NATIONAL ENVIRONMENTAL LABORATORY ACCREDITATION CONFERENCE (NELAC)

ON-SITE LABORATORY ASSESSMENT

RADIOCHEMISTRY CHECKLIST (17 PAGES TOTAL)

LABORATORY: _____

Physical Address: _____

_____

Mailing Address: _____
(if different from above)

_____

Telephone Number: _____ Facsimile Number: _____

E-mail address: _____

INSPECTED BY: (Name) (Affiliation)

_____ _____

_____ _____

_____ _____

INSPECTION DATES: _____

LABORATORY TECHNICAL DIRECTORS AND MANAGEMENT:
(Name) (Title)

_____ _____

_____ _____

_____ _____

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_____ _____

GENERAL INSTRUCTIONS: Before each item is a blank line and a NELAC Standard citation in **Bold Numerals**.

Place a check mark (__----) in the blank if the laboratory meets the NELAC Standard referenced.

Place an X-mark (X) in the blank if the Standard is not met and the laboratory must devise an acceptable Plan of Correction and estimated completion date. **The NELAC Standard reference must be cited in in the on-site assessment report.**

Mark **"N/A"** in the blank if the NELAC Standard is not applicable to this laboratory, either because of the nature of its business mission, because of the analytical tests it performs, or because of the situation never ever happening.

Notes:

40 CFR Parts 122.21(g)(7), 122.21(h)(4) 122.41(j)(4), 403.7(b)(2)(v), 403.12(b)(5)(vi), 403.12(g)(4), & 501.15(b)(10)(iv) mandate the use of test methods approved in **40 CFR Part 136**

40 CFR Part 141.25(a) mandates the use of test methods contained therein for compliance with the **Safe Drinking Water Act**

Photocopy **pages 4-14** as necessary to assess **each individual Radiochemistry test method**

Pages 15-17 provide a **quick reference guide** to counting techniques & chemical reagents that are required for each test method; this guide **does not substitute for training** of on-site laboratory assessors **required under NELAC**

If the laboratory appears to meet a particular NELAC Standard but does not have the documentation to back up its claim, use the following:

_____ **5.0** Does the laboratory have **all items** identified in NELAC Chapter 5 Quality Systems **available** for on-site inspection or data audit

____ **D.4.3(b)** Does the laboratory use the results of **proficiency test sample analysis** to evaluate its ability to **produce accurate data**

____ **D.4.6(b)** Does the laboratory **report each Radiochemistry test result** with the **associated measurement uncertainty**

____ **D.4.6(b)** Does the laboratory have **documented procedures** for determining measurement uncertainties that are **consistent with mandated methods or regulations**

____ **D.4.8** Has the laboratory maintained a **radiological control program** that addresses analytical radiological control

____ **D.4.8** Does the radiological control program address procedures for **segregating samples** with potentially **widely varying levels of radioactivity**

____ **D.4.8** Does the radiological control program explicitly define how low-level & high-level samples will be identified, segregated, & processed in order to **prevent sample cross-contamination**

____ **D.4.8** Does the radiological control program include the measures taken to **monitor & evaluate background activity or contamination** on an on-going basis

____ **5.5.6.4(d)** Do **all** containers of prepared standards & reference materials bear a **unique identifier, expiration date, & link** to its specific **preparation record**

____ **5.5.6.4(e)** Are procedures in place to ensure that prepared reagents **meet the requirements of the test method** (see the scientific discipline & technology checklists for specific requirements)

Note: Reagents of appropriate quality must be selected and used. In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than specified in the test method shall not be used. Checks of the container label to verify that the purity of the reagents complies with the test method must be documented.

Note: See pages 15-17 for specific chemical reagent requirements in each test method

____ **5.5.6.4(f)** Do containers of **prepared reagents** bear a **preparation date**

____ **5.5.6.4(f)** Is the expiration date for each prepared reagent **defined on the container or documented elsewhere** as indicated in the laboratory's quality manual or SOP

____ **D.4.7(a)** Does the laboratory's **quality control program** establish & **maintain provisions for radionuclide standards**

____ **D.4.7(a)(1)** Does the laboratory obtain reference standards **from NIST or suppliers** who participate in supplying **NIST standards or NIST traceable** radionuclides

____ **D.4.7(a)(1)** Does the laboratory purchase Radionuclide reference standards from commercial suppliers who **conform to ANSI N42.22** to assure the quality of their products

____ **D.4.7(a)(1)** For reference standards purchased **outside the United States**, are the reference standards **traceable back to each country's national standards laboratory**

____ **D.4.7(a)(2)** Do the reference standards **have certificates of calibration** whose content is as described in **ANSI N42.22 – 1995, Section 8**, Certificates

____ **D.4.7(a)(3)** Does the laboratory **not use** a value for the **activity** of the reference traceable standard **other than the decay-corrected certified value**

____ **D.4.7(a)(3)** Does the laboratory have a written procedures for **handling, storing, & establishment of expiration dates** for reference standards

____ **D.4.7(b)** Are all chemical reagents used in the laboratory **analytical reagent grade or better** in quality

____ **5.5.8.3.1(a)(2)** Has the laboratory **checked samples** for **proper preservation** (e.g. pH<2 with Nitric Acid, 4 degrees Celsius, 6-month holding time) prior to or during sample preparation or analysis

COMMENTS:

RADIOCHEMISTRY TEST METHODS

TEST METHOD EVALUATED: _____

____ **5.5.4.1.2(a)** Does the laboratory have an **in-house methods manual** for each accredited **analyte** or **method**
Note: This manual may consist of copies of published or referenced test methods

____ **5.5.4.1.2(b)** Does the laboratory **clearly indicate** in its methods manual **any modifications** made to the referenced test method and **describe any changes or clarifications** where the referenced test method is ambiguous or provides insufficient detail

Does each test method in the in-house methods manual include or reference, where applicable:

____ **5.5.4.1.2(b)(1)** **Identification** of the test method
____ **5.5.4.1.2(b)(2)** Applicable **matrix or matrices**
____ **5.5.4.1.2(b)(3)** **Method Detection Limit**
____ **5.5.4.1.2(b)(4)** **Scope & application**, including components to be analyzed
____ **5.5.4.1.2(b)(5)** **Summary** of the test method
____ **5.5.4.1.2(b)(6)** **Definitions**
____ **5.5.4.1.2(b)(7)** **Interferences**
____ **5.5.4.1.2(b)(8)** **Safety**
____ **5.5.4.1.2(b)(9)** **Equipment & supplies**
____ **5.5.4.1.2(b)(10)** **Reagents & standards**
____ **5.5.4.1.2(b)(11)** Sample **collection, preservation, shipment, & storage**
____ **5.5.4.1.2(b)(12)** **Quality control**
____ **5.5.4.1.2(b)(13)** **Calibration & standardization**
____ **5.5.4.1.2(b)(14)** **Procedure**
____ **5.5.4.1.2(b)(15)** **Data Analysis & Calculations**
____ **5.5.4.1.2(b)(16)** Method **performance**
____ **5.5.4.1.2(b)(17)** Pollution **prevention**
____ **5.5.4.1.2(b)(18)** **Data assessment & acceptance criteria** for quality control measures
____ **5.5.4.1.2(b)(19)** **Corrective actions** for out-of-control data
____ **5.5.4.1.2(b)(20)** Contingencies for **handling out-of-control or unacceptable data**
____ **5.5.4.1.2(b)(21)** **Waste management**
____ **5.5.4.1.2(b)(22)** **References**
____ **5.5.4.1.2(b)(23)** **Tables, diagrams, flowcharts, validation data**

____ **D** Does the laboratory have **procedures** for developing **acceptance/rejection criteria** for each Radiochemistry test method

____ **D** Does the laboratory ensure that the **essential standards** outlined in Appendix D are incorporated into the method manuals and/or Quality Manual

____ **5.5.9.2(d)** **D** Does the laboratory's **Radiochemistry data** indicate that the **quality control protocols** in the test methods manual **are being followed**

COMMENTS:

RADIOCHEMISTRY TEST METHOD EVALUATED: _____

____ **5.5.5.2.2.1(a)** Does the laboratory's **test method SOP** include or reference details of the **initial instrument calibration procedures**

Note: This includes calculations, integrations, & associated statistics

Note: If the test method is referenced for initial instrument calibration procedures, the laboratory must **have this method & make it available** for review

____ **5.5.5.2.2.1(b)** Does the laboratory retain **sufficient raw data records to permit reconstruction** of the initial instrument calibration

Note: Examples of such data records include calibration date, test method, instrument, analysis date, each analyte name, analyst initials or signature, concentration & response, calibration curve or response factor, and unique equation or coefficient used to reduce instrument responses to concentration

____ **5.5.5.2.2.1(c)** Does the laboratory **quantitate sample results** only from the **initial instrument calibration** and not from any continuing instrument calibration verifications, unless required by regulation, method, or program

____ **5.5.5.2.2.1(d)** Does the laboratory **verify all initial instrument calibrations** with a standard obtained from a **second manufacturer or lot** if the lot can be demonstrated from the manufacturer as prepared independently from other lots

Note: When commercially available, traceability shall be to a national standard

____ **5.5.5.2.2.1(e)** Has the laboratory established **criteria for the acceptance** of an initial instrument calibration

Note: Examples include linear regression correlation coefficient & response factor %RSD

Note: The acceptance criteria must be **appropriate** to the calibration technique employed

____ **5.5.5.2.2.1(f)** For purposes of establishing the **working calibration range**, is the lowest calibration standard concentration the **lower limit of quantitation**

____ **5.5.5.2.2.1(f)** Is all data reported **below the lower limit of quantitation** reported using **defined qualifiers** or flags or **explained in the case narrative**

____ **5.5.5.2.2.1(g)** Is the highest calibration standard the **highest concentration** for which **quantitative data are to be reported**

____ **5.5.5.2.2.1(g)** Is all data reported **above the highest calibration standard** reported using **defined qualifiers** or flags or **explained in the case narrative**

Note: For instrument technologies (e.g., ICP, ICP/MS) with validated techniques from manufacturers or methods employing standardization with a zero point & a single-point calibration std., the following must occur:

____ **5.5.5.2.2.1(h)(1)** Prior to the analysis of samples, are the **zero point & single point calibration analyzed**, and the **linear range of the instrument established** by analyzing a series of standards, one of which must be at the lowest quantitation level

Note: Sample results within the established linear range will not require data qualifier flags

____ **5.5.5.2.2.1(h)(2)** Are the zero point & single point calibration standard analyzed **with each analytical batch**

____ **5.5.5.2.2.1(h)(3)** Is a standard corresponding to the **limit of quantitation** analyzed with each analytical batch & meet established acceptance criteria

____ **5.5.5.2.2.1(h)(4)** Is the **linearity verified** at a frequency established by the test method and/or the manufacturer

RADIOCHEMISTRY TEST METHOD EVALUATED: _____

____ **5.5.5.2.2.1(h)** Does the laboratory report measured concentrations **outside the working calibration range** as having **less certainty** & using **defined qualifiers or flags or explained in the case narrative**

____ **5.5.5.2.2.1(h)** Is the **lowest calibration standard above the limit of detection** for each analyte

____ **5.5.5.2.2.1(i)** Does the laboratory **perform corrective actions** & reanalyze all associated samples if the initial instrument calibration results are **outside established acceptance criteria**

____ **5.5.5.2.2.1(i)** When reanalysis is not possible, does the laboratory **report sample data** associated with unacceptable initial instrument calibrations **with appropriate data qualifiers**
Note: NELAC Standards 5.5.5.2.2.1(h) & (i) may need to be assessed **in conjunction with** the Quality Systems **data audit**

____ **5.5.5.2.2.1(j)** Does the laboratory have a standard operating procedure for **determining the number of points** for establishing the initial instrument calibration
Note: NELAC 5.5.5.2.2.1(h),(i),(j) are not applicable to Radiochemistry, except for mass attenuation in gas-proportional counting & sample quench in liquid scintillation counting

____ **5.5.5.2.2.1(j)** Does the laboratory use a **minimum of two calibration standards** (not including blanks or a zero standard) for performing an initial instrument calibration
Note: This Standard applies if a reference or mandated method does not specify the number of calibration standards
Note: One of the standards must be at the limit of quantitation
Note: This Standard does not apply to instrument technologies for which it has been established by methodologies & procedures that a zero & a single point standard are appropriate for calibrations (see Section 5.5.5.2.2.1(h))

____ **5.5.5.2.2** Do the laboratory's initial & continuing instrument calibration verifications meet the requirements in **mandated test methods & regulations**
Note: If it is not apparent which standard is more stringent, then the requirements of the regulation or the mandated test method are to be followed

____ **D.4.4(a)(1)** Are the Radiochemistry **analytical instruments calibrated prior to initial use**, when the instrument is **placed back into service after malfunctioning**, & when the instrument's **response has changed** as determined by performance check or when the instrument's response exceeds predetermined acceptance criteria for the instrument quality control

____ **D.4.4(a)(2)** Does the laboratory perform **instrument calibrations with appropriate reference standards**
Note: "Appropriate" means meeting the requirements in NELAC Section D.4.7(a)

____ **D.4.4(a)(2)** Do the reference standards have the **same general characteristics** (i.e. geometry, density, homogeneity, etc.) as the **associated samples**

____ **D.4.4(a)(3)** Does the laboratory method manual **specify a frequency of calibration** (e.g., monthly), or a frequency of observations plotted on the associated control or tolerance chart that serves as the basis for calibration, if such a frequency is not specified in the test method

RADIOCHEMISTRY TEST METHOD EVALUATED: _____

____ **5.5.5.10** Does the laboratory **verify** the validity of the initial calibration by a **continuing instrument calibration verification** with **each analytical batch, prior to sample analyses,** whenever an initial instrument calibration is not performed on the day of analysis

____ **5.5.5.10(a)** Are the **details** of the continuing instrument calibration verification **procedure, calculations, & associated statistics** included or referenced in the **test method SOP**

____ **5.5.5.10(b)** Is calibration verified **for each compound, element, or other discrete chemical species**

____ **5.5.5.10(c)(1)** Is the instrument calibration verification performed at the **beginning & end** of **each analytical batch**

Note: Only **one** verification needs to be performed at the beginning of the analytical batch if an **internal standard** is used

____ **5.5.5.10(c)(2)** Is the instrument calibration verification performed whenever **it is expected** that the analytical system **may be out of calibration** or might not meet the verification acceptance criteria

____ **5.5.5.10(c)(3)** Is the instrument calibration verification performed if the **time period** for calibration or the most previous calibration verification **has expired**

____ **5.5.5.10(c)(4)** Is the instrument calibration verification performed for analytical systems that **contain a calibration verification requirement**

____ **5.5.5.10(d)** Does the laboratory retain **sufficient raw data records** to permit **reconstruction** of the continuing instrument calibration verification

Note: Such records include test method, instrument, analysis date, name of each analyte, concentration & response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations

____ **5.5.5.10(d)** Does the laboratory's continuing calibration verification records **explicitly connect** the continuing verification data to the initial instrument calibration

____ **5.5.5.10(e)** Has the laboratory established **criteria for the acceptance** of a continuing instrument calibration verification (e.g. relative percent difference)

____ **5.5.5.10(e)** Does the laboratory **perform corrective actions** if the continuing instrument calibration verification results are **outside established acceptance criteria**

____ **5.5.5.10(e)** Does the laboratory perform a **new initial instrument calibration** if the routine corrective action procedures **fail to produce** a **second consecutive** (immediate) calibration **verification within acceptance criteria**

Note: Alternatively, the laboratory can demonstrate acceptable performance after correction with **2 consecutive calibration verifications**

____ **5.5.5.10(e)** If the laboratory has not verified calibration, do sample analyses **not occur** until the analytical system **is calibrated or calibration verified**

Note: For sample data associated with an **unacceptable** calibration **verification**, the results **must be flagged** but the data may be useable under the following special conditions:

- **Non-detects** for analytes in associated samples where the acceptance criteria for the continuing calibration verifications are **exceeded high**
- Any test result for an analyte that indicates **exceedence of a maximum regulatory limit** or decision level, when the acceptance criteria for the continuing calibration verification for that analyte is **exceeded low**

TEST METHOD EVALUATED: _____

____ **D.4.4(b)** Does the laboratory conduct **performance checks** using **appropriate check sources**

____ **D.4.4(b)** Does the laboratory monitor the performance checks with **control charts or tolerance charts to ensure** that the instrument is **operating properly** & that the **detector response** (and thus the instrument calibration) **has not changed**

____ **D.4.4(b)** Is the **same check source** used in the calibration verification checks **as is used** in the preparation of the **tolerance chart or control chart at the time of calibration**

____ **D.4.4(b)** Do the check sources **provide adequate counting statistics** for a relatively short count time

____ **D.4.4(b)(1)** For **gamma spectroscopy systems**, are performance checks for efficiency & energy calibration and for peak resolution **performed on each day of use**

____ **D.4.4(b)(2)** For **alpha spectroscopy systems**, are the performance checks for **energy calibration performed weekly**

____ **D.4.4(b)(2)** For **alpha spectroscopy systems**, are the performance checks for **counting efficiency conducted at least monthly**

____ **D.4.4(b)(3)** For **gas-proportional & liquid scintillation counters**, are the performance checks for **counting efficiency conducted each day of use**

Note: For sample batches that **uninterruptedly count for more than a day**, a performance check can be conducted at the **beginning & end of the batch** as long as this time interval is **no greater than one week**

Note: Instrument calibration verification does not directly verify secondary calibrations (e.g. mass efficiency curve or quench curve)

____ **D.4.4(b)(4)** For **scintillation counters**, is the calibration verification check for **counting efficiency performed each day of use**

____ **D.4.4(c)** Does the laboratory perform **background measurements** on a regular basis & **monitored using control charts or tolerance charts** to ensure its capability to meet required data quality objectives

Note: These measured background values **may be subtracted from the total measured activity** in the determination of the sample activity

____ **D.4.4(c)(1)** For **gamma spectroscopy systems**, are background measurements performed at least **monthly**

____ **D.4.4(c)(2)** For **alpha spectroscopy systems**, are background measurements performed at least **monthly**

____ **D.4.4(c)(3)** For **gas-proportional counters**, are background measurements performed **weekly**

____ **D.4.4(c)(4)** For **scintillation counters**, are background measurements performed **each day of use**

____ **D.4.4(d)** Does the laboratory have a written procedure for **monitoring** radiation measurement instrumentation **for radioactive contamination**

____ **D.4.4(d)** Do these procedures for monitoring radioactive contamination **indicate the frequency** of the monitoring & **include criteria** that initiates corrective action

COMMENTS:

TEST METHOD EVALUATED: _____

____ **5.5.4.2.2(a)** Has the laboratory performed a **satisfactory demonstration of method capability** prior to the
C.1 acceptance & institution of this test method
D.4.3(a)
Note: Demonstrations of capability are done in an applicable & available **clean matrix sample** in a quality system matrix where **no target analytes or interferences present** at concentrations that impact the results of a specific test method
Note: These following steps are **may not be applicable** for tests with which **spiking is not an option** and for which Quality Control samples are **not readily available**
Note: Actual sample spike results, such as **4 consecutive matrix spikes** (or quality control samples of analytes that do not lend themselves to spiking), within the **last 12 months** may be used to meet this Standard
Note: A demonstration of capability is **not required** in cases where samples are analyzed with this test method in use by the laboratory **before July 1999** & where there have **been no significant changes** in instrument type, personnel, or test method, in which case the analyst's documentation of continued proficiency is acceptable (the laboratory must have records on file to show that a demonstration of capability is not required)
Note: Continuing demonstration of method performance, per the QC requirements in App. D (e.g., laboratory control samples), is required thereafter

____ **C.1** Does the laboratory **document** in its Quality Manual **other adequate approaches** to **Demonstration of Capability** if the procedure below is **not required** by the mandated test method or regulation and if the laboratory **elects not to perform** this procedure

____ **C.1(a)** Is the **quality control sample** used for this Demonstration of Capability obtained from an **outside source**
Note: If an outside source is not available, the laboratory may prepare this sample with stock standards that are **prepared independently** from those used in instrument calibration

____ **C.1(b)** Are the analytes diluted in a volume of **clean matrix** sufficient to prepare **4 aliquots** at the **specified concentration** or to a concentration approximately **1-4 times** the **limit of quantitation**

____ **C.1(c)** Are **at least 4 such aliquots prepared & analyzed** according to the test method
Note: These analyses may occur either concurrently or over a period of days

____ **C.1(d)** Does the laboratory **calculate the mean recovery** in the appropriate reporting units & the **standard deviation** of the population sample (n-1) in the same reporting units for **each parameter of interest** using **all of the analysis results obtained**
Note: When it is not possible to assess mean & standard deviation, such as **for presence-absence & logarithmic values,** the laboratory must assess performance against established & documented criteria

____ **C.1(e)** Are the mean and standard deviation for each parameter **compared** to the corresponding **acceptance criteria for precision & accuracy** in the test method (if applicable) or in laboratory-generated acceptance criteria (if the method or analyte is non-standard)

____ **C.1(e)** Does the laboratory consider the performance unacceptable & **not analyze actual samples** for parameters that **fail the acceptance criteria**

____ **C.1(f)** When one or more parameters **fail** at least one of the **acceptance criteria**, does the analyst:
- **Locate & correct** the source of the problem, then **repeat the test** for all parameters of interest, OR
- **Repeat the test** for all parameters that failed to meet criteria

Note: Repeated failure from employing the second option above indicates a general problem with the entire measurement system, and the analyst must then perform the first option above

RADIOCHEMISTRY TEST METHOD ASSESSED: _____

____ **C.1** Is an **initial evaluation** performed for **all analytes to be added** to an existing accredited test method (for analytes not currently found on the laboratory's list of accredited analytes)

____ **5.5.2.6(c)(3)** Does each Analyst have **documentation** of **continued proficiency** by at least **one of the following once per year:**

- Acceptable performance of a **blind sample** (single blind to the analyst)
- Another **demonstration of capability or initial measurement system evaluation**
- Successful performance of a blind performance sample on a **similar test method** using the **same technology** (the acceptable limits must be determined prior to analysis)
- At least **4 consecutive** laboratory **control samples** with **acceptable levels** of precision & accuracy (acceptable limits for precision & accuracy must be determined prior to analysis)
- Analysis of **authentic samples** that have been analyzed by **another trained analyst** with **statistically identical results**

____ **5.5.4.2.2(d)** **C.2** Does the laboratory use the **NELAC-specified certification statement** to document the **completion of each Demonstration of Capability** (initial & continuing)

____ **C.2** Are copies of these certification statements retained in the **personnel records** of each **employee performing the test method**

____ **5.5.4.2.2(d)** **C.1** Does the laboratory **retain all associated supporting data** necessary to **reproduce the analytical results** summarized in the appropriate certification statement

____ **5.5.4.2.2(e)** **C.1** **D.4.3(a)** Does the laboratory **complete a demonstration of capability each time** there is a **change** in **instrument type** (e.g., different detection technique), **personnel, or test method**

____ **5.5.4.2.2(f)** Does the laboratory **fully document** the achievement of **demonstration of capability requirements** for each **specialized work cell**

Note: A work cell is defined as a group of analysts with specifically defined tasks that together perform the test method

____ **5.5.4.2.2(g)** Does the laboratory demonstrate & document acceptable performance through **acceptable continuing performance checks** (e.g laboratory control samples) **each time** that **membership** in a work cell **changes**

____ **5.5.4.2.2(g)** Do the **new members** of the work cell **work with experienced analysts** in the specialty area

____ **5.5.4.2.2(g)** Does the laboratory **repeat a Demonstration of Capability** with the new work cell if the **first 4 continuing performance checks** following the change in personnel **produce a failure** in any sample batch acceptance criteria

____ **5.5.4.2.2(g)** Is the **Demonstration of Capability repeated** if the entire **work cell is changed or replaced**

____ **5.5.4.2.2(h)** Is the **performance of the work cell** as a group **linked to the training records** of the **individual members** of the work cell

____ **5.1.1** Does the laboratory's **procedure for demonstrating its capability** to perform the method, the **analyst's capability** to perform the method, or the **acceptance criteria** for precision & accuracy **comply with the requirements specified** in the **mandated test method**

Note: See page xx for such Demonstration of Capability procedural requirements & acceptance criteria

TEST METHOD EVALUATED: _____

____ **D** Does the laboratory **assess & evaluate** all **quality control measures** on an **on-going basis**

____ **D** Does the laboratory **use** quality control **acceptance criteria** to determine the **validity of the data**
Note: Acceptance criteria from method blanks (**D.4.1(a)**), laboratory control samples (**D.4.1(b)(1)**), matrix spikes (**D.4.1(b)(2)**), replicate sample analyses (**D.4.2**), tracer recoveries (**D.4.1(c)(1)**), & carrier recoveries (**D.4.1(c)(2)**) are the minimum criteria to be used to determine batch and/or individual sample result acceptance

____ **D.4.1(a)(1)** Does the laboratory analyze **method blanks** at a frequency of at least **one per preparation batch**

____ **D.4.1(a)(1)** Does the laboratory **assess the method blank result** against specific **acceptance criteria** in the laboratory **test method manual**

____ **D.4.1(a)(1)** If method blank acceptance criteria are not met, does the laboratory **take the corrective action & follow the contingencies specified** in its test method manual

____ **D.4.1(a)(1)** If method blank acceptance criteria are not met, are test results **reported with appropriate data qualifying codes**

____ **D.4.1(a)(1)** Are the occurrence of failed method blank acceptance criteria & corrective actions taken **noted in the laboratory test report**

____ **D.4.1(a)(2)** For **gamma spectroscopic methods**, generally a non-destructive analysis, is a method blank prepared using a **calibrated counting geometry** similar to that used for the samples
Note: The container of appropriate volume can be **empty or filled to similar volume**, to simulate partially gamma attenuation due to a sample matrix

____ **D.4.1(a)(3)** Does the laboratory **not subtract the method blank result from the sample results** in the associated preparation or analytical batch (unless permitted by the test method or regulatory program)
Note: Correction factors (e.g. instrument background, analyte presence in tracer, reagent impurities, peak overlap, etc.) may be applied to all analyzed samples & internal quality control samples as long as these correction factors don't depend on the required method blank result for the associated analytical batch

____ **D.4.1(a)(4)** Is the method blank prepared with **similar aliquot size** to that of the routine samples for analysis, with the method blank result & acceptance criteria calculated in a manner that compensates for sample results based on differing aliquot size

____ **D.4.1(b)(1)** Does the laboratory perform at least **one Laboratory Control Sample** per **preparation batch**

____ **D.4.1(b)(1)** Does the laboratory **assess the laboratory control sample result** against specific **acceptance criteria** in the laboratory **test method manual**

____ **D.4.1(b)(1)** If laboratory control sample acceptance criteria are not met, does the laboratory **take the corrective action & follow the contingencies specified** in its test method manual

____ **D.4.1(b)(1)** Are the occurrences of failed laboratory control sample acceptance criteria & the actions taken **noted in the laboratory test report**

TEST METHOD EVALUATED: _____

____ **D.4.1(b)(2)** Does the laboratory analyze at least **one matrix spike** per **preparation batch**, for Radiochemistry methods that **include a chemical separation process without the use of a carrier** or an internal standard (and for which there is sufficient sample to do so)

____ **D.4.1(b)(2)** Are matrix spikes performed **for gross alpha, gross beta, & tritium** analyses on **aqueous samples** (even though a chemical separation process is not involved)

____ **D.4.1(b)(2)** Does the laboratory **assess the matrix spike recovery** against specific **acceptance criteria** in the laboratory **test method manual**

____ **D.4.1(b)(2)** If matrix spike sample acceptance criteria are not met, does the laboratory **take the corrective action & follow the contingencies specified** in its test method manual

____ **D.4.1(b)(2)** Are the occurrences of failed matrix spike acceptance criteria & the actions taken **noted in the laboratory test report**

____ **D.4.1(b)(2)** Is the **lack of sufficient sample aliquot size** to perform a matrix spike noted in the laboratory report

____ **D.4.1(b)(3)** Is the activity of the laboratory control sample **at least 5 times the limit of detection** & at a level **comparable to routine samples** when such information is available if activities are expected to exceed 5 times the limit of detection

____ **D.4.1(b)(4)** Is the activity of the matrix spike analyte(s) **greater than 5 times the detection limit**

____ **D.4.1(b)(5)** Are the laboratory standards used to prepare the laboratory control sample & matrix spike from a **source independent** of the laboratory standards used for instrument calibration

____ **D.4.1(b)(6)** Is the matrix spike prepared by adding a known activity of target analyte **after subsampling** if required **but before any chemical treatment** (e.g., chemical digestion, dissolution, separation, etc.)

____ **D.4.1(b)(6)** When more than one analyte isotope is present above a specified limit of detection, does the laboratory **assess each isotope** against the specified acceptance criteria

Note: When a Radiochemical test method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g. Plutonium, Pu-238, & Pu-239 by alpha spectroscopy), only one of the analyte isotopes needs to be included in the laboratory control or matrix spike sample at the indicated activity level

____ **D.4.1(b)(7)** When gamma spectroscopy is used to identify & quantitate more than one analyte isotope, does the laboratory control sample **contain isotopes that represent the low** (e.g. Am-241), **medium** (e.g. Cs-137), & **high** (e.g. Co-60) **energy range** of the analyzed gamma spectra

Note: The selected isotopes do nor necessarily need to bracket the calibrated energy range or the range over which isotopes are identified & quantitated

____ **D.4.1(b)(8)** Is the laboratory control sample **prepared with similar aliquot size** to that of routine samples for analysis

COMMENTS:

TEST METHOD EVALUATED: _____

____ **D.4.1(c)(1)** For Radiochemistry test methods that utilize a tracer (i.e. internal standard), does **each sample result** have an associated **tracer recovery calculated & reported**

____ **D.4.1(c)(1)** Is the tracer added to the sample **after subsampling** if required **but before any chemical treatment** (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method

____ **D.4.1(c)(1)** Does the laboratory **assess the tracer recovery** against specific **acceptance criteria** in the laboratory **test method manual**

____ **D.4.1(c)(1)** If the tracer recovery acceptance criteria are not met, does the laboratory **take the corrective action & follow the contingencies specified** in its test method manual

____ **D.4.1(c)(1)** Are the occurrences of failed tracer recovery acceptance criteria & actions taken **noted in the laboratory test report**

____ **D.4.1(c)(2)** For Radiochemistry test methods that utilize a carrier for recovery determination, does **each sample result** have an associated **carrier recovery calculated & reported**

____ **D.4.1(c)(2)** Is the carrier added to the sample **after subsampling** if required **but before any chemical treatment** (e.g., chemical digestion, dissolution, separation, etc.) unless otherwise specified by the method

____ **D.4.1(c)(2)** Does the laboratory **assess the carrier recovery** against specific **acceptance criteria** in the laboratory **test method manual**

____ **D.4.1(c)(2)** If the carrier recovery acceptance criteria are not met, does the laboratory **take the corrective action & follow the contingencies specified** in its test method manual

____ **D.4.1(c)(2)** Are the occurrences of failed carrier recovery acceptance criteria & actions taken **noted in the laboratory test report**

____ **D.4.2(a)** Does the laboratory perform at least **one replicate sample analysis** per **preparation batch**, where there is sufficient sample to do so

Note: For low-level samples (<3 times the detection limit) LCS duplicate or matrix spike duplicate may be used to assess reproducibility within a preparation batch

____ **D.4.2(a)** Does the laboratory **assess the result of replicate sample analysis** against specific **acceptance criteria** in the laboratory **test method manual**

____ **D.4.2(a)** If replicate sample precision acceptance criteria are not met, does the laboratory **take the corrective action & follow the contingencies specified** in its test method manual

____ **D.4.2(a)** Are the occurrences of failed replicate acceptance criteria & the actions taken **noted in the laboratory test report**

COMMENTS:

TEST METHOD EVALUATED: _____

____ **D.4.5(a)** Has the laboratory **determined detection limits** prior to sample analysis

____ **D.4.5(a)** Has the laboratory determined the detection limit each time there is **significant change in the test method or instrument type**

____ **D.4.5(b)** Are the **procedures employed** for the detection limit determination **documented & consistent** with mandated test method or regulatory requirements

____ **5.1.1** Do the laboratory's detection limits **fulfill the requirements** of **mandated test methods** or **regulations** (i.e. reliably & consistently **below** any SDWA **Maximum Contaminant Levels**, RCRA **Toxicity Characteristics**, CWA **effluent limitations**, or other concentration levels upon which regulatory decisions are based)

Note: SDWA Detection Limit requirements in **40 CFR Part 141.25(c)**

Ra-226 + Ra-228	1.0 pCi/L
Gross Alpha	3.0 pCi/L
Tritium	1000 pCi/L
Sr-89	10 pCi/L
Sr-90	2.0 pCi/L
I-131	1.0 pCi/L
Cs-134	10 pCi/L
Gross Beta	4.0 pCi/L
Other Radionuclides	One-tenth the concentration determined to produce total body or organ dose of 4 millirem/year

Note: Other regulations (including state regulations) & permits may contain additional requirements for **Reporting Limits, Minimum Levels, Lower Limits of Detection,** & other criteria

Note: **NELAC 2000** now requires Method Detection Limits be done **prior to sample analyses** & each time there is **significant change** to test method or instrument type

Note: **NELAC 2000** requires the MDL procedure to be **documented & consistent** with mandated test methods or regulations

____ **5.1.1** Does the laboratory fulfill **additional requirements** specified in the **mandated test method** or **regulation**

SM7120B, 6a requires replicate sample analysis for every 10 samples analyzed

COMMENTS: List analytes for which the above Standards or requirements for measurement sensitivity have not been fulfilled

Gross Alpha, Gross Beta
EPA 900.0; EPA-600/4-75-008, p.1; EPA 00-01; EPA (1979), p.1; SM7110B; USGS R-1120-76

Thin end-window proportional counter; internal proportional & Geiger counters as alternate Beta counters
Counting pan, solids loading needs to be < 5 mg/cm2 to prevent Alpha radiation self-absorption
SDWA: Natural Uranium or Thorium-230 as Gross Alpha standards
Plutonium-239 or Americium-241 also as Gross Alpha standards
Cesium-137 or Strontium-90 as Gross Beta standards

Gross Alpha
EPA 900.1; EPA 00-02; SM7110C

Alpha scintillation counter or low-background proportional counter
Barium Chloride & Ferric Nitrate as carriers
Sulfuric Acid & Ammonia-water, to co-precipitate Alpha-emitters with Barium Sulfate & Iron(III) Hydroxide
SDWA: Natural Uranium, Thorium-230, or Americium-241 as Gross Alpha standards

Radioactive Cesium
EPA 901.0; EPA-600/4-75-008, p.4; SM7500Cs B; ASTM D2459-72; USGS R-1111-76

Low-background Beta (gas-proportional) counter or Gamma spectrometer
Cesium Chloride as carrier
Ammonium Phosphomolybdate, to precipitate radioactive Cesium
Chloroplatinic Acid, to convert cesium precipitate to Cs2PtCl6

Photon Emitters
EPA 901.1; EPA (1979), p.92; SM7120B; ASTM D3649-91; USGS R-1110-76; DOE 4.5.2.3; DOE GA-01-R

Gamma spectrometer with Tl-activated NaI detector or Li-drifted Ge detector

Radioactive Iodine – EPA 902.0; EPA-600/4-75-008, p.6; SM7500I B

Low-background Beta (gas-proportional) counter or Beta-Gamma coincidence counter or Gamma spectrometer
Potassium Iodate as carrier
Sodium Sulfite, to reduce iodine-species to iodide
Silver Nitrate, to precipitate iodide
Zinc powder, to purify silver iodide precipitate
Palladium Chloride & Sulfuric Acid, to re-precipitate iodide as PdI2

Radioactive Iodine – SM7500I C; ASTM D4785-93

Low-background Beta (gas-proportional) counter or Beta-Gamma coincidence counter or Gamma spectrometer
Sodium Iodide or Potassium Iodide as carrier
Sodium Bisulfite, to convert all iodine species to iodide (before anion exchange & after extraction)
Anion Exchange Resin, to concentrate iodide species
Sodium Hypochlorite, to elute iodide from column (as iodate at this point)
Hydroxylamine, to convert iodate from anion-exchange column to iodine prior to CCl4 extraction
Carbon Tetrachloride extraction solvent
Palladium Chloride or Copper(I) Chloride, to precipitate iodide as PdI2 or CuI, respectively

Radioactive Iodine – EPA-600/4-75-008, p.9; SM7500I D

Low-background Beta (gas-proportional) counter or Beta-Gamma coincidence counter or Gamma spectrometer
Potassium Iodide as carrier
Tartaric Acid & Nitric Acid, to distill HI into Sodium Hydroxide scrubber solution
Sodium Nitrite, to oxidize iodides to iodine
Carbon Tetrachloride extraction solvent
Sodium Bisulfite, to reduce iodine back to iodide & re-extract into aqueous phase
Palladium Chloride, to precipitate iodide as PdI2

Radium-226 – EPA 903.0, EPA-600/4-75-008, p.13; EPA Ra-03; SM7500Ra B; ASTM D2460-97; USGS R-1140-76

Internal gas-flow proportional counter, Alpha scintillation counter, or thin end-window gas-flow proportional Counter
Radium-226 standard
Lead Nitrate & Barium Chloride as carriers
Sulfuric Acid, to coprecipitate radium-lead-barium sulfates
EDTA & Ammonia-water, to redissolve precipitate
Acetic Acid at pH 4.5, to precipitate radium & barium sulfates (Pb & other natural Alpha emitters stay in sol'n)

Radium-226 – EPA 903.1; EPA-600/4-75-008, p.16; EPA Ra-04; EPA (1979), p.19; SM7500Ra C; ASTM D3454-97; DOE Ra-05; USGS R-1141-76

Alpha scintillation counter, with Ag-activated ZnS phosphor coating & SnO4-coated glass window
Radium-226 standard
Barium Chloride as carrier
Barium-133 as tracer (ASTM D3454-91)
Sulfuric Acid, to precipitate barium & radium sulfates
HF & Flux (fusion of Barium Sulfate, Potassium & Sodium Carbonates, & Sodium Borate), to remove silicates
Hydrogen Peroxide, to decompose insoluble radium compounds
Phosphoric Acid, to remove sulfites
Ascarite & Magnesium Perchlorate sorbents, for Radon-222 Emanation Assembly (3 weeks recommended)

Radium-228 – EPA 904.0; EPA-600/4-75-008, p.24; EPA Ra-05; EPA (1979), p.19; SM7500Ra D; USGS R-1142-76

Gas-flow internal proportional counter, or thin end-window proportional counter
Barium Chloride, Lead Nitrate, & Strontium Nitrate / Yttrium Oxide as carriers
Sulfuric Acid, to coprecipitate radium-lead-barium sulfates
EDTA & Ammonia-water, to redissolve precipitate (DTPA (Diethylenetriaminepentaacetic Acid) used in Ra-05)
Acetic Acid at pH 4.5, to precipitate radium & barium sulfates (Pb & other natural Alpha emitters stay in sol'n)
(In-growth of Actinium-228 from Radium-228 for 36 hours)
Ammonium Sulfide, to remove precipitate lead carrier
(pH 3 Acetic Acid used to re-precipitate Ra & Ba sulfates in Ra-05)
Sodium Hydroxide, to coagulate yttrium hydroxide (supernatant solution analyzed for Ra-226) (omitted in Ra-05)
Ammonium Oxalate, to carry the Actinium on yttrium oxalate precipitate
(Bis(2-ethylhexyl)phosphoric Acid in Heptane used to extract supernatent Actinium in Ra-05)
(If Ra-228 absent, Ra-226 may be determined by EPA 903.0 above; if Ra-228 present, radon emanation is used)

Strontium-89 & Strontium-90

EPA 905.0; EPA-600/4-78-008, p.29; EPA Sr-04; EPA (1979), p.65; SM7500Sr B; USGS R-1160-76; DOE Sr-01, Sr-02

Gas-flow internal proportional (low-background Beta) counter, or thin end-window proportional counter
Strontium Nitrate, Barium Nitrate, Yttrium Oxide, & Cerium(III) Nitrate / Zirconyl Chloride / Iron(III) Chloride as carriers
Sodium Carbonate & Sodium Hydroxide, to precipitate Strontium
Nitric Acid, to redissolve Strontium & separate it from SiO2, BaSO4, etc.
Fuming Nitric Acid, to precipitate Sr(NO3)2 & separate it from calcium interference
Ammonia-water, to precipitate rare earth hydroxides (converted Yttrium-90 Hydroxide later)
(Conversion of Strontium-90 to Yttrium-90 begins after removal of rare earth oxides, allow in-growth for 2 weeks)
Sodium Chromate & pH 5.5 Acetate Buffer, to precipitate barium
(Strontium re-precipitated as SrCO3 & can be counted to produce the Total Radiostrontium result)
Ammonium Oxalate, to convert yttrium hydroxide to yttrium oxalate precipitate for counting
Tributyl Phosphate extraction solvent (extract Y-90 as alternative to precipitation as hydroxide (oxalate))

Tritium

EPA 906.0; EPA-600/4-78-008, p.34; EPA H-02; EPA (1979), p.87; SM7500(3H) B; ASTM D4107-91; USGS R-1171-76

Liquid scintillation coincidence-type spectrometer
Sodium Hydroxide, to distill Tritium & not radiocarbon or radioiodine
Potassium Permanganate, to oxidize organic matter
Scintillation Solution (1,4-Di-2-(5-phenyloxazolyl)benzene (POPOP); 2,5-Diphenyloxazole (POP); Naphthalene; & 1,4-Dioxane)

Natural Uranium – EPA 908.0; SM7500U B

Gas-flow proportional counter or Alpha scintillation counter
Iron(III) Chloride as carrier
Uranyl Acetate standard
Ammonia-water, to co-precipitate iron(III) hydroxide & Uranium (boil sample first to remove carbonates)
Anion-exchange resin (wash out iron & plutonium with HI in HCl)
Hydrochloric Acid, to elute Uranium
Nitric Acid, to convert Uranium to its nitrate & to remove HIO3

Natural Uranium – EPA 908.1; SM7500U C (17th ed.); ASTM D2907-97; USGS R-1180-76, R-1181-76; DOE U-04

Fluorimeter (320-370 nm excitation wavelength, 530-570 nm fluorescence)
Sodium Fluoride & Lithium Fluoride Flux (ASTM D2907-91 direct method)
Aluminum Nitrate & Diammonium Hydrogen Phosphate, to co-precipitate aluminum & uranyl phosphates
Nitric Acid, to redissolve precipitate
Magnesium Nitrate, to salt the aqueous phase
Ethyl Acetate extraction solvent (MIBK in ASTM D2907-91)
Sodium Fluoride, Sodium Carbonate, & Potassium Carbonate Flux
Uranous-Uranium Oxide standard (U3O8)

Natural Uranium – EPA 00-07; EPA (1979), p.33; SM7500U C (18th ed.); ASTM D3972-97; USGS R-1182-76; DOE U-02

Alpha spectrometer
Uranium-232 as tracer
Iron(III) Chloride as carrier
Ammonia-water, to co-precipitate iron(III) hydroxide & Uranium (boil sample first to remove carbonates)
Anion-exchange resin (wash out iron & plutonium with HI in HCl)
Hydrochloric Acid, to elute Uranium
Electrolyte Solution (Ammonium Sulfate at pH 3.5)
Stainless-steel disk & electrode system, to electrodeposit Uranium

Natural Uranium – ASTM D5174-97

Laser Phosphorimeter
Nitric Acid & Hydrogen Peroxide, to digest sample
Uranium Complexant (e.g. phosphoric acid)