**Analyte List**: 90Sr

**Analytical Protocol Specifications**

**Analysis Limitations**: Perform direct measurement of analyte. Analysis of progeny allowed if radioactive equilibrium is established at laboratory from freshly isolated parent.

**Matrix**: Raw Cow’s Milk (fat content to vary)

**Possible Interferences**: Fresh beta-emitting, fission-product nuclides if purification steps are inadequate or non-existent.

**Concentration Range**: 1 to 50 pCi/L **Action Level**: 8 pCi/L

**Method Validation Level**: MARLAP Levels A, C, or D as applicable. See Attachment C for details.

**MQOs**: A required method uncertainty (*u*MR) of 0.5 pCi/L at 8 pCi/L and a relative method uncertainty (φMR) of 6.25% at > 8pCi/L

|  |  |  |
| --- | --- | --- |
| **QC Samples** | | |
| **Type** | **Frequency** | **Evaluation Criteria** |
| Method blank | 1 per batch | See Attachment B |
| Duplicate | 1 per batch | See Attachment B |
| Matrix Spike\* | 1 per batch | See Attachment B |
| Laboratory Control Sample | 1 per batch | See Attachment B |

|  |  |
| --- | --- |
| **Analytical Process Requirements** | |
| **Activity** | **Special Requirements** |
| Field Sample Preparation and Preservation | Sample size > 3.5 L; Preserve on ice or with 5 mL of 37% formaldehyde / L sample |
| Sample Receipt and Inspection | Rad survey samples upon receipt. Return sample receipt acknowledgment letter with date of receipt at lab. Cross index list for Sample ID and assigned Lab ID. Visually inspect containers upon receipt to ensure integrity and normal sample appearance. COC documentation applies. |
| Laboratory Sample Preparation | Take sufficient aliquant of sample after gamma-ray spectrometry analysis (see separate requirements in the gamma spectroscopy APS). Keep 1 liter as backup until analytical results have been approved by project manager. |
| Sample Dissolution | None |
| Chemical Separations | Isolate Sr by cation exchange resin, or precipitation of Sr from soured or dry-ashed milk. Separation from Ca is essential. Rare earth and Ba scavenging steps are necessary to eliminate possible interferences from fresh fission products. |
| Preparing Sources for Counting | Final source mount to accommodate nuclear instrumentation. |
| Nuclear Counting | Acceptable counting instrumentation includes: Liquid Scintillation Counter, Gas Proportional Counter or Solid State Beta Detector.  Detection. Method must discriminate against potential 89Sr  interference by physical means and/or calculation. |
| Data Reduction and Reporting | See Attachment A |
| Sample Tracking Requirements | Chain-of-Custody |
| Other - Chemical Yielding | Gravimetric (must have 99% Ca removal) or 85Sr tracer with > 90% Ca removal. |

\* Spiking range provided in Attachment B

**Attachment A**

**Data Reduction and Reporting Requirements**

**Data Reduction**

1. Calculation of Sr-90 activity or concentration (pCi/L) can be based on the quantification of Sr-90 and/or Y- 90, with proper addressing of decay and ingrowth of Y-90.
2. Calculation of the associated combined standard uncertainty (pCi/L) of the 90Sr concentration.
3. Calculation of the MDC, in terms of pCi/L, shall be sample specific using the detector efficiency and background, counting time, decay and ingrowth factors, Sr yield and sample volume used for the analysis.
4. Calculation of critical level, in terms of pCi/L, shall be sample specific.
5. Calculation of gross, net and background count rate, detector efficiency, chemical yield, decay and ingrowth factors for each sample.
6. Initial review and approval of data reduction equations shall be established during a desk or onsite audit as part of the lab approval/contracting process.
7. No changes in the equations used in data reduction shall be initiated without prior approval of the project manager.

**Data Reporting**

1. For each sample, the following sample specific parameters shall be reported:

Batch #, Sample ID, Lab ID, sample collection (reference) date, sample receipt date, estimated (or actual) sample volume received, 90Y separation date, counting date, cross reference to batch QC samples, SOP used, analyst, data reviewer and report date.

1. For each sample, the following sample processing parameters or factors shall be reported:

Gross, net and background count rates, detector efficiency, sample volume processed, 90Sr decay factor, 90Y decay and ingrowth factors (and separation and count times), and chemical yield factor.

1. For each sample the following calculated information will be reported:

critical level, MDC, 90Sr concentration and associated combined standard uncertainty (CSU).

1. Batch quality control results for the laboratory control sample (LCS), method blank, duplicate sample and matrix spike sample shall be reported with each batch of samples:

Reporting data to include:

LCS - calculated sample and prepared spike concentration with associated CSUs, and percent difference between sample result and known value

Duplicate samples - calculated concentrations with associated CSU for both samples

Matrix spike - calculated sample and known spike concentration with associated CSUs, and percent difference between sample results

1. A “Narrative” shall be provided with each batch of samples that describes processes used and any problems encountered or discrepancies noted, including the possible effect on the quality of specific results and actions taken to remedy the problem if recurrent.
2. Reports shall be provided electronically and as a hard copy. An electronic data format will be provided.

**Attachment B**

**Batch Quality Control Sample Evaluation Criteria**

A “batch” of samples is defined as 20 or fewer samples not including the QC samples. The results of the batch QC samples shall be evaluated according to the equations provided below. It should be noted that no action is to be taken when a “not-to-exceed” limit stated below is exceeded for an individual sample. However, if trending of the results indicates multiple results or a trend of results exceeds a limit, stop processing samples and take action to identify and correct the root cause of the problem. Sample processing may resume when corrective actions have been shown to be effective in eliminating the cause of the problem. It is expected that the Laboratory’s QA officer and project manager shall provide oversight on the sample processing and track the batch QC results.

**Laboratory Control Sample**

The 90Sr spike concentration of an LCS shall be between 10 and 20 pCi/L and the spiking uncertainty should be

≤ 5%. The percent deviation (%D) for the LCS analysis is defined as

%*D*  *SSR*  *SA* 100% 1)

*SA*

where

SSR is the measured result (spiked sample result) and SA is the spike activity (or concentration) added.

The %D control limit is ± 3 φ*MR* × 100% or ±19%. For long-term trending, the %D results should be plotted graphically in terms of a quality control chart with the expected mean %D value of zero.

**Duplicate Samples**

The acceptance criteria for duplicate analysis results depends on the analyte concentration of the sample, which is determined by the average *x* of the two measured results *x*1 and *x*2.

*x*  *x*1  *x*2 2)

2

When *x* < 8, the control limit for the absolute difference | *x*1 – *x*2 | is 4.24 *u*MR, or 2.1. When *x* ≥ 8 pCi/L, the control limit for the *relative percent difference* (RPD), defined as,

RPD  *x*1  *x*2 100% 3)

*x*

is 4.24 φMR × 100% or 27 %. For long-term trending, the absolute difference and RPD results should be plotted graphically in terms of a quality control chart with an expected absolute difference and RPD mean values of zero.

**Attachment B (Continued)**

**Batch Quality Control Sample Evaluation Criteria**

**Matrix Spikes**

The acceptance criteria for matrix spikes uses the “*Z* score,” defined below, as the test for matrix spikes. The pre-existing activity (or concentration) must be measured and subtracted from the activity measured after spiking as shown in equations 4) and 5). The 90Sr spike concentration of a matrix spike shall be between 10 and 20 pCi/L and the spiking uncertainty should be ≤ 5%.

*Z*  4)

*SSR*  *SR*  *SA*

*φ SSR*2  max(*SR*,*UBGR*)2

*MR*

*Z*  *SSR*  *SR*  *SA* 5)

0.0625 *SSR*2  max(*SR*,8)2

where:

SSR is the spiked sample result, SR is the unspiked sample result,

SA is the spike concentration added (total activity divided by aliquant mass), and max(SR,8) denotes the maximum of SR and 8 pCi/L.

The control limit for *Z* is set at ± 3. It is assumed that the uncertainty of SA is negligible with respect to the uncertainty of SSR. For long-term trending, the Z results should be plotted graphically in terms of a quality control chart with a Z value of zero as the expected mean value.

**Method Blanks** When an aliquant of a blank material is analyzed, the target value is zero. However, the measured value may be either positive or negative. The applicable control limit for blank samples shall be within ± 3 *u*MR or ± 1.5 pCi/L. For long-term trending, the blank results should be plotted graphically in terms of a quality control chart with an expected mean value of zero.

**Attachment C**

**Method Validation Requirements**

Prior to processing any milk samples, the laboratory is required to validate its 90Sr in cow's milk radioanalytical method according to the specifications stated in MARLAP Chapter 6. The level of method validation will depend on whether the laboratory has a previously validated method for 90Sr in milk (Level A), will modify a previously validated 90Sr method for a milk matrix (Level C) or must newly develop or adapt a method for 90Sr in cow's milk (Level D). The laboratory shall submit the method validation documentation to the project manager for review and approval prior to the acquisition of a laboratory contract. A summary of the method validation criteria is presented below for the three validation levels.

Level A method validation pertains to a previously validated method for 90Sr in milk. No additional testing is required if the method previously has been successfully validated and the available method validation documentation has been reviewed and approved by the project manager. Documentation of method validation should conform to the specifications provided below.

Level C method validation is to be conducted when a validated 90Sr method for a non-milk matrix is modified for applicability for the milk matrix, e.g., when the EPA 905 90Sr in water method is modified for use with a milk matrix. A method validation plan should be developed and documented. Validation Level C requires the preparation and analysis of five replicate cowmilk samples (internal performance testing samples) spiked at three different concentrations. For this project the three levels of 1, 10, 20 pCi/L (or within ± 15% of the values) should be used in the validation process. Each sample result for the lowest level (below the action level) must be within ± 2.9 *u*MR or ± 1.45 pCi/L of the spiked concentration value. Each sample result from the two higher spiked levels (above the action level) must be within ± 2.9 φMR × 100% or ± 18% of the spiked concentration value. Documentation of method validation should conform to the specifications provided below.

Level D method validation is to be conducted when a new method is specifically developed or adapted from the literature for the project’s 90Sr in milk application. Validation Level D requires the preparation and analysis of seven replicate cow's milk samples (internal performance testing samples) spiked at three different concentrations. For this project the three levels of 1, 10, 20 pCi/L (or within ± 15% of the values) should be used in the validation process. Each sample result for the lowest level (below the action level) must be within ±

3.0 *u*MR or ± 1.5 pCi/L of the spiked concentration value. Each sample result from the two higher spiked levels (above the action level) must be within ± 3.0 M< R × 100% or ± 19% of the spiked concentration value. Documentation of method validation should conform to the specifications provided below.

**Method Validation Documentation**

Documentation to be submitted to the project manager includes: Method Validation Plan, Method Number, Analyst(s) analyzing the samples, spiked concentration values, experimental results and comparison to the acceptable performance criteria for the validation level.

This page intentionally blank.

**MARLAP TABLE E.6 — Example of a proposal evaluation plan**

**Proposal Evaluation**

*Objective*: To ensure impartial, equitable, and comprehensive evaluation of proposals from contractors desiring to accomplish the work as outlined in the Request for Proposals and to assure selection of the contractor whose proposal, as submitted, offers optimum satisfaction of the government’s objective with the best composite blend of performance, schedules, and cost.

*Basic Philosophy*: To obtain the best possible technical effort which satisfies all the requirements of the procurement at the lowest overall cost to the government.

**Evaluation Procedures**

1. Distribute proposals and evaluation instructions to Evaluation Committee.
2. Evaluation of proposals individually by each TEC member. Numerical values are recorded with a concise narrative justification for each rating.
3. The entire committee by group discussion prepares a consensus score for each proposal. Unanimity is attempted, but if not achieved, the Chairperson shall decide the score to be given.
4. A Contract Evaluation Sheet listing the individual score of each TEC member for each proposal and the consensus score for the proposal is prepared by the Chairperson. The proposals are then ranked in descending order.
5. The Chairperson next prepares an Evaluation Report which includes a Contract Evaluation Sheet, the rating sheets of each evaluator, a narrative discussion of the strong and weak points of each proposal, and a list of questions which must be clarified at negotiation. This summary shall be forwarded to the Contracting Officer.
6. If required, technical clarification sessions are held with acceptable proposers.
7. Analysis and evaluation of the cost proposal will be made by the Contracting Officer for all proposals deemed technically acceptable. The Chairperson of the TEC will perform a quantitative and qualitative analysis on the cost proposals or those firms with whom cost negotiations will be conducted.

**Evaluation Criteria**

The criteria to be used in the evaluation of this proposal are selected before the RFP is issued. In accordance with the established agency policy, TEC members prepare an average or consensus score for each proposal on the basis of these criteria and only on these criteria.

A guideline for your numerical rating and rating sheets with assigned weights for each criteria are outlined next under Technical Evaluation Guidelines for Numerical Rating.

**MARLAP TABLE E.6 (Continued) — Example of a proposal evaluation plan**

**Technical Evaluation Guidelines for Numerical Rating**

1. Each item of the evaluation criteria will be based on a rating of 0 to 10 points. Therefore, each evaluator will score each item using the following guidelines:
   1. *Above normal*: 9 to 10 points (a quote element which has a high probability of exceeding the expressed RFP requirements).
   2. *Normal*: 6 to 8 points (a quote element which, in all probability, will meet the minimum requirements established in the RFP and Scope of Work).
   3. *Below normal*: 3 to 5 points (a quote element which may fail to meet the stated minimum requirements, but which is of such a nature that it has correction potential).
   4. *Unacceptable*: 0 to 2 points (a quote element which cannot be expected to met the stated minimum requirements and is of such a nature that drastic revision is necessary for correction).
2. Points will be awarded to each element based on the evaluation of the quote in terms of the questions asked.
3. The evaluator shall make no determination on his or her own as to the relative importance of various items of the criteria. The evaluator must apply a 0 to 10 point concept to each item without regard to his or her own opinion concerning one item being of greater significance than another. Each item is given a predetermined weight factor in the Evaluation Plan when the RFP is issued and these weight factors must be used in the evaluation.