4740.2120 QUALITY CONTROL CRITERIA FOR RADIOCHEMISTRY.

Subpart 1. **Scope.** This part applies to laboratories performing radiochemistry testing on environmental samples. All requirements in this part must be incorporated into the laboratory's standard operating procedures unless otherwise directed by the approved method. The quality control requirements specified by the laboratory's standard operating procedures manual must be followed. All quality control measures must be assessed and evaluated on an ongoing basis and quality control acceptance criteria must be used to determine the validity of the data.

Subp. 2. **Method blanks.**

A. A laboratory must analyze at least one method blank per batch. The method blank result must be evaluated according to the acceptance criteria in the laboratory's standard operating procedures manual.

B. When the method blank acceptance criteria are not met, a laboratory must take corrective action. The occurrence of a failed method blank and the actions taken must be noted in the laboratory report.

C. In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry, the method blank must be of similar counting geometry that is empty or filled to similar volume with ASTM Type II water to partially simulate gamma attenuation due to the sample matrix.

D. A laboratory must not subtract results of method blank analysis from the sample results in the associated batch unless permitted by the approved method. This does not preclude the application of any correction factor, such as instrument background, analyte presence in tracer, reagent impurities, peak overlap, or calibration blank, to all analyzed samples, both program- or project-submitted and internal quality control samples. However, the correction factors must not depend on the required method blank result in the associated analytical batch.

E. The method blank sample must be prepared with similar aliquot size to that of the routine samples for analysis whenever possible.

Subp. 3. **Laboratory control sample.**

A. Laboratory control samples must be performed at a frequency of one per batch. The results of the analysis must be one of the quality control measures to be used to assess the batch. The laboratory control sample result must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified laboratory control sample acceptance criteria are not met, the specified corrective action and contingencies must be followed. The occurrence of a failed laboratory control sample acceptance criterion and the actions taken must be noted in the laboratory report.

B. The activity of the laboratory control sample must:

(1) be two to ten times the detection limit; or

(2) at a level comparable to that of routine samples if the sample activities are expected to exceed ten times the detection limit.

C. The laboratory standards used to prepare the laboratory control sample must be from a source independent of the laboratory standards used for instrument calibration, if available.

D. The matrix spike must be prepared by adding a known activity of target analyte. When a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope, such as plutonium, Pu 238 and Pu 239, using alpha spectrometry, only one of the analyte isotopes need be included in the laboratory control sample. When more than one analyte isotope is added to the laboratory control sample, each isotope must be assessed against the specified acceptance criteria.

Subp. 4. **Matrix spikes.**

A. Matrix spikes must be performed at a frequency of one per batch for those methods that do not utilize an internal standard or carrier for which there is a chemical separation process and when there is sufficient sample to do so. The exceptions are gross alpha, gross beta, and tritium, which require matrix spikes for aqueous samples. The results of the analysis must be one of the quality control measures to be used to assess the sample results acceptance. The matrix spike result must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified matrix spike acceptance criterion is not met, the corrective actions specified in the laboratory's standard operating procedures must be followed. The occurrence of a failed matrix spike acceptance criterion and the actions taken must be noted in the laboratory report. The lack of sufficient sample aliquot size to perform a matrix spike must be noted in the laboratory report.

B. The activity of the analytes in the matrix spike must be greater than ten times the detection limit.

C. The laboratory standards used to prepare the matrix spike must be from a source independent of the laboratory standards used for instrument calibration, if available.

D. The matrix spike must be prepared by adding a known activity of target analyte. When a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope, such as plutonium, Pu 238 and Pu 239, using alpha spectrometry, only one of the analyte isotopes need be included in the matrix spike sample. When more than one analyte isotope is added to the matrix spike, each isotope must be assessed against the specified acceptance criteria.

E. When gamma spectrometry is used to identify and quantitate more than one analyte isotope, the laboratory control sample and matrix spike must contain isotopes that represent the low (americium-241), medium (cesium-137), and high (cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples, the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

F. The matrix spike sample must be prepared with similar aliquot size to that of the routine samples of analyses.

Subp. 5. **Tracer.** For those approved methods that allow or require the use of a tracer, that is, internal standard, each sample result must have an associated tracer recovery calculated and reported. The tracer recovery for each sample result must be one of the quality control measures used to assess the associated sample result acceptance. The tracer recovery must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified tracer recovery acceptance criteria are not met, corrective actions specified in the laboratory's standard operating procedures must be followed. The occurrence of a failed tracer recovery and the corrective actions taken must be noted in the laboratory report.

Subp. 6. **Carrier.** For those approved methods that allow or require the use of a carrier, each sample must have an associated carrier recovery calculated and reported. The carrier recovery for each sample must be one of the quality control measures used to assess the associated sample result acceptance. The carrier recovery must be assessed against the specific acceptance criteria specified in the laboratory standard operating procedures manual. When the specified carrier recovery acceptance criteria are not met, the corrective actions specified in the laboratory's quality assurance manual must be followed. The occurrence of failed carrier recovery acceptance criteria and the actions taken must be noted in the laboratory report.

Subp. 7. **Analytical variability; reproducibility for radiochemistry testing.**

A. A laboratory must analyze replicate samples at least once per batch when there is sufficient sample to do so. The results of the analysis must be one of the quality control measures used to assess sample results acceptance. The replicate result must be assessed against the specific acceptance criteria specified in the laboratory's standard operating procedures manual.

B. When the specified replicate acceptance criteria are not met, the corrective actions specified in the laboratory's standard operating procedures manual must be followed. The occurrence of failed replicate acceptance criteria and the actions taken must be noted in the laboratory test results.

C. If sample concentrations are expected to contain analytes of interest below three times the detection limit, a laboratory may substitute replicate laboratory control samples or replicate matrix spiked samples for replicate samples in item A. The replicate result must be assessed against the specific acceptance criteria specified in the laboratory's standard operating procedures manual. When the specified replicate acceptance criteria are not met, the corrective actions specified in the laboratory's standard operating procedures manual must be followed. The occurrence of failed replicate acceptance criteria and the actions taken must be noted in the laboratory test results.

Subp. 8. **Instrument calibration.**

A. Radiochemistry analytical instruments must be calibrated prior to first use in sample analysis.

B. Calibration must be verified when:

(1) the instrument is serviced;

(2) the instrument is moved; and

(3) the instrument settings have been changed.

C. The standards used for calibration must have the same general characteristics, that is, geometry, homogeneity, and density, as the associated samples.

D. The calibration must be described in the laboratory's standard operating procedures manual.

Subp. 9. **Continuing calibration verification.**

A. Calibration verification checks must be performed using appropriate check standards and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the calibration has not changed.

B. The same check standards used in the preparation of the tolerance chart or control chart at the time of calibration must be used in the calibration verification of the instrument.

C. The check standards must provide adequate counting statistics for a relatively short count time. The sources must be sealed or encapsulated to prevent leakage and contamination of the instrument and laboratory personnel.

D. For alpha and gamma spectroscopy systems, the instrument calibration verification must include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

E. For gamma spectroscopy systems, the calibration verification checks for efficiency and energy must be performed at least weekly along with performance checks on peak resolution.

F. For alpha spectroscopy systems, the calibration verification check for energy must be performed at least weekly and the performance check for counting efficiency must be performed at least monthly for each day the instrument is used for sample analysis.

G. For gas-proportional and scintillation counters, the calibration verification check for counting efficiency must be performed each day of use.

Subp. 10. **Background radiation measurement.**

A. Background radiation measurements must be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives.

B. Background radiation measurement values must be subtracted from the total measured activity in the determination of the sample activity.

C. For gamma spectroscopy systems, background radiation measurements must be performed at least monthly.

D. For alpha spectroscopy systems, background radiation measurements must be performed at least monthly.

E. For gas-proportional counters, background radiation measurements must be performed at least weekly.

F. For scintillation counters, background radiation measurements must be performed each day of use.

Subp. 11. **Instrument contamination monitoring.** A laboratory must have a written procedure for monitoring radiation measurement instrumentation for radioactive contamination. The procedure must indicate the frequency of the monitoring and must indicate criteria that initiate corrective action.

Subp. 12. **Detection limits.**

A. Detection limits must be determined before sample analysis and must be redetermined each time there is a significant change in the test method or instrument type.

B. The procedures employed must be documented and consistent with published references.

Subp. 13. **Quality of standards and reagents.**

A. The quality assurance manual must describe the procurement, use, and storage of radioisotope standards.

B. Reference standards that are used in a radiochemical laboratory must be obtained from the National Institute of Standards and Technology (NIST), EPA, suppliers of NIST standards or NIST traceable radioisotopes, or suppliers located outside of the

United States. Reference standards must be traceable back to the appropriate country's national standards laboratory.

C. Reference standards must be accompanied with a certificate of calibration that describes traceability to NIST or another country's national standards laboratory, when appropriate.

D. Laboratories must consult with the supplier if the laboratory's assessment of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory must not use a value other than the decay-corrected certified value.

E. All reagents used must be analytical reagent grade or better.

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