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MULTI-AGENCY RADIATION SURVEY AND SITE INVESTIGATION MANUAL (MARSSIM)

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APPENDIX N

Data Validation Using Data Descriptors

Data validation is often defined by six data descriptors:

1) reports to decision maker
2) documentation
3) data sources
4) analytical method and detection limit
5) data review
6) data quality indicators

The decision maker or reviewer examines the data, documentation, and reports for each of the six data descriptors to determine if performance is within the limits specified in the DQOs developed during survey planning. The data validation process should be conducted according to procedures documented in the QAPP.

N.1 Reports to Decision Maker

Data and documentation supplied to the decision maker should be evaluated for completeness and appropriateness and to determine if any changes were made to the survey plan during the course of work. The survey plan discusses the surveying, sampling, and analytical design and contains the QAPP and DQOs. The decision maker should receive all data as collected plus preliminary and final data reports. The final decision on qualifying or rejecting data will be made during the assessment of environmental data. All data, including qualified or rejected data, should be documented and recorded even if the data are not included in the final report.

Preliminary analytical data reports allow the decision maker to begin the assessment process as soon as the surveying effort has begun. These initial reports have three functions.

1) For scoping or characterization survey data, they allow the decision maker to begin to characterize the site on the basis of actual data. Radionuclides of interest will be identified and the variability in concentration can be estimated.

2) They allow potential measurement problems to be identified and the need for corrective action can be assessed.

3) Schedules are more likely to be met if the planning of subsequent survey activities can begin before the final data reports are produced.

N.2 Documentation

Three types of documentation should be assessed: (1) field operation records; (2) laboratory records; and (3) data handling records (EPA 1997a).

N.2.1 Field Operation Records

The information contained in these records documents overall field operations and generally consists of the following:

- *Field measurement records*. These records show that the proper measurement protocol was performed in the field. At a minimum, this documentation should include the names of the persons conducting the activity, measurement identification, measurement locations, measurement results, maps and diagrams, equipment and SOP used, and unusual observations. Bound field notebooks are generally used to record raw data and make references to prescribed procedures and changes in planned activities. Data recording forms might also be used. A document control system should be used for these records to control attributes such as formatting to include pre-numbered pages with date and signature lines.

- *Sample tracking records*. Sample tracking records (*e.g.*, chain-of-custody) document the progression of samples as they travel from the original sampling location to the laboratory and finally to disposal (see Section 7.7).

- *QC measurement records*. QC measurement records document the performance of QC measurements in the field. These records should include calibration and standards' traceability documentation that can be used to provide a reproducible reference point to which all similar measurements can be correlated. QC measurement records should contain information on the frequency, conditions, level of standards, and instrument calibration history.

- *Personnel files*. Personnel files record the names and training certificates of the staff collecting the data.

- *General field procedures*. General field procedures (*e.g.*, SOPs) record the procedures used in the field to collect data and outline potential areas of difficulty in performing measurements.

- *Deficiency and problem identification reports*. These reports document problems and deficiencies encountered as well as suggestions for process improvement.

- *Corrective action reports*. Corrective action reports show what methods were used in cases where general field practices or other standard procedures were violated and include the methods used to resolve noncompliance.

N.2.2 Laboratory Records

The following list describes some of the laboratory-specific records that should be compiled if available and appropriate:

- *Laboratory measurement results and sample data.* These records contain information on the sample analysis used to verify that prescribed analytical methods were followed. The overall number of samples, sample identification, sample measurement results, any deviations from the SOPs, time of day, and date should be included. Sample location information might also be provided.

- *Sample management records*. Sample management records should document sample receipt, handling and storage, and scheduling of analyses. The records will verify that sample tracking requirements were maintained, reflect any anomalies in the samples (*e.g.*, receipt of damaged samples), and note proper log-in of samples into the laboratory.

- *Test methods*. Unless analyses were performed exactly as prescribed by SOPs, this documentation will describe how the analyses were carried out in the laboratory. This documentation includes sample preparation and analysis, instrument standardization, detection and reporting limits, and method-specific QC requirements. Documentation demonstrating laboratory proficiency with each method used could also be a part of the data reporting package, particularly for subcontracted work.

- *QC measurement records*. These include the general QC records, such as initial demonstration of capability, instrument calibration, routine monitoring of analytical performance, calibration verification, *etc.*, considered in Section 7.3 for selecting a radioanalytical laboratory. Project-specific information from the QC checks such as blanks, spikes, calibration check samples, replicates, splits, and so on should be included in these reports to facilitate data quality analysis.

- *Deficiency and problem identification reports*. These reports document problems and deficiencies encountered as well as suggestions for process improvement.

- *Corrective action reports*. Corrective action reports show what methods were used in cases where general laboratory practices or other standard procedures were violated and include the methods used to resolve noncompliance. Corrective action procedures to replace samples violating the SOP also should be noted.

N.2.3 Data Handling Records

Data handling records document protocols used in data reduction, verification, and validation. Data reduction addresses data transformation operations such as converting raw data into reportable quantities and units, using significant figures, calculating measurement uncertainties, *etc.* The records document procedures for handling data corrections.

N.3 Data Sources

Data source assessment involves the evaluation and use of historical analytical data. Historical analytical data should be evaluated according to data quality indicators and not the source of the data (*e.g.*, analytical protocols may have changed significantly over time). Data quality indicators are qualitative and quantitative descriptors used in interpreting the degree of acceptability or utility of data. Historical data sources are addressed during the Historical Site Assessment, and are discussed in Section 3.4.1.

N.4 Analytical Method and Detection Limit

The selection of appropriate analytical methods based on detection limits is important to survey planning. The detection limit of the method directly affects the usability of the data because results near the detection limit have a greater possibility of false negatives and false positives. Results near the detection limit have increased measurement uncertainty. When the measurement uncertainty becomes large compared to the variability in the radionuclide concentration, it becomes more difficult to demonstrate compliance using the guidance provided in MARSSIM.

The decision maker compares detection limits (*i.e.*, minimum detectable concentrations; MDCs) with radionuclide-specific results to determine their effectiveness in relation to the DCGL. Assessment of preliminary data reports provides an opportunity to review the detection limits early and resolve any detection sensitivity problems. When a radionuclide is reported as not detected, the result can only be used with confidence if the MDCs reported are lower than the DCGL.

If the DCGL is less than or equal to the MDC, and the radionuclide is not detected, report the actual result of the analysis. Do not report data as "less than the detection limit." Even negative results and results with large uncertainties can be used in the statistical tests described in Chapter 8. Results reported as "<MDC" cannot be fully used and, for example, complicate even such simple analyses as calculating an average. When the MDC reported for a radionuclide is near the DCGL, the confidence in both identification and quantitation may be low. Information

concerning non-detects or detections at or near MDCs should be qualified according to the degree of acceptable uncertainty.

N.5 Data Review

Data review begins with an assessment of the quality of analytical results and is performed by a professional with knowledge of the analytical procedures. Only data that are reviewed according to a specified level or plan should be used in the quantitative site investigation. Any analytical errors, or limitations in the data that are identified by the review, should be noted. An explanation of data qualifiers should be included with the review report.

All data should receive some level of review. Data that have not been reviewed should be identified, because the lack of review increases the uncertainty in the data. Unreviewed data may lead to Type I and Type II decision errors, and may also contain transcription errors and calculation errors. Data may be used in the preliminary assessment before review, but should be reviewed at a predetermined level before use in the final survey report.

Depending on the survey objectives, the level and depth of the data review varies. The level and depth of the data review may be determined during the planning process and should include an examination of laboratory and method performance for the measurements and radionuclides involved. This examination includes

- evaluation of data completeness
- verification of instrument calibration
- measurement of precision using duplicates, replicates, or split samples
- measurement of bias using reference materials or spikes
- examination of blanks for contamination
- assessment of adherence to method specifications and QC limits
- evaluation of method performance in the sample matrix
- applicability and validation of analytical procedures for site-specific measurements
- assessment of external QC measurement results and QA assessments

A different level or depth of data review may be indicated by the results of this evaluation. Specific data review procedures are dependent upon the survey objectives and should be documented in the QAPP.

N.6 Data Quality Indicators

The assessment of data quality indicators presented in this section is significant to determine data usability. The principal data quality indicators are precision, bias, representativeness, comparability, and completeness (EPA 1997a). Other data quality indicators affecting the RSSI process include the selection and classification of survey units, Type I and Type II decision error rates, the variability in the radionuclide concentration measured within the survey unit, and the lower bound of the gray region (see Section 2.3.1).

Of the six principal data quality indicators, precision and bias are quantitative measures, representativeness and comparability are qualitative, completeness is a combination of both qualitative and quantitative measures, and accuracy is a combination of precision and bias. The selection and classification of survey units is qualitative, while decision error rates, variability, and the lower bound of the gray region are quantitative measures.

The major activity in determining the usability of data based on survey activities is assessing the effectiveness of measurements. Scanning and direct measurements taken during survey activities and samples collected for analysis should meet site-specific objectives based on scoping and planning decisions.

Determining the usability of analytical results begins with the review of QC measurements and qualifiers to assess the measurement result and the performance of the analytical method. If an error in the data is discovered, it is more important to evaluate the effect of the error on the data than to determine the source of the error. The documentation described in Section N.2 is reviewed as a whole for some criteria. Data are reviewed at the measurement level for other criteria.

Factors affecting the accuracy of identification and the precision and bias of quantitation of individual radionuclides, such as calibration and recoveries, should be examined radionuclide by radionuclide. Table N.1 presents a summary of the QC measurements and the data use implications.

N.6.1 Precision

Precision is a measure of agreement among replicate measurements of the same property under prescribed similar conditions. This agreement is calculated as either the range or the standard deviation. It may also be expressed as a percentage of the mean of the measurements such as relative range (for duplicates) or coefficient of variation.

Table N.1 Use of Quality Control Data

Quality Control Criterion	Effect on Identification When Criterion is Not Met	Quantitative Bias	Use
Spikes (Higher than expected result)	Potential for incorrectly deciding a survey unit does not meet the release criterion (Type II decision error)	High	Use data as upper limit
Spikes (Lower than expected result)	Potential for incorrectly deciding a survey unit does meet the release criterion[a] (Type I decision error)	Low	Use data as lower limit
Replicates (Inconsistent)	None, unless analyte found in one duplicate and not the other—then either Type I or Type II decision error	High or Low[b]	Use data as estimate—poor precision
Blanks (Contaminated)	Potential for incorrectly deciding a survey unit does not meet the release criterion (Type II decision error)	High	Check for gross contamination or instrument malfunction
Calibration (Bias)	Potential for Type I or Type II decision errors	High or Low[b]	Use data as estimate unless problem is extreme

[a] Only likely if recovery is near zero.
[b] Effect on bias determined by examination of data for each radionuclide.

For scanning and direct measurements, precision may be specified for a single person performing the measurement or as a comparison between people performing the same measurement. For laboratory analyses, precision may be specified as either intralaboratory (within a laboratory) or interlaboratory (between laboratories). Precision estimates based on a single surveyor or laboratory represent the agreement expected when the same person or laboratory uses the same method to perform multiple measurements of the same location. Precision estimates based on two or more surveyors or laboratories refer to the agreement expected when different people or laboratories perform the same measurement using the same method.

The two basic activities performed in the assessment of precision are estimating the radionuclide concentration variability from the measurement locations and estimating the measurement error attributable to the data collection process. The level for each of these performance measures

should be specified during development of DQOs. If the statistical performance objectives are not met, additional measurements should be taken or one (or more) of the performance parameters changed.

Measurement error is estimated using the results of replicate measurements, as discussed in Chapter 6 for field measurements and Chapter 7 for laboratory measurements. When collocated measurements are performed (in the field or in the laboratory) an estimate of total precision is obtained. When collocated samples are not available for laboratory analysis, a sample subdivided in the field and preserved separately can be used to assess the variability of sample handling, preservation, and storage along with the variability in the analytical process, but variability in sample acquisition is not included. When only variability in the analytical process is desired, a sample can be subdivided in the laboratory prior to analysis.

Summary statistics such as sample mean and sample variance can provide as assessment of the precision of a measurement system or component thereof for a project. These statistics may be used to estimate precision at discrete concentration levels, average estimated precision over applicable concentration ranges, or provide the basis for a continual assessment of precision for future measurements. Methods for calculating and reporting precision are provided in *EPA Guidance for Quality Assurance Project Plans* (EPA 1997a).

Table N.2 presents the minimum considerations, impacts if the considerations are not met, and corrective actions for precision.

N.6.2 Bias

Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction. Bias assessments for radioanalytical measurements should be made using personnel, equipment, and spiking materials or reference materials as independent as possible from those used in the calibration of the measurement system. When possible, bias assessments should be based on certified reference materials rather than matrix spikes or water spikes so that the effect of the matrix and the chemical composition of the contamination is incorporated into the assessment. While matrix spikes include matrix effects, the addition of a small amount of liquid spike does not always reflect the chemical composition of the contamination in the sample matrix. Water spikes do not account for either matrix effects or chemical composition of the contamination. When spikes are used to assess bias, a documented spiking protocol and consistency in following that protocol are important to obtaining meaningful data quality estimates.

Table N.2 Minimum Considerations for Precision, Impact if Not Met, and Corrective Actions

Minimum Considerations for Precision	Impact When Minimum Considerations Are Not Met	Corrective Action
Confidence level as specified in DQOs. Power as specified in DQOs. Minimum detectable relative differences specified in the survey design and modified after analysis of background measurements if necessary One set of field duplicates or more as specified in the survey design. Analytical duplicates and splits as specified in the survey design. Measurement error specified.	Errors in decisions to act or not to act based on analytical data. Unacceptable level of uncertainty. Increased variability of quantitative results. Potential for incorrectly deciding a survey unit does meet the release criterion for measurements near the detection limits (Type I decision error).	For Surveying and Sampling: Add survey or sample locations based on information from available data that are known to be representative. Adjust performance objectives. For Analysis: Analysis of new duplicate samples. Review laboratory protocols to ensure comparability. Use precision measurements to determine confidence limits for the effects on the data. The investigator can use the maximum measurement results to set an upper bound on the uncertainty if there is too much variability in the analyses.

Activity levels for bias assessment measurements should cover the range of expected contaminant concentrations, although the minimum activity is usually at least five times the MDC. For many final status surveys, the expected contaminant concentration is zero or background, so the highest activity will be associated with the bias assessment measurements. The minimum and maximum concentrations allowable in bias assessment samples should be agreed on during survey planning activities to prevent accidental contamination of the environment or an environmental level radioanalytical laboratory.

For scanning and direct measurements there are a limited number of options available for performing bias assessment measurements. Perhaps the best estimate of bias for scanning and direct measurements is to collect samples from locations where scans or direct measurements were performed, analyze the samples in a laboratory, and compare the results. Problems associated with this method include the time required to obtain the results and the difficulty in

obtaining samples that are representative of the field measurement to provide comparable results. A simple method of demonstrating that analytical bias is not a significant problem for scanning or direct measurements is to use the instrument performance checks to demonstrate the lack of analytical bias. A control chart can be used to determine the variability of a specific instrument and track the instrument performance throughout the course of the survey. Field background measurements can also be plotted on a control chart to estimate bias caused by contamination of the instrument.

There are several types of bias assessment samples available for laboratory analyses as discussed in Chapter 7. Field blanks can be evaluated to estimate the potential bias caused by contamination from sample collection, preparation, shipping, and storage.

Table N.3 presents the minimum considerations, impacts if the considerations are not met, and corrective actions for bias.

Table N.3 Minimum Considerations for Bias, Impact if Not Met, and Corrective Actions

Minimum Considerations for Bias	Impact When Minimum Considerations Are Not Met	Corrective Action
Matrix spikes to assess bias of non-detects and positive sample results if specified in the survey design. Analytical spikes as specified in the survey design. Use analytical methods (routine methods whenever possible) that specify expected or required recovery ranges using spikes or other QC measures. No radionuclides of potential concern detected in the blanks.	Potential for incorrectly deciding a survey unit does meet the release criterion (Type I decision error): if spike recovery is low, it is probable that the method or analysis is biased low for that radionuclide and values of all related samples may underestimate the actual concentration. Potential for incorrectly deciding a survey unit does not meet the release criterion (Type II decision error): if spike recovery exceeds 100%, interferences may be present, and it is probable that the method or analysis is biased high. Analytical results overestimate the true concentration of the spiked radionuclide.	Consider resampling at affected locations. If recoveries are extremely low or extremely high, the investigator should consult with a radiochemist or health physicist to identify a more appropriate method for reanalysis of the samples.

N.6.3 Accuracy

Accuracy is a measure of the closeness of an individual measurement or the average of a number of measurements to the true value (EPA 1997a). Accuracy includes a combination of random error (precision) and systematic error (bias) components that result from performing measurements. Systematic and random uncertainties (or errors) are discussed in more detail in Section 6.8.1.

Accuracy is determined by analyzing a reference material of known contaminant concentration or by reanalyzing material to which a known concentration of contaminant has been added. To be accurate, data must be both precise and unbiased. Using the analogy of archery, to be accurate one's arrows must land close together and, on average, at the spot where they are aimed. That is, the arrows must all land near the bull's eye (see Figure N.1).

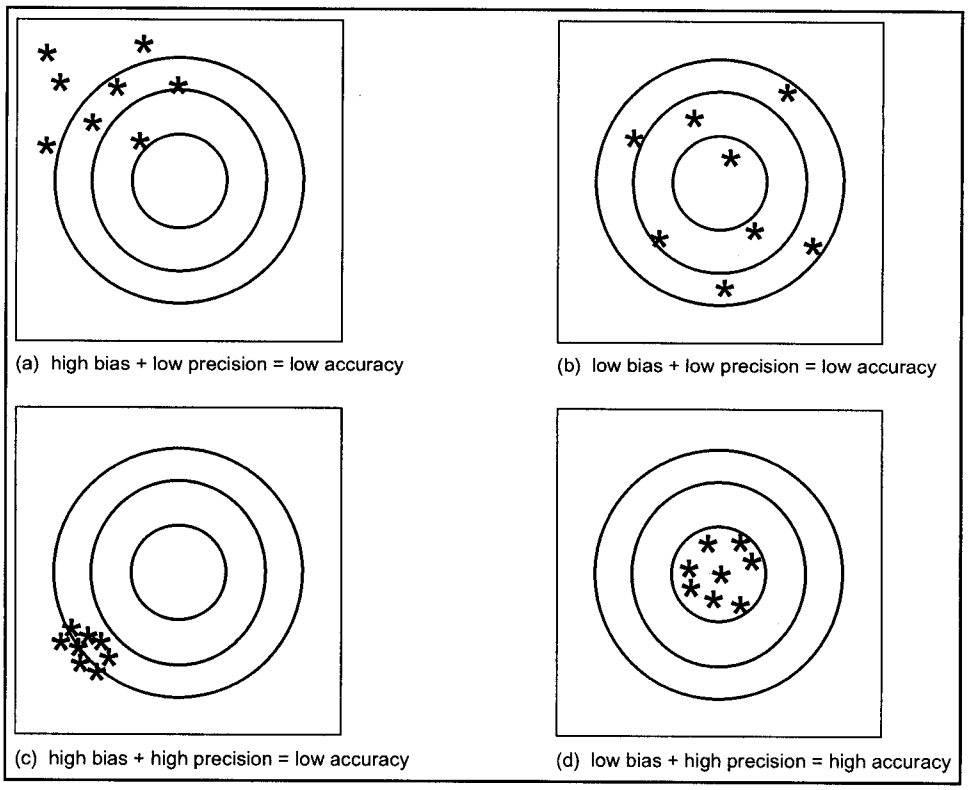

Figure N.1 Measurement Bias and Random Measurement Uncertainty

Accuracy is usually expressed either as a percent recovery or as a percent bias. Determination of accuracy always includes the effects of variability (precision); therefore, accuracy is used as a combination of bias and precision. The combination is known statistically as mean square error. Mean square error is the quantitative term for overall quality of individual measurements or estimators.

Mean square error is the sum of the variance plus the square of the bias. (The bias is squared to eliminate concern over whether the bias is positive or negative.) Frequently it is impossible to quantify all of the components of the mean square error—especially the biases—but it is important to attempt to quantify the magnitude of such potential biases, often by comparison with auxiliary data.

N.6.4 Representativeness

Representativeness is a measure of the degree to which data accurately and precisely represent a characteristic of a population parameter at a sampling point or for a process condition or environmental condition. Representativeness is a qualitative term that should be evaluated to determine whether *in situ* and other measurements are made and physical samples collected in such a manner that the resulting data appropriately reflect the media and contamination measured or studied.

Representativeness of data is critical to data usability assessments. The results of the environmental radiological survey will be biased to the degree that the data do not reflect the radionuclides and concentrations present at the site. Non-representative radionuclide identification may result in false negatives. Non-representative estimates of concentrations may be higher or lower than the true concentration. With few exceptions, non-representative measurements are only resolved by additional measurements.

Representativeness is primarily a planning concern. The solution to enhancing representativeness is in the design of the survey plan. Representativeness is determined by examining the survey plan. Analytical data quality affects representativeness since data of low quality may be rejected for use.

Table N.4 presents the minimum considerations, impacts if the considerations are not met, and corrective actions for representativeness.

N.6.5 Comparability

Comparability is the qualitative term that expresses the confidence that two data sets can contribute to a common analysis and interpolation. Comparability should be carefully evaluated to establish whether two data sets can be considered equivalent in regard to the measurement of a specific variable or groups of variables.

Table N.4 Minimum Considerations for Representativeness, Impact if Not Met, and Corrective Actions

Minimum Considerations for Representativeness	Impact When Minimum Considerations Are Not Met	Corrective Action
Survey data representative of survey unit. Documented sample preparation procedures. Filtering, compositing, and sample preservation may affect representativeness. Documented analytical data as specified in the survey design.	Bias high or low in estimate of extent and quantity of contaminated material. Potential for incorrectly deciding a survey unit does meet the release criterion (Type I decision error). Inaccurate identification or estimate of concentration of a radionuclide. Remaining data may no longer sufficiently represent the site if a large portion of the data are rejected, or if all data from measurements at a specific location are rejected.	Additional surveying or sampling. Examination of effects of sample preparation procedures. Reanalysis of samples, or resurveying or resampling of the affected site areas. If the resurveying, resampling, or reanalyses cannot be performed, document in the site environmental radiological survey report what areas of the site are not represented due to poor quality of analytical data.

Comparability is not compromised provided that the survey design is unbiased, and the survey design or analytical methods are not changed over time. Comparability is a very important qualitative data indicator for analytical assessment and is a critical parameter when considering the combination of data sets from different analyses for the same radionuclides. The assessment of data quality indicators determines if analytical results being reported are equivalent to data obtained from similar analyses. Only comparable data sets can be readily combined.

The use of routine methods (as defined in Section 7.6) simplifies the determination of comparability because all laboratories use the same standardized procedures and reporting parameters. In other cases, the decision maker may have to consult with a health physicist and/or radiochemist to evaluate whether different methods are sufficiently comparable to combine data sets.

There are a number of issues that can make two data sets comparable, and the presence of each of the following items enhances their comparability (EPA 1997a).

- two data sets should contain the same set of variables of interest.
- units in which these variables were measured should be convertible to a common metric.
- similar analytic procedures and quality assurance should be used to collect data for both data sets
- time of measurements of certain characteristics (variables) should be similar for both data sets
- measuring devices used for both data sets should have approximately similar detection levels
- rules for excluding certain types of observations from both samples should be similar
- samples within data sets should be selected in a similar manner
- sampling frames from which the samples were selected should be similar
- number of observations in both data sets should be of the same order of magnitude

These characteristics vary in importance depending on the final use of the data. The closer two data sets are with regard to these characteristics, the more appropriate it will be to compare them. Large differences between characteristics may be of only minor importance depending on the decision that is to be made from the data.

Table N.5 presents the minimum considerations, impacts if they are not met, and corrective actions for comparability.

N.6.6 Completeness

Completeness is a measure of the amount of valid data obtained from the measurement system, expressed as a percentage of the number of valid measurements that should have been collected (*i.e.*, measurements that were planned to be collected).

Completeness for measurements is calculated by the following formula:

$$\%Completeness = \frac{(Number\ of\ Valid\ Measurements)\ x\ 100}{Total\ Number\ of\ Measurements\ Planned}$$

Completeness is not intended to be a measure of representativeness; that is, it does not describe how closely the measured results reflect the actual concentration or distribution of the contaminant in the media being measured. A project could produce 100% data completeness (*i.e.*, all planned measurements were actually performed and found valid), but the results may not be representative of the actual contaminant concentration.

Table N.5 Minimum Considerations for Comparability, Impact if Not Met, and Corrective Actions

Minimum Considerations for Comparability	Impact When Minimum Considerations Are Not Met	Corrective Action
Unbiased survey design or documented reasons for selecting another survey design. The analytical methods used should have common analytical parameters. Same units of measure used in reporting. Similar detection limits. Equivalent sample preparation techniques. Analytical equipment with similar efficiencies or the efficiencies should be factored into the results.	Non-additivity of survey results. Reduced confidence, power, and ability to detect differences, given the number of measurements available. Increased overall error.	For Surveying and Sampling: Statistical analysis of effects of bias. For Analytical Data: Preferentially use those data that provide the most definitive identification and quantitation of the radionuclides of potential concern. For quantitation, examine the precision and accuracy data along with the reported detection limits. Reanalysis using comparable methods.

Alternatively, there could be only 70% data completeness (30% lost or found invalid), but, due to the nature of the survey design, the results could still be representative of the target population and yield valid estimates. The degree to which lack of completeness affects the outcome of the survey is a function of many variables ranging from deficiencies in the number of measurements to failure to analyze as many replications as deemed necessary by the QAPP and DQOs. The intensity of effect due to incompleteness of data is sometimes best expressed as a qualitative measure and not just as a quantitative percentage.

Completeness can have an effect on the DQO parameters. Lack of completeness may require reconsideration of the limits for decision error rates because insufficient completeness will decrease the power of the statistical tests described in Chapter 8.

For most final status surveys, the issue of completeness only arises when the survey unit demonstrates compliance with the release criterion and less than 100% of the measurements are determined to be acceptable. The question now becomes whether the number of measurements is sufficient to support the decision to release the survey unit. This question can be answered by constructing a power curve as described in Appendix I and evaluating the results. An alternative

method is to consider that the number of measurements estimated to demonstrate compliance in Chapter 5 was increased by 20% to account for lost or rejected data and uncertainty in the calculation of the number of measurements. This means a survey with 80% completeness may still have sufficient power to support a decision to release the survey unit.

Table N.6 presents the minimum considerations, impacts if the considerations are not met, and corrective actions for completeness.

Table N.6 Minimum Considerations for Completeness, Impact if Not Met, and Corrective Actions

Minimum Considerations for Completeness	Impact When Minimum Considerations Are Not Met	Corrective Action
Percentage of measurement completeness determined during planning to meet specified performance measures.	Higher potential for incorrectly deciding a survey unit does not meet the release criterion (Type II decision error). Reduction in power. A reduction in the number of measurements reduces site coverage and may affect representativeness. Reduced ability to differentiate site levels from background. Impact of incompleteness generally decreases as the number of measurements increases.	Resurveying, resampling, or reanalysis to fill data gaps. Additional analysis of samples already in laboratory. Determine whether the missing data are crucial to the survey.

N.6.7 Selection and Classification of Survey Units

Selection and classification of survey units is a qualitative measure of the assumptions used to develop the survey plan. The level of survey effort, measurement locations (*i.e.*, random vs. systematic and density of measurements), and the integrated survey design are based on the survey unit classification. The results of the survey should be reviewed to determine whether the classification used to plan the survey is supported by the results of the survey.

If a Class 3 survey unit is found to contain areas of contamination (even if the survey unit passes the statistical tests), the survey unit may be divided into several survey units with appropriate classifications, and additional surveys planned as necessary for these new survey units.

Class 3 areas may only require additional randomly located measurements to provide sufficient power to release the new survey units. Class 2 and Class 1 areas will usually require a new survey design based on systematic measurement locations, and Class 1 areas may require remediation before a new final status survey is performed.

If a Class 2 survey unit is determined to be a Class 1 survey unit following the final status survey and remediation is not required, it may not be necessary to plan a new survey. The scan MDC should be compared to the $DCGL_{EMC}$ to determine if the measurement spacing is adequate to meet the survey objectives. If the scan MDC is too high, a new scan survey using a more sensitive measurement technique may be available. Alternatively, a new survey may be planned using a new measurement spacing or a stratified survey design may be implemented to use as much of the existing data as possible.

N.6.8 Decision Error Rates

The decision error rates developed during survey planning are related to completeness. A low level of completeness will affect the power of the statistical test. It is recommended that a power curve be constructed as described in Appendix I, and the expected decision error rates compared to the actual decision error rates to determine if the survey objectives have been accomplished.

N.6.9 Variability in Contaminant Concentration

The variability in the contaminant concentration (both in the survey unit and the reference area) is a key parameter in survey planning, and is related to the precision of the measurements. Statistical simulations show that underestimating the value of σ (the standard deviation of the survey unit measurements) can greatly increase the probability that a survey unit will fail to demonstrate compliance with the release criterion.

If a survey unit fails to demonstrate compliance and the actual σ is greater than the σ used during survey planning, there are several options available to the project manager. If the major component of variability is measurement uncertainty, a new survey can be designed using a measurement technique with higher precision or a lower MDC to reduce variability. If samples were collected as part of the survey design, it may only be necessary to reanalyze the samples using a method with higher precision rather than collect additional samples. Alternatively, the number of measurements can be increased to reduce the variability.

If the variability is due to actual variations in the contaminant concentration, there are still options available. If there is a high variability in the reference area, it may be appropriate to demonstrate the survey unit is indistinguishable from background. NUREG 1505 (NRC 1997b) provides guidance on determining whether this test is appropriate and performing the statistical tests. If the variability is caused by different contaminant distributions in different parts of the site (*i.e.*, changing soil types influences contaminant concentrations), it may be appropriate to redefine the survey unit boundaries to provide a more homogeneous set of survey units.

N.6.10 Lower Bound of the Gray Region

The lower bound of the gray region (LBGR) is used to calculate the relative shift, which in turn is used to estimate the number of measurements required to demonstrate compliance. The LBGR is initially set arbitrarily to one half the $DCGL_W$. If this initial selection is used to design the survey, there is no technical basis for the selection of this value. This becomes important because the Type II decision error rate (β) is calculated at the LBGR.

For survey units that pass the statistical tests, the value selected for the LBGR is generally not a concern. If the survey unit fails to demonstrate compliance, it may be caused by improper selection of the LBGR. Because the number of measurements estimated during survey planning is based on the relative shift (which includes both σ and the LBGR), MARSSIM recommends that a power curve be constructed as described in Appendix I. If the survey unit failed to demonstrate compliance because of a lack of statistical power, an adjustment of the LBGR may be necessary when planning subsequent surveys.

es/er/ms-5

Evaluation of Radiochemical Data Usability

J.G. Paar
University of Tennessee, Knoxville/Oak Ridge National Laboratory

D.R. Porterfield
Chemical Science and Technology Division
Los Alamos National Laboratory

Environmental Restoration Division
P.O. Box 2003
Oak Ridge, TN 37831-7294

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Environmental Management Activities at the
EAST TENNESSEE TECHNOLOGLY PARK
Oak Ridge, Tennessee 37831
managed by
LOCKHEED MARTIN ENERGY SYSTEMS, INC.
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U.S. DEPARTMENT OF ENERGY
under contract DE-AC-05-84OR21400

PREFACE

Environmental remedial decisions require the acquisition of data of known and sufficient quality for intended use. Data verification and validation (V&V) are two of the tools that ensure a level of quality assurance in data usability. When applied to radiochemistry, no national standard currently covers V&V concepts adequately, and the need for a document of this type was recognized by most of the Department of Energy Complex. This document was developed through intersite cooperation and provides a reasonable approach for the evaluation of radioanalytical data for purposes of environmental remediation but can also be applied to data intended for non-remedial purposes.

This document is intended to provide a framework onto which implementing procedures can be written. It is a stand-alone document for the purposes of data evaluation; however, sufficient laboratory deliverables must exist to enable the V&V tools to be used. It is recommended that if V&V procedures are written based on this document, that corresponding specifications for analytical laboratory deliverables are also written.

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D. Bottrell	DOE-EM 263
D. Carden	DOE-ORO
J. Chambers	FERMCO
B. Crandall	WSRC
A. Daniel	MMES
J. Fisk	LANL
J. Griggs	EPA-NAREL
L. Hook	MMES
J. Karnofsky	
T. Killeen	WSRC
C.K. Liu	DOE-RL
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M. Schwab	BHC
J. Warren	EPA/QAMS
R. Wells	INEL
L. Withrow	Quanterra
C. Wrotriiak	Dames & Moore

I. PURPOSE 1

II. REFERENCES 1

III. DEFINITIONS 3

IV. GENERAL INFORMATION 6
- A. Relation to Laboratory Statement of Work 6
- B. Reporting Recommendations 6
- C. Qualification of Data 7
- D. Problem Identification 7
- E. Data Verification 7
  - 1. Verification Report 8
- F. Analytical Data Validation 8
  - 1. Validation Report 8

V. PROCEDURE 8
- A. Custody of Samples and Sample Documentation 9
- B. Holding Time and Turn-around Time 9
- C. Sample Preservation 10
- D. Instrument Calibration 11
  - 1. Scope 11
  - 2. Mechanism for Initial Calibration Evaluation 12
  - 3. General Technical Specifications for Initial Calibration 12
    - a. Check Source Statistics 12
    - b. Radioactive Sources 13
    - c. Control Criteria 13
    - d. Establishment of control points 13
    - e. Control of Background 14
    - f. Geometry 14
    - g. Background subtraction 14
    - h. Recalibration 14
  - 4. Specific Technical Specifications for Calibration Verification 15
  - 5. Initial Calibration for Gross Alpha and Gross Beta by Gas Proportional Systems 15
  - 6. Calibration Verification for Gross Alpha and Gross Beta by Gas Proportional Systems 17
  - 7. Initial Calibration for Liquid Scintillation 18
  - 8. Calibration Verification for Liquid Scintillation 19
  - 9. Initial Calibration for Alpha Spectrometry 21
  - 10. Calibration Verification for Alpha Spectrometry 22
  - 11. Initial Calibration for Gamma Spectrometry 24
  - 12. Calibration Verification for Gamma Spectrometry 25
- E. Quality-indicator samples 27
  - 1. Total Propagated Uncertainty (TPU) 28
  - 2. Standard Traceability 29
  - 3. Laboratory Control Sample (LCS) 29
  - 4. Laboratory Duplicate 31
  - 5. Matrix Spike (MS) 32
  - 6. Method Blank 34

F. Chemical Yield - Tracers and Carriers 36
G. Required Detection Limits (RDL) 37
H. Nuclide Identification and Quantification 37
1. Test for Detection 37
2. Negative Results 38
3. Sample Result Recalculation 38
I. Instrument-Specific Sample Considerations 38
1. Sample Analytical Parameters for Gas Proportional Counting 39
2. Spectral Interpretation (alpha and gamma spectrometry) 39

VI. APPENDIXES 41

I. PURPOSE

This procedure provides a framework for implementation of radiochemical data verification and validation for environmental remediation activities. It has been developed through participation of many individuals currently involved in analytical radiochemistry, radiochemical validation, and validation program development throughout the DOE complex. It should be regarded as a guidance to use in developing an implementable radiochemical validation strategy.

This procedure provides specifications for developing and implementing a radiochemical validation methodology flexible enough to allow evaluation of data useability for project-specific Data Quality Objectives (DQO). Data produced by analytical methods for which this procedure provides limited guidance are classified as "non-routine" radionuclides and methods, and analyses by these methods may necessitate adoption of modified criteria from this procedure.

This procedure is applicable to radionuclide contaminants routinely analyzed by common radioanalytical methods primarily in aqueous and soil/sediment matrices for Environmental Restoration (ER) activities. This procedure is applicable to radionuclide data produced through radioanalytical methods which use instrumentation for detecting activity. This procedure is not applicable to mass spectrometric or fluorimetric methodologies. An example listing of radionuclides and general methodologies to which this procedure applies is provided in Appendix D.

Specifications in this procedure should be incorporated into appropriate project documentation such as the Sampling and Analysis Plan or Project Work Plan and into contractual Statements of Work (SOW) between the project and the analytical laboratories. This procedure shall be used as a baseline used to create project-specific procedures and checklists needed to perform radiochemical data verification and validation.

In this procedure, the word "shall" is used to denote a requirement; the word "should" is used to denote a recommendation; and the word "may" is used to denote permission, neither a requirement nor a recommendation. In conformance to this procedure, all analyses shall be performed in accordance with its requirements, but not necessarily with its recommendations; however, justification must be documented for deviations from recommendations.

II. REFERENCES

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III. DEFINITIONS

Affected Sample Result: a sample result that is considered to be significantly influenced by a quality deficiency, and is qualified, accordingly, through analytical data validation.

Analytical Batch: An analytical batch is a group of sample aliquots analyzed together on the same instrument detector system.

Analytical Data Validation: a technically based analyte and sample specific process that extends the qualification process beyond method or contractual compliance and provides level of confidence in the data that an analyte is presentor absent and if present, the associated variability. Data validation is a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data that may result in physical qualification of the data. Data validation occurs prior to drawing a conclusion from the body of data.

Analytical Data Verification: a process of evaluating the completeness, correctness, consistency, and compliance of a set of facts against a standard or contract. Data verification is defined as a systematic process, performed by either the data generator or by an entity external to the data generator.

Calibration Verification: Calibration verification, as described in this procedure, is defined as a periodic evaluation of instrumentstandardization established during initial calibration. Using tolerance or statistical control charts, calibration verification an alert the instrument user of the occurrence of out-of-control instrumental conditions.

Carrier: A carrier is a stable element/compound, introduced into the sample preparation/analysis process, that will behave chemically similar to the analyte isotope(s). It is by virtue of this chemical similarity that the carrier will "carry" the analyte isotope(s) through the samplepreparation/analysis process. The amount of the carrier recovered at the end of the analysis compared to that added initially is often used in the calculation of the final result.

Correctable Problem: Correctable problems are deficiencies within data packages which may be rectified through consultation with the laboratory. Correctable problems may be revealed during both data verification and data validation.

- correctable problems revealed during verification are those deficiencies that can be addressed by obtaining additional information from the laboratory
- correctable problems revealed during validation are those deficiencies with analyses that can be solved by either a second preparation and/or analysis of a sample.

Counting uncertainty: Counting uncertainty, as described in this procedure, is defined as the statistical sample standard deviation, which is an approximation of the population standard deviation, and is numerically defined as the square root of the number of counts obtained from a detector. This relationship holds true, provided that thedistribution that the counts follows the Poisson distribution. Units for counting uncertainty are the same as for the reported result and the MDC.

Initial Calibration: Initial calibration, as described in this procedure, is defined as the standardization of an instrument used in radioactivity detection against a traceable radioactive source(s) of known identity and quantity. This standardization prevails until such time as analytical conditions are deemed out of acceptable tolerance or statistical control limits.

Holding Time: Holding time, as described in this procedure, is defined as the period of time between sample collection and sample activity detection.

Minimum Detectable Activity (MDA) : The amount of a radionuclide, which if present in a sample, would be detected with a β probability of non-detection while accepting a probability, α, of erroneously detecting that radionuclide in a appopriate blank sample. For this procedure, the α and β probabilities are both set at 0.05.

Minimum Detectable Concentration (MDC): The MDA expressed in concentration units relative to the sample weight or volume.

Non-correctable problem: Non-correctable problems are those deficiencies, within data packages that cannot be addressed through additional laboratory submittals, and sample results must stand as-is.

Non-Correctable Problem: Non-correctable problems are deficiencies within data packages which preclude the evaluation of data quality by predefined criteria.Non-correctable problems may be revealed during both data verification and data validation.

Preparation Batch: A preparation batch is a group of sample aliquots prepared together at the same time using the same method and related to the same quality-indicator samples.

Quality Control Chart: For purposes of this procedure, a quality control chart is used to determine if the response of the instrument has changed statistically; the magnitude the statistical response change

may or may not be significant when compared to the required precision and accuracy criteria for the overall analytical technique.

Quality-indicator Sample: Quality-indicator samples are those samples made ready in the laboratory which provide direct or indirect evaluation of the status of analytical system and resulting data quality. Collectively, quality indicator samples are the laboratory control sample, laboratory duplicate, matrix spike, and method blank.

Reporting Batch: A reporting batch is a group of sample results reported together in a single data package. The reporting batch may be comprised of samples prepared and analyzed together in the same preparation batch or samples prepared and analyzed in different preparation or analytical batches.

Required Detection Limit (RDL): The RDL is a contractually-specified detection limit (MDA or MDC) which, under typical analytical circumstances, should be achievable.

Standard Reference Material (SRM): A material or substance of one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. The SRM is characterized by the U.S. National Institute of Standards and Technology (NIST) or other certified testing authority, and issued with a certificate providing the results of the characterization.

Tolerance Chart: For purposes of this procedure, a tolerance chart is based upon maintaining a change of instrument response to a tolerance level judged acceptable to meet overall quality requirements for the technique; a tolerance level should never be more restrictive than what is statistically possible.

Total Propagated Uncertainty (TPU): The addition of the square root of the sum of the squares of random components of the individual uncertainties, plus the magnitude of the estimated individual systematic relative uncertainties. TPU may include uncertainties introduced through field sampling and analytical laboratory procedures. For the purposes of this procedure, TPU includes only those random and systematic uncertainties associated only with laboratory preparation and analysis. Refer to Appendix C for a full description of TPU.

$$TPU = \sqrt{\Sigma R_i^2 + \Sigma s_j^2}$$

R	=	random components of individual relative uncertainties
s	=	magnitude of the estimated individual systematic relative uncertainties

Traceable Reference Material (TRM): A NIST prepared standard reference material or a sample of known activity or concentration prepared from a NIST standard reference material (derived standard material).

Tracer: A tracer is a radioactive isotope, introduced into the sample preparation/analysis process, that will behave chemically similar to the analyte isotope(s). The tracer isotope is of the same element as the analyte isotope(s) except where the decay mode, half-life, or availability dictate the use of the isotope

of a different element. The activity of tracer detected at the end of the analysis compared to that added initially is used in the calculation of the final result.

Turn-around Time: Turn-around time is contractually-specified as the amount of time which elapses between laboratory receipt of the raw samples and subsequent data receipt by the client.

Well Characterized Reference Material (WCRM): The WCRM may be derived from a field sample which has been well characterized through multiple analyses providing a high level of confidence of the activity level in the sample. The WCRM may be submitted to NIST for characterization and classification as a TRM.

IV. GENERAL INFORMATION

A. Relation to Laboratory Statement of Work

Implementation of this procedure depends on the agreement of work to be performed by analytical laboratory in the form of a project-specific laboratory Statement of Work (SOW). While it is outside the scope of this procedure to provide requirements for the radioanalytical laboratory, validation will be expedited if laboratory deliverables are consistent with the evaluation requirements of this procedure.

B. Reporting Recommendations

All samples should be reported with a background-subtracted sample result. The counting uncertainty, TPU, and MDC must be reported at the same level of confidence (e.g. ±2s at 95%).

C. Qualification of Data

When qualification conditions are met, a qualifier and reason code shall be physically or electronically associated with theaffected result(s). If more than one reason code is applied, each must be separated by commas. The listing of available codes is presented in Appendix B. If data are not qualified, a character (e.g. an equals sign, orother alphanumeric symbol different from a qualifier) is entered on the sample result line. As directed or as appropriate, reason codes may be applied without qualifiers.

D. Problem Identification

Problems identified through data verification and validation are separated into two categories: correctable problems and non-correctable problems.

i. Correctable problems

- The first category contains those problems which may be rectified through consultation with the laboratory.
- The second subcategory contains those problems are those that can be rectified by either a second preparation and/or analysis of a sample.

ii. *Non-Correctable Problem:* Non-correctable problems are deficiencies within data packages which preclude the evaluation of data quality by predefined criteria.

E. Data Verification

Data verification is defined as a systematic process, performed by either the data generator or by an entity external to the data generator, which results in evaluation of the completeness, correctness, consistency, and compliance of a data set against a standard or contract.

If verification is performed by the data generator, a project-level surveillance must be established by which the performance of the verification process is evaluated.

Data verification, at the project level, may be conducted either by the data verifier or by the data validator, whichever expedites the review process. If verification is conducted independently of the validator, it includes two activities. The first activity entails inventory of the data package to ensure compliance with the contract and statement of work, in terms of the required deliverables. The second activity entails to inform the validator that a non-correctable problem has occurredand that data may need qualification. If verification is conducted by the validator, the first activity is conducted similarly, but the second activity may result in prompt qualification of data.

Data verification should provide a mechanism for problem resolution with the laboratory; it should not be an after-the-fact identification of non-correctable deficiencies.

1. Verification Report

A verification report is written by thedata verifier and takes, as input, the steps in this procedure that are listed as "Verification." The data verifie does not qualify data if verification criteria are not met but indicates in the verification report the circumstances surrounding correctable and non-correctable problems in the data package which will be transferred to the validator for possible qualification. The verification report must be in a standard format, and must remain a part of the analytical data package. An example of a verification report is included in Appendix F.

F. Analytical Data Validation

Analytical data validation, including field andlaboratory data review, is defined as a systematic process, performed external from the data generator, which applies a defined set of performance-based criteria to a body of data which may result in qualification of the data. Data validation is not performed by the analytical laboratory. Data validation provides a levebf assurance, based on a technical evaluation, that an analyte is present or absent,and if present, the level of uncertainty associated with the measurement, and occurs prior to drawing a conclusion from a body of data. Analytical data validation for radiochemistry includes a technical review of a laboratory data package covering the evaluation of quality-indicator samples, the identification and quantitation of analytes, and the effect of deficiencies in quality control on analytical sample data.

1. Validation Report

A validation report that includes the results of validation activities must be completed by the validator and takes, as input, the verification report and the steps in this procedure that are listed as "Validation." A validation report must be completed for every reporting batch that passes through validation. To expedite writing the validation report, comments must include explanations for qualification only if the reason codes do not adequately describe justification for qualification. Comments on data qualification for which reason codes adequately describe qualification reason are not necessary.

Both the verification and validation reports must be completed regardless of who performs the reviews.

V. PROCEDURE

The steps in this section describe the processes of radiochemical data verification and validation.

Refer to Appendix A for qualifier and reason code descriptions. Refer to Appendix B for guidance on qualification due to multiple quality deficiencies.

A. Custody of Samples and Sample Documentation

While verification/validation cannot asure a sample has truly been in custody from the field to the final result, an evaluation of field and laboratory chains of custody will provide the best indicator. A sample is defined as being in custody if any of the following conditions are met:

- it is within one's possession;

- it is within one's view, after being in one's possession;
- it was in one's possession and then was secured to prevent tampering;
- it is placed in a designated secure area.

1. Verification

Custody of the samples reported in the data package should be verified by tracing the signature record on both field and laboratory chains of custody. Verify that all samples on the chains of custody are present in the reporting batch. If there is a break in custody of the sample(s), representativeness of the samples may be in question. Indicate this problem in the verification report.

Verification of sample documentation includes result report header checks for accuracy from the COC. If header information is incorrect when compared to the COC, the verifier shall mark through the incorrect header entry and pen in the correct entry, placing initials and date next to the correction. If sample identity is in question, every attempt should be made to verify the true identity of each sample, else sample representativeness is in doubt.

2. Validation

If sample origin and identity can not be substantiated, sample data may be qualified "R."

B. Holding Time and Turn-around Time

Holding time is defined for radiochemical data verification and validation as the period of time between sample collection and sample activity detection. Samples must be analyzed within a holding time precluding significant decay of short-lived target radionuclides. Holding time should be contractually specified to the laboratory. If holding times are not contractually satisfied, and if other technical corrective actions, such as increased count times or increased sample aliquot volumes or weights, have not been performed to compensate for long holding times, , the RDL may be exceeded

Physical characteristics and matrix influences must also be considered when setting holding times. These characteristics must be considered when planning for validation implementation. The following table presents commonly-analyzed radionuclides on the DOE Complex that have characteristics affecting holding time establishment decisions:

Table 5.1 Physical and Matrix-related Characteristics

Nuclide	Physical or Matrix-related Characteristic
Anionic	Volatile when placed in acid solution
^{3}H	Volatile
^{99}Tc	Volatile

The following table presents commonly-analyzed radionuclides on the DOE Complex particularly susceptible to holding time and RDL exceedences due to short half-life:

Table 5.2 Commonly-Analyzed Short Half-Life radionuclides

Nuclide	$T_{½}$
^{131}I	8.04 days
^{222}Rn	3.82 days
^{210}Po	138 days
^{89}Sr	50.5 days

1. Verification

If the sample MDC is reported at a level greater than the RDL when the sample result is less than the MDC or ±2s counting uncertainty, and no justification is provided for not reanalyzing at a longer count time or greater sample aliquot, data are noncompliant with the contractual RDL. Note the occurrence of RDL exceedence in the verification report.

Turn-around time is evaluated as strictly a contractual issue An agreement should be established between the client and the laboratory concerning turn-around times for reported data. If turn-around times are not satisfied, a contractual mechanism should provide for appropriate action.

2. Validation

It should be realized that RDL exceedence may occur for reasons other than excessive radionuclide disintegration. Reference should be made to other potential factors such as count time and aliquot size to apply the appropriate reasoncode(s). The affected results are not qualified, but are flagged only with the reason code "Q10" if reanalysis can not be performed or deemed by the project as unnecessary.

C. Sample Preservation

When appropriate, or in the absence of known preservation techniques, the preservatives and container types listed in the following table should be used for aqueous samples.

Table 5.3 Preservation and Container Requirements

Nuclide or Group	Preservative	Container
Gross Alpha or Beta	Concentrated HCl or HNO_3 to pH≤ 2	Plastic or Glass
^{226}Ra or ^{228}Ra	Concentrated HCl or HNO_3 to pH≤ 2	Plastic or Glass
U, Th	Concentrated HCl or HNO_3 to pH≤ 2	Plastic or Glass
^{89}Sr / ^{90}Sr	Concentrated HCl or HNO_3 to pH≤ 2	Plastic or Glass
^{131}I	HCl/ 2M $NaHSO_3$	Plastic or Glass
^{3}H	None	Glass

^{99}Tc	Concentrated HCl or HNO_3 to pH≤ 2	Plastic or Glass
Gamma Emitters	Concentrated HCl or HNO_3 to pH 2	Plastic or Glass

United States Environmental Protection Agency. *Manual for the Certification of Laboratories Analyzing Drinking Water, Criteria and Procedures Quality Assurance, Third Edition.* EPA/570/9-90/008.

1. Verification

If the proper preservative and/or container type have not been used, note the problem in the verification report.

2. Validation

If samples have not been preserved properly in the field or have been stored in an improper container, qualify those sample results < MDC "UJ." Sample results ≥MDC may not require qualification; however "J" may be placed if necessary.

If samples with radionuclides amenable to preservative with acid have not been acidified in the field, but have been acidified in the laboratory prior to subsampling, qualification may not be necessary. The matrix and container type will not affect the radioactive characteristics of the radionuclides in the sample. For this reason, neglecting to acidify samples prior to shipment to a laboratory should not necessarily result in qualification. However, as radionuclides will adhere to the container walls, acidification (of aqueous samples) either during the sampling event or at the laboratory prior to subsampling is critical to ensure that all radioactive components are in solution, and the representativeness of the sample is maintained.

D. Instrument Calibration

1. Scope

It is outside the scope of this document to prescribe calibration requirements for the laboratory. This section provides recommended frequencies, performance and evaluation criteria, based on existing ANSI standards. Decisions regarding the deliverable requirements for calibration data must be made during the project planning phase and communicated to the laboratory in the SOW. Decisions regarding calibration evaluation strategy should be influenced by the strategy outlined in this section in order to provide a consistent approach to data evaluation with respect to calibration and to expedite the verification and validation processes.

2. Mechanism for Initial Calibration Evaluation

All calibration data are essential for project records from the standpoint of defending the conditions under which samples were analyzed; however, evaluation of initial calibration data should not involve verification and validation with each reporting batch.

To provide a mechanism for initial calibration evaluation at the project-level, the following approach should be taken.

a. Laboratory Statements of Work must incorporate calibration definitions, frequency, and quality control criteria.

b. Upon award of a contract, the laboratory must provide to the project all initial calibration data for all detector systems to be used under the contract. At this time, the control status of initial calibration can be evaluated. The contract must require that the laboratory update the project with changes in calibration status. Tracking numbers for the initial calibration must be included in all reporting batches for reference purposes.

c. If the necessary deliverables for initial calibration cannot be provided by the laboratory, two potential options exist:

   i. if missing deliverables do not significantly affect the ability to evaluate sample data, the project may accept the initial calibration data

   ii. if missing deliverables either present a significant nonconformance for project records retention or preclude the ability to evaluate sample data, the project may request the laboratory to perform a new initial calibration in accordance with the deliverables in this section.

3. General Technical Specifications for Initial Calibration

The following technical specifications apply to all initial calibrations and calibration verifications, independent of counting instrumentation category.

a. Check Source Statistics

The instrument calibration sources should provide adequate counting statistics (less than 1% Poisson statistic uncertainty) over the time period for which the source is to be counted. However, the source shall not be so radioactive as to cause 1) pulse pileups, 2) dead time that is significantly different from that to be expected from routine analysis, or 3) gain shift in the case of pulse height analyzer systems.

b. Radioactive Sources

Commercially prepared and sealed standards shall not be used after their stated expiration dates, which are based on radionuclide half-life or physical form of the standard, e.g. sealed source or plated planchet. Standards prepared at the laboratory or those purchased without expiration dates should be replaced yearly.

The standard source(s) used in initial calibration shall be NIST-traceable Standard Reference Materials, or equivalent; however, source(s) used in calibration verification are not required to

be NIST-traceable, unless measurements of these sources are directly used in calculation of analytical sample data results.

c. Control Criteria

The scope of this document does not include prescriptive requirements for calibration, however, quality of analytical data is highly dependent on control of the calibration process. To facilitate a framework for defining control of the calibration process, the three following strategies may be incorporated dependant on what instrumentation is being used:

i. Tolerance charts may be established based on consideration of specific performance characteristics of the instrument and complexity of the matrix of samples that will be analyzed. The required precision of tolerance charts must never be more restrictive than that of a quality control chart.

ii. Statistical quality control charts may be established based only on a level of confidence considered necessary for statistical quality control.

iii. Fixed limits may be used by consideration of percent deviation from a known value. With some radiochemical methodologies (e.g. alpha and gamma spectroscopy) establishment of tolerance or statistical quality control charts may provide unrealistic precision goals (e.g. 5% RPD may exceed a ±3s control chart limit but still provide adequate instrumental precision). In these cases evaluation of measured values using a percent deviation approach may provide realistic evaluation of detector precision.

d. Establishment of control points

Establishment of control points for use with a tolerance or statistical quality control chart may be approached in two differing strategies, fixed range or moving range.

i. Fixed range control charts are established by acquiring a predetermined number of points, with associated mean and standard deviation, and comparing subsequent data point acquisitions to those statistical descriptors. This allows for evaluation of instrumental control over time, but may not represent true precision over time.

ii. Moving range control charts are established by acquiring a predetermined number of points, with associated mean and standard deviation, and as subsequent points are acquired, they are included for an up-to-date evaluation of system precision. In using a moving range control chart, only the most recent 20 points are considered in establishing statistical descriptors. The use of moving range control charts allows for real-time evaluation of detector control, but does not allow for evaluation of detector control in relation to initial calibration.

e. Control of Background

The control limits for check sources and backgrounds (where applicable) shall be established using a minimum of 20 sequentially measured data points. .For extended background count periods, a series of at least 10 single background measurements is acceptable. No samples, subject to these specifications may becounted until these warning and control limits have been established.

Background count time should be equal to or greater than sample count time unless precluded by extended low level sample count times, in which case background count time may be less than sample count time.

f. Geometry

With all methods of detection, thecalibration counting geometry used should be the same as that used with the analytical samples.

g. Background subtraction

Calibration data should be background-subtracted, whether data is used in generation of efficiencies, cross-talk, or resolution evaluation.

h. Recalibration

Recalibration should only be necessary in the event that the instrument/system has malfunctioned and the repaired equipment has responded to a QC test in a fashion that the tolerance level of a control chart has been exceeded, i.e.the operating or response characteristics of the instrument/system have changed more than the tolerance/control limits permit. Detector calibration is verified according to frequencies that will satisfy contractual criteria, and according to criteria defining the warrant of corrective action.

4. Specific Technical Specifications for Calibration Verification

a. Calibration verification data must besubmitted with each reporting batch and will be evaluated at that frequency. If samples within a reporting batch are from separate counting batches, calibration verification documentation must be included for all counting batches.

b. Each reporting batch submitted from the laboratory to the project should contain control charting data related to calibration verification for all detectors used in the analyses of the analytical samples.

c. Calibration verification is performed and monitored with tolerance or QC charts for instrumental parameters specific to each typeof detector. If the daily check source count result exceeds the tolerance limits or $\pm 3\sigma$ control limit of the appropriate 20 points, the laboratory should recount the check source to verify the out of control condition. If the recount again

exceeds the control limit, the system is considered out of control, and no samples shall be run on that system until it is brought back into control. If the recount is in control a third count shall be done and if in control, analytical sample counting may continue, otherwise no samples shall be analyzed on that system until it is brought back into control. Any samples counted after the last in-control check standard must be recounted except for those where decay has eliminated that radionuclide.

d. If calibration verification data exceed the tolerance limits or the $\pm 3\sigma$ control limits, reference must be made to quality control sample data in the data package to determine the extent of calibration nonconformance on the analytical batch. Exceedence of control limits may not constitute qualification of data; but conversely, excessive control limit exceedence may affect all data in an analytical batch, justifying qualification.

5. Initial Calibration for Gross Alpha and Gross Beta by Gas Proportional Systems

Initial calibration review consists of evaluations of efficiency, background, and cross talk.

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Summary report(s) for both gross alpha and gross beta including the following:
- dates of calibration and planchet preparation;
- mass attenuation curves including mas of salts, planchet diameter, efficiencies at each mass, and mathematical relationships developed from self absorption curves;
- matrix residue identity used for curve establishment — e.g. ASTM Type II water;
- identities of nuclides used for calibration;
- geometries;
- backgrounds;
- charts of voltage plateaus;
- number of counts accumulated in each channel for each standard;
- count times for all analyses;
- calculated activities for all analyses;
- cross talk factors

ii. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate;
iii. All raw data supporting initial calibration;
iv. Certificates for standards used in calibration.

b. Frequency

An initial calibration for gas proportional counters should be performed when out-of-control conditions, indicated from control charting, necessitate recalibration of the instrument or if the project requests recalibration based on deliverable deficiencies.

c. Performance Criteria

The following criteria are recommended in the establishment of initial calibration mass attenuation curves for gas proportional counters:

i. Each alpha and beta calibraton standard shall be counted to an accumulation of 10,000 counts
ii. Each mass attenuation curve should consis of at least 10 points well distributed throughout the mass range unless instrument specific programs designate otherwise
iii. Operating voltage on the plateau shall be established to achieve a cross talk of α into the β channel of < 10%, and β into the α channel of <1%
iv. The instrument background must be checked at the time of initially calibrating the detector.
v. The matrix residue used in establishment of the mass attenuation curves must be reasonably well matched to the expected sample matrix.
vi. Plated planchets used must be less than 3 years old
vii. The maximum calibration planchet density for alpha and beta counting should be $\leq$ 5 mg/cm^2.
viii. The sources used for the determination of cross talk should be of similar geometry and isotope content to that of the analytical samples; however, when performing analyses for gross measurement, a standard isotope source is acceptable (e.g. ^{241}Am for gross alpha , and ^{137}Cs, ^{90}Sr/^{90}Y for beta calibration).
ix. Standard activity shall be decay corrected (if applicable) prior to calculation of instrument efficiency.

6. Calibration Verification for Gross Alpha and Gross Beta by Gas Proportional Systems

Calibration verification consists of acceptable efficiency and background data

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Matrix residue identity used for check source;
ii. Date of preparation of planchets used in calibration verification;
iii. Number of counts in each channel for each mass-efficiency calibration standard;
iv. Calibration points including mass on planchet and attenuation factor;
v. Tolerance chart or statistical control chart of the the appropriate 20 efficiencies with $\pm 3\sigma$ limits with residue weights for each efficiency verification;
vi. Tolerance chart or statistical control chart of the appropriate 20 instrument backgrounds with $\pm 3\sigma$ limits;
vii. Listing of X/Y coordinates used in constructing the control charts;
viii. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate;
ix. Geometries used in analysis.

b. Frequency

i. Calibration verificationmust occur on a daily basis, or prior to use. The only exception to this specification is when performing low level counts with extended count times precluding the verification of calibration on a dailybasis. In this case, calibration may be verified on a weekly basis.

ii. Both the operating voltage (on the plateau) and the crosstalk shall be checked on a semi-annual basis.

c. Performance Criteria

i. Each alpha and beta verification standard shall be counted to an excess of 10,000 counts

ii. The check source count result for both alpha and beta should be maintained on a control chart with the mean and $\pm 3\sigma$ limits plotted. If the daily check source count result, for either alpha or beta, exceeds the tolerance or statistical 3σ control limit, the checks source must be recounted to verify the measurement was correct. If the recount again exceeds the control limit, the system is considered out of control, no samples shall be run on that system until it is brought back into control. If the recount is in control a third count shall be done and, if in control, analytical sample counting may continue, otherwise no samples shall be run on that system until it is brought back into control. Any samples counted after the last in-control check standard shall be recounted.

iii. Efficiency for both alpha and beta must remain within tolerance or statistical $\pm 3\sigma$ control limits of the appropriate 20 efficiencies.

iv. Instrument background for both alpha and beta must be maintained on a control chart with the mean and $\pm 3\sigma$ limits plotted.

v. Following gas changes, gas should be allowed to flow for a period of time necessary to purge the system; check source and background counts must be performed following a gas change

d. Verification

Verification of completeness of deliverables must be performed prior to validation. If deliverables are found to be inconsistent with the listing in this section, contact the laboratory to request the additional information. If the information can not be obtained, indicate this in the verification report as a non-correctable problem

e. Validation

If the frequency of calibration verification does not meet the frequency specifications, place the appropriate reason code on the affected data. Control charts shall be evaluated for out-of-control conditions. If any of these conditions are met, reference must be made to the laboratory case narrative for justification for analyzing analytical samples under non-compliant conditions; QC samples should be evaluated to determine if the non-compliant check source is indicative of a systemic problem or if it is a chance occurrence. If the occurrence is considered a chance occurrence, and samples do not seem affected, no qualification action is necessary. If the occurrence is considered systemic, and sample results seem to be affected, sample results may be qualified "J" or "R" based on specific conditions in the analytical batch.

If standard source(s) have aged greater than the expiration date on the certificate(s), affected sample results should be qualified "J," and qualified "R" using professional judgement.

7. Initial Calibration for Liquid Scintillation

Initial calibration review consists of acceptable efficiency and background data

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Summary report(s) including dates of calibration, geometries, efficiency, background, quench levels, count times for all analyses, number of counts accumulated for each standard, measured activities for all analyses;
ii. QC source identity;
iii. Matrix used for efficiency curve establishment (e.g. ASTM Type II water);
iv. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate;
v. Calibration points including quench level and measured results;
vi. All raw data supporting initial calibration;
vii. Certificates for standards used in calibration.

b. Frequency

An initial calibration for liquid scintillation systems should occur when out-of-control conditions, indicated from control charting, necessitate recalibration of the instrument or if the project requests recalibration based on deliverable deficiencies.

c. Performance Criteria

i. The quench curve standard's vial characteristics (glass or plastic) and volume shall be similar to that of the samples to be counted.

ii. The efficiency standard shall be counted prior to each analytical batch and shall be counted to a low counting error (less than 1% at 3σ, or 100,000 counts) at least twice. The average of the efficiencies shall be used to calculate the activity of the samples. The standard need not be prepared daily, but shall be replaced if a decrease in efficiency is noted or phase separation is apparent in the cocktail.

iii. The tritiated water solution prepared from the flame-sealed NIST standard reference material (or equivalent) shall be replaced or recalibrated every 6 months with a fresh standard.

iv. For those labs using the internal standard method of quench correction, the tritium standard used to spike the samples shall be recalibrated or replaced on an annual basis.

v. For those labs using an externalquench monitor and a variable quench protocol, a minimum of ten standards, with quench's well distributed over the appropriate range, shall be used to

determine the quench curve. Each standard in the quench set will be counted to accumulate a minimum of 100,000 counts.

8. Calibration Verification for Liquid Scintillation

Calibration verification consists of acceptable efficiency and background data.

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Matrix used for check source (e.g. ASTM Type II water);
ii. Calibration point(s) including quench level, number of counts for each point, and measured result(s);
iii. Tolerance chart or statistical control chart of the appropriate 20 efficiencies with $\pm\sigma$ limits
iv. Tolerance chart or statistical control chart of the appropriate 20 instrument backgrounds with $\pm 3\sigma$ limits;
v. Listing of X/Y coordinates used in constructing the control charts;
vi. Geometries used in analysis;
vii. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate.

b. Frequency

Calibration verification must occur on a daily basis, or prior to use. The only exception to this specification is when performing low level counts with extended count times precluding the verification of calibration on a daily basis. In this case, calibration may be verified on a weekly basis.

c. Performance Criteria

i. On each day that samples willbe counted, an unquenched flame-sealed check source vial, shall be counted prior to sample counting. In excess of 10,000 counts of the respective activity shall be accumulated.

ii. On each day that samples will be counted, the background count rate shall be determined by counting a vial free of the analyte(s) of interest. The duration of this background count shall be as long, if not longer, than thatfor the analytical samples. This determination shall be made separate from any vials that are counted to meet method blank requirements of the respective statement of work.

iii. For those labs that quench correct the background activity, each day that samples will be counted, a vial of the quenched background shall be counted to ensure control of the instrument background. The quench level shall lie within the tolerance limits of the set of 20 used in establishing initial calibration.

iv. For tritium analyses, the laboratory shall show that thewater used for the background, to be free of tritium activity (by comparison to an EPA blank water or through other means).

v. If the daily check sourcecount result or the background count rate result exceeds the 3 sigma limit, recount to verify the out of control condition. If the recount again exceeds the control limit, the system is out of control, no samples shall berun on that system until it is brought back into control. If the recount is in control a third count shall be done and if in control analytical sample counting may continue, otherwise no samples shall be analyzed on that system until it is brought intocontrol. Any samples analyzed after the last in-control check standard shall be reanalyzed.

d. Verification

Verification of completeness of deliverables must be performed prior to validation. If deliverables are foundto be inconsistent with the listing in this section, contact the laboratory to request the additional information. If the information can not be obtained, indicate this in the verification report.

e. Validation

If the frequency of calibration verification does not meet the frequency specifications, place the appropriate reason code on the affected data. Control charts shall be evaluated for of out-of-control conditions. If any of these conditions are met, reference must be made to the laboratory case narrative for justification for analyzing analytical samples under non-compliant conditions; QC samples should be evaluated to determine if the non-compliant check source is indicative of a systemic problem or if it is a chance occurrence. If the occurrence is considered a chance occurrence, and samples do not seem affected, no qualification action is necessary. If the occurrence is considered systemic, and sample results seem to be affected, sample results may be qualified "J" or "R" based on specific conditions in the analytical batch.

If standard source(s) have aged greater than the expiration date on the certificate(s), affected sample results should be qualified "J," and qualified "R" using professional judgement.

9. Initial Calibration for Alpha Spectrometry

Special note for alpha spectrometry calibration. Where a method or Statement of Work do not require the determinationof tracer recovery, the determination of detector counting efficiency is not necessary. In this circumstance, the following specifications for efficiency calibration are not applicable.

Initial calibration review consists of acceptable efficiency, background, and peak resolution data.

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Summary report(s) including dates of calibration,efficiency, background, peak(s) resolution, count times for all analyses, number of counts accumulated in each channel for each standard, measured activities for all analyses;
ii. Matrix used for efficiency establishment (e.g. ASTM Type II water);
iii. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate;

iv. Calibration points including efficiency, energy, and peak resolution for relevant peaks;
v. All raw data supporting initial calibration;
vi. Certificates for standards used in calibration;

b. Frequency

An initial calibration for alpha spectrometry systems should occur when out-of-control conditions, indicated from control charting, necessitate recalibration of the instrument or if the project requests recalibration based on deliverable deficiencies.

c. Performance Criteria

i. A single check source may be used for efficiency calibrations, and should consist of ^{241}Am, ^{238}Pu, ^{239}Pu, or ^{244}Cm. A running average of up to 5 efficiencies for one or more of these radionuclides may be used as the efficiency for sample calculations.

ii. At least two, preferably three, isotopesshall be used for energy calibrations. The isotopes used should be from those listed above.

iii. Radionuclides should be selected which have energies that will limit the detection of attenuated alpha events of higherenergies. The sources should be of a high enough quality as to limit the amount of tailing created by attenuation. Peaks outside the ROI need not be identified.

10. Calibration Verification for Alpha Spectrometry

Calibration verification consists of acceptable efficiency, background, and peak resolution data.

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Tolerance chart or statistical control chart of the appropriate 20 efficiencies, peak energies or channel numbers with $\pm 3\sigma$ limits;
ii. Background results;
iii. Resolution demonstration of relevant peak(s);
iv. Listing of peak energies;
v. Listing of X/Y coordinates used in constructing the control charts;
vi. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate;
vii. Geometries used in analysis.

b. Frequency

Calibration verification will be performed weekly prior to analytical sample counting, unless analytical conditions warrant more frequent verification.

The background count rate shall be determined at least once per month by counting a clean mount of the same geometry to sample mounts. The duration of this background count shall be at least 12 hours.

c. Performance Criteria

i. The check source shall be counted a sufficient period of time to accumulate an excess of 2,000 counts in each region of interest. This check source count will be used to monitor counting efficiency, peak energy, and peak resolution. The region of interest of a minimum of one isotope from the following (^{241}Am, ^{238}Pu, ^{239}Pu, or ^{244}Cm – ^{234}U and ^{238}U are also recommended) shall be used to monitor these performance parameters.

ii. If the daily check source count result exceeds the 3 sigma limit, recount to verify the out of control condition. If the recount again exceeds the control limit, the system is out of control, no samples shall be run on that system until it is brought back into control. If the recount is in control a third count shall be done and if in control analytical sample counting may continue, otherwise no samples shall be analyzed on that system until it is brought into control. Any samples analyzed after the last in-control check standard shall be reanalyzed.

iii. The isotope(s) radionuclides/isotopes chosen for evaluation of peak resolution should be the same as used in the initial calibration. The resolution of the detector shall be sufficient to minimize the tailing of counts from peaks of higher energy into regions of interest (ROI) of lower energy peaks.

iv. The background count rate of a detector used for alpha spectrometry will naturally increase in those regions of interest corresponding to radionuclides present in the counted samples. This background count rate is corrected for in the result calculations and thus has little impact on the quality of the reported data. However, it is possible that over time this background count rate could increase to a level where the RDL of some analytes are impacted, unless the lab has corrected for this with longer counting times. If this problem should be observed, the corrective action of cleaning or replacing the detector should be discussed with the laboratory.

d. Verification

Verification of completeness of deliverables must be performed prior to validation. If deliverables are found to be inconsistent with the listing in this section, contact the laboratory to request the additional information. If the information can not be obtained, indicate this in the verification report.

e. Validation

If the frequency of calibration verification does not meet the frequency specifications, place the appropriate reason code on the affected data. Control charts shall be evaluated for of out-of-control conditions. If any of these conditions are met, reference must be made to the laboratory case narrative for justification for analyzing analytical samples under non-compliant conditions; QC samples should be evaluated to determine if the non-compliant check source is indicative of a systemic problem or if it is a chance occurrence. If the occurrence is considered a chance occurrence, and samples do not seem affected, no qualification action is necessary. If the occurrence is considered systemic, and sample

results seem to be affected, sample results may be qualified "J" or "R" based on specific conditions in the analytical batch.

If standard source(s) have aged greater than the expiration date on the certificate(s), affected sample results should be qualified "J," and qualified "R" using professional judgement.

11. Initial Calibration for Gamma Spectrometry

Initial calibration review consists of acceptable peak energy, efficiency, background and peak resolution data

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Summary report(s) including dates of calibration (energy, efficiency, background, peak(s) resolution), geometry, count times for all analyses, number of counts accumulated in each channel for each standard, measured activities for all analyses;
ii. Matrix used in the geometry standard (e.g. epoxy);
iii. Density of standard;
iii. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate;
iv. Calibration points including efficiency, energy, and peak resolution for relevant peaks;
v. All raw data supporting initial calibration;
vi. Certificates for standards used in calibration.

b. Frequency

An initial calibration for gamma spectrometry systems should occur when out-of-control conditions, indicated from control charting, necessitate recalibration of the instrument or if the project requests recalibration based on deliverable deficiencies. An initial energy/efficiency calibration for each geometry in conjunction with the preparation of a tolerance or QC chart shall be performed, thus linking the calibration to the tolerance/QC chart.

The energy/efficiency calibration of gamma spectrometers shall be performed at least semi-annually. The energy/efficiency calibration standard shall have at least three gamma emitting energies. If there are only 3 energies in the calibration standard, then the difference between the energies shall be at least 500 keV with one energy being less than 300 keV. Energy calibration photopeaks shall have 1332 keV (cobalt-60) Full Width at Half Maximum (FWHM) values of less than 4 keV for HPGe and Ge(Li) detectors. Where NaI detectors are used for an analysis, as permitted by the method/SOW, the FWHM shall be less than 1.5 times the instrument manufacturer's stated specification for FWHM.

c. Performance Criteria

i. Where the method is not specific to a single radionuclid using a single energy (e.g. cesium-137 using only 661 keV), the calibration source shall have several emissions over an energy range of about 0 - 2000 keV.

The source shall not be used for calibration for more than 4 half-lives beyond the calibration date indicated on the certificate of calibration.

ii. The calibration source shall be counted to accumulation of an excess of 20,000 counts in each region of interest.

iii. The energy calibration standard shall have at least three gamma emitting energies. The difference between the energies shall be at least 500 keV with one energy being less than 300 keV. Energy calibrationphotopeaks shall have Full Width at Half Maximum (FWHM) values of less than 4 keV.

12. Calibration Verification for Gamma Spectrometry

Calibration verification consistsof acceptable peak energy, efficiency, background and peak resolution data.

a. Deliverables

The basic deliverables required to complete the checks are described in this section:

i. Tolerance chart or statistical control chart of the appropriate 20 efficiencies with $\pm\sigma$ limits
ii. Tolerance chart or statistical control chartof the appropriate 20 relevant peak energies with $\pm 3\sigma$ limits;
iii. Resolution demonstration of relevant peak(s);
iv. Listing of X/Y coordinates used in constructing the control charts;
v. Evidence of decay correction of standards prior to calculation of efficiencies, if appropriate;
vi. Geometries used in analysis.

b. Frequency

i. Energy, efficiency and resolutioncalibration verification must occur on a daily basis or prior to use. The only exception to this specification is when performing low level counts with extended count times precluding the verification of calibration on a daily basis. In this case, calibration may be verified on a weekly basis.

ii. The background count rate for the representative geometries shall be determined at least once per week. Two alternatives should be consideredin counting the background rate: by counting each representative geometry filled to the respective volume with distilled or deionized water, or counting without a representative geometry, an empty cave. The duration of the background count shall be as long, if not longer, as that of the respective samples.

c. Performance Criteria

i. The check source used to verify energy calibration should include radionuclides with energies covering the expected energy range of the radionuclides of interest. It is recommended that low, mid, and highenergy radionuclides be included for verification of energy, efficiency, and peak resolution.

ii. Energies of the low, mid and high energy radionuclides should fall within ± 0.2keV of the initial calibration energies.

iii. The check source shall be counted to accumulation of an excess of 20,000 counts in the low, mid, and high energy ranges of interest. Examples of checksources which will cover common radionuclide energy ranges are listed in the previous section.

These emissions shall not be used for calibration verification for more than 4 half-lives beyond the calibration date indicated on the certificate of calibration.

iv. If the daily check source count result for counting efficiency at a low, mid, and high points exceeds the tolerance chart or statistical 3σ control limit, recount to verify the out of control condition. If the recount again exceeds the control limit, the system is out of control, no samples shall be run on that system until it is brought back into control. If the recount is in control a third count shall be done and if in control sample counting may continue, otherwise no samples shall be run on that system until it is brought back into control. Any samples counted after the last in-control check standard shall be recounted.

v. Peak resolution from the low, mid, and high energy ranges should be < ± 1 FWHM from the respective peaks in the initial calibration.

d. Verification

Verification of completeness of deliverables must be performed prior to validation. If deliverables are found to be inconsistent with the listing in this section, contact the laboratory to request the additional information. If the information can not be obtained, indicate this in the verification report.

e. Validation

If the frequency of calibration verification does not meet the frequency specifications, place the appropriate reason code on the affected data. Control charts shall be evaluated for of out-of-control conditions. If any of these conditions are met, reference must be made to the laboratory case narrative for justification for analyzing analytical samples under non-compliant conditions; QC samples should be evaluated to determine if the non-compliant check source is indicative of a systemic problem or if it is a chance occurrence. If the occurrence is considered a chance occurrence, and samples do not seem affected, no qualification action is necessary. If the occurrence is considered systemic, and sample results seem to be affected, sample results may be qualified "J" or "R" based on specific conditions in the analytical batch.

If standard source(s) have aged greater than the expiration date on the certificate(s), affected sample results should be qualified "J," and qualified "R" using professional judgement.

E. Quality-indicator samples

quality-indicator samples are evaluated during data validation to determine the control of the analytical method, and under what conditions the usability of data has been affected.

The strategy by which quality-indicator samples are evaluated involves an evaluation of whether the difference between expected and measured results is statistically significant when compared to their Total Propagated Uncertainties (TPU). The mathematical relationships presented in the following sections are compared to a factor corresponding to a statisticalevel of confidence. When the relationship exceeds the factor, the two results differ at that statistical level of confidence whenompared to their TPU.

The statistical assumption inherent in these tests is that sample results are drawn from normally-distributed populations with estimated means and known variances.Factors in the TPU relationship may originate from populations which are not necessarily normally distributed (e.gcounting uncertainty). However, use of sample results and TPU, assuming approximation to the normal distribution, provides a reasonable and appropriate approach to evaluating control of analytical conditions. Presented in this procedure are statistical decision-making levels at 95% and 99% levels of confidence (decision-making factors are 1.96 and 2.58, respectively). Projects may choose other levels of confidence and decision-making factors based on project DQOs, with the realization that qualification decisions made through validation will be at differing levels of confidence and conservatism. The following table provides examples of these decision-making factors which are applied as decision-making tools through this procedure:

Table 5.4 Examples of Confidence Levels for Qualification Decision-Making

Confidence Level, %	Decision-making factor	Decision-making level
50	0.68	more conservative
67	1.00	
75	1.15	
90	1.645	
95	1.960	
99	2.575	less conservative

adapted from Taylor, J.K. Quality Assurance of Chemical Measurements. Lewis Publishers, 1987.

Listed in this section are guidance for qualification for single quality-indicator samples being outside control criteria based on a 95% and 99% level of confidence. Analytical samples should not be rejected

based on a singular quality control sample. Effects of other QC sample deficiencies must be considered to evaluate whether conditions are such to justify rejection of data. Appendix B provides analytical decision-making guidance for situations where multiple quality deficiencies are encountered. A strategy to incorporate into laboratory SOWs is to require corrective action if the $\pm 2.58\sigma$ limits are exceeded. If the limits are exceeded again, data may be reported, but are subject to qualification through validation. The laboratory case narrative should reflect the corrective action.

The tests in this section are meaningful only if the radioanalytical method functions properly. If a method is deemed seriously out of control, the tests in this section are not appropriate, and no further validation needs to be done; all results may be considered unusable.

1. Total Propagated Uncertainty (TPU)

The tests presented in this section rely heavily upon evaluation of uncertainty associated with radioanalytical results. The factors presented in the TPU relationship in appendix D must be communicated to the laboratory prior to sample analysis in order for the laboratory to provide the relevant components of the TPU. The random factors in the TPU relationship include counting uncertainty, and net countrate uncertainty; the remaining terms comprise the systematic factors. Many laboratories choose to report uncertainties separately as total random and total systematic. These factors are acceptable to use in the tests in this section providing that the components of the uncertainties are recognized.

In the event that not all the requested uncertainties are available, the magnitude of TPU must be evaluated considering which components are the dominant factors in the relationship. At relatively low count rates, the random components will likely be the dominant factors; and at high rates, systematic components may be dominant. Communication with the laboratory in the evaluation of TPU in absence of all components is crucial in evaluating the tests presented in this section.

2. Standard Traceability

Standards used in the preparation of quality control samples (laboratory control samples, matrix spikes) or sample-specific spikes (tracers or carriers) shall be shown to be traceable to a reliable source (e.g. NIST, IAEA).

a. Verification

Verification of the identity of the standard used in quality control sample preparation, or sample-specific spiking shall be performed by tracing the standard control number from the certificate to the quality control sample preparation documentation.

b. Validation

If a standard is determined to be not traceable, a qualifier is not required, but the reason code "E02" must be placed next to sample results that are in the same preparation batch with the affected QC sample.

3. Laboratory Control Sample (LCS)

The purpose of an LCS is to monitor the accuracy of the preparation and analysis of the analytical samples, provided that LCS is fully homogenized prior to preparation and analysis. The LCS must be the same matrix type as the analytical samples (e.g., water, soil). Exceptions should be made in cases of novel matrices (e.g. sludge, oil, biota). The spike in the LCS should be of a level near that of the analytical samples, or contractually specified at a predetermined level (e.g. 20 times the MDC may be appropriate).

Three types of LCS material have been identified as being appropriate to evaluate laboratory process accuracy:

- Traceable Reference Material (TRM)
- Standard Reference Material (SRM)
- Well Characterized Reference Material (WCRM)

a. Deliverables

The basic deliverables necessary to complete the checks are described in this section:

- Traceability certificate(s) for TRM and/or SRM with uncertainty associated with the standard.
- Measured result of LCS expressed in activity/unit weight or volume.
- TPU for LCS expressed in activity/unit weight or volume.
- MDC of LCS expressed in activity/unit weight or volume.
- Expected result of LCS expressed in activity/unit weight or volume.
- If samples within a reporting batch originate from separate preparation batches, validation requires that laboratory control sample results be included from all appropriate preparation batches.

b. Frequency

The preparation of the LCS occurs in the laboratory simultaneously with the preparation batch of analytical samples. The LCS must be analyzed on the same detection system as the samples with which it was prepared and using the same analysis conditions as with the samples. The MDC of the LCS must be less than the specified RDL. The LCS should be prepared at a frequency of one per batch of up to 20 analytical samples.

c. Verification

Verify that one LCS was prepared at the same time with the analytical samples, is of the same matrix, and has been analyzed with an analytical batch of up to 20 analytical samples. If these criteria are not met, state as a non-correctable problem in the verification report.

d. Validation

If LCS data are not reported for a sample(s), place the reason code "L05" on the sample results for the affected preparation batch.

LCS - Test for bias

The normalized difference between the measured LCS and expected LCS results given by the following relationship is used in testing the null hypothesis that the measured and true results of the LCS do not differ significantly when compared to their respective TPU..

$$\frac{LCS_{meas.} - LCS_{exp.}}{\sqrt{TPU^2_{meas.} + TPU^2_{exp.}}}$$

$LCS_{meas.}$ = Measured LCS result
$LCS_{exp.}$ = Expected result of LCS
$TPU_{meas.}$ = Total Propagated Uncertainty of measured result
$TPU_{exp.}$ = Total Propagated Uncertainty of expected result

If the normalized difference is between 1.96 and 2.58, or between -1.96 and -2.58, qualify sample results ≥ MDC "J," as the conclusion is reached that the spiked and expected results differ at the 5% level of significance. Qualify results < MDC "J" only if the normalized difference shows a negative bias. If the normalized difference is greater than 2.58 or less than -2.58, consider the effects of deficiencies in other quality-indicator samples prior to qualifying sample results "R," as at the 1% level of significance, the conclusion is reached that the spiked and expected results are different enough to indicate a significant positive or negative bias. If multiple quality deficiencies are encountered, qualify using the guidance provided in Appendix B.

4. Laboratory Duplicate

The purpose of a laboratory duplicate is to monitor the precision of the analytical method, provided the sample is fully homogenized prior to preparation and analysis. The laboratory duplicate is a randomly chosen split of an analytical sample into two aliquots prior to sample preparation. To provide for relevancy of laboratory duplicate data, the sample chosen should have measurable activity (i.e. > MDC and 2σ counting uncertainty); however, the test provided in this section may be performed on results < MDC or 2σ counting uncertainty. In analytical methods where no sample preparation or separation is performed (e.g. gamma spectrometry), a sample recount may be performed in lieu of a laboratory duplicate, although qualification under these conditions should be based on instrumental performance, as most gamma spectrometry entails minimal sample preparation.

a. Deliverables

The basic deliverables necessary to complete the checks are described in this section:

- Measured result of sample expressed in activity/unit weight or volume.
- Measured result of duplicate expressed in activity/unit weight or volume.
- MDCs of sample and duplicate expressed in activity/unit weight or volume.
- TPU of sample and duplicate expressed in activity/unit weight or volume.

- If samples within a reporting batch originate from separate preparation batches, validation requires that laboratory duplicate results be included from all appropriate preparation batches.

b. Frequency

The preparation of the laboratory duplicate occurs in the laboratory simultaneously with the preparation batch of analytical samples. Analyses of the laboratory duplicate must proceed using the same analysis conditions used with the samples. The laboratory duplicate should be prepared at a frequency of one per preparation batch of up to 20 analytical samples.

c. Verification

Verify that one laboratory duplicate was prepared at the same time with the analytical samples, is of the same matrix, and has been analyzed with a preparation batch of up to 20 analytical samples. If there criteria are not met, state this as a non-correctable problem in the verification report.

d. Validation

If laboratory duplicate data are not reported for a sample(s), place the reason code "D04" on the sample results for the affected preparation batch.

Duplicate - Statistical test for difference between sample and duplicate

The normalized absolute difference between the sample and laboratory duplicate, given by the relationship below is used in testing the null hypothesis that the results do not differ significantly when compared to their respective TPU.

$$\frac{S - D}{\sqrt{TPU_S^2 + TPU_D^2}}$$

S	=	Sample result
D	=	Laboratory duplicate result
TPU_s	=	Total Propagated Uncertainty of the sample
TPU_D	=	Total Propagated Uncertainty of the duplicate

If the sample or laboratory duplicate results are less than their respective MDC, the results may still be used in this relationship to determine precision.

If the normalized absolute difference is greater than 1.96, qualify the affected sample results "J," as at the 5% level of significance, the conclusion is reached that the sample and laboratory duplicate differ. If multiple quality deficiencies are encountered, qualify using the guidance provided in Appendix B.

5. Matrix Spike (MS)

The purpose of a matrix spike is to measure the effect of interferences from the sample matrix that will preclude accurate quantitation by the instrumentation, provided that samples are fully homogenized prior to preparation and analysis. The matrix spike is a split of a field-originating analytical sample in which one half of the split is spiked with a known amount of radionuclide of interest prior to sample preparation.

Due to difficulties in homogenization of solid samples for gamma analyses, a matrix spike may not present useful information. Matrix spikes may present useful information for aqueous samples; however, should not be used for energies <100KeV.

Matrix spikes may not be required for methods where a carrier or tracer is used, provided that the tracer chosen is chemically similar to the radionuclide of interest. Matrix effects will be detected through tracer recovery; however, difficulty may be experienced in ascertaining that poor recovery is due to matrix effect or through losses in separation.

a. Deliverables

The basic deliverables necessary to complete the checks are described in this section:

- Traceability certificate(s) for standard with uncertainty associated with the standard.
- Measured result of the unspiked sample in activity/unitweight or volume.
- Measured result of MS expressed in activity/unitweight or volume or volume
- Expected result of the MS expressed in activity/unitweight or volume.
- MDCs of unspiked sample and MS expressed in activity/unitweight or volume.
- TPU of unspiked sample, and MS expressed in activity/unitweight or volume.
- If samples within a reporting batch originate from separate preparation batches, validation requires that matrix spike results be included from all preparation batches.

b. Frequency

The preparation of the matrix spike occurs in the laboratory simultaneously with the preparation batch of analytical samples. If notably different matrices are present within the same preparation batch, matrix spikes should be prepared for each matrix. Analyses of the matrix spike must proceed using the same analysis conditions used with the samples. The matrix spike should be prepared at a frequency of one per preparation batch of up to 20 analytical samples.

c. Verification

Verify that one matrix spike was prepared at the same time with the analytical samples, was of the same matrix as the analytical samples, and has been analyzed with a preparation batch of up to 20 analytical samples. If this frequency is not met, state this as a non-correctable problem.

d. Validation

If matrix spike data are not reported for a sample(s), place the reason code "M05" on the sample results for the affected preparation batch.

Matrix Spike - Test for matrix-induced bias

The normalized difference between the spike result (SSR-SR) and expected value (ER) of the MS given by the following equation is used in testing the null hypothesis that the expected and measured results of the MS do not differ significantly when compared to their respective TPU.

$$\frac{(SSR - SR) - ER}{\sqrt{TPU_{SSR}^2 + TPU_{SR}^2 + TPU_{ER}^2}}$$

SSR	=	Spiked Sample Result
SR	=	Sample Result (unspiked)
ER	=	Expected Result (spike amount)
TPU	=	Total Propagated Uncertainty

If the normalized difference is between 1.96 and 2.58, or between -1.96 and -2.58, qualify sample results ≥ MDC "J," as the conclusion is reached that the spiked and expected results differ at the 5% level of significance. Qualify sample results < MDC "UJ" only if the normalized difference shows a negative bias. If the normalized difference is greater than 2.58 or less than -2.58, consider the effects of deficiencies in other quality-indicator samples prior to qualifying sample results "R," as at the level of significance of 1%, the conclusion is reached that the spiked and expected results differ to a point indicative of a significant positive or negative matrix-induced bias.,

Consideration should be given to the similarity in matrix type among samples in the preparation batch batches. If the matrices differ notably (particularly in soil particle size) qualification may be placed on only the sample associated with the matrix spike. If matrices do not differ notably, qualification may be placed on all samples in the preparation batch. If multiple quality deficiencies are encountered, qualify using the guidance provided in Appendix B.

6. Method Blank

The purpose of a method blank is to monitor the presence of contamination of the analyte of interest in the sample preparation and analysis processes. The method blank is a laboratory-generated sample of the same matrix as the analytical samples but in absence of the analyte of interest.

a. Deliverables

The basic deliverables necessary to complete the checks are described in this section:

- Measured result of method blank expressed in activity/unit weight or volume.
- MDC of method blank expressed in activity/unit weight or volume.
- TPU of method blank expressed in activity/unit weight or volume.

- If samples within a reporting batch originate from separate preparation batches, validation requires that method blank results be included from all preparation batches.

b. Frequency

The preparation of the method blank occurs in the laboratory simultaneously with the preparation batch of analytical samples. Analyses of the method blank must proceed using the same analysis conditions used with the samples. The method blank should be prepared at a frequency of one per batch of up to 20 analytical samples.

c. Verification

Verify that all method blank activities are less than their MDC and 2s counting uncertainty. If method blank activity is greater than the MDC or 2s counting uncertainty, note this in the verification report.

Verify that one method blank was prepared at the same time with the analytical samples, is of the same matrix, and has been analyzed at a frequency of one per preparation batch of up to 20 analytical samples. If these criteria are not met, state this as a non-correctable problem in the verification report.

d. Validation

If method blank data are not reported for a sample(s), place the reason code "B06" on the sample results for the affected preparation batch.

If either of the following conditions are met, no actions outlined in this section need to be performed:

- method blank result is less than its MDC or less than its 2s counting uncertainty

- method blank result is greater than its MDC with the sample result less than its MDC.

Validation of the method blank tests whether blank contamination is of a level which may be indicative of systematic contamination through sample preparation.

If both the method blank and sample activity are greater than their respective MDC or 2s counting uncertainty, the following test shall be used in determining the effect of possible blank contamination on sample results.

The normalized absolute difference between the method blank and a sample result, given by the relationship below, is used in testing the null hypothesis that the sample and the method blank do not differ significantly when compared to their respective TPU.. This test may be used as long as the method blank is reported in terms of activity per unit weight or volume consistent with the sample results.

$$\frac{|S - B|}{\sqrt{TPU_S^2 + TPU_B^2}}$$

S	=	Sample result
B	=	Method blank result
TPU	=	Total Propagated Uncertainty

If the normalized absolute difference is greater than 2.58, no qualification is necessary, as at the 1% level of significance, the conclusion is reached that the method blank and sample differ significantly. If the normalized absolute difference is between 1.96 and 2.58, qualify sample results ≥ MDC "J," the sample and method blank differ at the 5 % level of significance (sample results < MDC do not require qualification). If the normalized absolute difference is between 0 and 1.96, consider the effects of deficiencies in other quality-indicator samples prior to qualifying sample results "R," the conclusion is reached that the method blank and sample results differ at the 1% level of significance. If multiple quality deficiencies are encountered, qualify using the guidance provided in Appendix B.

F. Chemical Yield - Tracers and Carriers

Tracers and carriers are used in radiochemical separations methods to provide evaluation of chemical separation. Chemical yield is evaluated through the recovery of chemical species spiked into samples. Yield is evaluated radiometrically with a tracer and gravimetrically with a carrier. Each sample is spiked with either a carrier or tracer, and sample results are adjusted for yields greater or less than 100%.

Generally, a low yield is indicative of losses of tracer and radionuclide of interest through sample separation, and recoveries greater than expected (> 100%) are indicative of instrumental problems or contamination, as carriers fortified into samples are not expected to be recovered at levels greater than spiked.

1. Verification

Verify that for applicable analyses, one carrier or tracer recovery is reported for each sample. If a carrier or tracer percent recovery is not reported for each sample, contact the laboratory for submittal of this data. If the data can not be provided, state this as a non-correctable problem in the verification report.

As yield decreases, the MDC may elevate to a point at which the RDL is exceeded, and analytical results are contractually noncompliant. If the laboratory has not initiated corrective action, for samples in which the MDC exceeds the RDL, the project may choose to contact the laboratory for sample rework. If rework is not feasible, indicate the noncompliant data in the verification report.

2. Validation

If yield is not reported for a sample(s), place the reason code "Y04" on the sample result.

Yield is validated based on percent recovery of the spiked nuclide. Low yield may be indicative of increased uncertainty in the sample result. Criteria for qualification should be based on what magnitude of correction has been applied to the sample result (e.g. 20% recovery implies a sample result correction of 5), although a point of debate exists concerning useability of radionuclide data with yields near 0%.

Yield criteria may also be established from existing sample yield data from previous sampling at the site, if this data are available.

Sample results should not be qualified based on yield alone. Sample yield should be evaluated in reference to chemical yield of quality-indicator samples. If yield is generally low throughout the preparation batch, but recoveries of target radionuclides in the LCS are acceptable, data may be accepted without qualification;however, if quality control sample yield is generally low, sample results with low yield may need qualification.

G. Required Detection Limits (RDL)

1. Verification

For all samples, the MDC must be less than the contractual RDL. RDLs are identified and communicatedto the laboratory in the laboratory SOW. If the MDC is greater than the RDL, this may indicateuse of a small sample size, inadequate count times, or matrix problems. All sample results shall be reviewed to determine RDL compliance. In cases where the MDC is greater than the RDL, the verifier should consult the laboratory case narrative to evaluate the reason for the noncompliance; the project may request a reanalysis.

H. Nuclide Identification and Quantification

1. Test for Detection

a. Validation

The test for detection of a radionuclide includes two distinct steps.

i. The first step is to evaluate whether a sample result is greater than its MDC.

ii. The second step is to evaluate whether the sample result is greater than its 2s counting uncertainty.

If the sample result is not greater than its MDC or less than its 2s counting uncertainty, qualify the sample result "U."

If the sample result is less than its MDC (assuming that the MDC is reported at the 95% confidence level) the data user is accepting the probability of 5% of a false negative result. If the sample result is less than its 2s counting uncertainty, at the 95% confidence level, the radionuclide result is not different from zero.

In placing the "U" qualifier, special attention must be paid to instances where nominal conditions are not followed (i.e. sample aliquot size, count time). Sample results which are less than their MDC, but greater than their 2s counting uncertainty may have been counted for a period of time long enough to be considered detected. Sample results which are greater than the MDC, but less than the 2s counting uncertainty may not have been counted for a period of time long enough to be considered detected.

2. Negative Results

a. Validation

Negative results that have absolute values greater than their 2s counting uncertainty are an indication that the instrument background has shifted. The implication of unstable background is a possible negative bias in the sample result. Qualification due to very negative sample results must take into account evaluation of calibration verification background and quality-indicator samples that may indicate a bias. Qualifiers should be placed using professional judgement when applied due to the occurrence of negative results, and the rationale for placement must be fully described in the validation report.

3. Sample Result Recalculation

The accuracy and consistency of sample result calculation by the laboratory can be addressed through two different techniques. The application of each strategy depends on the laboratory's ability to minimize transcription during reporting, and how familiar the project is with the performance of the laboratory.

a. If sample results are produced primarily through software processing and minimal transcription is performed in the laboratory, the data system(s) can be evaluated during an audit or surveillance by performing two different tests on the software: 1) supply the data system a consistent set of input designed to provide a consistent set of output, and 2) supply the data system a set of nonconforming data to test the error detection routines. An evaluation of the laboratory's software configuration control and security is also necessary. If reporting software has been developed for customized use in the laboratory, additional confidence is gained through the laboratory's adherence to the *IEEE Standard for Software Verification and Validation Plans* 1012-1986 and through maintenance of a software verification and validation plan. Through this technique, a high level of confidence can be gained in the laboratory's reporting techniques and will result in a minimal need for manual recalculation of sample results.

b. If the laboratory has a high rate of manual transcription in generation of sample results, the project may choose to manually recalculate sample results at a determined frequency. If sample results cannot be reproduced through manual calculation, contacting the laboratory may be necessary to resolve the problem. Qualifiers should be placed using professional judgement.

Activity and TPU equations provided in appendix C are useful for providing the basic structure for calculating radioanalytical results. Modifications to the equations may be needed in method-specific cases. All calculations used must be contractually agreed upon between the project and the laboratory prior to sample analysis, and iterated in the laboratory SOW.

I. Instrument-Specific Sample Considerations

1. Sample Analytical Parameters for Gas Proportional Counting

a. Validation

Equations to approximate a parametric relationship between standard weight and activity are detector-specific. They are developed from the mass attenuation curves by the laboratory and are applied to calculate sample-specific efficiencies. If recalculating sample results, efficiencies used must be sample-specific.

A representative sample aliquot must be chosen to ensure the dissolved solid content of the sample falls within the mass range of the appropriate curve. Sample results for which aliquot weight is outside the attenuation curve should be qualified "J" if not reanalyzed with a smaller aliquot.

2. Spectral Interpretation (alpha and gamma spectrometry)

a. Gamma Spectrometry

Identification of the radionuclides associated with the measured gamma-ray energies and intensities is largely performed by gamma-ray spectral-analysis algorithms. These algorithms automatically locate peaks in spectra by applying a user-definable set of criteria. Based upon this set of criteria, the peaks in a spectrum are distinguished from the Compton edges, backscatter peaks, and the numerous features created by the background radiation and detection process. The algorithms automatically calculate the peak areas and correct them for the system's detection efficiency as a function of gamma energy. The identification of the radionuclides that produced the peaks is accomplished by comparison to the information in a customizable radionuclide analysis library. For all peaks identified in a spectrum (that correspond to a radionuclide in the analysis library), the algorithms calculate the activities of the identified radionuclides in the sample. However, the use of peak search, identification, and quantification algorithms does not preclude the need for laboratory data review by an experienced spectroscopist.

The resulting quality of the gamma spectrometry data will largely depend upon the ability of the spectroscopist to establish a set of analysis criteria to be used by the algorithms with perform the location, identification and quantification of features within spectra. The quality of this process is greatly influenced by the spectral library. For this reason, it is recommended that the library originate from a reliable source and software be controlled with respect to changes in identification criteria. Multiple libraries are often constructed based upon expected interferences and the level of activity in the samples. The multiple libraries reduce the number of unidentified peaks due to summation events and interferences within the sample matrix. The goal of reducing the number of unidentified peaks within a spectrum, while not compromising the analysis routines by the inclusion of extremely low abundance branching probabilities, helps limit the uncertainty of detection of those radionuclides which emit more than one photon.

The data package should include both identified and unidentified peaks. This will support the evaluation of instrumental system control.

b. Evaluation of Gamma Spectra

The assurance of radionuclide identification and quantitation by properly functioning algorithms, through reasonable resolution criteria, and by an evaluation by an experienced radiochemist will provide a level

of certainty in the quality of the data. Validation of data produced under these conditions should be minimal, i.e. a thorough review of all spectra including radionuclide identification and recalculation of sample results should not be necessary. A recommendation is to browse sample-specific spectra for changes in energies positions of target radionuclides and for significant peak overlap.

Radiochemical data produced under substandard or unknown conditions for identification and quantification warrant a closer inspection. Recommended criteria for evaluating gamma spectra in relation to radionuclide identification and resolution are as follows:

i. Where more than one isotope of a single Naturally Occurring Radioactive Material (NORM) series are reported, reported results for that series should demonstrate secular equilibrium.

ii. For soil samples, two peaks that are almost always observed are the 511 keV annihilation peak and the 1460 keV peak of ^{40}K. The appearance of these peaks at the respective energy, and the respective peak shape should be checked.

iii. If ^{40}K is quantitated in the analysis, the reported value should be checked against the activity expected in site's soil (if those data are available).

iv. For isotopes identified and/or quantitated with two or more gamma energies (i.e. 1173 and 1332 keV for ^{60}Co) the count rate at each energy can be observed to ensure that the count rate at each respective energy is reasonable to confirm the presence of the isotope.

c. Alpha Spectra

As with gamma spectrometry, the alpha spectrometry can require the use of algorithms for the location, identification, and quantification of peaks created by radionuclides with spectra. While many of the analysis algorithms are the same for alpha spectrometry as gamma spectrometry, there are fundamental differences which effect the validation of data. These differences which are inherent to the mode of the decay, sample preparation techniques, and phenomena associated to the detection and quantification require validation. As with gamma spectrometry, if algorithms and operational criteria are employed by the laboratory, then they should be reviewed by a competent spectroscopist and/or radiochemist.

Alpha spectrometry has limitations due to the alpha particles ability to transfer energy at an extremely high rate. This phenomenon is responsible for alpha particles being significantly attenuated by the sample matter, the mass of the radionuclide itself, and the molecules of air between the detector and the sample. If the mass of the samples is not reduced or the radionuclides are present in appreciable amounts, then likelihood of receiving meaningful data is reduced due to attenuation. Therefore, samples for alpha spectrometry frequently require extensive radiochemical preparations, with the sample being placed in vacuums and in close proximity to the detectors. Typically, these separation/preparation techniques isolate one or two target nuclides, thus reducing the possible attenuation which occurs at high masses.

This separation process presents the possibility that contaminating elements could break through the chemical separation process. If chemical breakthrough were to occur, these interferences could erroneously be incorporated into the quantification of the radioisotope of interest.

Unlike gamma spectrometry, the spectra resulting from properly prepared alpha sources/samples are relatively simple, since the peaks are near Gaussian, the background is very low, and detector efficiency is almost uniform for the 3.0 MeV to 6.5 MeV energy range. Frequently, the use of Range of Interest (ROI) are integrated to determine gross activity which can be adjusted for background and then corrected for chemical recovery, decay, aliquot, and detection efficiency to produce concentration. A ROI peak report, including all information needed to manually recalculate results, is useful to evaluate data, as well as the spectra itself.

d. Evaluation of Alpha Spectra

Alpha spectra is not generated through the application of algorithms; consequently the approach to validation differs from that of gamma spectrometry. The following two tests should be performed on alpha spectral data:

i. target peaks should be in the energy range of interest

ii. peak tailing should not significantly overlap peaks at lower energies.

Professional judgement should be used in qualification of sample data based on spectral interferences or peak misidentification. Communication with the laboratory is essential to obtaining reliable identification/quantitation of suspect radionuclides.

VI. APPENDIXES

Appendix A Qualifiers and Reason Codes
Appendix B Qualification Tables for Multiple Quality Deficiencies
Appendix C Calculations and Equations
Appendix D Common Radionuclides and Analytical Methodologies
Appendix E Recommended Surveillance Considerations for Gamma Algorithms

APPENDIX A

DATA VALIDATION QUALIFIERS AND REASON CODES

Radiological Analytical Data

Reason codes must be used with all qualifiers placed on analytical data.

U Nuclide considered not detected above the reported MDC or 2σ counting uncertainty

J Nuclide identified; the associated numerical value is approximated

UJ Nuclide not detected above the reported MDC or 2σ counting uncertainty and a quality deficiency affects the data making the reported data more uncertain

R Result is not usable for its intended purpose

= "Equals" sign, indicates that no qualifier is necessary

DATA VALIDATION QUALIFICATION CODES

Radiological Analytical Data

Method Blank

B01 Concentration of contaminant in the method blank at a level => the qualification level.
B02 Method blank was not the same matrix as the analytical samples.
B03 Gross contamination exists.
B04 Blanks were not analyzed at appropriate frequency.
B05 Sample not significantly different than radiochemical method blank.
B06 Blank data not reported.
B07 Other (describe in comments)

Calibration

C01 Initial calibration sequence was not followed as appropriate.
C02 Calibration was not performed at the appropriate frequency.
C03 Calibration data not reported
C04 Calibration not performed.
C05 Chemical resolution criteria were not satisfied.
C06 Standard curve was established with fewer than the required number of standards
C07 Instrumental system determined to be out of control

C08 Other (describe in comments)

Laboratory Duplicate

D01 Significant difference between sample and duplicate.
D02 Laboratory duplicate was not analyzed at the appropriate frequency
D03 Laboratory duplicate data not reported
D04 Other (describe in comments)

Evidentiary Concerns

E01 Custody of Sample in Question.
E02 Standard not Traceable.
E03 Other (describe in comments)

General

G01 Professional judgement was used to qualify the data.
G02 Other (describe in comments)

Holding Times

H01 Holding times were exceeded.
H02 Holding times were grossly exceeded.
H03 Samples were not preserved properly.
H04 Other (describe in comments)

Laboratory Control Sample

L01 LCS recovery above upper control limit.
L02 LCS recovery below lower control limit.
L03 LCS was not analyzed at appropriate frequency.
L04 LCS not the same matrix as the analytical samples.
L05 LCS data not reported
L06 Other (describe in comments)

Matrix Spike and MS/MSD

M01 MS recovery above upper control limit.
M02 MS recovery below lower control limit.
M03 MS not analyzed at the appropriate frequency
M04 MS data not reported
M05 Other (describe in comments)

Instrument Performance

P01 High background levels or a shift in the energy calibration were observed
P02 Extraneous peaks were observed.
P03 Loss of resolution was observed.
P04 Peak-tailing or peak splitting that may result in inaccurate quantitation were observed
P05 Instrument performance not analyzed at the appropriate frequency
P06 Other (describe in comments)

Quantitation

Q01 Peak Misidentified
Q02 Target analyte affected by interfering peak.
Q03 Qualitative criteria were not satisfied.
Q04 Cross contamination occurred.
Q05 No raw data were provided to confirm quantitation.
Q06 MDC > RDL
Q07 Inappropriate aliquot sizes were used.
Q08 Sample result < MDC.
Q09 Sample result < 2s uncertainty.
Q10 Negative result.
Q11 Compounds were not adequately resolved.
Q12 Sample geometry different from calibration geometry.
Q13 Sample weight greater than greatest weight on mass attenuation curve.
Q14 Other (describe in comments)

Radiochemical Yield

Y01 Radiochemical tracer yield was above the upper control limit
Y02 Radiochemical tracer yield was below the lower control limit
Y03 Radiochemical tracer yield was zero.
Y04 Radiochemical yield data was not present.
Y05 Other (describe in comments)

APPENDIX B

Mapping for Multiple Quality Deficiencies

These tables represent validation qualification decisions made based on radioanalytical data quality considerations only. Data quality needs (e.g. risk assessment, remediation technologies) must be considered when using the guidance in these tables. For example, quantitative data needs may necessitate that data be rejected due to multiple quality deficiencies, but qualititative data needs may indicate that the same data should only be qualified estimated.

Table table B.1 provides a mapping scenario for qualification guidance. For example, if the Laboratory Control Sample shows a high bias, and chemical yield is also high, choose the letters "A" and "E" and reference Table B.2 for guidance on qualification based on those quality deficiencies. The differing separation lines in table B.1 indicate relationships among the quality indicators QC samples and yield. Double lines indicate an "and" function, in that any combination is possible. Thick lines indicate that a single QC sample can only indicate a bias in one direction.

Table B.1

Lab Control Sample		Matrix Spike		Chemical Yield		Method Blank	Lab Duplicate
High Bias	Low Bias	High Bias	Low Bias	High	Low		
A	B	C	D	E	F	G	H

Table B.2
Qualification Guidance for 2 Simultaneous Quality Deficiencies

Combination	Qualification	
	< MDC	≥ MDC
AC	none	R
AD	UJ	J
AE	none	R
AF	UJ	J
AG	none	J
AH	none	J
BC	UJ	J
BD	R	J
BE	UJ	J
BF	R	J
BG	UJ	J
BH	UJ	J
CE	none	R
CF	UJ	J
CG	none	R
CH	none	R
DE	UJ	J
DF	R	J
DG	UJ	R
DH	UJ	R
EG	none	R
EH	none	J
FG	UJ	J
FH	UJ	J
GH	UJ	J

Table B.3
Qualification Guidance for 3 Simultaneous Quality Deficiencies

Combination	Qualification	
	< MDC	≥ MDC
ACE	none	R
ACF	UJ	R
ACG	none	R
ACH	none	R
ADE	UJ	R
ADF	R	R
ADG	UJ	R
ADH	UJ	R
AEG	none	R
AEH	none	R
AFG	UJ	R
AFH	none	R
BCE	UJ	R
BCF	UJ	R
BCG	UJ	R
BCH	UJ	R
BDE	R	R
BDF	R	R
BDG	R	R
BDH	R	R
BEG	UJ	R
BEH	UJ	R
BFG	R	R
BFH	R	R
CEG	none	R
CEH	none	R
CFG	UJ	R

CFH	UJ	R
DEG	UJ	R
DEH	UJ	R
DFG	R	R
DFH	R	R
EGH	none	R
FGH	UJ	R

Table B.4
Qualification Guidance for 4 Simultaneous Quality Deficiencies

Combination	Qualification	
	< MDC	≥ MDC
ACEG	none	R
ACFG	UJ	R
ACEH	none	R
ACFH	UJ	R
ADEG	UJ	R
ADFG	R	R
ADEH	UJ	R
ADFH	R	R
BCEG	UJ	R
BCFG	R	R
BCEH	UJ	R
BCFH	R	R
BDEG	R	R
BDFG	R	R
BDEH	R	R
BDFH	R	R
CEGH	none	R
CFGH	UJ	R
DEGH	UJ	R
DFGH	R	R

Appendix C

Pages 1-6 Provide Equations For Method Blank Corrected Sample Concentrations

Pages 7-12 Provide Equations For Sample Concentrations Without Blank Subtraction

The equations in this appendix are meant to be used as the basis for developing method-specific applications.

Sample Activity Concentration — (Method Blank Corrected Sample Concentrations)

$$ACT_S = \frac{NCR_S}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

ACT_S = Sample Activity Concentration (pCi/g or pCi/L)
NCR_S = Net Sample Count Rate in cpm
2.22 = Factor for Converting dpm to pCi
EFF = Detector Efficiency (Fraction)
ALI = Sample Aliquot Volume or Mass (g or L)
ABN_S = Abundance Fraction of the Emissions Used for Analyte Identification/Quantification
R = Sample Tracer/Carrier (Chemical) Recovery
λ = Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute λ]
t = Time from Sample Collection to Radionuclide Separation or Mid-point of Count Time (Same units as half-life)
CF = Other Correction Factors as Appropriate (i.e, Ingrowth factor, Self-absorption Factor, etc.)

Net Sample Count Rate (NCR) and σ_{NCR}— (Method Blank Corrected Sample Concentrations)

$$NCR = \left(\frac{C_{GS}}{T_{GS}} - \frac{C_{SB}}{T_{SB}}\right) - \left[\left(\frac{C_{GB}}{T_{GB}} - \frac{C_{BB}}{T_{BB}}\right) * \left(\frac{EFF_{SD}}{EFF_{BD}}\right) * \left(\frac{R_S}{R_B}\right)\right]$$

Where:

C_{GS} = Sample Counts
T_{GS} = Sample CountTime (minutes)
C_{SB} = Background Counts
T_{SB} = Background CountTime (minutes)
C_{GB} = Gross Method Blank Counts
T_{GB} = Gross Method Blank CountTime (minutes)
C_{BB} = Method Blank Background Counts
T_{BB} = Method Blank Background CountTime (minutes)
EFF_{SD} = Efficiency of the Sample Detector
EFF_{BD} = Efficiency of the Method Blank Detector
R_S = Sample Tracer/Carrier Recovery Fraction
R_B = Method Blank Tracer/Carrier Recovery Fraction

$$\sigma^2_{NCR} = \left[\frac{C_{GS}}{T^2_{GS}} + \frac{C_{SB}}{T^2_{SB}}\right] + \left\{\left[\frac{C_{GB}}{T^2_{GB}} + \frac{C_{BB}}{T^2_{BB}}\right] * \left(\frac{EFF_{SD}}{EFF_{BD}}\right)^2 * \left(\frac{R_S}{R_B}\right)^2\right\}$$
$$+ \left\{\left[\left(\frac{C_{GB}}{T_{GB}} - \frac{C_{BB}}{T_{BB}}\right) * \left(\frac{EFF_{SD}}{EFF_{BD}}\right) * \left(\frac{R_S}{R_B}\right)\right]^2 * \left[\left(\frac{\sigma_{EFF_{SD}}}{EFF_{SD}}\right)^2 + \left(\frac{\sigma_{EFF_{BD}}}{EFF_{BD}}\right)^2 + \left(\frac{\sigma_{R_S}}{R_S}\right)^2 + \left(\frac{\sigma_{R_B}}{R_B}\right)^2\right]\right\}$$

$$\sigma_{NCR} = \sqrt{\sigma^2_{NCR}}$$

Calculation of Recovery - Radiometric — (Method Blank Corrected Sample Concentrations)

$$R = \frac{\left(\frac{C_{GT}}{T_{GT}} - \frac{C_{TB}}{T_{TB}}\right)}{EFF * ABN_T * AMT_T} = \frac{NCR_T}{EFF * ABN_T * AMT_T}$$

Where:

R = Tracer Recovery
C_{GT} = Gross Count of Tracer
T_{GT} = Tracer CountTime (minutes)
C_{TB} = Background Count of Tracer [Region of Interest (ROI)]
T_{TB} = Background CountTime (minutes)
EFF = Detector Efficiency Fraction
AMT_T = Amount of Tracer Activity Added (dpm)
NCR_T = Net Count Rate of Tracer (cpm)
ABN_T = Abundance Fraction of the Tracer Emissions used for Quantification of the Tracer

Note: It is assumed that the tracer half-life is long enough to be an insignificant uncertainty contributor. If the tracer has a relatively short half-life, then it must be considered and these equations modified. Likewise, uncertainty in the time is also considered to be an insignificant contributor.

Calculation of RE_R - Radiometric— (Method Blank Corrected Sample Concentrations)

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 + \left(\frac{\sigma_{EFF}}{EFF}\right)^2 + \left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2$$

$$\left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 = \frac{\left(\frac{C_{GT}}{T_{GT}^2} + \frac{C_{TB}}{T_{TB}^2}\right)}{NCR_T^2}$$

$$\left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2 = \left(\frac{\sigma_{STS_T}}{STS_T}\right)^2 + \left(\frac{\sigma_{MASS_T}}{MASS_T}\right)^2 + \left(\frac{\sigma_{VOL_T}}{VOL_T}\right)^2 + \Sigma\left(\frac{\sigma_{DIL_T}}{DIL_T}\right)^2 + \Sigma\left(\frac{\sigma_{ALI_T}}{ALI_T}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

RE_R = Relative Error of the Tracer Recovery
σ_R = Standard Deviation of the Tracer Recovery
σ_{NCR_T} = Standard Deviation of the Tracer's Net Count Rate
σ_{EFF} = Standard Deviation of the Detector Efficiency
σ_{AMT_T} = Standard Deviation of the Amount of Tracer Activity Added
σ_{STS_T} = Standard Deviation of the Amount of Trace Activity Taken for Stock Tracer Solution (provided with certificates received with standards)
σ_{MASS_T} = Standard Deviation of the Mass of Standard Solution Used to Prepare Stock Tracer Solution
σ_{DIL_T} = Standard Deviation of theVolume(s) of the Dilution(s) Made to Prepare the Working Tracer Solution
σ_{VOL_T} = Standard Deviation of the Volume of the Stock Tracer Solution
σ_{ALI_T} = Standard Deviation(s) of the Aliquot(s) of Tracer Solution(s) Diluted to Prepare Working Tracer Solution
C_{GT} = Gross Count of Tracer
T_{GT}^2 = Square of Tracer CountTime (minutes)
C_{TB} = Background Count of Tracer [Area or Region of Interest (ROI)]
T_{TB}^2 = Square of Background CountTime (minutes)
R = Tracer Recovery
NCR_T = Net Count Rate of the Tracer
EFF = Detector Efficiency
AMT_T = Amount of Tracer Activity Added (dpm)
STS_T = Amount of Tracer Activity (dpm) in Stock Tracer Solution
$MASS_T$ = Mass (grams) of Standard Solution Used to Prepare Stock Tracer Solution
VOL_T = Volume of Tracer Solution Added
DIL_T = Volume(s) of Dilution(s) Made to Prepare the Working Tracer Solution
ALI_T = Aliquot(s) of Tracer Solution(s) Taken to Prepare Serial Tracer Solution Dilution(s)

Note: Certificates, such as those from NIST, may give two or even three sigma uncertainty. Only one sigma should be used for STS_T.

Calculation of Recovery and RE_R - Gravimetric — (Method Blank Corrected Sample Concentrations)

$$R = \frac{WT_c}{CONC_{CS} * VOL_{CS}}$$

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{PPT.\ WT.}}{PPT.\ WT.}\right)^2 + \left(\frac{\sigma_{CONC_{CS}}}{CONC_{CS}}\right)^2 + \left(\frac{\sigma_{VOL_{CS}}}{VOL_{CS}}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

R = Carrier Recovery
RE_R = Relative Error in Recovery
WT_c = Weight of Carrier Present in Final Precipitate
$CONC_{cs}$ = Concentration of Carrier Solution
VOL_{cs} = Volume of Carrier Solution Added
$\sigma_{PPT.\ WT.}$ = Standard Deviation in Weight of Precipitate
σ_{CONC} = Standard Deviation in Carrier Concentration
σ_{VOL} = Standard Deviation in Carrier Volume
PPT. WT. = Weight of Final Carrier Precipitate

Counting Uncertainty (CU) at the 95% Confidence Level

$$CU_{2\sigma} = \frac{1.96 * (\sigma_{NCR_S})}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

σ_{NCR_S} = Standard Deviation of the Net Sample Count Rate
EFF = Detector Efficiency
ALI = Sample Aliquot Volume or Mass
R = Sample Tracer/Carrier Recovery
ABN_S = Abundance Fraction of the Emissions Used for Analyte Identification/Quantification
λ = Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute λ]
t = Time from Sample Collection To Radionuclide Separation or Mid-point of CountTime (Same units as half-life)
CF = Other Correction Factors as Appropriate (i.e, Ingrowth factor, Self-absorption Factor, etc.)

Sample Activity Concentration Total Propagated Uncertainty (TPU)—(Method Blank Corrected Sample Concentrations)

$$TPU_{1\sigma} = \sigma_{ACT} = \frac{\sqrt{\sigma_{NCR_S}^{\ 2} + (NCR)^2 * \left(RE_{EFF}^2 + RE_{ALI}^2 + RE_R^2 + \Sigma\ RE_{CF}^2\right)}}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

- EFF = Detector Efficiency
- ALI = Sample Aliquot Volume or Mass
- R = Sample Tracer/Carrier Recovery
- ABN_S = Abundance Fraction of the Emissions Used for Identification/Quantification
- $\sigma^2_{NCR_S}$ = Variance of the Net Sample Count Rate
- NCR = Net Sample Count Rate
- RE^2_{EFF} = Square of the Relative Error of the Efficiency Term
- RE^2_{ALI} = Square of the Relative Error of the Aliquot
- RE^2_R = Square of the Relative Error of the Sample Recovery
- RE^2_{CF} = Square of the Relative Error of Other Correction Factors
- λ = Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute λ]
- t = Time from Sample Collection to Radionuclide Separaiton or Mid-Point of Count Time (Same units as half-life)
- CF = Other Correction Factors as Appropriate (i.e, Ingrowth factor, Self-absorption Factor, etc.)

Sample Activity Concentration — Sample Concentrations <u>Without Blank Subtraction</u>

$$ACT_B = \frac{NBCR_S}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

ACT_B = Sample Activity Concentration Without Method Blank Subtraction
$NBCR_S$ = Net Sample Background-Corrected Count Rate
2.22 = Factor for Converting dpm to pCi
EFF = Detector Efficiency
ALI = Sample Aliquot Volume or Mass
ABN_S = Abundance Fraction of the Emissions Used for Identification/Quantification
R = Sample Tracer/Carrier Recovery
λ = Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute λ]
t = Time from Sample Collection to Radionuclide Separation or Mid-point of Count Time (Same units as half-life)
CF = Other Correction Factors as Appropriate (i.e., Ingrowth factor, Self-absorption Factor, etc.)

Net Sample Count Rate ($NBCR_S$) and σ_{NBCR_S} — Sample Concentrations <u>Without Blank Subtraction</u>

$$NBCR_S = \left(\frac{C_{GS}}{T_{GS}} - \frac{C_{SB}}{T_{SB}} \right)$$

Where:
$NBCR_S$ = Net Background Corrected Count Rate
C_{GS} = Sample Counts
T_{GS} = Sample Count Time (minutes)
C_{SB} = Background Counts
T_{SB} = Background Count Time (minutes)

σ_{NBCR_S}

$$\sigma_{NBCR_S} = \left[\frac{C_{GS}}{T_{GS}^{\ 2}} + \frac{C_{SB}}{T_{SB}^{\ 2}} \right]^{\frac{1}{2}}$$

Calculation of Minimum Detectable Concentration (MDC) - general formula

$$MDC = \frac{4.65 * \sqrt{b/T}}{K} + \frac{2.71}{K * T}$$

Where:

b= background count rate
T =Sample Count Time (minutes)
K = instrument-specific and sample-specific correction factors (e.g. ALI * $e^{-\lambda t}$ * R * EFF_S * ABN_S)

In using the above equation, the background and sample count times are either equivalent, or the background count time is greater than sample count time

Calculation of Recovery and RE_R - Radiometric — Sample Concentrations <u>Without Blank Subtraction</u>

$$R = \frac{\left(\frac{C_{GT}}{T_{GT}} - \frac{C_{TB}}{T_{TB}}\right)}{EFF * ABN_T * AMT_T} = \frac{NCR_T}{EFF * ABN_T * AMT_T}$$

Where:

R = Tracer Recovery
C_{GT} = Gross Count of Tracer
T_{GT} = Tracer CountTime (minutes)
C_{TB} = Background Count of Tracer Region of Interest (ROI)
T_{TB} = Background CountTime (minutes)
EFF = Detector Efficiency
AMT_T = Amount of Tracer Activity Added (dpm)
NCR_T = Net Count Rate of Tracer (cpm)
ABN_T = Abundance Fraction of the Tracer Emissions used for Quantification of the Tracer

RE_R - Radiometric — Sample Concentrations <u>Without Blank Subtraction</u>

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 + \left(\frac{\sigma_{EFF}}{EFF}\right)^2 + \left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2$$

$$\left(\frac{\sigma_{NCR_T}}{NCR_T}\right)^2 = \frac{\left(\frac{C_{GT}}{T_{GT}^2} + \frac{C_{TB}}{T_{TB}^2}\right)}{NCR_T^2}$$

$$\left(\frac{\sigma_{AMT_T}}{AMT_T}\right)^2 = \left(\frac{\sigma_{STS_T}}{STS_T}\right)^2 + \left(\frac{\sigma_{MASS_T}}{MASS_T}\right)^2 + \left(\frac{\sigma_{VOL_T}}{VOL_T}\right)^2 + \Sigma\left(\frac{\sigma_{DIL_T}}{DIL_T}\right)^2 + \Sigma\left(\frac{\sigma_{ALI_T}}{ALI_T}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

RE_R = Relative Error of the Tracer Recovery
σ_R = Standard Deviation of the Tracer Recovery
σ_{NCR_T} = Standard Deviation of the Tracer's Net Count Rate
σ_{EFF} = Standard Deviation of the Detector Efficiency
σ_{AMT_T} = Standard Deviation of the Amount of Tracer Activity Added
σ_{STS_T} = Standard Deviation of the Amount of Trace Activity Taken for Stock Tracer Solution (provided with certificates received with standards)
σ_{MASS_T} = Standard Deviation of the Mass of Standard Solution Used to Prepare Stock Tracer Solution
σ_{DIL_T} = Standard Deviation of theVolume(s) of the Dilution(s) Made to Prepare the Working Tracer Solution
σ_{VOL_T} = Standard Deviation of the Volume of the Stock Tracer Solution
σ_{ALI_T} = Standard Deviation(s) of the Aliquot(s) of Tracer Solution(s)Diluted to Prepare Tracer Working Solution
C_{GT} = Gross Count of Tracer
T_{GT}^2 = Square of Tracer Count Time
C_{TB} = Background Count of Tracer ROI
T_{TB}^2 = Square of Background Count Time
R = Tracer Recovery
NCR_T = Net Count Rate of the Tracer
EFF = Detector Efficiency
AMT_T = Amount of Tracer Activity Added (dpm)
STS_T = Amount of Tracer Activity (dpm) in Stock Tracer Solution
$MASS_T$ = Mass (grams) of Standard Solution Used to Prepare Stock Tracer Solution
VOL_T = Volume of Tracer Solution Added
DIL_T = Volume(s) of Dilution(s) Made to Prepare the Working Tracer Solution
ALI_T = Aliquot(s) of Tracer Solution(s) Taken to Prepare Serial Tracer Solution Dilution(s)

Note: Certificates, such as those from NIST, may give two or even three sigma uncertainty. Only one sigma should be used for σ_{STS_T}.

Calculation of Recovery and RE_R - Gravimetric — Sample Concentrations <u>Without Blank Subtraction</u>

$$R = \frac{WT_c}{CONC_{CS} * VOL_{CS}}$$

$$(RE_R)^2 = \left(\frac{\sigma_R}{R}\right)^2 = \left(\frac{\sigma_{PPT.\ WT.}}{PPT.\ WT.}\right)^2 + \left(\frac{\sigma_{CONC_{CS}}}{CONC_{CS}}\right)^2 + \left(\frac{\sigma_{VOL_{CS}}}{VOL_{CS}}\right)^2$$

$$RE_R = \sqrt{\left(\frac{\sigma_R}{R}\right)^2}$$

Where:

R = Carrier Recovery
RE_R = Relative Error in Recovery
WT_C = Weight of Carrier Present in Final Precipitate
$CONC_{CS}$ = Concentration of Carrier Solution
VOL_{CS} = Volume of Carrier Solution Added
$\sigma_{PPT.\ WT.}$ = Standard Deviation in Weight of Precipitate
σ_{CONC} = Standard Deviation in Carrier Concentration
σ_{VOL} = Standard Deviation in Carrier Volume
PPT. WT. = Weight of Final Carrier Precipitate

Counting Uncertainty (CU) at the 95% Confidence Level

$$CU_{B_{2\sigma}} = \frac{1.96 * (\sigma_{NBCR_S})}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

σ_{NBCR_S} = Standard Deviation of the Net Background Corrected Count Rate
EFF = Detector Efficiency
ALI = Sample Aliquot Volume or Mass
R = Sample Tracer/Carrier Recovery
ABN_S = Abundance Fraction of the Emissions Used for Identification/Quantification
λ = Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute λ]
t = Time from Sample Collection To Radionuclide Separation or Mid-point of Count Time (Same units as half-life)
CF = Other Correction Factors as Appropriate (i.e, Ingrowth factor, Self-absorption Factor, etc.)

Sample Activity Concentration Total Propagated Uncertainty (TPU)—
Sample Concentrations <u>Without Blank Subtraction</u>

$$TPU_{B_{1\sigma}} = \sigma_{ACT} = \frac{\sqrt{\sigma_{NBCR_S}^{\ 2} + (NBCR_S)^2 * \left(RE_{EFF}^2 + RE_{ALI}^2 + RE_R^2 + \Sigma\ RE_{CF}^2\right)}}{2.22 * EFF * ALI * R * ABN_S * e^{-\lambda t} * CF}$$

Where:

- EFF = Detector Efficiency
- ALI = Sample Aliquot Volume or Mass
- R = Sample Tracer/Carrier Recovery
- ABN_S = Abundance Fraction of the Emissions Used for Analyte Identification/Quantification
- $\sigma^2_{NBCR_S}$ = Variance of the Net Background Corrected Count Rate
- $NBCR_S$ = Net Background Corrected Count Rate
- RE^2_{EFF} = Square of the Relative Error of the Efficiency Term
- RE^2_{ALI} = Square of the Relative Error of the Aliquot
- RE^2_R = Square of the Relative Error of the Sample Recovery
- RE^2_{CF} = Square of the Relative Error of Other Correction Factors
- λ = Analyte Decay Constant - ln 2/(half-life) [Same units as the half-life used to compute λ]
- t = Time from Sample Collection to Radionuclide Separation or Mid-Point of Count Time (Same units as half-life)
- CF = Other Correction Factors as Appropriate (i.e, Ingrowth factor, Self-absorption Factor, etc.)

NOTE: For methods using a tracer or carrier, the inclusion of efficiency and recovery terms in the equation above may result in overestimation of the TPU.

APPENDIX D

Listing of Common Nuclides/Methodologies

Source of radionuclide identities: DOE National Sample Tracking System, FY 1994

Analysis Type	Matrix						Instrumentation					Source
	Water	Soil	Fly Ash	Solid	Air	Biota	Gas Proportional	Liquid Scintillation	α Spectrometry	γ Spectrometry	α Scintillation	
Gross α/β	X	X					X					2&4
^{3}H	X	X				X		X				2
Pu isotopic	X	X	X		X	X			X			2
U Isotopic	X	X	X		X	X			X			2
^{241}Am	X	X	X			X			X			2
^{90}Sr	X		X	X			X	X				2&4
$^{223,224,226}Ra$	X										X	3
^{226}Ra	X			X							X	2
^{228}Ra	X						X					2
^{99}Tc	X							X				1
Th Isotopic	X	X	X		X	X	X		X			2
^{129}I	X									X		4
Cm Isotopic	X						X					3
^{89}Sr	X		X	X			X	X				2&4

^{14}C	X						X					2
^{237}Np	X						X				X	3
^{222}Rn												
^{210}Pb	X			X		X	X					2
^{210}Po						X			X			2
^{79}Se	X							X				4
Gamma Emitters	X									X		3

Sources :

1. "Technetium-99 Analysis Using Extraction Chromatography"
2. "Eastern Environmental Radiation Facility Radiochemistry Procedures Manual"
3. United States Environmental Protection Agency. *Prescribed Procedures for Measurement of Radioactivity in Drinking Water.* EPA-600/4-80-032.
4. *DOE Methods for Evaluating Environmental and Waste Management Samples DOE/EM-0089T, Rev.* 2, October, 1994.

Appendix E
Recommended Surveillance Considerations for Gamma Algorithms

Radionuclide identification algorithms used by the laboratory should be evaluated during laboratory audits. Consideration should be given to the following:

i. the width of the energy-search windows for radionuclide identification to ensure a minimum of peak misidentifications,

ii. the criteria by which the laboratory evaluates sample-specific peak resolution,

iii. documentation of the source of the isotope identification library used by the source [Most software packages come with a master library which users can pare down to one more useful in day to day analyses.],

iv Training records of those persons(s) using the peak identification respective software,

v. SOP documentation of the parameters used by the software in the analysis of gamma spectra,

vi. existence of SOP documentation for the internal review of the reported data,

vii. If the software package is capable of using pre-programmed macros to automate the spectral analysis, evaluate the existence of SOP documentation of these macros,

viii. If the software package uses various flags to denote peak quality or analysis results, the analyst using the software should be able to explain the meaning of the flags and their impact on data quality.

Laboratory Data Validation Guidelines For Evaluating Radionuclide Analyses

Prepared By:

Thomas L. Rucker, PhD. and C. Martin Johnson, Jr.

of

Science Applications International Corporation (SAIC)
An Employee-Owned Company
301 Laboratory Road
P.O. Box 2501
Oak Ridge, TN 37830

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TABLE OF CONTENTS

Page

INTRODUCTION 1

PRELIMINARY REVIEW 2

RADIONUCLIDE PROCEDURE 2

I. Holding Times 3

II. Calibration 5

III. Blanks 9

IV. Sample Specific Chemical Recovery 10

V. Laboratory Control Sample 12

VI. Matrix Spike Sample Analysis 13

VII. Duplicate Analysis Samples 14

VIII. Field Duplicates Analysis 16

IX. Radionuclide Quantitation and Implied Detection Limits 16

X. Chemical Separation Specificity (Alpha Spectrometry) 18

XI. Target Radionuclide List Identification (Gamma Spectroscopy) 19

XII. Tentatively Identified Radionuclides (Gamma Spectroscopy) 21

XIII. Systems Performance 23

XIV. Overall Assessment of Data for a Case 24

GLOSSARY A: Data Qualifier Definitions 26

GLOSSARY B: Additional Terms 27

REFERENCES 30

FORMS 31

TABLE OF FORMS

Page

Cover Page 32

FORM 1: Radiometric Analysis Results 33

FORM 2: Blank Summary 34

FORM 3: Chemical Recovery 35

FORM 4: Laboratory Control Sample 36

FORM 5: Matrix Spike Sample Rcovery 37

FORM 6: Duplicates 38

INTRODUCTION[1]

This document is designed to offer guidance in laboratory evaluation and validation of radioanalytical data. It is the intent of this document to provide data validation guidelines for radioanalytical data equivalent to those provided by the Environmental Protection Agency (EPA) under its Contract Laboratory Protocol (CLP) guidance. Therefore, the format used is similar to that contained in reference 2. In some aspects, it is equivalent to a standard operating procedure (SOP). In more subjective areas, only general guidance is offered due to the complexities and uniqueness of data relative to specific samples.

Four terms are used throughout this document: shall indicates a requirement for the data validator, must indicates a requirement for the data, should indicates a recommendation, and may indicates an acceptable practice (neither a requirement nor a recommendation).

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

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

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

All data reviews shall have a cover sheet. If mandatory actions are required, they should be specifically noted on this sheet. In addition, this sheet is to be used to summarize overall deficiencies requiring attention, as well as general laboratory performance and any discernible trends in the quality of the data. (This sheet is not a replacement for the data review.) Sufficient supplementary documentation shall accompany the sheet to clearly identify the problems associated with a case.

---

[1]Largely adapted from reference 2's introduction.

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PRELIMINARY REVIEW[2]

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

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

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

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

   * If applicable

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

RADIONUCLIDE PROCEDURE

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

---

[2]Largely adapted from reference 2's preliminary review.

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1. Holding times (CCS - Lab holding times only)
2. Calibration
   - Initial (CCS)
   - Continuing (CCS)
3. Blanks (CCS)
4. Sample specific chemical recovery (CCS)
5. Laboratory control sample (CCS)
6. Matrix spike (CCS)
7. Field duplicates
8. Duplicate sample (CCS)
9. Radionuclide quantitation and implied detection limits
10. Chemical separation specificity
11. Target radionuclide list identification
12. Tentatively identified radionuclides
13. System performance (CCS)
14. Overall assessment of data for a case

I. HOLDING TIMES

A. Objective

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

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

B. Criteria

The following technical requirements for sample holding times and preservation have only been established for water matrices. Due to limited information concerning holding times for soil samples, it is left to the

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professional judgment of the data reviewer whether to apply water holding time criteria to soil samples.

1. Tritium solutions: 6 months, with no preservative and stored in glass.

2. Carbon-14: 6 months, cool to 4°C and slightly basic with NaOH.

3. Iodine solutions: 6 months, with no preservatives.

4. Radon-222: 4 days, cool to 4°C and stored in glass with teflon-lined septum.

5. Cesium: 6 months, when preserved to pH <2 in hydrochloric acid.

6. Plutonium: 6 months, when preserved in 2M nitric acid.

7. Other radionuclides: 6 months, when preserved to pH <2 in nitric or hydrochloric acid.

C. Evaluation Procedure

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

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

D. Action

1. If criteria for holding times and preservation are not met, qualify all results as estimated (J).

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

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

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II. CALIBRATION

A. Objective

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable quantitative data. Initial and continuing calibration demonstrates that the instrument is capable of acceptable performance at the beginning of the calibration period and routine calibration verification and system checks document that the initial calibration is still valid.

B. Criteria

Calibrations and routine system checks are to be performed according to the following schedule:

1. Initial and Continuing Calibration (Gross α, Gross β, Gamma Spec.)

   a. Gas Flow Proportional and Other Gross Counting Measurements

   The gross counting systems must be efficiency calibrated for each alpha and beta counting geometry at least annually or when the daily performance check indicates an unacceptable change in system efficiency. Self-absorption curves must be developed at least annually, or if the absorption correction is included in an efficiency curve, a new curve must be developed when the daily performance check indicates an unacceptable change in system efficiency. A plateau curve and alpha/beta cross-talk factors must be established, and a performance check must be made after each P-10 counting gas bottle change. If an unacceptable change has occurred due to the new counting gas, then the new gas must be replaced. If, out of necessity, counting is performed using undesirable counting gas, a special calibration must be performed for that gas.

   The counter background must be established quarterly or when the routine performance check indicates an unacceptable change in instrument background. If desired, matrix or batch blanks may be used for background subtraction rather than counter background.

   b. Liquid Scintillation Spectrophotometer (Tritium)

   Efficiency quench curves must be established for the liquid scintillation spectrophotometer for each radionuclide to be counted at least annually or when the daily performance check indicates an unacceptable change in system efficiency.

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Instrument high voltage, gain, energy calibration, or quench indicator calibration must be adjusted using standard instrument calibration sources prior to calibration and routinely (usually daily) thereafter in order to maintain valid quench calibrations over the year. An efficiency calibration is not required when comparative measurement or internal standardization is used. However, calibration verification shall be performed.

The counter background must be established quarterly or when the routine performance check indicates an unacceptable change in instrument background. Background quench curves must be established for each radionuclide to be counted unless matrix or batch blanks are used for background subtraction.

c. Spectroscopy Measurements

Energy versus channel calibration must be established for spectroscopy systems at least quarterly or when the daily performance check indicates an unacceptable change in energy gain or zero offset.

Resolution versus energy calibration must be established for gamma spectroscopy systems quarterly or when the daily performance check indicates an unacceptable change in system resolution.

Spectroscopy systems must be efficiency calibrated for each counting geometry at least annually or when the daily performance check indicates an unacceptable change in system efficiency. Efficiency versus energy curves must be established for gamma spectroscopy systems for the energy region of interest with particular attention to energy regions where the efficiency depends strongly on energy. Single efficiency values may be used for alpha spectroscopy systems for alpha energies between 4.0 and 6.0 MeV. An efficiency calibration is not required when comparative measurement or internal standardization is used. However, calibration verification must be performed.

Spectroscopy system background determinations must be established quarterly or when the routine performance check indicates an unacceptable change in system background.

d. Lucas Cells and Radon Flask Counting Systems

Each Lucas Cell and radon flask counting system must be efficiency calibrated with the Department of Energy

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Environmental Measurements Laboratory Radon Program at least annually or when calibration verification shows an unacceptable change in efficiency. A high voltage plateau curve must be established at least annually or if there are any unusual events affecting the power supply or the counting instrument.

The Lucas Cell and radon bubbler background must be established before each use.

2. Routine Calibration Verification

a. Gas Flow Proportional and Other Gross Counting Measurements

The efficiency calibration of gross counting systems must be checked using alpha and beta (if applicable) check sources each day that the system is used. The net alpha and beta counts (corrected for decay) and the alpha/beta cross-talk must be recorded and should be plotted on a QC chart daily.

The counter background must be checked each day that the system is used. The background alpha and beta counts must be recorded and should be plotted on a QC chart daily.

Acceptable tolerances must be established for each QC chart based on system performance and analytical requirements. Maximum tolerance limits of ± 10% of the value determined at time of calibration are recommended for efficiency verification control charts; otherwise, tolerance limits of ± 3 standard deviations are recommended. When tolerance limits are exceeded, recalibration is required.

b. Liquid Scintillation Spectrophotometers

The efficiency of liquid scintillation spectrophotometers must be checked using a calibration source each day that the system is used. The net counts (corrected for decay) must be recorded and should be plotted on a QC chart daily.

The counter background must be checked each day that the system is used. The background alpha and beta counts must be recorded and should be plotted on a QC chart daily.

Acceptable tolerances must be established for each QC chart based on system performance and analytical requirements. Maximum tolerance limits of ± 10% of the value determined at time of calibration are recommended for efficiency verification control charts; otherwise, tolerance limits of

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± 3 standard deviations are recommended. When tolerance limits are exceeded, recalibration is required.

c. Spectroscopy Systems

The energy, resolution, and efficiency must be monitored using a calibration source each day that the system is used and must be compared against the initial calibration values. The check source must have both low- and high-energy peaks. The centroid energy, full width at half maximum (FWHM), and net counts under each calibration peak (corrected for radioactive decay) must be recorded and should be plotted on a QC chart daily.

The counter background must be checked at least once every 20 counting periods. The net count rate in each background peak (gamma) or region (alpha) must be recorded and should be plotted on a QC chart. The background check count rate is compared to the established background spectra used for background subtraction. Acceptable tolerances must be established for each background peak or region. When tolerance limits are exceeded or when new background peaks are identified, a new background must be established for the system.

Acceptable tolerances must be established for each QC chart based on system performance and analytical requirements. Maximum tolerance limits of ± 10% are recommended for efficiency calibration verification control charts and ± 1 FWHM for energy calibration verification control charts; otherwise, tolerance limits of ± 3 standard deviations are recommended. All control limits are relative to the value determined at time of calibration. When tolerance limits are exceeded, recalibration must be performed.

d. Lucas Cells and Radon Flask Counting Systems

The efficiency calibration of the Lucas Cells must be checked using a NIST traceable radium source in a radon bubbler 1 every 20 times it is used. The counts (corrected for decay) must be recorded and should be plotted on a QC chart.

The dark current background must be checked at least weekly or when a new high voltage plateau has been established. The dark current background must be recorded and should be plotted on a QC chart.

Acceptable tolerances must be established for each QC chart based on system performance and analytical requirements.

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Maximum tolerance limits of ± 10% of the value determined at time of calibration are recommended for efficiency verification control charts; otherwise, tolerance limits of ± 3 standard deviations are recommended. When tolerance limits are exceeded, recalibration is required.

C. Evaluation Procedure

1. Verify that the instrument was calibrated each time the instrument was setup and at the required frequency as stated above. Evaluate the shape and smoothness of high voltage plateaus, efficiency versus energy curves, and quench curves.

2. Observe the QC charts and verify that proper limits have been established and that recalibration was performed whenever the limits were exceeded.

3. Verify, at a minimum, 10% of the calibration calculations. If errors are found in the calculations, verify more calculations by using professional judgment to see the extent of the errors.

D. Action

1. If the specified calibration and/or verification frequency is not followed, the efficiency or quench curves are not smooth, or the QC results fall outside the appropriate tolerance limits, qualify the results for all samples analyzed between acceptable calibration verifications as estimated (J).

2. When significant errors are found in the calculations, flag all affected results that allow a bias of between 10-20% as estimated (J), and greater than 20% as unusable (R).

III. BLANKS

A. Objective

Blank analysis results are assessed to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks applies to any blank associated with the samples. If problems with <u>any</u> blank exist, all data associated with the case shall be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

B. Criteria

At least one blank must be analyzed for every matrix, every batch, or for every 20 samples (5% of samples), whichever is more frequent. The result of all blanks must be recorded and should be plotted on a QC chart at least

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daily for each method. Acceptable tolerances must be established for each QC chart based on system performance and analytical requirements. Tolerance limits of ± 3 standard deviations are recommended.

When average blanks or instrument backgrounds are subtracted to determine net counts, the net blank result must be less than the associated uncertainty. Contamination shall be suspected when the net blank result is larger than the associated uncertainty.

C. Evaluation Procedures

Review the results reported on the Blank Summary (Form 2) and evaluate the blank control charts as well as the raw data for all blanks. Verify that the results were accurately reported and that tolerance limits were not exceeded. Verify that net blank results are less than the associated uncertainty.

D. Action

for Gross α Gross β, all blank results are > than uncertainty

If the blank QC results fall outside the appropriate tolerance limits or if the net blank results are not less than the associated uncertainty, qualify the results for all associated samples that are less than 10 times the blank value as estimated (J).

IV. SAMPLE SPECIFIC CHEMICAL RECOVERY

A. Objective akin to "surrogates"

Laboratory performance on individual samples subject to chemical process and separation is established by means of spiking with tracer quantities of other radioisotopes of the same element or carrier quantities of the inactive isotope of the same or a chemically similar element. All samples are spiked prior to sample preparation. The evaluation of the results of these spikes is not necessarily straightforward. The sample matrix may produce interferences. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the review and validation of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

B. Criteria

1. Sample specific recoveries must be within limits as per applicable scope of work (SOW). Generally, recoveries of 50-100% are considered acceptable. However, lower recoveries may be typical for some matrices and the acceptable lower limit may be lowered in such instances. Each chemical tracer percent recovery (CT %R) must be recorded and should be plotted on a QC chart for each radionuclide and method and fall within the prescribed limits.

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2. The quantity of tracer material used must be adequate to provide a maximum of 10% uncertainty at the 95% confidence level in the measured recovery.

C. Evaluation Procedure

1. Review Form 3 and verify that sample specific recoveries fall within the control limits.

2. Check the raw data to verify that sample specific recoveries are accurately reported on Form 3. Recalculate, at a minimum, 10% of the sample specific recoveries (CT %R) using the following equation:

$$CT\ \%R = \frac{CT_{Found}}{CT_{True}} * 100$$

Where:

CT_{Found} = concentration (in pCi/L for aqueous; pCi/kg for solid) of each analyte measured in the analysis of LCS solution

CT_{True} = concentration (in pCi/L for aqueous; pCi/kg for solid) of each analyte in the LCS source.

3. Check spike levels to verify that sufficient levels are used to provide adequate precision for recovery determination.

4. Evaluate recovery to verify that limits specified in SOW are met.

D. Action

For sample specific recoveries out of specification, the following approaches are suggested based on a review of all data from the case, especially considering the apparent complexity of the sample matrix:

1. For sample specific recoveries, qualify results for the appropriate radionuclides in all associated samples as follows:

   a. 50-100%: acceptable for use
   b. 100-150%: estimated (J)
   c. 20-50%: estimated (J)
   d. <20%: unusable (R)
   e. >150%: unusable (R)

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2. When significant errors are found in the calculations, flag all affected results that allow a bias of between 10-20% as estimated (J), and greater than 20% as unusable (R).

V. LABORATORY CONTROL SAMPLE

A. Objective

The laboratory control sample (LCS) serves as a monitor of the overall accuracy and performance of all steps in the analysis, including the sample preparation. For the following limits to apply, the LCS must contain greater than 10 times the radionuclide's detection limit activity.

B. Criteria

1. At least one LCS must be analyzed for every matrix, every batch, or for every 20 samples (5% of samples), whichever is more frequent.

2. All aqueous LCS results must fall within the control limits of 80-120% recovery of the standard value.

3. All solid LCS results must fall within the control limits of 70-130% recovery of the standard value.

4. All LCS results must be recorded and should be plotted on a QC chart according to sample type and radionuclide and fall within the prescribed limits.

C. Evaluation Procedure

1. Review Form 4 and verify that results fall within the control limits.

2. Check the raw data (counter printout, strip charts, bench sheets, etc.) to verify the reported recoveries on Form 4. Recalculate, at a minimum, 10% of the LCS percent recoveries (LCS %R) using the following equation:

$$LCS\ \%R = \frac{LCS_{Found}}{LCS_{True}} * 100$$

Where:

LCS_{Found} = concentration (in pCi/L for aqueous; pCi/kg for solid) of each analyte measured in the analysis of LCS solution

LCS_{True} = concentration (in pCi/L for aqueous; pCi/kg for solid) of each analyte in the LCS source.

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D. Action

1. Aqueous LCS

a. If the LCS %R for any analyte falls within the range of 50-80%, or 120-150%, qualify results for that radionuclide in all associated samples as estimated (J).

b. If LCS %R are <50% or >150%, qualify results for that radionuclide in all associated samples as unusable (R).

2. Solid LCS

a. If the LCS %R for any analyte falls within the range of 40-70% or 130-160%, qualify results for that radionuclide in all associated samples as estimated (J).

b. If LCS %R are <40% or >160%, qualify results for that radionuclide in all associated samples as unusable (R).

VI. MATRIX SPIKE SAMPLE ANALYSIS

A. Objective

The matrix spike sample (MSS) analysis provides information about the effect of each sample matrix on the digestion and measurement methodology. MSSs are required when sample specific chemical recovery mechanisms are not available and the samples undergo a chemical process.

B. Criteria

1. At least one MSS must be analyzed for every matrix, every batch, or for every 20 samples (5% of samples), whichever is more frequent, when sample specific chemical recovery mechanisms are not available and the samples undergo a chemical process.

2. Samples identified as field blanks must not be used for spiked sample analysis.

3. Matrix spike sample percent recovery (MSS %R) must be within the limits of 80-120% for aqueous matrix and 70-130% for solid matrix samples. However, spike recovery limits do not apply when sample concentration exceeds the spike concentration by a factor of 4 or more.

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4. The MSS %R of the matrix spike must be recorded and should be plotted on a QC chart and fall within the prescribed limits.

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C. Evaluation Procedure

1. Review Form 5 and verify that results fall within the specified limits.

2. Check raw data and recalculate, at a minimum, 10% of the %R using the following equation to verify that the results were correctly reported on Form 5.

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

Where:

SSR = Spiked Sample Result

SR = Sample Result

SA = Spike Added

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

D. Action

1. If the spike recovery is between 120% and 150% (130% and 160% for solids) or between 50% and 80% (40% and 70% for solids), qualify the results for that radionuclide for associated samples as estimated (J).

2. If spike recovery is <50% (<40% for solids) or >150% (>160% for solids), qualify the results for that radionuclide for associated samples as unusable (R).

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

VII. DUPLICATE ANALYSIS SAMPLES

A. Objective

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

B. Criteria

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

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2. At least one duplicate must be analyzed for every matrix, every batch, or for every 20 samples (5% of samples), whichever is more frequent.

3. The duplicate analyses results must be in agreement when the 2 standard deviations (95% confidence limit) uncertainties are considered. For this to be true, the duplicate error ratio (DER) as defined in the following equation must be less than 1. The DER must be recorded and should be plotted on QC charts with a control limit set at 1.

Duplicate Error Ratio

$$DER = \frac{|S - D|}{(2\sigma_S + 2\sigma_D)}$$

Where,

S = First Sample Value (original)

D = Second Sample Value (duplicate)

$2\sigma_S$ = First Sample Uncertainty

$2\sigma_D$ = Second Sample Uncertainty.

C. Evaluation Procedure

1. Review Form 6 and verify that DER results are less than 1.

2. Check, at a minimum, 10% of the duplicate results and recalculate the DER values. Use the above equation to verify that DER results have been correctly reported on Form 6.

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

D. Action

1. If DER for a particular radionuclide is greater than 1, qualify the results for that radionuclide in all associated samples of the same matrix as estimated (J).

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

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VIII. FIELD DUPLICATES ANALYSIS

A. Objective

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

B. Criteria

There are no review criteria for field duplicate analyses comparability.

C. Evaluation Procedures

Samples that are field duplicates should be identified using sample field sheets. The reviewer should compare the results reported for each sample and calculate the DER.

D. Action

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

IX. RADIONUCLIDE QUANTITATION AND IMPLIED DETECTION LIMITS

A. Objective

The objective is to ensure that the reported quantitation results are accurate and that the required detection limits have been met. When detection limit requirements are not met, the data quality objectives may not have been met. All results shall be evaluated relative to the uncertainty associated with the analysis.

B. Criteria

1. Radionuclide quantitation must be calculated according to the appropriate procedures specified in the contractual SOW.

2. Detection limits specified in the specific procedures must be met unless other detection limits are specified in the SOW.

3. Analytical uncertainties must be reported with all results in order to qualify the data. Results and uncertainties must be reported for all required analyses regardless of the size or sign of the result. The reported uncertainty must include all uncertainties associated with the

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analysis. If the reported uncertainty only includes counting uncertainty, this fact must be documented in the case narrative.

4. For solid samples a minimum of 100 grams must be homogenized prior to subsampling an aliquot for analysis. Homogenization of the entire sample is recommended for all samples and is required for liquid samples with more than one phase. The minimum homogenized sample aliquot size used for analysis must be 1 g for dry solids or 1 ml for liquid samples, although further dilution may be performed after chemical dissolution or extraction.

5. When samples are dry mounted for counting, mounting aliquots must be selected in order to keep the dry mounted weight to ≤ 5 mg/cm^2 for alpha analyses and ≤ 10 mg/cm^2 for beta analyses.

C. Evaluation Procedures

1. The raw data shall be examined to verify the correct calculation of sample results reported on Form 1 by the laboratory.

   a. Examine the raw data for any anomalies (i.e., omissions, legibility, etc.). Recalculate, at a minimum, 10% of the results to verify proper calculation. If calculation errors are found, recalculation of more results may be required in order to determine the extent of the error.

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

   c. Verify that all analytical uncertainties have been propagated and reported or otherwise documented.

   d. Verify that appropriate aliquot sizes have been used for sample preparation (Form 7) and mounting.

2. Verify that uncertainties (Form 1) have been reported for all results.

3. Check the detection limits by verifying that, for blanks or any other samples that have an uncertainty greater than the result, the 2σ uncertainty multiplied by 1.65 is less than or equal to the specified detection limit. 1.65

Note: Net negative results that have uncertainties greater than their absolute value indicate the sample count was less than background. Net positive results that have uncertainties larger than the results indicate the sample count was less than the critical level or less than 95% confidence of positive detection.

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D. Action

1. When significant errors are found in the calculations, flag all affected results that allow a bias of between 10-20% as estimated (J), and greater than 20% as unusable (R). If errors are found in the calculation of the uncertainties or they have been rounded improperly, flag the uncertainty as estimated (JE).

2. When inappropriate aliquot sizes are used, flag all affected results as estimated (J).

3. For net negative results that have uncertainties smaller than their absolute value, flag the data as unusable (R). This is an indication of improper blank subtraction.

4. When detection limits are not met, flag the data as detection limit exceeded (DL).

5. When analytical uncertainties are not reported, flag the results as estimated (J).

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

X. CHEMICAL SEPARATION SPECIFICITY (Alpha Spectrometry)

A. Objective

Chemical separation specificity is the contract laboratory's ability to separate various radionuclides by chemical separation techniques. The chemical separation specificity can be verified for alpha spectroscopy measurements by observation of the alpha energy spectrum.

B. Criteria

1. There are not to be any radionuclides that interfere with the quantitation of the radionuclide of interest once the chemical separation process has been completed.

2. Energy of the radionuclide of interest must be within 40 keV of the observed peak energy.

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C. Evaluation

1. Check that the energy of the observed peak of interest is within 40 keV of the energy for the radionuclide of interest.

2. Check the energy spectra for any peaks that overlap or that have associated peaks that may interfere with the peak of the radionuclide of interest.

3. When interfering radionuclides are present and can be corrected for from associated peaks in the spectrum, check to see if the peak area for the radionuclide of interest has been properly corrected.

D. Action

1. If the energy of the peak of interest is more than 40 keV from the energy for the radionuclide of interest, qualify the results as unusable (R).

2. If the alpha energy spectra contains any peaks that overlap with or have associated peaks that may interfere with the peak of the radionuclide of interest and it is impossible to correct for the interference, qualify the results as unusable (R).

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

XI. TARGET RADIONUCLIDE LIST IDENTIFICATION (Gamma Spectroscopy)

A. Objective

The target radionuclide list (TRL) contains those radionuclides for which a quantitative analysis is required. Therefore, net quantitation with uncertainties must be provided for all TRL radionuclides (whether or not the radionuclide is identified in the peak search and identification). This is accomplished by determining the net area in the region associated with the radionuclide when the radionuclide is not detected by the computerized peak search routine. When a peak is detected for the radionuclide, positive identification is achieved through the use of the following criteria.

B. Criteria

1. The target radionuclide energy must be within 2 keV of the observed peak.

2. There are not to be any radionuclide gamma peaks that interfere with the quantitation of the radionuclide of interest. If there is an interference, the radionuclide of interest result must be corrected to

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negate the interfering radionuclide's contribution to the radionuclide of interest's gamma peak.

C. Evaluation Procedure

1. Check that the energy of the identified peaks is within 2 keV of the standard library energy for the identified radionuclide.

2. Verify that net peak areas and associated uncertainties have been obtained for all TRL radionuclides not meeting the above criteria.

3. Check the energy spectra for any peaks that overlap or that have associated peaks that may interfere with the peak of the radionuclide of interest.

4. When interfering radionuclides are present and can be corrected for from associated peaks in the spectrum, check to see if the peak area for the radionuclide of interest has been properly corrected.

D. Action

Qualify the data according to the following:

1. For TRL radionuclides that are not detected in the computerized peak search, qualify the net peak area results as described in Section IX.

2. For TRL radionuclide peaks that are detected but fail to meet the positive identification criteria, flag the data as rejected (R).

3. If improper methods are used, flag the data as estimated (J).

4. If the gamma energy spectra contains any peaks that overlap with or have associated peaks that may interfere with the peak of the radionuclide of interest and it is impossible to correct for the interference, qualify the results as unusable (R).

5. If the results can be and have not been properly corrected for the interfering radionuclide, qualify the data as unusable (R).

If any discrepancies are found, the laboratory may be contacted by the designated representative to obtain additional information that may resolve any differences. If a discrepancy remains unresolved, the reviewer shall decide which value is the best value. Under these circumstances, the reviewer may determine whether qualification of data is warranted.

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XII. TENTATIVELY IDENTIFIED RADIONUCLIDES (Gamma Spectroscopy)

A. Objective

One of the objectives of the criteria for gamma spectroscopy qualitative analysis is to minimize the number of erroneous identifications of radionuclides. An erroneous identification can either be a false positive (reporting a radionuclide present when it is not) or a false negative (not reporting a radionuclide that is actually present). It is much easier to detect false positives than false negatives, because more information is available due to the requirement for submittal of data supporting positive identification. Negatives, or nondetected radionuclides, on the other hand, represent an absence of data and are, therefore, much more difficult to assess.

Gamma spectra peaks in radionuclide analyses that are not TRL isotopes are potential tentatively identified radionuclides (TIRs). TIRs must be qualitatively identified by a radionuclide spectra library computer search and the identifications assessed by the data reviewer.

B. Criteria

1. Identified radionuclide energy must be within 2 keV of the observed peak.

2. Gamma spectra of the sample radionuclide and the standard radionuclide library must match according to the following criteria:

   a. 50% of total abundance of all gamma peaks listed in the standard radionuclide library <u>must</u> be present in the sample spectrum.

   b. The sampling to count time must not be greater than 10 half lives of the identified radionuclide.

3. Radionuclide concentrations present in the gamma spectra must be consistent with related radionuclides (e.g., when daughter radionuclides are expected to be in equilibrium with parents, detection of both provides confirmation of identification).

4. All peaks greater than three standard deviations of the background identified radionuclides spectrum shall be considered and accounted for.

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5. Guidelines for tentative identification are as follows:

   a. Peaks present in the sample spectrum but not in the reference spectrum shall be reviewed for possible background contamination or interference.

   b. When the above criteria are not met, but the data reviewer or gamma spectral interpretation specialist judges the identification to be correct, the data reviewer may report the identification.

   c. If the data reviewer judges the identification to be uncertain or there are extenuating factors affecting radionuclide identifications, the TIR result may be reported as "unknown."

C. Evaluation Procedure

1. Check the raw data to verify that the laboratory has generated a computer library search for all required peaks in the spectra (samples and blanks).

2. Examine the blank spectra to verify that TIR peaks present in samples are not found in blanks. When a low-level non-TRL radionuclide that is a common artifact or laboratory contaminant is detected in a sample, a thorough check of blank spectra may require examining for peaks that are less than the critical level but present in the blank spectra at similar counting time.

3. Examine all gamma peaks in every sample and blank spectra.

4. Check that expected related radionuclides are present.

   Note: Since TIR computer library searches often yield several candidate radionuclides having close matching peaks, consider all reasonable choices.

   Note: The reviewer should be aware of common laboratory artifacts/contaminants and their sources (e.g., radon and thoron daughters in the air, etc.). These may be present in blanks and not reported as sample TIRs.

D. Action

1. All verified TIRs shall be listed in the evaluation report.

2. All TIR results without supporting data shall be flagged as tentatively identified with estimated concentrations (JN).

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3. All TIRs without quantitation shall be flagged as tentatively identified (N).

4. General actions related to the review of TIR results are as follows:

   a. If it is determined that a tentative identification of a non-TRL radionuclide is not acceptable, the tentative identification shall be flagged as rejected (R) or changed to an appropriate identification.

   b. If all peaks were not library searched, the designated representative could request these data from the laboratory.

5. TIR results that are not sufficiently above the level in the blank should not be reported. (Dilutions, sample size, and counting times shall be taken into account when comparing the amounts present in blanks and samples).

6. When a radionuclide is not found in any blanks, but is a suspected artifact of common laboratory contamination, the result may be flagged as unusable (R).

7. In deciding whether a library search result for a TIR represents a realistic identification, professional judgment shall be exercised. If there is more than one reasonable match, the result may be reported as "either radionuclide X or radionuclide Y."

8. Other case-specific factors may influence TIR judgments. If a sample TIR match is poor but other samples have a TIR with a good library match and similar gamma peaks, identification information may be inferred from the other sample TIR results.

9. Physical constants, such as half life, shall be factored into professional judgment of TIR results.

XIII. SYSTEMS PERFORMANCE

A. Objective

During the period following instrument performance QC checks (e.g., blanks, tuning, calibration, etc.), changes may occur in the system that degrade the quality of the data. While this degradation would not be directly shown by QC checks until the next required series of analytical QC runs, a thorough review of the ongoing data acquisition may yield indicators of instrument performance.

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B. Criteria

Some examples of instrument performance indicators for various factors are shown below. (Note: This is not an exhaustive list.)

1. Abrupt, discreet shifts in background or detector response may indicate contamination and/or gain or threshold changes.

2. Poor spectroscopy performance affects both qualitative and quantitative results. Indications of substandard performance include:

   a. High background levels or shifts in energy calibration.

   b. Extraneous peaks.

   c. Loss of resolution.

   d. Peak-tailing or peak splitting that may result in inaccurate quantitation.

C. Evaluation Procedure

Evaluate the raw data for each sample to determine if unexpected activity, extraneous peaks, loss of resolution, or loss of expected background peaks has occurred.

D. Action

Continued analytical activity with degraded performance suggests lack of attention or professional experience. Based on the instrument performance indicators, the data reviewer shall decide if the system has degraded to the point of affecting data quality or validity. If data quality may have been affected, data shall be qualified using the reviewer's best professional judgment.

XIV. OVERALL ASSESSMENT OF DATA FOR A CASE[3]

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

[3]This section was taken from the "Overall Assessment of Data for a Case" section of reference 2.

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data reviewer would be greatly assisted in this endeavor if the data quality objectives were provided. The cover form and supplementary documentation shall be included with the review."

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GLOSSARY A
Data Qualifier Definitions

J - The associated numerical value is an estimated quantity.

JN - Presumptive evidence of the presence of the radionuclide at an estimated quantity.

N - Presumptive evidence of the presence of the radionuclide with no estimation of quantity.

DL - Detection limit requirements not met. Data quality objectives may not be met.

R - The data are unusable (radionuclide may or may not be present). Resampling and reanalysis is necessary for verification.

JE - Uncertainty is an estimated quantity. This estimated flag applies only to the uncertainty and has no reflection on the quality of the result.

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GLOSSARY B
Additional Terms

Calibration Curve - An analytical curve based on the pulse energy, detector efficiency, energy absorbance, or other measured characteristic obtained from standard sources and a reagent blank.

Calibration Source - A radionuclide source counted daily to verify the calibration of a counting system.

Case - A finite (usually predetermined) number of samples collected over a given time period for a particular site. A case consists of one or more sample delivery group(s).

Chemical Tracer - A trace quantity of a different radioisotope of the same element or a carrier quantity of an inactive isotope of the same or a chemically similar element.

Contract Compliance Screening (CCS) - A process in which the analytical data is reviewed for contractual compliance.

Critical Level (CL) - The net count rate that must be exceeded before there is a specific degree of confidence that the sample contains any measurable radioactive material above background.

Customer Required Detection Limit (CRDL) - The minimum concentration in a given matrix type that a customer will accept of a radionuclide that can be measured and reported with a specific degree of confidence that the radionuclide activity is greater than zero.

Duplicate - Two aliquots taken from a homogenized sample and analyzed as individual samples. These are used to determine the precision of the method.

Duplicate Error Ratio - The ratio of the difference between the duplicate results to the sum of the two standard deviation uncertainties for duplicate results.

Field Blank - A sample of radionuclide-free media which is taken to the field in sealed containers and transferred from one vessel to another at the sampling site and preserved with the appropriate reagents. This serves as a check on reagent and environmental contamination. These blanks are treated as actual samples but may not be used for matrix spikes or sample duplicates.

Field Duplicate - Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

Full Width at Half Maximum (FWHM) - the width of the distribution at a level which is half the maximum ordinate of the peak.

Holding Times - The time between the date of collection of sample and the date of sample analysis.

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Laboratory Control Sample (LCS) - A control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents, and analytical methods employed for the unknown samples being analyzed. The results from the analysis of the controls are plotted and compared to control limits to determine the usability of the data.

Matrix Spike Sample (MSS) - An aliquot of sample spiked with a known concentration of target radionuclide(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix. (Some Federal Regulations require that data be corrected for spike recovery prior to reporting. Environmental Protection Agency recommends a minimum of 10 times the method detection limit or 2 to 4 times the measured quantity.)

Method Blank - A radionuclide-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process and should not be used for matrix spikes or sample duplicates.

Method Detection Limit (MDL) - The minimum concentration of a radionuclide that can be measured and reported with a specific degree of confidence that the radionuclide's activity is greater than zero and is determined for analysis of a sample in a given matrix type. MDL is equivalent to LLD, MDA, etc.

Percent Recovery (%R) - The fractional amount of the known activity of the radionuclide of interest that was obtained in the analysis.

Quality Control (QC) - An aggregate of activities designed to ensure adequate quality of analytical data.

QC Chart - A graphic representation on which the values obtained on the analysis of backgrounds, blank, calibrations, and laboratory control samples are plotted sequentially. The chart usually consist of a central line and two control limit lines parallel to the central line. The distribution of the plotted values with respect to the control limits provide valuable visual and statistical information on the quality of the analyses.

Quench Curve - A plot of efficiency versus degree of quenching for quenched standards.

Quenching - A reduction in the pulse height from the output of the photomultiplier tube due to physical or chemical processes occurring during or after the deposition of energy by the ionizing particle in the scintillator. Quenching reduces the scintillation efficiency and hence produces a loss in counting efficiency.

Standard Operating Procedure (SOP) - Established or prescribed methods to be followed routinely for the performance of designated operations or in designated situations.

Statement of Work (SOW) - A detailed description of work to be performed by a contracted laboratory or facility.

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Target Radionuclide List (TRL) - A listing of radionuclides for which a quantitative analysis is required. Therefore, net quantitation with uncertainties must be provided for all TRL radionuclides whether or not the radionuclide is identified in the computerized peak search and identification routine.

Tentatively Identified Radionuclide (TIR) - A detected radionuclide not on the Target Radionuclide List.

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FORMS

ENVIRONMENTAL SURVEY
COVER PAGE
RADIOMETRIC DATA ANALYSIS PACKAGE

Lab Name: _____ Case No.: _____
Method Type: _____ SDG No.: _____
Matrix: _____

Sample Numbers

Client ID	Lab ID No.

Comments: _____

_____

_____

Release of the data contained in this data package has been authorized by the laboratory manager or the manager's designee, as verified by the following signature:

_____

Manager, Radiological Laboratory
Date: _____

FORM 1

RADIOMETRIC ANALYSIS RESULTS

Date: _____

Lab Name: _____

Case No.: _____

Method Type: _____

SDG No.: _____

Client Sample No.	Lab ID No.	Sample Type	Radio-nuclide	Result	Q	Uncertainty	Units	Analysis Date	Method No.	Instrument ID

Legend shall be attached

Comments: _____

BLANK SUMMARY
FORM 2

Lab Name: _____ Contract: _____

Lab Sample ID: _____ SDG No.: _____

Blank Matrix (soil/water): _____ Case No.: _____

Concentration Units (pCi/L or pCi/g): _____

Radiochemical Determination	Method Blank	Field Blank	Trip Blank

Comments: _____

CHEMICAL RECOVERY
FORM 3

Lab Name: _____

Contract: _____

Radiochemical Determination: _____

Case No.: _____

Sample Matrix (soil/water): _____

SDG No.: _____

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

Spike Units (pCi or mg) _____

Sample Identification	Chemical Tracer	Chemical Tracer Result (CT_{Found})	Chemical Tracer Added (CT_{True})	CT % R	Q

Comments: _____

LABORATORY CONTROL SAMPLE
FORM 4

Lab Name: _____ Contract: _____

Lab Sample ID: _____ Case No.: _____

Solid LCS Source: _____ SDG No.: _____

Aqueous LCS Source: _____

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

Radiochemical Determination	Aqueous (pCi/L)			Solid (pCi/g)		
	True	Found	LCS %R	True	Found	LCS %R

Comments: _____

MATRIX SPIKE SAMPLE RECOVERY
FORM 5

SAMPLE NO.

Lab Name: _____

Contract: _____

Lab Sample ID: _____

Case No.: _____

Matrix (soil/water): _____

SDG No.: _____

% Solids for Sample: _____

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

Concentration Units (pCi/L or pCi/g): _____

Radiochemical Determination	Spiked Sample Result (SSR)	Sample Result (SR)	Spike Added (SA)	MSS %R	Q

Comments: _____

DUPLICATES
FORM 6

SAMPLE NO.

Lab Name: _____

Contract: _____

Lab Sample ID: _____

Case No.: _____

Matrix (soil/water): _____

SDG No.: _____

% Solids for Sample: _____

% Solids for Duplicate: _____

$$DER = \frac{|S - D|}{(2\sigma_S + 2\sigma_D)}$$

Radiochemical Determination	Sample Result (S)	Sample Uncertainty ($2\sigma_S$)	Duplicate Result (D)	Sample Uncertainty ($2\sigma_D$)	DER	Q

Comments: _____

FORM 7
PREPARATION LOG

Lab Name: _____
Case No.: _____
Method: _____

Contract: _____
SDG No.: _____

Sample No.	Preparation Date	Weight (gram) or Volume (ml) of Sample Homogenized	Weight (gram) or Volume (ml) Aliquot of Homogenized Sample Processed	Dilution Factor (Fraction of aliquot counted)