QCP3

PREPARATION, CONTROL, AND TRACEABILITY OF STANDARDS

1.0 PURPOSE

To establish procedures for the preparation, control, and traceability of radioactive standards used by the radiochemical and radiophysics laboratories.

2.0 RESPONSIBILITIES

2.1 Laboratory Manager or designee

2.1.1 Identify needs for new/additional laboratory chemistry standards and initiate purchase requisitions.

2.1.2 Authorize purchase of standard materials for laboratory operations by approving a purchase requisition.

2.1.3 Maintain a logbook for certified and working standards with the supporting certificates and associated paperwork. The logbook may or may not be electronic.

2.1.4 Ensure that the data required in steps 4.13 or 5.10 are recorded in the logbook and/or any required paperwork.

2.1.5 Review and approve, in the logbook, when a working standard is prepared.

2.1.6 Provide the proper training to analysts for the preparation of standards for laboratory operations.

2.1.7 If the half life of the isotope of interest is less than five years, determine the expiration date of the standard as required in steps 4.3 and 5.3, otherwise, there is no expiration date for the standard and this must be noted on the label for the standard.

2.1.8 Maintain storage area for all standards and tracers.

2.1.9 Initiate disposal of standards, as appropriate, due to questionable content, damage, expiration, etc.

2.1.10 Provide the proper training to analysts for the preparation of specific media and geometries for counting room standards.

2.1.11 When a revision is made to this procedure, review of standards and tracers is to be performed to ensure previously prepared solutions meet the requirements of the new procedure.

2.1.12 When changes are required as a result of the review process in 2.1.11, the changes will be properly documented in the appropriate logbook and the labels of the affected standards and tracers will be updated.

2.2 Analyst

2.2.1 Use only those standards and tracers approved by the manager or his designee and are located in the designated storage area for active standards and tracers. If there is a question concerning whether or not a standard or tracer is active, consult the manager or his designee.

2.2.2 After training, prepare standards and/or tracers following the steps in this procedure.

2.2.3 Fill in the appropriate standard and/or tracer information in the appropriate standard and/or tracer logbook for review and approval by the manager or his designee, as required by this procedure.

3.0 TRACEABILITY

3.1 All standards, used for calibration of laboratory and radiometric operations, shall be traceable to the National Institute of Standards and Technology (NIST).

3.2 For the purposes of this procedure, standards as they are received from the supplier, i.e. undiluted, are certified standards; dilutions of certified standards are called working standards; Working standards are either primary working standards made directly from a certified standard or a secondary working standard made from a primary working standard. Either type of working standard may be used to spike various media, matrices, or special geometries, in which case the spiked sources are considered working standards.

4.0 PREPARATION OF PRIMARY WORKING STANDARDS

4.1 Select the desired certified standard (based on radionuclide, concentration, matrix, etc.). Take necessary protective precautions based on activity levels specified in QCP6 for contamination control. Contact the safety office before handling any activity greater than 1 μCi.

4.2 Calculate the desired dilution; obtain a clean volumetric flask.

4.3 Determine the standard's expiration date as follows:

The standard's expiration date is at the end of three half-lives. Standards with half-lives which are greater than five years do not require calculation of an expiration date. When an expiration date is not required for a standard, this must be noted on the label for the standard.

4.4 Determine the appropriate diluting agent from the standard certificate.

4.5 Check the integrity of the flask that is to be used to dilute the standard. Weigh the empty flask, add water to the fill line, and weigh the flask with water. These data are recorded in the logbook for the isotope of interest. The net weight of the water in grams should be approximately equal to the volume of the flask in milliliters, considering the density and temperature.

4.6 Break the top off of the ampoule/container (it may be necessary to etch the neck of an ampoule to obtain a clean break). Use caution to keep foreign matter, including finger prints, from the container surface.

CAUTION: Broken glass offers potential for internal contamination. Use extreme caution while handling broken glass.

4.7 Using a small plastic pycnometer with a narrow capillary end, uptake as much of the solution as possible from the ampoule.

4.8 Weigh the pycnometer with the solution on an analytical balance. Record the weight in the logbook for the isotope of interest.

4.9 Transfer the solution to the volumetric flask.

4.10 Weigh and record the weight of the pycnometer in the logbook for the isotope of interest.

4.11 Bring the solution up to volume using the diluting solution. Either, add a stirring bar to the container and stir to mix or place a stopper on the flask and invert and shake several times to mix the solution.

4.12 Label the container with radionuclide, standard number, diluting solution, activity concentration, two sigma uncertainty, and expiration date, if necessary. If the solution has no expiration date, this will be noted in the logbook of the solution and on the label of the solution.

4.13 Record the following information in the logbook and/or any required paperwork for isotope of interest:

- Name of radionuclide
- Vendor name
- Standard Identification number
- Standard matrix
- Standard reference date
- Standard expiration date, refer to 4.3
- All pertinent measured values
  1) pycnometer and solution weight
  2) pycnometer empty weight
  3) final dilution volume
- All pertinent calculations and calculated values

1) solution weight
2) diluted solution activity on reference date

- Name of person performing dilution and date performed

4.14 Notify the Laboratory Manager or designee the standard preparation is complete and that the information associated with the standard is ready for review and approval.

4.15 Store the primary working standard in the designated limited access area or cabinet.

4.16 Continued acceptability of primary standards and tracers will be monitored by evaluation of secondary working standard and tracers. When secondary working standard and tracers cannot be prepared within the acceptance limits of the uncertainties, the primary solution or solutions will be taken out of service or retested according to section 9.0 of this procedure. Head space in the standard or tracer container will be monitored during each use and reduced volumes transferred to a container with a capacity close to the remaining solution volume.

5.0 PREPARATION OF SECONDARY WORKING STANDARDS AND TRACERS

5.1 Select the desired primary working standard (based on radionuclide, concentration, matrix, etc.). Take necessary protective precautions based on activity levels specified in QCP6 for contamination control.

5.2 Calculate the desired dilution; obtain a clean diluting container.

5.3 Determine the standard's expiration date as follows:

The standard's expiration date is at the end of three half-lives. Standards with half-lives which are greater than five years do not require an expiration date. When there is no expiration data required for a standard, this must be noted on the label for the standard.

5.4 Determine the appropriate diluting agent from the standard certificate.

5.5 Check the integrity of the flask that is to be used to dilute the standard. Weigh the empty flask, add water to the fill line, and weigh the flask with water. The net weight of the water in grams should be approximately equal to the volume of the flask in milliliters, considering the density and temperature.

5.6 Select a pipette that will deliver the desired volume of the primary working standard. Check the pipette by weighing water of the desired volume and record the weight of the water in the logbook for the isotope of interest. The net weight of the water in grams should be approximately equal to the volume of the pipette in milliliters, considering the density and temperature.

5.7 Using the selected pipette dispense the required volume of the working standard into a tarred flask.

5.8 Bring the solution up to volume using the diluting solution. Either add a stirring bar and stir to mix or place a stopper on the flask and invert and shake several times to mix the solution.

5.9 Label the container with radionuclide, standard number, diluting solution, activity concentration, two sigma uncertainty, and expiration date, if necessary.

5.10 Record the following information in the logbook and/or any required paperwork for isotope of interest:

- Name of radionuclide
- Vendor name
- Standard Identification number
- Standard matrix
- Standard reference date
- Standard expiration date, to refer 5.3
- All pertinent measured values
  1) flask and solution weight
  2) flask tare weight
  3) final dilution volume
- All pertinent calculations and calculated values
  1) solution weight
  2) diluted solution activity on reference date
- Name of person performing dilution and date performed

5.11 Notify the Laboratory Manager or designee the standard preparation is complete and that the information associated with the standard is ready for review and approval.

5.12 Store the secondary working standard or tracer in the designated limited access area or cabinet.

5.13 Continued acceptability of working standards and tracers will be monitored by evaluation of process control chart trends for the measured to known ratios of standards and for the tracer yield percentages. When unacceptable trends are noted the solution will either be taken out of service or retested according to section 9.0 of this procedure. Head space in the standard or tracer container will be monitored during each use and the volume transferred to a container with a capacity close to the remaining solution volume

6.0 CALCULATIONS

6.1 All calculations are to be documented in a logbook, which may be either electronic or bound, for the isotope of interest.

6.2 All activities and/or concentrations are to be stated in picocuries (pCi) or pCi/unit.

6.3 Since the activity and/or concentration of standards are sometimes stated in

Becquerel (Bq) and/or Bq/unit, all conversions of activity and/or concentration from Bq to pCi will be performed using the conversion factor 0.037 Bq/pCi.

7.0 VERIFICATION OF STANDARDS AND TRACERS

7.1 Once the standards and tracers have been prepared, a minimum of 6 and up to 10 aliquots will be prepared to verify the activity of the solution.

7.2 The aliquots that are used to verify the activity of the solution should be prepared with minimum processing to ensure the integrity of each aliquot.

7.3 Once the aliquots are prepared, the aliquots are submitted to the count room to be measured on the appropriate counting instrument.

7.4 After sample counting has been completed, the activity of each aliquot will be determined. The average of the aliquot activities will be compared to the activity of the solution provided by the vendor on the certificate to determine if the solution has been properly prepared.

7.5 If the measured activity of the solution aliquots is in agreement with the vendor provided certificate activity value at the two-sigma uncertainty level, the solution is ready for use in routine work.

7.6 The Laboratory Manager or designee will review and approve the standard and/or tracer information before usage.

7.7 After the solution is determined ready for use, the 1 sigma standard deviation of the 6-10 measurements will be propagated with the 1 sigma uncertainty supplied by the vendor, and the 1 sigma uncertainty associated with the half life to generate an analytical uncertainty appropriate for the solution. The result will be converted to 2 sigma for use in the propagation of individual sample concentration uncertainties.

7.8 If it is necessary to use a solution that is not NIST traceable, the solution will be standardized against another solution that is NIST traceable using methodology that is appropriate for the type of emission. The methodology will be documented as part of the verification process. The Laboratory Manager or designee will review and approve the methodology to ensure the solution can be properly standardized.

8.0 CONTROL OF STANDARDS

8.1 The Laboratory Manager or designee will have control of the area and/or cabinet in which all certified and working standards are stored. The analytical staff will have access to the storage area.

8.2 Upon receipt of a standard, the following information shall be entered into the logbook for isotope of interest.

- Name of Radionuclide
- Vendor Name
- Standard Identification Number
- Standard Matrix
- Standard Reference Date
- Standard Expiration Date, refer to 4.3

8.3 Notify the Laboratory Manager or designee the standard preparation is complete and that the information associated with the standard is ready for review and approval.

8.4 Access to all standards will be controlled. Certified and working standards will be stored in a designated location with access limited to the laboratory staff. These standards may be removed from this area for the following reasons:

- Preparation of calibration standards for the various counting instrumentation.
- Dispensing tracer solutions for those analytical procedures requiring internal tracers for the determination of chemical recovery.
- Preparation of spikes and matrix spikes for QC requirements.

8.5 Working standards must be appropriately identified and labeled. Working standards will be stored in the designated limited access area or cabinet.

8.6 Standards will be removed for disposal when the expiration date is reached or the standard is no longer considered dependable for any reason.

9.0 STANDARD AND TRACER QUALITY

9.1 The quality of the standards will be monitored through the evaluation of process control charts associated with the radioanalytical procedures. When this monitoring process indicates a problem with a standard or a tracer, the solution will be replaced when it is certain that NIST traceability cannot be maintained or for any other reason that brings the integrity of the solution into question.

9.2 All standards and tracers will be verified once each year using three verification measurements. The verification will be deemed acceptable if the verification value is within 5% of the decay corrected concentration. The two sigma value used for the 95% confidence interval of the mean shall not exceed 10% of the mean value of the three verification measurements. The verification results for each standard and tracer will be maintained in a logbook.

9.3 The precision and bias of each standard will be monitored through quarterly evaluation of the process control data.

9.4 At no time can a standard or tracer have certification value other than the value on the original certification with corrections for weighing and solution dilutions for routine use in an analytical procedure.

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STANDARDS, TRACERS, AND WORKING SOLUTION LOG

Isotope: _____ Recommended Half Life: _____

Vendor: _____ Half Life Uncertainty (1s): _____

Certified Standard ID Number: _____ Matrix: _____

Stated Activity of Certified Standard: _____ Date Received: _____

Stated Uncertainty of Certified Standard (1s): _____ Expiration Date (only if half life is less than five years): _____

Reference Date: _____

Primary Working Standard:_____ OR Secondary Working Standard:_____

Primary Working Standard ID Numer:_____ Secondary Working Standard ID Number:_____

Dilution Calculations for Primary Working Standard

	Mass		Uncertainty	Relative Error (RE)
Pycnometer + Solution(g):				
Pycnometer(g):				
Solution(g):				

Dilution Volume:_____ Diluent:_____

Vol. Flask + Water (g): _____

Vol. Flask (g): _____ Volumetric Flask OK to Use?_____

Solution (g): _____

Use space below to hand calculate the activity of the primary working standard

Activity in pCi/mL of primary working solution on reference date:

Concentration	TPU (1σ)	Relative Error
_____	_____	_____

Dilution Calculations for Secondary Working Standard

Primary Working Standard ID number used to prepare this Secondary Working Standard:_____

Volume and Mass of Primary Working Standard used to prepare this Secondary Working Standard:_____

Dilution Volume:_____ Diluent:_____

Vol. Flask + Water (g): _____

Vol. Flask (g): _____ Volumetric Flask OK to Use?_____

Solution (g): _____

Pipette Check Pipette OK to Use?_____

Volume water: _____

Weight water: _____

Use space below to hand calculate the activity of the secondary working standard

Activity in pCi/mL of secondary working solution on reference date:

Concentration	TPU (1σ)	Relative Error
_____	_____	_____

Prepared By:_____ Date:_____

Reviewed By:_____ Date:_____

The label must meet the requirements of steps 4.3 for Primary Working Standards or 5.3 for Secondary Working Solutions and Tracers.

Prepared By:_____ Date:_____

Reviewed By:_____ Date:_____