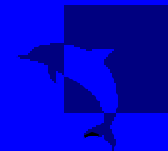


Sample handling and radiochemical analysis

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& I W Croudace
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University
of Southampton

Topics to discuss

- Sample storage and preservation
- Gross alpha/beta measurements
- The importance of dissolution
- Tritium analysis
- Non-radiometric techniques
- Quality control

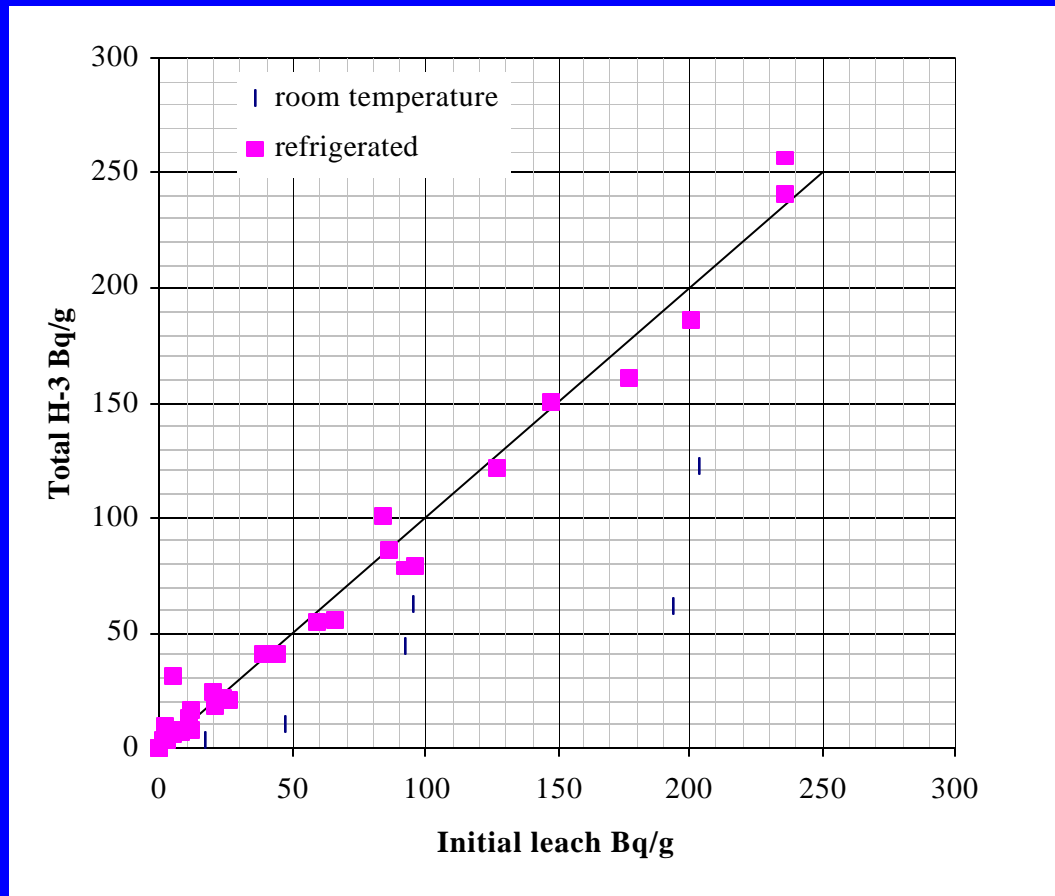
- Communication
- Specification
- Implementation
- Review

Sample storage

Sample preparation

- Storage of sample
 - to prevent sample degradation
(*e.g. volatilisation of ^3H , ^{129}I*)
 - archiving of remaining sample
- Homogenisation and sub-sampling
 - to ensure that a representative sub sample is analysed

Loss of ^3H



Room temperature samples
stored for 13 days

Gross alpha/beta analysis

Gross alpha / beta screening

A screening technique only

Results will be dependent on...

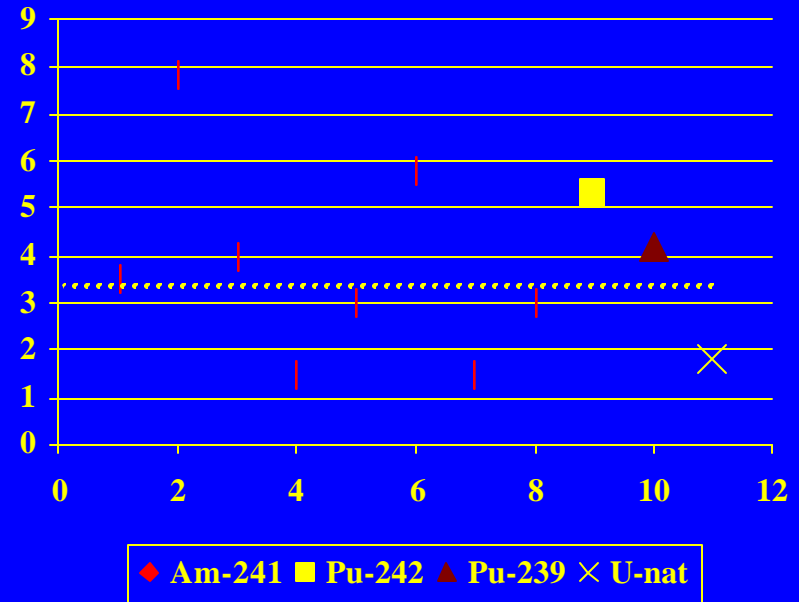
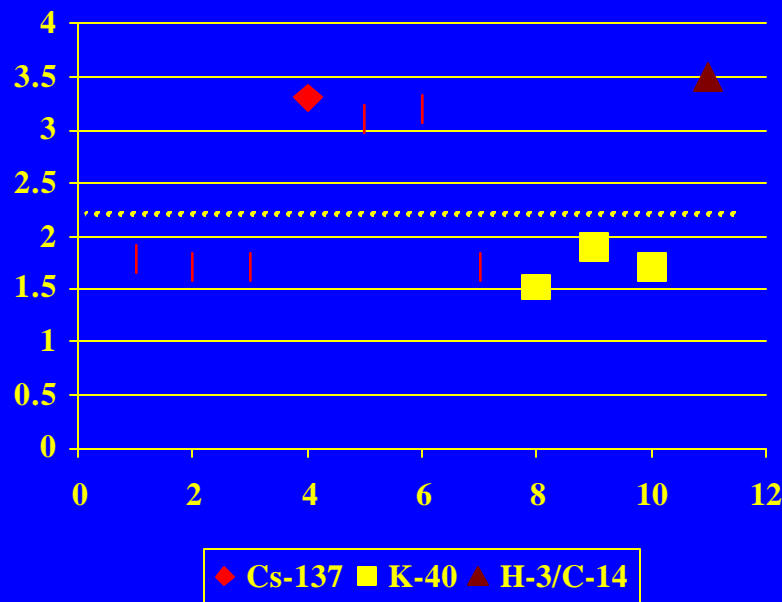
- The sample composition
- The measurement technique used
- The nuclides present
- The calibration nuclides used

Gross alpha / beta intercomparison

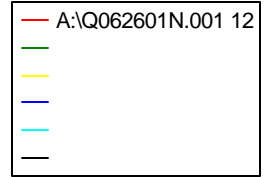
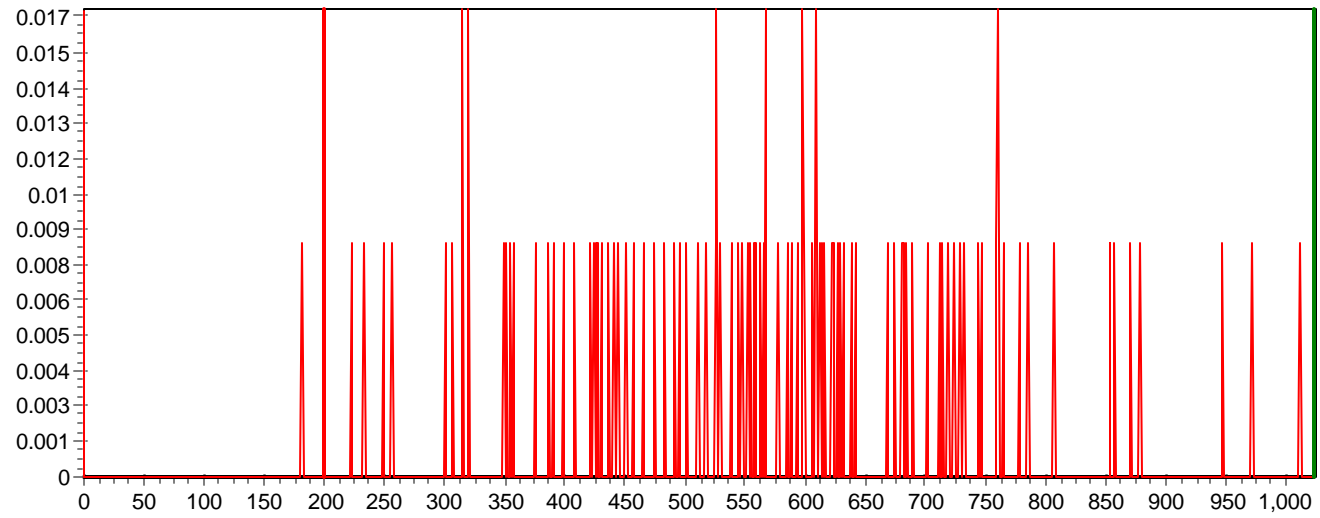
1998, nine of the top UK laboratories.

Using a well characterised, homogenised sediment

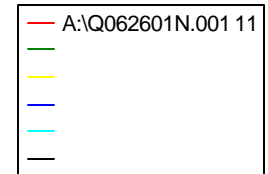
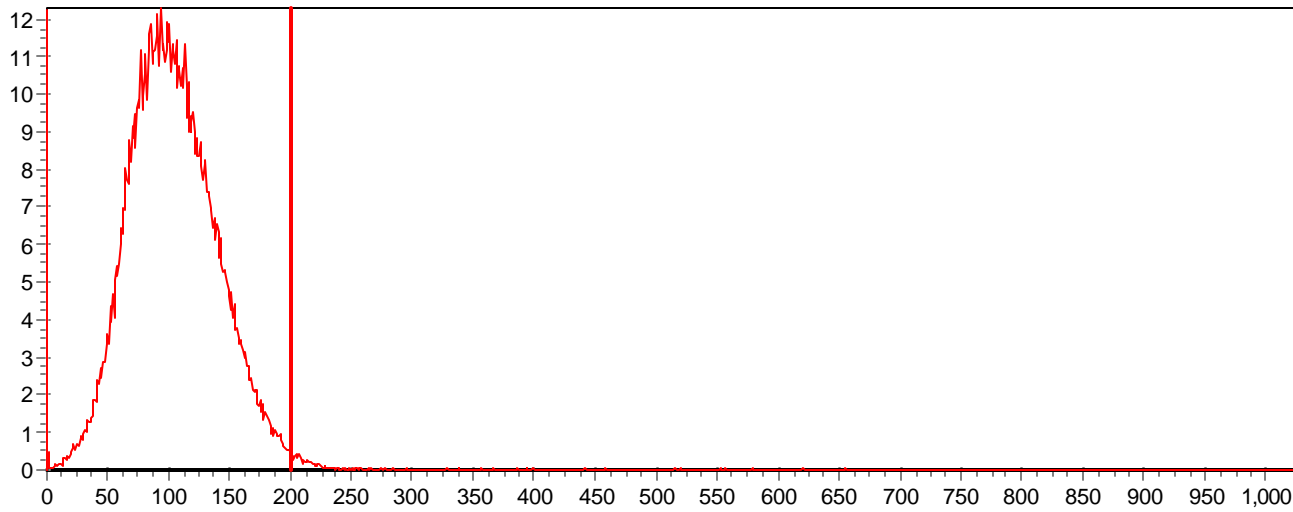
Replicates were internally consistent to 20%, but...



Sample Spectrum



Sample Spectrum



Sample dissolution

Options

- Leaching using a specific reagent
 - Total digestion
 - Fusion
-
- The choice will depend on the type of study and analyte being determined

Sample dissolution

• Leaching 6.78 ± 0.01 Bq/kg

• Fusion 14.26 ± 0.03 Bq/kg

^{238}U in NIST 4357

Tritium analysis

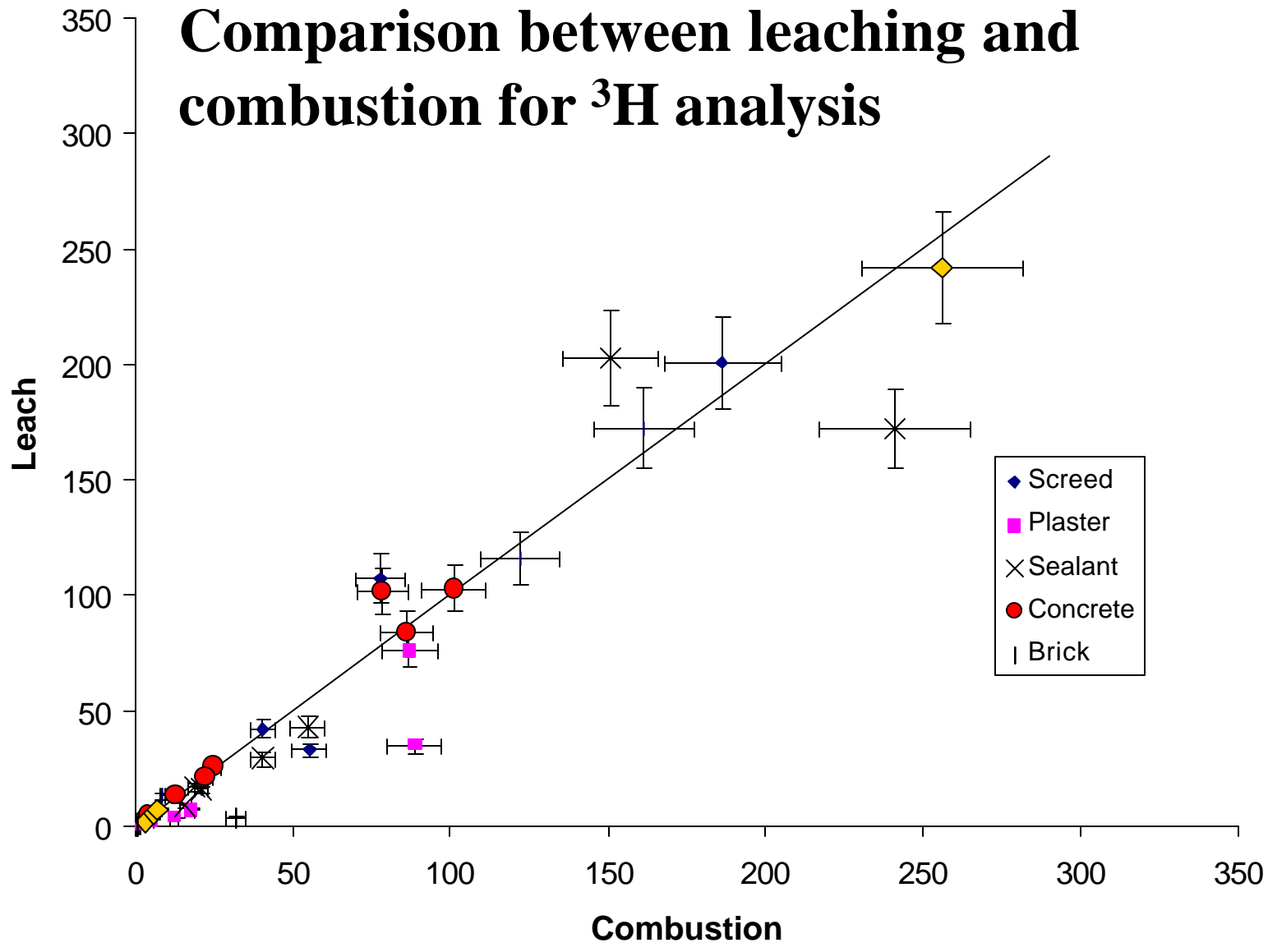
Tritium analysis

- Specific problems with ^3H analysis
- Highly dependent on the form of ^3H
(HTO, organically-bound ^3H , labile ^3H)

Measurement of ^3H

- HTO by distillation (sometimes alkaline)
- Leachable / exchangeable ^3H
- Total ^3H by oxidation (thermal or chemical)

Comparison between leaching and combustion for ^3H analysis



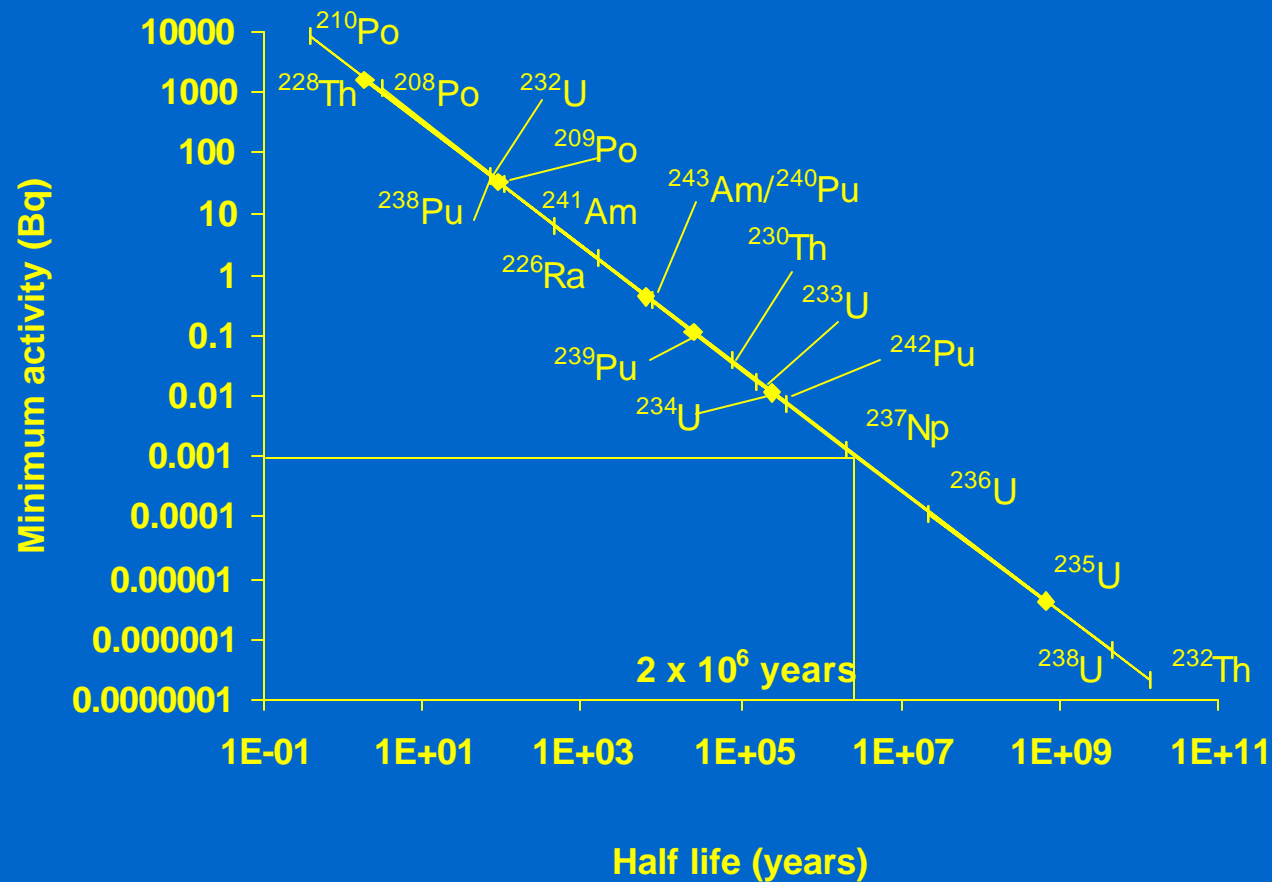
Use of non-radiometric techniques



Advantages of mass spectrometry

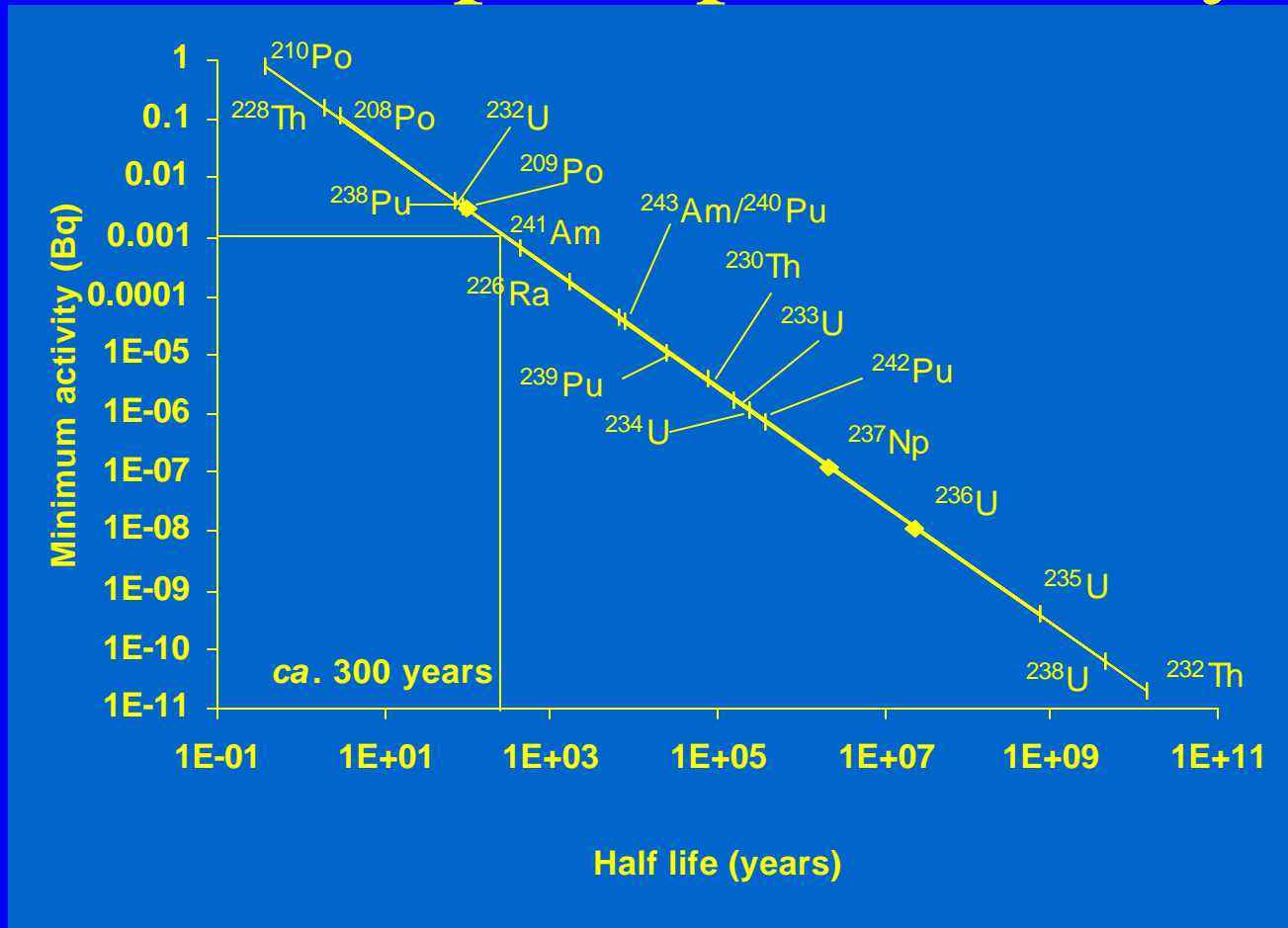
- Often rapid analyses
- Improved data quality
(e.g. ^{238}U : ^{235}U ratios)
- Permits analyses that are not possible radiometrically
(e.g. ^{239}Pu and ^{240}Pu separately)
- Improved sensitivity for long lived nuclides

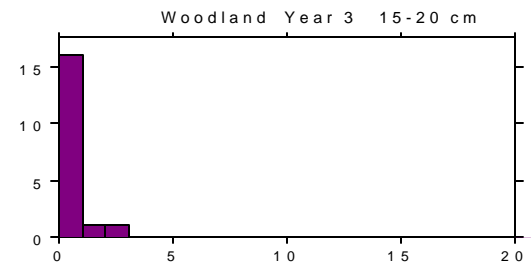
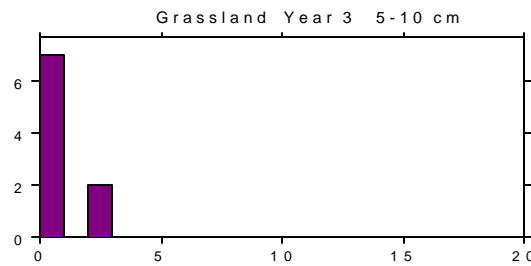
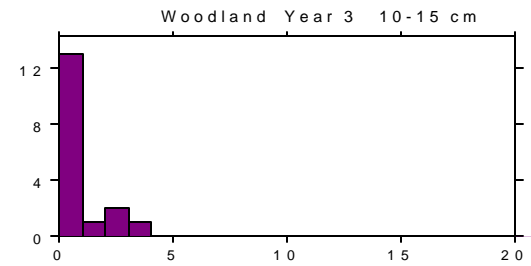
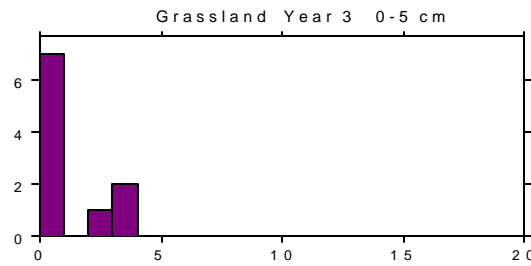
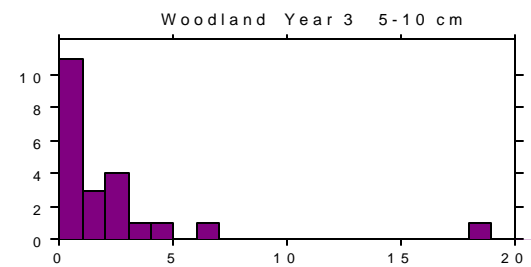
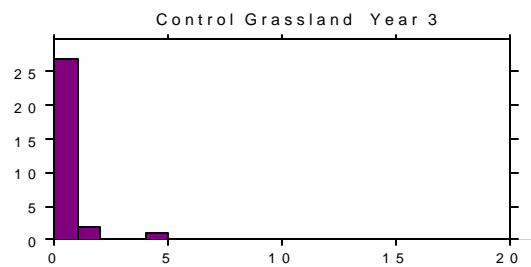
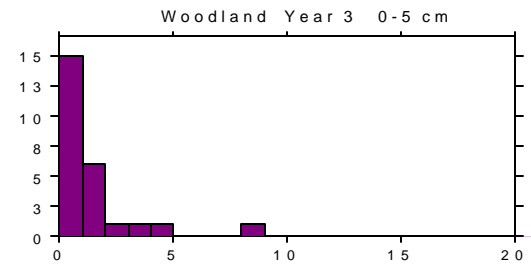
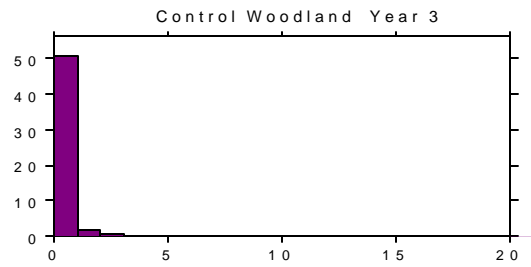
Sensitivity of ICPMS versus alpha spectrometry



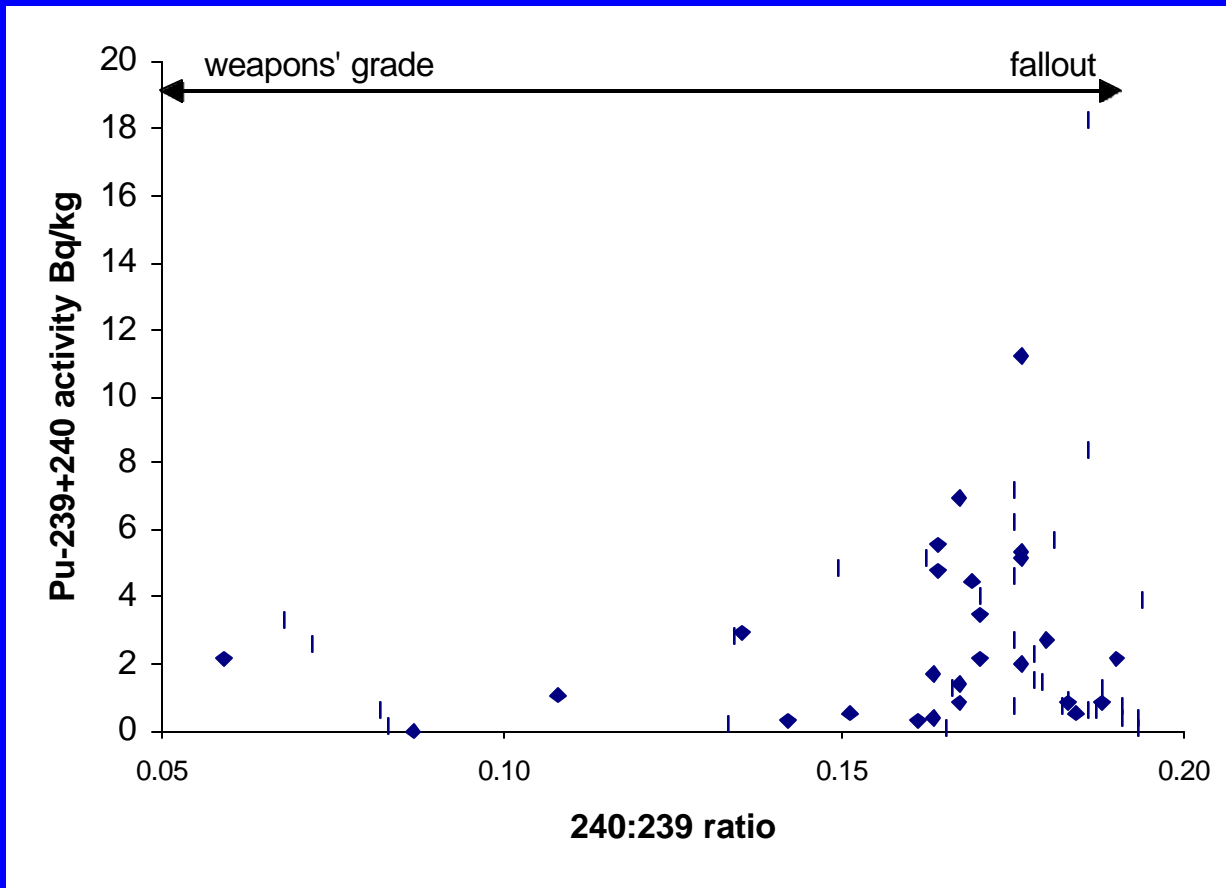
Assuming 10ppt limit
5ml solution

Sensitivity of MC-ICPMS versus alpha spectrometry





Pu source apportionment

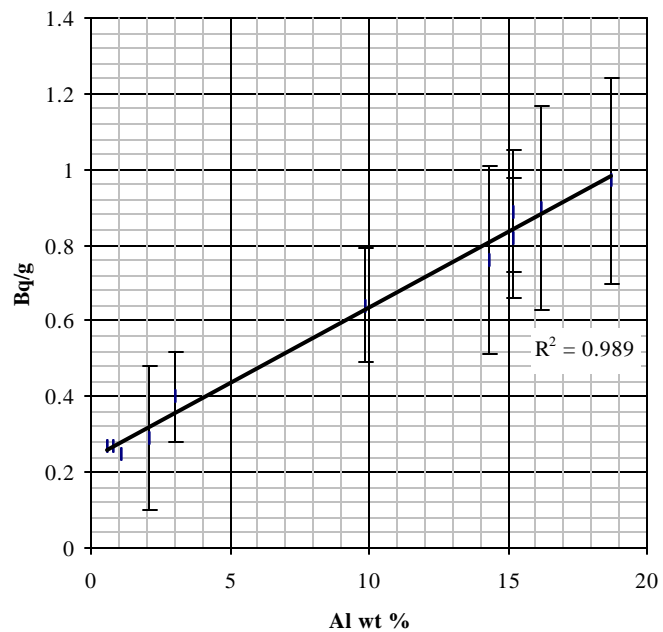


Elemental characterisation

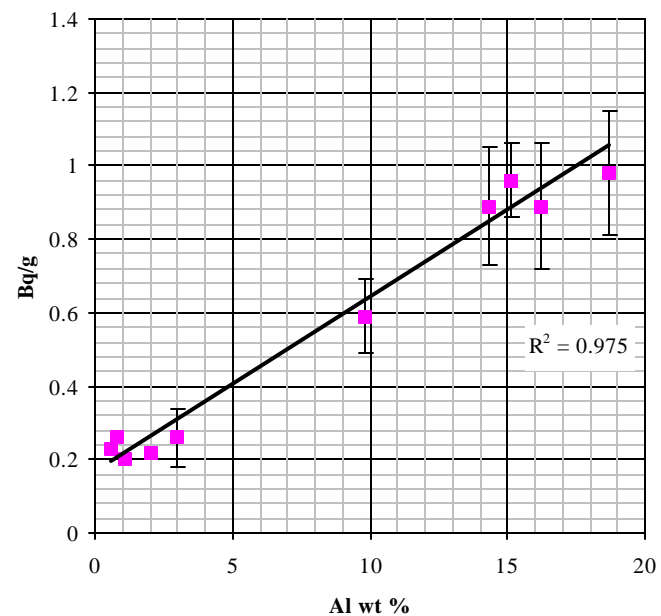


Alpha/beta vs Al concentration

Gross alpha versus Al content



Gross beta versus Al content



Quality control

- Reference materials
- In-house standards
- Method blanks

- How many will be run ?

Summary - I

There are a wide range of analytical techniques available for radionuclide analysis

To ensure that the data is 'fit for purpose' it is vital that the most appropriate technique is used

To ensure this the analytical laboratory must be involved at the initial stages of project development

Summary - II

Agree on the following technical aspects

- The most appropriate analytical technique for the nuclides being investigated
- Specific sampling and storage requirements and the amount of sample required
- Sub-sampling, preparation and dissolution procedures
- Potential interferences
- The required sensitivity, precision and detection limits
- Methods for calculating uncertainties and detection limits
- Quality control