

Introduction

The processes involved in the preparation of environmental samples for radiochemical and radiometric assay are an integral part of the measurement process.

Preparation objectives

The overall objective for preparation is to provide the "analyst" with a suitable aliquot of the collected environmental sample for further chemical separation, purification and chemical determination. More specifically, samples should be:

- Fully identifiable
- Provided with the appropriate supporting data (e.g. dry/wet ratios)
- Representative of the original environmental matrix
- Free from contamination (including from other samples)
- Provided in the required form (e.g. edible or other fraction)
- Provided in the required physical state and/or geometry
- Provided to the required customer time-scale



Main processes to facilitate sample preparation

Process (for samples)	Typical Function
Wet Processing	Obtaining a specific fraction for analysis e.g. edible fraction
Dry Processing	Removing water content by controlled drying
Homogeneity Processing	Providing a representative sample by grinding or milling
Ash Processing	Removal of organic material by use of a muffle furnace
Geometry processing	Providing an appropriate counting source e.g. disc or tub



2. Development of ⁹⁹Tc analysis method - Effect of ashing temperature

Recovery of ⁹⁹ Tc (%)	Ashing temperature °C	Recovery of KReO ₄ (%)
100.9 ± 1.2	400	100.2 ± 1.7
99.6 ± 1.5	450	100.0 ± 1.9
98.3 ± 1.8	500	98.6 ± 2.0
87.5 ± 5.4	550	82.2 ± 1.2
41.0 ± 2.2	600	48.8 ± 2.0

B. R. Harvey, K. J. Williams, M. B. Lovett & R. D. libbett (1992). Determination of ⁹⁹Tc in environmental material with rhenium as a yield monitor. J. Radioanal. Nucl. Chem., Articles 158 417-436.

Testing the methodology

I. Demonstration that Preparation processes do not affect assay

Sub sample	Species	Collection Date	Wet weight taken g	²³⁹⁺²⁴⁰ - Pu mBq kg ⁻¹ (wet)	²³⁸ - Pu mBq kg ⁻¹ (wet)	²³⁹⁺²⁴⁰ - Pu / ²³⁸ - Pu
Sellafield 1 #	Crab	10/05/02	206.60	196 ± 3.56%	39.5 ± 6.84%	4.96 ± 7.17%
Sellafield 2 #	Crab	10/05/02	201.90	203 ± 3.69%	46.7 ± 6.68%	4.36 ± 7.03%
Sellafield 3 *	Crab	10/05/02	222.05	190 ± 3.28%	41.2 ± 6.07%	4.61 ± 6.37%

Errors quoted are ± 1σ percentage counting propagated errors only

Tracer added to wet sample, wet ashed and analysed

* Dried and ashed, and tracer added prior to analysis

3. Recent inter-comparison exercises for ¹³⁷Cs determinations in solid materials

Inter-comparison Exercise	Date	Material	CEFAS Result Bq kg ⁻¹ ± 1σ	Target Value Bq kg ⁻¹ ± 1σ	All Reported Results (Range) Bq kg ⁻¹
FSA	2000	Dried Lobster Dried Mussel Dried Liver	95.7 ± 3.1 407 ± 12 1085 ± 32	101.5 ± 15.3 405.4 ± 80.8 1165 ± 175	82.0 - 118.9 305 - 516 930 - 1310
IAEA-414	2001	Dried Fish	5.26 ± 0.28	5.16 ± 0.30	4.3 - 6.3
IAEA Almera Proficiency	2001	Spiked Soil	34.09 ± 0.76	34.9 ± 1.0	NA
AIWG ICL01/03	2002	Dried Grass Dried Cabbage	363 ± 6 18.9 ± 0.5	370 ± 14 19.3 ± 0.7	326 - 404 17.9 - 21.6