QCP5

ANALYTICAL QUALITY CONTROL AND SAMPLE FLOW

1.0   PURPOSE

To provide a procedure for maintaining effective sample flow and Quality Control (QC) samples for analytical radiochemical procedures. Client specifications are followed as requested.

2.0   RESPONSIBILITIES

Laboratory Staff will maintain control of sample flow, incorporate QC samples in analyses, and evaluate QC results.

3.0   PROCEDURE

   3.1   Non-destructive analyses

        3.1.1   Samples flow through non-destructive analyses on an ongoing basis.

        3.1.2   Quality Control

Quality Control Activity	Frequency	Acceptance Criteria
Background: empty chamber count	Weekly	Within 3 σ of established limits for defined regions of interest and for full spectrum background
Reproducibility Check: count reference material of known activity	Daily	Within 3 σ of known

Analyses for which quality control results do not meet these guidelines will be evaluated by the laboratory staff in conjunction with cognizant project staff. Information such as data end use and sample matrix characteristics will be used to determine whether reanalysis is necessary. In all such cases, explanatory comments will be added to the data sheets and project files.

   3.2   Chemical analyses

3.2.1   Sample Flow/Batching

Samples flow through chemical procedures in batches. Batches are used to monitor sample flow and ensure quality control. Upon receipt of a Laboratory Work Request, the analyst establishes batches as follows:

C   Batches consist of samples to be analyzed by the same procedure for a common set of parameters.

C   Batches may range from 1-20 samples, based on the number of analyses requested, sample matrix, analytical parameters, and the level of QC required.

C   The Database (DB) automatically assigns a batch identification (ID) number. This number is the next sequential number in the DB. The ID number for the batch, sample identification, and associated QC samples (as required by step 3.2.2), are recorded in the DB.

Batches will be handled as follows:

C   Analyze samples in a continuous, sequential manner; do not interrupt by processing samples from other batches. Analyze in the same area of the laboratory.

C   Use the same lots of reagents, if possible.

3.2.2   Quality Control

Type	Frequency	Acceptance Criteria
Method Blank	One per batch	Established process control limits
Laboratory Control Standard (LCS)	One per batch	Within 50% of known value for gross alpha/beta and for non-routine procedures. Within 20% of known value for all other routine procedures.
Chemical Recovery (Including BMO analyses)	Per sample or at least 1 per batch	Isotopic 30 - 110% Stable 40 - 110%

C   Method Blank - An analytical control, consisting of all reagents and internal standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

C   Laboratory Control Standard (LCS) - NIST traceable materials or other industry accepted standards and reference materials (e.g., NRM, TRM).

C   Chemical Recovery/Yield - for chemical analyses. This is a measurement of the fraction or percent of analyte present at the completion of the procedure.

C   The review of the analytical data will include persistent negative data for a batch and negative data for a single sample that is outside the negative 3 sigma limit. The review of negative data will determine if the cause of the negative data is related to a systematic error or a random error. If the cause is systematic, it shall be corrected before submitting data. If the cause is random, it shall be documented in the Case Narrative for data submitted under a DOE Sample Management Office statement of work.

3.2.3   Matrix spike, matrix spike duplicate, and/or duplicate samples may be analyzed to demonstrate sample characteristics or to meet DOECAP QC requirements. The following equations are used to evaluate the validity of the analyses.

Matrix spikes

$$MS\%Rec = \frac{|MS - S|}{MSKnown} * 100$$

$$U_{MS\%Rec} = MS\%REC * \sqrt{RMS^2 + RS^2 + RMSKnown)^2}$$

Matrix spikes are considered as being accepted if the result of the calculations is ± 25 percent of the known concentration.

Matrix spikes

$$MSD\%Rec = \frac{|MSD - MS|}{MSDKnown} * 100$$

$$U_{MSD\%Rec} = MSD\%REC * \sqrt{(\frac{U_{MS}}{MS})^2 + (\frac{U_{MSD}}{MSD})^2 + (\frac{U_{MSKnown}}{MSKnown})^2}$$

Matrix spike duplicates are considered as being accepted if the result of the calculations is ± 25 percent of the known concentration.

Duplicates:

$$RER = \frac{|S - D|}{\sigma_{95D} + \sigma_{95S}}$$

Radiochemical replicate determinations shall agree when the 95 percent confidence level uncertainties are considered. That is, the RER shall be less than or equal to one. This control criterion is not applied, and reanalysis or data qualification are not required, when both of the measured values are less than their associated MDCs.

Where:

MS	=	Matrix Spike measured concentration	
S	=	Sample concentration	
σ_{95S}	=	Sample 2σ Uncertainty	
D	=	Duplicate concentration	
σ_{95D}	=	Duplicate 2σ Uncertainty	
MSKnown	=	Matrix Spike Known concentration	
MS%Rec	=	Matrix Spike percent recovery	
MSD	=	Matrix Spike Duplicate measured concentration	
MSDKnown	=	Matrix Spike Duplicate Known concentration	
MSD%Rec	=	Matrix Spike Duplicate percent recovery	
RMS	=	1σ relative uncertainty of MS concentration	
RS	=	1σ relative uncertainty of Sample concentration	
RMSKnown	=	1σ relative uncertainty of MS Known concentration	
RMSDKnown	=	1σ relative uncertainty of MSD Known concentration	
RER	=	Relative Error Ratio	
$U_{MS\%Rec}$	=	1σ Uncertainty of Matrix Spike percent recovery	
$U_{MSD\%Rec}$	=	1σ Uncertainty of Matrix Spike Duplicate percent recovery	

3.2.4   When analyses are complete samples are placed in an active storage area pending archival or other disposition.

4.0   SAMPLE FLOW

The diagram on the next page outlines typical sample flow.

IEAV Sample Flow and Control Diagram

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                    ┌─────────────────────────┐
                    │ Sample collected or      │
                    │ received from other      │
                    │ organization             │
                    └───────────┬─────────────┘
                                ▼
                    ┌─────────────────────────┐
                    │ Initiate chain of custody│
                    └───────────┬─────────────┘
                                ▼
                    ┌─────────────────────────┐
                    │ Screen for contamination │
                    │ level                    │
                    └───────────┬─────────────┘
                                ▼
                    ┌─────────────────────────┐
                    │ Log-in                   │
                    └───────────┬─────────────┘
                                ▼
                    ┌─────────────────────────┐
                    │ Custody and samples      │
                    │ transferred to laboratory│
                    └───────────┬─────────────┘
                                ▼
  ┌──────────────┐    Yes   ◇ Special
  │ Prepare      │◄─────────◇ precautions
  │ procedures   │          ◇ necessary
  └──────┬───────┘              │ No
         └──────────────────────▼
                    ┌─────────────────────────┐
                    │ Laboratory work request  │
                    │ submitted                │
                    └───────────┬─────────────┘
                                ▼
                    ┌─────────────────────────┐
                    │ Assemble batches         │
                    └───────────┬─────────────┘
                                ▼
                    ┌─────────────────────────┐
                    │ Prepare and analyze      │
                    │ samples                  │
                    └───────────┬─────────────┘
                                ▼
                         ◇ Sample    Yes ────────────►┌──────────────┐
                         ◇ consumed                   │ Complete     │
                             │ No                     │ disposition  │
                             ▼                        │ form         │
                         ◇ Sample    Yes ────────────►│              │
                         ◇ returned                   └──────┬───────┘
                         ◇ to originator                     │
                             │ No                            │
                             ▼                               │
                         ◇ Portion of  Yes ──►┌────────────┐ │
                         ◇ sample removed      │ Note on    │ │
                             │                 │ sample     │ │
                             │ No              │ labels     │ │
                             ▼                 └────────────┘ │
                         ◇ Sample to be  ──►┌──────────┐  ┌─────────┐
                         ◇ archived          │ Complete │  │ Archive │
                             │               │ archival │  └─────────┘
                             │               │ request  │
                             │               └──────────┘
                             │                Yes  ┌──────────────┐
                             │ No             ◄─────│ Periodic     │
                             ▼                      │ review for   │
                    ┌──────────────────┐            │ disposal     │
                    │ Obtain disposal  │            │ option       │
                    │ approval         │            └──────────────┘
                    └────────┬─────────┘
                             ▼
                    ┌──────────────────┐
                    │ Dispose of sample│
                    └────────┬─────────┘
                             ▼
                    ┌──────────────────┐
                    │ Notify archival  │◄──────────
                    │ coordinator      │
                    └──────────────────┘
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