Analytical Protocol Specifications

Analyte List: ^{3}H

Analysis Limitations: **Don't let samples dry out**

Matrix: **Concrete core**

Possible Interferences: **Fission product β^- emitters**

Concentration Range: **$4.5x10^{-6}$ to $5.8x10^{-1}$ µCi/g**

Action Level: **$5x10^{-5}$ µCi/g**

Discrimination Level: **$4.5x10^{-6}$ µCi/g**

Method Validation Requirements: (Later)

MQOs:

u_{MR} = $[5.0x10^{-5} - 4.5x10^{-6}]/(2.326+2.326]$ = $9.8x10^{-6}$ µCi/g at less than $5x10^{-5}$ µCi/g

$\varphi_{MR} = 100x(9.8x10^{-6}/5x10^{-5}) = 19.6$ % at greater than $5x10^{-5}$ µCi/g

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QC Samples

Type	Frequency	Evaluation Criteria
Blank concrete sample that has been cured for at least two weeks	1 per batch	See attachment
Laboratory QC: Concrete sample made with tritiated water yielding ~**$5x10^{-5}$ µCi/g**	1 per batch	See attachment
Duplicate (will be sent with samples and labelled "Field Duplicate")	1 per batch	See attachment

Analytical Process Requirements*

Activity	Special Requirements
Field Sample Preparation and Preservation	Cores to be labelled so that the end closest to the water interface is marked "T" for top, the other end marked "B" for bottom. Core samples to be immediately placed in plastic containers with gas tight seals. Shipped at 4-8 °C.
Sample Receipt and Inspection	Sample containers checked for integrity and shipping containers monitored for temperature. Samples to be refrigerated upon receipt.
Laboratory Sample Preparation	Each core to be cut in half at laboratory. Top and bottom to be analyzed separately

Sample Dissolution	Each sample (top and bottom halves) to be split in half. One half to be equilibrated with a known quantity of distilled water. Other sample half to be analyzed for water content. {thus, for each core sample there are two samples and two analyzed for water content}
Chemical Separations	STS to be distilled from the concrete. First 20 mL to be discarded, next 100 mL to be collected.
Preparing Sources for Counting	----------
Nuclear Counting	Sufficient volume of distilled sample to be counted using liquid scintillation to achieve a detection limit of 4.5×10^{-6} µCi/g
Data Reduction and Reporting	----------
Sample Tracking Requirements	
Other	

Laboratory Control Sample Control Limits

Control value for LCS = %Deviation from true spike value

= 100x(Sample result – Spike value)/(Spike value)

Control limit = $3 \times \varphi_{MR} = \pm 60$ %

Laboratory Blank Control Limits

Control Limit = $3 \times u_{MR} = 3 \times 9.8 \times 10^{-6} = 2.9 \times 10^{-5}$ µCi/g

Matrix Spike Sample Control Limits

Control Limit value for Z = ± 3

Where Z is calculated for each matrix spike as follows

$$Z = \{SSR - SR - SA\}/[\varphi(SSR^2 + Max(SR, 5 \times 10^{-5} \text{ µCi/g})]$$

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 5×10^{-5} µCi/g.

Duplicate Sample Control Limits

The acceptance criterion for duplicate analysis results depends on the analyte concentration of the sample, which is determined by the average x of the two measured results $x1$ and $x2$.

When $x < 5x10^{-5}$ µCi/g, the control limit for the absolute difference $|x1 - x2|$ is 4.24 uMR, or $4.2x10^{-5}$ µCi/g.

When $x \geq 5x10^{-5}$ µCi/g, the control value for the *relative percent difference* (RPD), defined as,

$$RPD = 100x[x_1 + x_2]/x_{avg} \%$$

is compared to $4.24x\phi$ MR × 100% or 254 %. 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.

Decision Rule:

If the true concentration in the sample is less than the action level ($5x10^{-5}$ µCi/g) it may be released for unrestricted use. Otherwise, further remediation may be required.