

QSM 5.1 FAQs

DoD ELAP

➤ **FAQ – Holding Time - Hours**

For a test with a recommended maximum holding time measured in hours, the holding time shall be tracked by the hour.

- For example, an exceedance of holding time for a sample with a 48-hour holding time will occur when the 49th hour is reached (e.g., a sample with a 48-hour holding time collected at 830 AM on April 4th must be analyzed or extracted by 9 AM on April 6th, or an exceedance will be considered to have occurred).

➤ **FAQ – Holding Time - Days**

For a test with a recommended maximum holding time measured in days, the holding time shall be tracked by the day.

- For example, an exceedance of holding time for a sample with a 14-day holding time will occur when the 15th day is reached (e.g., a sample with a 14-day holding time collected at 830 AM on April 4th must be analyzed or extracted by 12 AM on April 19th, or an exceedance will be considered to have occurred).

➤ **FAQ – Holding Time - Months**

For a test with a recommended maximum holding time measured in months, the holding time shall be tracked by the month. One month is defined as 30 days.

- For example, an exceedance of holding time for a sample with a 6-month holding time will occur when 6 months have passed (e.g., a sample with a 6-month holding time collected at 830 AM on April 4th must be analyzed or extracted by 12 AM on October 1st, or an exceedance will be considered to have occurred).

➤ **FAQ – Canister Cleaning Table B-21**

Clarification:

- For accreditation purposes, the laboratory must have the ability (procedures in place) to certify each canister. If the project uses the QSM to define their requirements, then individual cans must be certified.

****Batch certification is only permitted if that is what the project requests****

PFAS

➤ PFAS FAQ of QAOS consideration

Do you have to prepare the entire aqueous sample received when preparing routine samples (i.e. by SPE)?

Yes, the entire sample that is received must be sent through the SPE cartridge. In addition, the bottle must be solvent rinsed and this rinsate must be sent through the SPE cartridge as well and included in the sample extract. The method blank (MB) and laboratory control sample (LCS) must be extracted in exactly the same manner (i.e., must include the bottle solvent rinse). It should be noted that a water rinse alone is not sufficient. This does not apply to samples with high concentrations of PFAS that are prepared using serial dilution and not SPE.

➤ Table B-15 FAQs

1. Question:

Regarding initial calibration internal standards selection:

Do you have to use the internal standard analyte with the closest retention time for analytes where a labeled analog is not available, if there is another internal standard analyte that is more chemically similar?

Answer:

No, for those analytes that a labeled analog is not available, if there is a more suitable internal standard analyte based on chemical similarity and retention time versus retention time only, that internal standard analyte can be used.

2. Question:

Regarding acceptance criteria for the Extracted Internal Standard Analyte:

If your instrument software system will not calculate recoveries based off of a true value, can you calculate recovery (50-150%) off of the area measured in the ICAL midpoint standard?

Answer:

Yes, if the instrument software system utilized does not allow for recoveries to be calculated off of a true value, recoveries can be based off of the area measured in the ICAL midpoint standard.

3. Question:

Regarding acceptance criteria for recoveries of the Injection Internal Standard Analyte:

If your instrument software system will not calculate recoveries based off of peak area, can you calculate recovery (50-150%) off of the true value?

Answer:

Yes, if the instrument software system utilized does not allow for recoveries to be calculated off of a peak area, recoveries can be based off of the area measured in the true value. The criteria used must be documented in the standard operating procedures utilized by the laboratory.

4. Question:

Do the bottle requirements listed in EDQW Fact Sheet, Rev. 1.2, Nov. 2106 titled "Bottle Selection and Other Considerations When Sampling For Per- and Polyfluoroalkyl Substances (PFAS)" apply to sample bottles used in the sample preparation and analysis processes in the laboratory?

Answer: Yes, the requirements to utilize only HDPE bottles also apply to bottles used in the preparation and extraction process when preparing QC samples. However, once the extraction has been performed and the PFAS are in solvent, the lab is not required to use HDPE supplies for extract storage, handling and analysis.

Reasoning: HDPE bottles are recommended for sampling because they have shown to have the least amount of adsorption compared to polypropylene. So, in water matrix, PFAS are adsorbed onto the walls of the container, and all QC samples must be treated the same as field samples. Sample extracts are comprised mainly of methanol, a solvent that can retain the analytes in solution (previous FAQ). Because of this, laboratories can use polypropylene in the lab and not have an issue. Plus, there are no HDPE supplies for some of these steps.

DOE

➤ FAQ: QSM Issue: Calibration Performance Checks

Current Text:

The following shall be implemented in addition to TNI section 1.7.1 b):

If a performance check fails, the laboratory can immediately analyze two additional consecutive performance checks (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed performance check and the two additional performance checks). Any corrective actions that change the dynamics of the system requires that all samples since the last acceptable performance check be reanalyzed....Both of these performance checks must meet acceptance criteria in order for the samples to be reported without reanalysis....If either of these two performance checks fail, the associated samples cannot be reported and must be reanalyzed....If the laboratory cannot immediately analyze two performance checks, perform corrective action(s) and repeat the performance check and all associated samples since the last successful performance check....Recalibration must occur if the above scenario fails. All affected samples since the last acceptable performance check must be reanalyzed.

Discussion:

Despite the title of the section in the 2009 TNI Standard, it is misleading to consider Instrument Performance Checks (IPCs) to be analogous to Continuing Calibration Verification (CCV) standards used for environmental chemistry methods. In the 2016 Voting Draft TNI Standard Module 6 this has been corrected, and there is an entirely separate section (1.7.1.3) for Calibration Verification. Essential differences between IPC checks for radiometric measurements and CCVs for environmental chemistry measurements include the following:

1. Unlike environmental chemistry calibration verification standards, the relationship of actual or true activity concentration in the IPC source to the instrument response is not evaluated. The IPC source check is not used for calibration or quantitation. As stated in MARLAP, Chapter 18:

Because the performance check's purpose is to demonstrate that the system's efficiency remains constant, the source's absolute disintegration rate need not be known, provided its purity can be established, its half-life is known, and its activity is sufficient to provide adequate precision. Accordingly, it is not necessary to use a NIST-traceable check source for this purpose. Check sources that are non-NIST-traceable can meet the precision objectives of the performance check and they are less expensive.

The only aspect of instrument performance that is evaluated is the trend or change in instrument response to the same IPC source analyzed over time.

2. IPC sources for some methods are run weekly or even monthly, but they are not routinely analyzed interspersed with samples being analyzed. As such, there are no inherently "associated samples," as there are with environmental chemistry CCVs.
3. Most importantly, the appropriate immediate corrective action for a failed IPC check is not complete recalibration. Instead, the necessary corrective action is what is listed further down in the same section for alpha spectroscopy:

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

Detector efficiency determinations are only one limited aspect of radiation counting equipment calibration. Calibration is typically a labor intensive multi-day effort that includes many other performance characteristics in addition to detector efficiency, such as:

- Channel versus energy calibration of alpha or gamma spectrometers,
- Mass-attenuation calibration of gas-flow proportional or x-ray detectors,
- Quench-efficiency calibration of liquid scintillation detectors,
- Mass-crosstalk calibration of gas-flow proportional detectors,

- Quench-crosstalk calibration of liquid scintillation detectors,
- Efficiency determination, and
- Background determination for each energy region of interest (ROI).

The statement for corrective action for alpha spectroscopy is accurate as far as it goes, and is arguably contradictory to the first six paragraphs of Section 1.7.1, perhaps this could be handled as a sort of clarification, rather than a revision.

Suggested Revision or Clarification:

“When results for instrument performance checks exceed predetermined acceptance criteria (i.e., limit of a statistical or tolerance chart or other QC parameters), the cause of the problem shall be investigated.”

If a performance check fails, the laboratory can immediately analyze two additional consecutive performance checks (immediately is defined as starting a consecutive pair within one hour; no samples can be run between the failed performance check and the two additional performance checks). This approach allows for spurious failures of analytes to be reported without reanalysis of samples. Both of these performance checks must meet acceptance criteria in order for the samples to be reported without reanalysis.

If either of these two performance checks fail, or if the laboratory cannot/does not immediately analyze two performance checks, perform corrective action(s) and repeat the performance check and all associated samples since the last successful performance check. Any corrective actions that change the dynamics of the system requires that all samples since the last acceptable performance check be reanalyzed.

If the problem is not corrected and indicates an intrinsic change in instrument response, then the instrument shall be recalibrated. All affected samples since the last acceptable performance check must be reanalyzed.”

➤ **FAQ: Gamma Spectroscopy**

Current Text:

Module 6, Section 1.8.5.b.i.1 Gamma Spectroscopy:

At least 10,000 net counts (total counts minus the Compton continuum and ambient background) shall be accumulated in each ISO/TNI/DoD/DOE QSM, July 2013 Module 6, Page 163 full-energy gamma-ray peak of interest used for the efficiency equation (ASTM D 3649-98a). Sodium Iodide Detectors: Refer to ANSI N42.12. Efficiencies shall be determined when there is a change in resolution, geometry, or system configuration (ASTM D 3649-98a).

Discussion:

The referenced gamma ANSI standard is out of date. The “new” N-type detectors are sensitive with constant efficiency below the Am-241/59 keV lower limit often seen in purchased energy-efficiency calibration standards since that was close to the functional lower end of the older detectors in the labs when the standard was written. We also note some labs with thin-window gamma detectors or labs which are doing single-isotope calibration curves below 59 keV. We observe labs with N-type detectors will calibrate down to 59 keV (Am-241) but use them below that based on manufacturer’s information which says the efficiency is constant. What is needed is a new criterion for low energy gamma to be added to the QSM. 1) If manufacturer’s information indicates that low-energy (below 59 keV) response is expected to be constant, it would be allowable to use the device down below that point on the curve ONLY if an LCS containing the isotope in question is utilized on every detector in a set to demonstrate that the response has remained as expected. 2) Otherwise, for use of a gamma detector at energies below the lowest calibration point, a single-isotope efficiency curve MUST be established that is isotope-specific.

Suggested Revision or Clarification:

a) Efficiency Calibration Requirements:

i) Each gamma spectrometry system shall be efficiency calibrated for the sample geometry and matrix with NIST traceable or accepted international standards or prepared from NIST/international traceable sources.

Germanium Detectors: Refer to ANSI N42.14 for guidance on isotope specific efficiency and efficiency as a function of energy calibrations. The efficiency calibration approach selected for broad spectrum gamma analysis must cover the energy range of the gamma ray peaks used for nuclide quantification. When establishing an efficiency curve as a function of energy, the efficiency calibration measurements shall be at least six peaks which cover the typical energy range of approximately 0.059 to 2 MeV. At least 10,000 net counts (total counts minus the Compton continuum and ambient background) shall be accumulated in each full-energy gamma-ray peak of interest used for the efficiency equation (ASTM D 3649-98a).

If the detector is to be used for emissions below the lowest energy of a broad spectrum calibration (e.g. below the 0.059 MeV criteria identified above), additional demonstration of acceptable calibration is required. Acceptable approaches include:

- 1) If manufacturer’s information indicates that low-energy response below the lowest energy in the calibration standard is expected to be constant, use of the detector below that point requires check sources or LCSs to contain the isotope to be quantified (or other isotope with lower emission energies). Acceptable recovery must be demonstrated for every detector used for that isotope analysis.
- 2) If low-energy response below the lowest energy calibration standard is not expected to be constant, use of a gamma detector at energies below the lowest calibration point requires that a single-isotope efficiency curve or separate low-energy curve bounding the energy of interest be established for that isotope.

In all cases, the laboratory must be able to demonstrate that sample matrix effects (including potential attenuation from sample containers) on low energy emissions have been accounted for.

Sodium Iodide Detectors: Refer to ANSI N42.12.

Efficiencies shall be determined when there is a change in resolution, geometry, or system configuration (ASTM D 3649-98a).

➤ **DOE Errata Document, QSM Revisions of Rev. 5.0, Rev. 3 (November 30, 2016)**

Requirement 1 - Laboratories shall develop and maintain procedures for sample receiving and login that minimizes changes in thermal preservation.

Requirement 2 – The laboratory shall document if thermal preservation is not maintained during sample receiving and login. The client will be notified in writing if thermal preservation is not maintained. This requirement is for environmental samples and does not apply to industrial hygiene samples (unless the IH method requires thermal preservation).

Requirement 3 – Subcontract laboratories performing analytical services (i.e. testing, data review, data processing, project management, IT support, etc.) for DOE shall be approved in writing by the appropriate DOE or subcontractor client prior to the commencement of work.

Note: EPA has recently promulgated temperature conditions of 0°C to 6°C, with a footnote “above freezing” and the QSM Rev. 5.0, Section 5.5.13.1 f) Table also requires the same temperature ranges. The existing LOI can be used or the revision may be applied.

Revisions to QSM Rev. 5.0 – Volume 1, Module 2, Quality Systems General Requirements

4.6.1 DoD/DOE (Requirement)

Do records for services and supplies that may affect the quality of environmental test include the following, where applicable:

- a) Date of receipt
- b) Expiration date;
- c) Date opened
- d) Source;
- e) Lot or serial number;
- f) Calibration and verification records; and
- g) Accreditation or certification scopes/certificates?

4.11.8 DoD/DOE (Requirement)

The laboratory shall have and use a record system for tracking corrective actions to completion and for analyzing trends to prevent the recurrence of the nonconformance.

Corrective actions developed to address findings during DOECAP audits must be implemented. Any changes to reviewed corrective action plans must be reviewed by the DOECAP Operations Team, as appropriate.

4.11.8 DoD/DOE (Guidance)

Willful avoidance of corrective actions may result in a DOECAP Priority I finding.

4.12.3 DoD/DOE (Requirement)

Are the following requirements implemented?

- i) Are individuals who are making corrections to records dating and initialing the corrections?
- ii) Are corrections due to reasons other than transcription errors specified?
- iii) Do records for changes made to data (either hardcopy or electronic) include the identification of the person who made the change, and the date and time of the change?

5.8.4 c) v) QSM DoD/DOE (Requirement)

Does the facility have a calibrated, backup radiological survey meter that can be used in the event that the other survey meter is inoperative or that the calibration has exceeded?

Addendum to QSM 5.8.9 DoD/DOE (Requirement)

The laboratory shall obtain certificates of disposal or destruction for all DOE samples and waste submitted to a TSDF

Revisions to QSM Rev. 5.0 - Radiochemical

1.8.2 Radon Scintillation (Lucas Cell)

a) Laboratory SOPs for sample analyses by Lucas Cell shall incorporate and adhere to ASTM D3454 (current version), *Standard Test Method for Radium-226 in Water*. Performance shall be in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. Reference is to the current version of the method. When references are updated, an implementation schedule shall be determined by the lab.

1.8.3 Liquid Scintillation Counting

c) Laboratory SOPs for methods using liquid scintillation counting shall incorporate and adhere to ANSI N42.15 (current version), American National Standard Check Sources for and Verification of Liquid Scintillation Systems. Performance shall be in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. References are for the current version. When references are updated, an implementation schedule shall be determined by the lab.

1.8.4 Gas Flow Proportional Counting

b) Instrument Calibration: Shall be performed in accordance with the requirements in ANSI N42.25 (current version), Calibration and Usage of Alpha/Beta Proportional Counters. Calibration shall be performed in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. References are for the current version. When references change, an implementation schedule shall be determined.

1.8.4 Gas Flow Proportional Counting (ALTERNATE)

b) Laboratory SOPs for sample analysis by gas flow proportional counting shall incorporate and adhere to ANSI N42.25 (current version), Calibration and Usage of Alpha/Beta Proportional Counters. Performance shall be performed in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. References are for the current version. When references change, an implementation schedule shall be determined.

1.8.5 Gamma Spectrometry

a) Sample Counting Requirements:

i) Laboratory SOPs for sample analysis by gamma spectrometry shall incorporate and adhere to ANSI N42.14 (current version), Calibration and Use of Germanium Spectrometers for the Measurement of Gamma Ray Emission Rate of Radionuclides, and/or ANSI N42.12 (current version), Calibration and Usage of Thallium-Activated Sodium Iodide Detector Systems for Assay of Radionuclides. . Performance shall be in accordance with the standard unless otherwise defined in this document or as documented by the laboratory and accepted by clients. References are for the current version. When references change, an implementation schedule will be determined.