>1/2</sub> – Radioactive half-life

**Normalized Difference for Replicate Analyses:**

$$\text{ND} = \frac{|X_1 - X_2|}{\text{TPU}}$$

Where:  
X<sub>1</sub> – Sample result  
X<sub>2</sub> – Sample result  
TPU – sample uncertainty @ 1 sigma (68.3%) level

**Linear Regression Equation for R<sup>2</sup>:**

$$y = b_0 + b_1x$$

**Percent Recovery (%R):**

$$\%R = \frac{\text{spikedresult} - \text{sampleresult}}{\text{spikeadded}} * 100$$

**Standard Deviation (sigma):**

$$\text{Standard Deviation (s)} = \sqrt{\frac{N}{t}}$$

Where:

N - Sample count rate (CPM)

T - Sample count time (minutes)

**Relative Error Ratio (RER):**

$$\text{RER} = \frac{|C_1 - C_2|}{\sum \text{counting uncertainties}} = Z_{\text{REP}} = \frac{|X_1 - X_2|}{\sqrt{u_c^2(x_1) + u_c^2(x_2)}}$$

Where for RER:

For duplicate analysis:

C<sub>1</sub> = Measured total activity off the first detection or the first sample aliquot

C<sub>2</sub> = Measured total concentration off the second detection or the duplicate sample aliquot

For MS/MSD analyses:

C<sub>1</sub> = Spike sample result minus sample result

C<sub>2</sub> = Spike sample result minus sample result

For Z<sub>REP</sub>:

X<sub>1</sub> and X<sub>2</sub> denotes two measure activity concentrations

u<sub>c</sub>(X<sub>1</sub>) and u<sub>c</sub>(X<sub>2</sub>) denotes the respective measure activity concentrations uncertainty

Total Propagated Uncertainty (TPU). The TPU is an estimated number that can be calculated by taking into account the effect of random and systematic uncertainties. An example of this equation is given below:

$$TPU_S = \sqrt{(U_E)^2 + (U_{Ab})^2 + (U_{t_{1/2}})^2 + (U_Y)^2 + (U_V)^2 + (U_{prep})^2}$$

where  $\sqrt{U_y^2 + U_v^2 + U_{PREP}^2} = 0.05$

Where:  $TPU_S$  = the uncertainty of the activity of the sample

$ACT_s$  = the activity in pCi/(units of volume)

$(U_E)^2$  = Uncertainty in efficiency = 0

$(U_{Ab})^2$  = Uncertainty in abundance = 0

$(U_{t_{1/2}})^2$  = Uncertainty in half-life

$(U_y)^2$  = Uncertainty in yield

$(U_v)^2$  = Uncertainty in volume

$(U_{prep})^2$  = Uncertainty in preparation

**EXAMPLE CALCULATIONS FROM MARLAP CHAPTER 18:**

**The following equations are measurement indicators.**

**Laboratory Replicates:**

$$Z_{\text{REP}} = \frac{|X_1 - X_2|}{\sqrt{u_C^2(x_1) + u_C^2(x_2)}}$$

Where:

$X_1$  and  $X_2$  denotes two measure activity concentrations

$u_c(X_1)$  and  $u_c(X_2)$  denotes the respective measure activity concentrations uncertainty

**Laboratory Control Sample:**

$$Z_{\text{LCS}} = \frac{x - d}{\sqrt{u_C^2(x) + u_C^2(d)}}$$

Where:

$x$  = measured value of the spiked sample

$d$  = spike concentration added

$u_c(x)$  and  $u_c(d)$  denotes the respective measure activity concentrations uncertainty

**Matrix Spike/Matrix Spike Duplicate**

$$Z_{\text{MS/MSD}} = \frac{x - x_0 - d}{\sqrt{u_C^2(x) + u_C^2(d)}}$$

Where:

$x$  = measured value of the spiked sample

$x_0$  = measured concentration of the unspiked sample

$d$  = spike concentration added

$u_c(x)$  and  $u_c(d)$  denotes the respective measure activity concentrations uncertainty