VOLUME 1, MODULE 6 Quality Systems for Radiochemical Testing

1.0 RADIOCHEMICAL TESTING

1.1 Introduction

1.2 Scope DoD/DOE (Clarification)

This DoD/DOE module references the radiochemical terms, definitions, and requirements contained in the 2009 TNI Standard *Module 6 Quality Systems for Radiochemical Testing*. However, it does not preclude the use of other terms, definitions, and requirements from the consensus document *Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual*, July 2004.

1.3 Terms and Definitions

1.4 Method Selection

1.5 Method Validation

1.5.2.1 DoD/DOE (Requirement)

The following shall be implemented in addition to 1.5.2.1.a) through d):

e) SOPs that incorporate equations to calculate the decision level and the minimum detectable concentration (or activity) must be documented and consistent with the mandated method or regulation.

1.5.2.1.1 DoD/DOE (Requirement)

a) The Minimal Detectable Activity (MDA) is the smallest amount of an analyte in a sample that will be detected with a probability β of non-detection (Type II error), while accepting a probability α of erroneously deciding that a positive (non-zero) quantity of analyte is present in an appropriate blank sample (Type I error). Confidence levels may be dictated by the project. For the purposes of this module and the equations below, the α and β probabilities are assumed to be 0.05. MARLAP utilizes the Minimum Detectable Concentration (MDC) term instead of MDA.

b) MDA Factors and Conditions - MDAs are determined based on factors and conditions such as instrument settings and matrix type, which influence the measurement. The MDA is used to evaluate the capability of a method relative to the required detection reporting limit (RL). Sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency shall be optimized to result in sample MDAs less than or equal to the RLs. If RLs are not achieved, then the cause shall be addressed comprehensively in the Case Narrative.

c) MDA Calculation - The basic MDA calculation shall be based on the concepts developed by L. A. Currie from his paper "Limits for Qualitative Detection and Quantitative Determination, *Analytical Chemistry*, March, 1968, Vol. 40, or from the MARLAP Manual Chapter 20. The following general equation derived from the work of L. A. Curie can be used to calculate the MDA.

i) With a Blank Population:

$$MDA = \frac{3.29 * s_b}{KT_S} + \frac{3}{KT_S}$$

K = efficiency * $e^{-\lambda t}$ * aliquot fraction * tracer recovery*
T_S = count time of the sample in minutes
s_b = standard deviation of the blank population where the blank population is in net blank counts in count time T_S

Use of blank populations for calculation of MDAs requires the selection of an implementation method, which includes but is not limited to:

Identification of blanks to be used in the population:

1. The number of blanks to use in the population;
2. How the blank population changes; and
3. Limitations on the deletion of blanks.

The method of implementation shall not introduce any statistical bias.

The appropriate blank subtraction shall be the mean blank value of the blank population.

The implementation of blank populations for calculation of MDAs shall be described in detail in a Standard Operating Procedure (SOP).

In the original Currie derivation, a constant factor of 2.71 was used. Since that time it has been shown and generally accepted that a constant factor of 3 is more appropriate. (*Multi Agency Radiation Survey & Site Investigation Manual*, Aug. 2000)

ii) General:

The above equation for MDA has the units of dpm/sample. Any other units will require appropriate conversion.

Site specific requirements may be provided for other MDA formulations.

MDAs for samples without a blank population can be determined if based on appropriate L. A. Currie or MARLAP calculations.

d) MDA Optimization: The laboratory shall optimize analysis parameters in order to achieve analyte MDAs less than or equal to the required detection threshold. Samples with elevated activities shall be handled according to the following requirements:

i) The appropriate aliquant size shall be determined based on the activity level in the sample. The aliquot shall be large enough to generate data, which meet the following criteria:

ii) The measurement uncertainty shall not be greater than 10% (1 sigma) of the sample activity.

iii) The MDA for the analysis shall be a maximum of 10% of the sample activity.

iv) If sample-specific MDAs are calculated and reported, that shall be clearly stated in the data package.

v) The definition of the MDA presupposes that an appropriate detection threshold (i.e., the decision level) has already been defined. The α probabilities assumed for the decision level shall also be used for the calculation of the MDA.

1.5.2.1.2 DoD/DOE (Requirement)

a) Decision Level (DL): In the context of analyte detection, the minimum measured value (e.g., of the instrument signal or the analyte concentration) required to give confidence that a positive (nonzero) amount of analyte is present in the material analyzed. The DL is sometimes called the critical level (Lc) or critical value (MARLAP). It is the quantity of analyte at or above which an *a posteriori* decision is made that a positive quantity of the analyte is present. Confidence levels may be dictated by the project. For this document, the probability of a Type I error (probability of erroneously reporting a detectable nuclide in an appropriate blank or sample) is assumed to be set at 0.05.

b) DL Factors and Conditions: DLs are determined *a posteriori* based on sample-specific sample size, count duration, tracer chemical recovery, detector background, blank standard deviation, and detector efficiency.

c) DL Calculation: The basic DL calculation shall be based on concepts developed by L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination, *Analytical Chemistry*, March, 1968, Vol. 40, or MARLAP Chapter 20. The following general equations can be used to calculate the decision level.

d) The DL can either be based on the Combined Standard Uncertainty (CSU) of the blank, or the standard deviation determined from a set of appropriate blanks.

i) With Blank Population:

When determined from the standard deviation of a set of appropriate blanks, the DL evaluates the level where the blank results will not exceed more than 5% of the time (or other specified level of confidence) and may be estimated by the following equation:

$$DL = \frac{(t \times S_B) + \overline{R_B}}{E \times R \times IDF \times W}$$

Where:

DL = the decision level in disintegrations per minute per unit volume or weight (dpm/unit);
S_B = the standard deviation of a set of appropriate blank net count rate after background subtraction for blanks counted for the same length of time as the sample;
R_B = the average blank count rate in counts per minute (cpm);
t = the student t factor for appropriate degrees of freedom and confidence level;
E = the fractional detector efficiency (c/d) for the sample;
R = the fractional chemical yield for the sample;
IDF = the ingrowth or decay factor for the sample; and
W = the weight or volume of the sample.

DLs are used as the default detection threshold. Alternatively, the client may use/specify detection thresholds that meet project/site-specific requirements.

DLs for samples without a blank population can be determined if based on appropriate L. A. Currie or MARLAP calculations using a CSU.

1.5.4 DoD/DOE (Requirement)

Measurement Uncertainties: Each result shall be reported with the associated measurement uncertainty as a combined standard uncertainty. The SOP for determining the measurement uncertainty must be consistent with mandated method and regulation.

Combined Standard Uncertainty: All measurement uncertainties shall be propagated and reported with each result. The formula for calculating the Combined Standard Uncertainty (CSU) of a result shall be documented in the appropriate SOP. The CSU shall include both systematic and random error. CSU is always 1 sigma. Results should be reported at the 95% confidence level, which is 2 sigma.

The uncertainty of a count may be estimated as the square root of counts except when there are zero (0) counts. In the case of zero (0) counts, the uncertainty of the count is assumed to be the square root of one count.

Systematic Error shall include, but is not necessarily limited to:

a) The errors from all measurement devices, such as, but not limited to pipettes and balances.

b) The uncertainty of known values of tracer solutions, calibration uncertainties, etc.

Random Error shall include, but is not necessarily limited to, the total random counting error associated with each sample and appropriately propagated when more than one variable is used to determine the result.

1.7.1 Instrument Calibration

a) Initial Calibration:

viii) Detection efficiency shall be determined with sources that are NIST- traceable or with sources prepared from NIST-traceable standards. When sources used for determinations for detection efficiency are prepared from NIST-traceable standards, they shall be "working reference materials" as defined in STD.ASTM C1128 (current version).

ix) For alpha spectrometry, a material balance check shall be done on each source to clearly demonstrate accountability of all activity by mass balance. The material balance check shall be done on the fraction remaining from the neodymium fluoride precipitation, or the electro-deposition plus all rinses from an adequate cleaning of any vessel used in the process. The estimated error in preparing the source shall be propagated into the error of the efficiency determination.

x) Check sources shall be used only to verify that efficiencies have not changed. They shall not be used to determine efficiencies.

b) Instrument Calibration Verification (Performance Checks)

i) For systems using sample changers and/or long count times that run more than a day, the energy calibration shall be checked before each analytical batch.

The Full-Width-Half-Maximum (FWHM) resolution of the alpha or gamma detector shall be evaluated prior to instrument use and following repair or loss of control (MARLAP 18.5.6.2). The measured FWHM resolution shall be trended.

Detector response (counting efficiency) determinations shall be performed when the check source count is outside the acceptable limits of the control chart (reference ANSI N42.23, Annex A5).

It is important to use calibration or QC sources that will not cause detector contamination from recoil atoms from the source.

For radon scintillation detectors, efficiency shall be verified at least monthly, when the system is in use.

c) Background Measurement

Background check measurements shall be conducted after calibration and monthly thereafter, and monitored for trends to ensure that a

Table – 16. Alpha Spectrometry					
QC Check	Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) (Energy, efficiency and FWHM peak resolution)	Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings (MARLAP 18.5.6.3).	Verify manufacturer's specifications for point source efficiency.(MARLAP) or Two calibration peaks that are: 1) ≥1000 keV apart; or 2) that bracket all peaks to be determined. Energy vs. channel slope equation <15 keV per channel. Full Width –Half Maximum (FWHM) ≤100 keV for each peak used for calibration. Minimum of 3,000 net counts in each peak.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Use traceable calibration source (CS) that matches sample test source (STS) configuration (type, size and position relative to the detector). May use same count for initial efficiency calibration. No samples may be run until energy and FWHM calibration criteria are met.

Table – 16. Alpha Spectrometry					
QC Check	Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	After initial calibration.	Determine peak location, resolution, and ROI/alpha peak efficiency (where counting efficiency is an analytical requirement) using at least two alpha peaks (MARLAP 18.5.6.3). or Observed peak centroid falls ≤20 keV from reference energy for each peak used in the initial energy calibration. FWHM ≤100 keV and within 30 keV of corresponding calibration peaks in initial energy calibration.	Repeat ICV to check for error. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate.	Flagging criteria are not appropriate.	Use a second-source standard that matches STS configuration (type, size and position relative to the detector) or pulsar for energy check only. Bracketing peaks may also be used that are >1000 keV apart. No samples may be run until calibration has been verified with a second source.
Continuing Calibration Verification (CCV) (Pulsar check)	Pulsar energy verification daily, prior to analysis of samples.	Energy response check shall have a tolerance limit set at ± 3% or control chart set at ± 3σ (MARLAP 18.5.6.3). or Observed peak centroid falls ≤20 keV from reference energy.	Recount and check control chart for trends. Determine cause, correct problem, and repeat CCV and all associated samples since last successful CCV.	Flagging criteria are not appropriate.	Pulsar check can be used to verify energy calibration when using radiotracers during analysis. No samples may be run until calibration has been verified.

Table – 16. Alpha Spectrometry					
QC Check	Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV) (Check source)	Daily source check verification prior to analysis of samples.	Response checks shall have a tolerance limit or control chart set at ± 3% or 3σ. (MARLAP 18.5.6.3) or Observed peak centroid falls ≤20 keV from reference energy for each peak used in the initial energy calibration. FWHM ≤100 keV and within 30 keV of corresponding calibration peaks in initial energy calibration.	Recount and check control chart for trends. Determine cause, correct problem, and repeat CCV and all associated samples since last successful CCV.	Flagging criteria are not appropriate.	Source check can be used to verify energy, FWHM and efficiency. No samples may be run until calibration has been verified.
Background Subtraction Count (BSC) Measurement	Prior to initial use or after initial calibration and monthly. (MARLAP 18.5.6.3)	Within ±3σ of mean activity of recent BSCs for each ROI to be determined (minimum of 3 BSC values).	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC and reanalyze all impacted samples since last acceptable BSC.	If reanalysis cannot be performed, apply B-flag where count rate <10 times that in the affected ROI(s) in the BSC.	BSC test source matches STS configuration (type, size and position relative to the detector). Activity must meet project objectives.
Instrument Contamination Check (ICC)	Performed weekly, at minimum, and after counting high activity samples. Count duration ≥ longest STS count.	Z_{Blank} ≤3 for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1)	Check control chart for trends and recount. Determine cause and correct problem. If background activity has changed, re-establish BSC and reanalyze all infected samples.	If reanalysis cannot be performed, apply Q-flag to all affected result since last acceptable ICC where the STS count rate in the impacted ROI is ≤5 times that of STS. Explain in the case narrative.	

Table – 16. Alpha Spectrometry					
QC Check	Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Performance Check (IPC)	Weekly with same check source used to establish control charts.	Minimum Peak Centroid: within 20 keV of mean of initial QC check data. Minimum Peak Resolution (FWHM): ≤100 keV and within 30 keV of mean energy of checks gathered at or prior to the initial energy calibration. Value of efficiency check within ±10% of mean efficiency in checks gathered at or prior to the initial calibration.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	Check source needed for energy only. QC check data must be established prior to or concurrent with initial energy calibrations. Minimum of 2,000 net counts in at least 2 peaks. Bracketing counts may be used for longer counts but samples may not be reported until bracketing counts meet criteria. Results may not be reported without a valid IPC. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table – 16. Alpha Spectrometry					
QC Check	Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch. (MARLAP 18.4.1)	$\lvert Z_{Blank} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Blank} \rvert \geq 2$. (MARLAP 18.4.1) or In-house control limits of ±3 σ of the mean.	Recount the blank to confirm results. Inspect MB control chart for indication of significant bias If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Blank matrices must be the same as the associated samples (i.e. radon free distilled or deionized water, representative solid material, physically and chemically identical filter media. With project approval and appropriate qualification and narration, report results with a count rate >5 times that of the affected ROI in the MB.
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$. (MARLAP 18.4.3) or Use in-house control limits of LCS ± 3 σ of the mean. In-house control limits may not fall more than 25% from the known LCS value.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Reprep and reanalyze the LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Qualification is only appropriate in cases where the samples cannot be reanalyzed. LCS matrices must be the same as the associated samples. LCS must be counted for a sufficient time to meet the required project minimum activity. Acceptance criteria for LCS recovery may be specified by the project.

Table – 16. Alpha Spectrometry					
QC Check	Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch. *(MS not required when chemical yield tracers or carriers are employed).*	If activity of the MS > 5 times the unspiked sample, $\lvert Z_{MS} \rvert \leq 3$. (MARLAP 18.4.3) or Within 60-140% recovery.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$. (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is <3; or the relative percent difference (RPD) is <25%.	Check for lab error. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Tracers (if used)	Added to each sample as isotopic yield monitor.	Isotopic yield within 30-110%. FWHM <100 keV and peak energy within ±40 keV of known peak energy.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Carriers (if used)	Added to each sample as chemical yield monitor.	Chemical yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-17. Gamma Spectrometry					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) (Energy, efficiency and FWHM peak resolution)	Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.2)	Verify manufacturer's specifications for gamma peak resolution. (MARLAP 18.5.6.2) Efficiency vs. energy for each geometry/matrix. 95% confidence limit of the fitted function: ≤8% over energy range. (MARLAP 18.5.6.2) or Peak energy difference is within 0.1 keV of reference energy for all points. Peak Full Width at Half Maximum (FWHM) < 2.5 keV at 1332 keV. Energy vs. channel slope equation shall be linear and accurate to 0.5 keV.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Traceable calibration source (CS) matches sample test source (STS) configuration (type, size, geometry and position relative to the detector). Minimum of 10,000 net counts in each peak in at least six calibration peaks that bracket the range of use. No samples may be run until all calibration criteria are met.
Initial Calibration Verification (ICV)	After ICAL for energy/efficiency and prior to analysis of samples	Observed peaks of second source standard fall within ± 10% of initial calibration value relative to energy, FWHM, and efficiency.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV as appropriate.	Flagging criteria are not appropriate.	Traceable second-source standard matches STS configuration (type, size, geometry and position relative to the detector). Minimum of 10,000 net counts in each peak in at least six calibration verification peaks that bracket the range of use. No samples may be run until calibration has been verified.

Table F-17. Gamma Spectrometry					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV) (Daily Check)	Daily or prior to use. When working with long count times or batch sequences that run more than a day, CCV is performed at the beginning and end of each analytical batch as long as it not longer than a week.	Verify peak shift within tolerance limit; verify efficiency within control parameters; verify resolution in tolerance limit. Response checks shall have a tolerance limit or control chart set at ± 3% or 3σ of the mean. (MARLAP 18.5.6.2) or Peak Energy/Efficiency: low, mid, and high energies within 10% of the initial calibration value; FWHM: low, mid, and high energies within 10% of initial FWHM value.	Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Background Subtraction Count Measurement (BSC) (Long count for subtracting background from blanks or test sources)	Immediately after ICAL and then performed on at least a monthly basis. (MARLAP 18.5.6.2)	Statistical test of successive counts and count rates for identified background peaks show no significant difference. (MARLAP 18.5.6.2)	Recount and check control chart for trends. Determine cause, correct problem, re-establish BSC. If background activity has changed, re-establish BSC and reanalyze or qualify all impacted samples since last acceptable BSC.	Apply B-flag to all results for specific nuclide(s) in all samples associated with the blank.	A detector's background should be determined immediately after calibration, with or without a counting container, depending on the inherent radionuclide activity levels in the counting container. The counting interval for the long count shall be between one and four times the nominal counting interval of the test sources. (MARLAP 18.5.6.2)

Table F-17. Gamma Spectrometry					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Contamination Check (ICC) (Short count for controlling gross contamination)	Daily or when working with long count times before and after each analytical batch. Check after counting high activity samples	No extraneous peaks identified (i.e., no new peaks in the short background spectrum compared to previous spectra); The tolerance limit or control chart: ± 3% or 3σ of the mean activity. (MARLAP 18.5.6.2)	Recount the background. If still out of control, locate and correct problem; reanalyze or qualify all impacted samples since last acceptable ICC. If background activity has changed, re-establish BSC and reanalyze samples.	If corrective action fails, apply Q-flag to all results for specific nuclide(s) in all samples associated with the BSC.	Integrate spectrum from ~50 - 2,000 keV to check for gross contamination. (MARLAP 18.5.6.2)
Method Blank (MB)	One per preparatory batch.	$\lvert Z_{Blank} \rvert \leq 3$ for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1) or No analytes detected > 2 times the blank Combined Standard Uncertainty (CSU). Blank result must not otherwise affect sample results.	Recount the blank to confirm results, unless all sample results are >5 times the blank activity. Inspect MB control chart for indication of significant bias. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	The results of method blanks typically are not used to correct sample activities, but only to monitor for contamination. (MARLAP 18.4.1) Blank matrices must be the same as the associated samples (i.e., radon free distilled or deionized water, representative solid material, physically and chemically identical filter media.) Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-17. Gamma Spectrometry					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$. (MARLAP 18.4.3) or Use in-house control chart limits of ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. If required, reprep and reanalyze the LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples and shall contain nuclides within the energy ranges of all those nuclides to be reported. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Qualification is only appropriate in cases where the samples cannot be reanalyzed.
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$. (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is <3; or the relative percent difference (RPD) is <25%.	Check for lab error. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table F-18 Gas Flow Proportional Counting					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration - Voltage Plateau (ICALV) (separate plateaus determined for alpha and beta activity)	Prior to initial use and after loss of control. (MARLAP 18.5.6.1)	Verify manufacturer's specifications. (MARLAP 18.5.6.1) Plot voltage vs. count rate to determine proper operating voltages. (MARLAP 18.5.6.1) or Slope of the plateau less than 5% over a range of 100V.	Correct problem, then repeat ICALV.	Flagging criteria are not appropriate.	Series of 1-minute counts in <50V steps from ~300V to ~1500V. No samples may be run until plateau calibration criteria are met.
Initial Calibration - Efficiency (ICALE)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)	Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using electroplated sources. (MARLAP 18.5.6.1) A 1σ counting uncertainty of ≤1% shall be achieved for all detector efficiency determinations. (MARLAP 18.5.6.1)	Correct problem, then repeat ICALE.	Flagging criteria are not appropriate.	Detector's counting efficiency, using traceable calibration sources, shall be determined for each radionuclide used to analyze test sources. No samples may be run until efficiency calibration criteria are met.
Initial Calibration – Cross-talk Factors (ICALCT)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)	Verify manufacturer's specifications for cross talk in alpha and beta channels. (MARLAP 18.5.6.1)	Correct problem, then repeat ICALCT.	Flagging criteria are not appropriate.	Determine crosstalk factors for each nuclide, matrix and method. For mass loaded test sources, determine crosstalk factors for the nuclide as a function of test source mass. No samples may be run until cross talk calibration criteria are met.

Table F-18 Gas Flow Proportional Counting					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration – Self-Absorption Curve (ICALSA)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.1)	For each radionuclide of interest, establish mathematical function (curve) of detector efficiency vs. source mass loading. 95% confidence limit of the fitted function (curve) over the calibration range to ≤10% and ≤5% uncertainty for alpha and beta, respectively. (MARLAP 18.5.6.1) or Best fit of data with correlation coefficient closest to 1.00 and the smallest standard error.	Correct problem, then repeat ICALSA.	Flagging criteria are not appropriate.	Minimum of seven mass attenuated standards. No samples may be run until mass attenuation calibration criteria are met.
Efficiency Calibration Verification (IECV)	After ICALE for alpha and beta and prior to analysis of samples.	A tolerance limit or control chart shall be established immediately after the initial counting efficiency calibration, and after instrument loss of control. A tolerance limit or control chart shall be set at ± 3% or 3σ of the mean. (MARLAP 18.5.6.1) or Value of second source calibration for each isotope within ±10% of initial calibration value.	Correct problem and verify second source standard. Rerun IECV. If that fails, correct problem and repeat ICALE.	Flagging criteria are not appropriate.	Use traceable second source standard that matches sample test source configuration (type, size, and position relative to the detector). No samples may be run until calibration has been verified.

Table F-18 Gas Flow Proportional Counting					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing Calibration Verification (CCV)	After a counting gas change and daily for short test-source counting intervals. For longer test-source counting times, a detector response check for a multi-sample shelf unit shall be conducted prior to test source counting, while a detector response check for a sequential sample counter shall be performed before and after the sample batch. (MARLAP 18.5.6.1)	Within tolerance or control chart limits ± 3% or 3σ of the mean.	Correct problem, rerun calibration verification. If that fails, then repeat ICALE. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	Minimum of 2,000 net counts for each energy level. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method Blank (MB)	One per preparatory batch.	$\lvert Z_{Blank} \rvert \leq 3$ for blank subtracted (net) activity in all ROIs. (MARLAP 18.4.1) or No analytes detected > 2 times the blank Combined Standard Uncertainty (CSU). Blank result must not otherwise affect sample results.	Recount the blank to confirm results, unless all sample results are >5 times the blank activity. Inspect MB control chart for indication of significant bias. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Blank matrices must be the same as the associated samples (i.e. radon free distilled or deionized water, representative solid material, physically and chemically identical filter media.

Table F-18 Gas Flow Proportional Counting					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Background Subtraction Count (BSC) Measurement (Long count for subtracting background from blanks or test sources)	Performed at least on a monthly basis. Determine alpha and beta background initially and after efficiency calibration. (MARLAP 18.5.6.1)	Use a statistical test to determine a change in the background count rate value. (MARLAP 18.5.6.4) or Within ±3σ of mean activity of recent BSCs (minimum of 3 BSCs).	Check control chart for trend and recount. Determine cause and correct problem. If background activity has changed, re-establish BSC. All samples following the last acceptable background measurement must be reanalyzed.	If reanalysis of samples is not possible, apply B-flag to all results in all samples associated with the failed blank.	Detector background measured using a contamination-free source mount. Activity must meet project requirements.
Instrument Contamination Check (ICC) (Short count for controlling gross contamination)	Daily or when working with long count times, before and after each analytical batch. Check after counting high activity samples	Use a statistical test to determine a change in the background count rate value. (MARLAP 18.5.6.4) or Within ±3σ of mean activity of recent BSCs (minimum of 3 BSCs).	Recount the background. If still out of control, locate and correct problem; reanalyze or qualify all impacted samples since last acceptable ICC. If background activity has changed, re-establish BSC and reanalyze samples.	If corrective action fails, apply Q-flag to all results for specific nuclide(s) in all samples associated with the BSC.	Develop detector response control chart immediately after calibration and loss of control.
Laboratory Control Sample (LCS)	One per preparatory batch.	$\lvert Z_{LCS} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{LCS} \rvert \geq 2$. (MARLAP 18.4.3) or Use in-house control chart limits of ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. If required, reprep and reanalyze the LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples and shall contain nuclides within the energy ranges of all those nuclides to be reported. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-18 Gas Flow Proportional Counting					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike (MS)	One per preparatory batch. *(MS not required when yield tracers are employed)*	If activity of the MS > 5 times the unspiked sample, $\lvert Z_{MS} \rvert \leq 3$. (MARLAP 18.4.3) or Within 60-140% recovery.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample Duplicate	One per preparatory batch per matrix.	$\lvert Z_{Dup} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Dup} \rvert \geq 2$. (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is <3; or the relative percent difference (RPD) is <25%.	Check for lab error. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Tracers (if used)	Added to each sample.	Recovery (isotopic yield) within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-19. Liquid Scintillation Counter Analysis					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL) (Efficiency, ROI)	Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings. (MARLAP 18.5.6.4)	Verify manufacturer's specifications for counting efficiency. (MARLAP 18.5.6.4) Establish beta energy ROIs for nuclides of interest. (MARLAP 18.5.6.4)	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Use appropriate reference radionuclide sources, typically unquenched LS cocktails tagged with ^{3}H and/or ^{14}C. No samples may be run until efficiency calibration criteria are met and energy ROIs are established for radionuclides of interest.
Method Calibration (QCAL) (Quench curve)	Prior to method application, matrix, and cocktail changes or if control of system cannot be re-established or demonstrated. (MARLAP 18.5.6.4) .	A mathematical function and quench curve shall be developed so that the 95 percent confidence limit of the function is ≤5% over the expected quench range of the sources. Individual calibration sources shall be counted to achieve ROI measurement uncertainty of ≤1%. (MARLAP 18.5.6.4) or Minimum 10,000 counts for each data point. Correlation coefficient for quench curve is > 0.995	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	When establishing a quench curve, a minimum of five calibration sources of different quench factors shall be used. No samples may be run until calibration criteria are passed.

Table F-19. Liquid Scintillation Counter Analysis					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Calibration (QCAL) (Standard Addition)	Once after each ICAL.	Statistically evaluate replicate test-source analyses. (MARLAP 18.5.6.4) or The duplicate error ratio (DER) between the sample and the duplicate is <3; or the relative percent difference (RPD) is <25%.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Add a spike to a duplicate processed sample or add a spike to a sample that has been counted and then recount. No samples may be run until calibration (Standard Addition) has been verified.
Initial Calibration Verification (ICV)	Once after each ICAL.	Value of each second source nuclide ± 10% of initial calibration value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Use a second source standard for each nuclide. No samples may be run until calibration has been verified.
Continuing Calibration Verification (CCV)	Counting efficiency performance check performed on day-of-use basis. Prior to use for short counting intervals. Before and after a test source batch for longer counting intervals. (MARLAP 18.5.6.4) For batch sequences that run more than a day, performance check is performed at the beginning and end of the batch, as long as it is not longer than a week.	Response checks should have a tolerance limit or control chart set at ± 3% or 3σ of the mean. (MARLAP 18.5.6.4)	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific nuclide(s) in all samples since the last acceptable calibration verification.	ROI for unquenched reference standards (typically 3H and/or 14C). Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table F-19. Liquid Scintillation Counter Analysis					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method Blank (MB)	One per preparatory batch.	$\lvert Z_{Blank} \rvert \leq 3$. Investigate recurrent results with $\lvert Z_{Blank} \rvert \geq 2$. (MARLAP 18.4.1) or In-house control limits of ±3 σ of the mean. With project approval and appropriate qualification and narration, report results with a count rate >5 times that of the affected ROI in the MB.	Recount the blank to confirm results, unless all sample results are >5 times the blank activity. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank. All samples following the last acceptable background measurement must be reanalyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Blank matrices must be the same as the associated samples (i.e. radon free distilled or deionized water, or representative of the material media.
Background Subtraction Count (BSC) Measurement (Unquenched blank)	Prior to initial use and monthly. (MARLAP 18.5.6.4)	Use a statistical test to determine a change in the unquenched background ROI count rate value. (MARLAP 18.5.6.4) or Within ±3σ of mean activity of recent BSCs for each ROI to be determined. (minimum of 3 BSCs)	Check control chart for trend and recount. Determine cause and correct problem. If background activity has changed, re-establish BSC and reanalyze all impacted samples since last acceptable BSC.	If reanalysis cannot be performed, apply B-flag to all results for specific nuclide(s) in all samples associated with the blank.	Unquenched sealed background vial not used for background subtraction. Activity must meet project objectives.
Method Background Measurement (MBM) (Quenched blank)	Each batch. (MARLAP 18.5.6.4)	Use a statistical test to determine a change in the quenched background ROI count rate value. (MARLAP 18.5.6.4) or Within ±3σ of mean activity of recent MBMs for each ROI to be determined. (minimum of 3 MBMs)	Check control chart for trends and recount. Determine cause, correct problem. If background activity has changed, re-establish MBM and reanalyze all impacted samples since last acceptable MBM.	If reanalysis cannot be performed, apply B-flag where count rate <10 times that in the affected ROI(s) in the MBM.	MBM test source matches STS configuration (type, size and position relative to the detector).

Table F-19. Liquid Scintillation Counter Analysis					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	$\|Z_{LCS}\| \leq 3$. Investigate recurrent results with $\|Z_{LCS}\| \geq 2$. (MARLAP 18.4.3) or Use in-house control chart limits of ± 3σ of the mean. In-house control limits may not fall more than 25% from the known LCS value. Acceptance criteria for LCS recovery may be specified by the project.	Recount the LCS to confirm results. Inspect LCS control chart for indication of significant bias. Reprep and reanalyze method LCS and all associated samples.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific nuclide(s) in all samples in the associated preparatory batch.	LCS matrices must be the same as the associated samples and shall contain nuclides within the energy ranges of all those nuclides to be reported. LCS must be counted for a sufficient time to meet the required project minimum activity. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch. *(MS not required when yield tracers or carriers are employed)*	If activity of the MS > 5 times the unspiked sample, $\|Z_{MS}\| \leq 3$. (MARLAP 18.4.3) or Within 60-140% recovery	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample Duplicate	One per preparatory batch per matrix.	$\|Z_{Dup}\| \leq 3$. Investigate recurrent results with $\|Z_{Dup}\| \geq 2$. (MARLAP 18.4.1) or The duplicate error ratio (DER) between the sample and the duplicate is <3; or the relative percent difference (RPD) is <25%.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table F-19. Liquid Scintillation Counter Analysis					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Tracers	Added to each sample as yield monitor.	Yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Carriers	Added to each sample as yield monitor.	Chemical yield within 30-110%.	Reanalysis of sample, including sample preparation.	For the specific nuclide(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. Flagging is only appropriate in cases where the samples cannot be reanalyzed.