1. Principle of Method

1.1. Samples which have been mounted according to USTUR 500 or 510 are measured for alpha activity using alpha spectrometry.

1.2. Detection limits for alpha activity are based on instrument background count data collected in region of interest areas associated with the alpha decay spectrum for each nuclide of interest, the recovery of the radiotracer, the counter efficiency, the branching ratio of the nuclide of interest, the length of the sample count, and the background time.

1.3. Multinuclide alpha secondary standards are used to energy- and efficiency-calibrate the alpha spectrometers according to USTUR 620.

1.4. Quality control methods are used to monitor counter efficiency and isotopic backgrounds to insure detectors remain in control. (See USTUR 620).

1.5. See USTUR 660 for samples to be measured by absolute analysis.

1.6. See USTUR 650 for samples to be measured by relative analysis.

2. Apparatus

2.1. Alpha spectrometer: 4 unit ORTEC Octete PC, 4096-channel analyzer.

2.2. Vacuum pump.

2.3. 450 mm² EG&G ORTEC ULTRA silicon surface barrier detectors.

2.4. Alphavision V4.02.

2.5. Maestro for Windows V.5.0.
2.6. Forceps.

3. Alpha Spectroscopy Overview

3.1. Each alpha spectrometer is set up with an energy range of 3.5 to 6 MeV in 512 channels.

3.2. Each detector is energy-calibrated using multinuclide secondary standards of $^{242}$Pu, $^{239}$Pu, and $^{241}$Am prepared according to USTUR 630. See USTUR 620 for the energy-calibration procedures. Table 1 shows the energy and channels for the calibration nuclides.

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<th>Isotope</th>
<th>Principal $\alpha$ Energy (keV)</th>
<th>Channel</th>
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<td>331</td>
</tr>
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<td>Am-241</td>
<td>5486</td>
<td>397</td>
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</table>

3.3. Each detector is efficiency-calibrated using the $^{242}$Pu content of the secondary alpha sources. See USTUR 620 for the efficiency-calibration procedure.

3.4. A 300,000 s background count typically is obtained and a control chart kept for each isotopic region of interest (ROI) for each detector. See USTUR 620 for background counting procedures and QA/QC methods associated with backgrounds.

3.4.1. All detectors to be used for analysis shall have had an acceptable background measurement within four weeks prior to beginning the analysis.

3.4.2. The last background measurement is used for background subtraction as defined in the equations in Section 5.

3.5. Samples are routinely measured for 150,000 s on shelf 2.

3.5.1. Tracers with impurities are discouraged from use for sample analysis. Tracers for which no impurity free source exists are used in some cases (Am-243 contains a small amount of Am-241).

3.5.2. Contamination in tracers is corrected by subtracting the fraction of isotopic activity from the net isotopic count rate as described in Section 5.
Example: The net counts in the ROI for Am-243 tracer was 2000 and the net counts for Am-241 was 100. This tracer is known to be contaminated with 0.17% (counts $^{241}$Am/counts $^{243}$Am) Am-241. Thus (2000 counts Am-243) x 0.0017 counts $^{241}$Am/counts $^{243}$Am = 3.4 counts which are subtracted from the net counts for Am-241 resulting in 96.6 counts.

3.6. Chambers for Th and U measurement are equipped with recoil suppression and are operated at a pressure of ~35 torr.

3.7. Blanks are run with each batch to monitor for reagent contribution as well as to provide monitoring for possible contamination during analysis.

3.7.1. QC charts of blank values are evaluated to insure blank measurements are in control.

3.7.2. Blanks are corrected for tracer recovery according to relative analysis procedures described in USTUR 650.

3.7.3. Blanks are corrected for known tracer contamination as described in Section 3.5.

3.7.4. Sample activities are corrected by subtracting the average isotopic blank value if necessary.

3.7.5. Batches with blanks deemed out of control (exceed 99% confidence limits) are evaluated for cause of discrepancy and re-run if possible. If the sample cannot be re-run, the blank value from the batch will be used for blank subtraction. Appropriate indication of the correction method used shall be indicated on the analysis form.

3.8. Samples are evaluated by measuring the activity of isotopes in a specific region of interest (ROI) following radiochemical separation and sample mounting.

3.9. Absolute analysis of a planchet is conducted according to USTUR 660. Absolute analysis is used for limited applications such as secondary source preparation and background counts.

3.10. Relative analysis of a sample is conducted according to USTUR 650. Relative analysis is the standard method used to evaluate USTUR radiochemical samples.

3.11. ROI’s and FWHM (full width at half-maximum)
3.11.1. The alpha emissions of most radionuclides are made up of several different alpha energies each with a specific decay probability. Table 1 shows the emission probabilities of selected radionuclides.

3.11.2. The size of the FWHM will dictate the size of the ROI, the number of peaks observable, and may cause the alpha decays from one isotope to overlap with another of the same element. Table 2 indicates the ROI changes as the FWHM increases. The effective branching ratio of a radionuclide may also change as the ROI expands to include minor alpha peaks.

4. Data Quality

The USTUR program requires high quality data in support of its efforts to provide improved understanding of biokinetic modelling, distribution studies, and other operations. Data quality criteria are provided by USTUR-Richland prior to sample analysis. Figures 2, 3, 4, and 5 may be useful in establishing these objectives.

5. Sample calculations for relative analysis

5.1. Calculation of aliquot activity ($A_a$)

$$A_a (dpm) = \frac{A_{0t} \cdot B_t (c_a t_b - c_a t_s - v_t (c_a t_b - c_b t_b) \cdot t_b)}{B_s (c_a t_b - c_b t_b)} \cdot \frac{\ln 2}{t_{1/2}} \cdot \frac{[d_c - d_a]}{365.25 \text{ days}}$$

where

- $A_{0t}$ = Activity of the tracer at date of certification (dpm)
- $T_{1/2}$ = Tracer half-life (years)
- $d_d$ = Date of detection (counting)
- $d_c$ = Date of certification (tracer)
- $c_s$ = Gross counts of sample
- $c_b$ = Background counts of sample
- $t_b$ = Background time of sample in seconds
- $c_{st}$ = Gross counts of tracer
- $c_{bt}$ = Background counts of tracer
- $B_t$ = Tracer branching ratio
- $B_s$ = Nuclide branching ratio
- $t_s$ = Sample count time in seconds
- $v_t$ = Relative count rate of contamination of nuclide in tracer.

5.2. Calculation of uncertainty in the activity for the aliquot ($\sigma_{A_a}$)

$$\sigma_{A_a} = \frac{A_{0t}}{A_a} \sqrt{\left(\frac{\sigma_{A_{0t}}}{A_{0t}}\right)^2 + \left(\frac{c_a t_b \cdot t_b^2 + c_a t_s \cdot t_s^2}{c_a t_b - c_b t_b}\right)^2 + \left(\frac{c_a t_b \cdot t_b}{c_a t_b - c_b t_b}\right)^2}$$

$USTUR\ 600-4$
\[ \sigma_{\text{A}} = \text{The uncertainty associated with the activity of the tracer at date of certification (dpm).} \]
\[ \sigma_v = \text{Uncertainty in relative contamination of nuclide in tracer.} \]

5.3. Calculation of blank corrected activity of the aliquot \( (A_c) \)
\[ A_c (\text{dpm}) = A_a - V_a, \text{ where} \]
\[ V_a = \text{the activity of the blank (dpm).} \]

5.4. Calculation of uncertainty associated with the blank corrected activity of the aliquot \( (\sigma_{A_c}) \)
\[ \sigma_{A_c} = \sqrt{\sigma_{A_a}^2 + \sigma_v^2}, \text{ where} \]
\[ \sigma_v = \text{the uncertainty associated with the activity of the blank value.} \]

5.5. Calculation of fractional recovery \( (R) \)
\[ R = \frac{\frac{C_{st}}{t_s} - \frac{C_{bt}}{t_b}}{B_t \cdot A_{bt} \cdot E} \cdot 60 \cdot \frac{s}{\text{min}} \cdot e^{-\left[\frac{\ln 2}{T_{1/2}} \cdot \left(\frac{d_d - d_e}{365.25 \text{ days}}\right)}\right]} \]

5.6. Calculation of decision limit for aliquot \( (L_{da}) \)
\[ L_{da} (\text{dpm}) = \frac{3.29 \cdot \sqrt{\frac{C_{da} \left(1 + \frac{t_a}{t_b}\right)}{t_b}} + 3}{B_s \cdot E \cdot R \cdot t_s} \left(\frac{60 \text{ dpm}}{Bq}\right), \text{ where} \]
\[ E = \text{The efficiency of the detector (Counter Eff.).} \]

5.7. Calculation of sample concentration result \( (S_R) \)
\[ S_R = \frac{A_c \cdot W_s}{W_a \cdot W_x} \left(\frac{1000 \text{ g}}{1 \text{ kg}}\right) \left(\frac{1 \text{ Bq}}{60 \text{ dpm}}\right), \text{ where} \]
\[ A_c = \text{Blank corrected activity of the aliquot (dpm)} \]
\[ W_s = \text{Total solution weight at time of preparation (g)} \]
\[ W_a = \text{Weight of aliquot used for analysis (g)} \]
\[ W_x = \text{Wet weight analyzed if soft tissue, or ashed weight analyzed if bone (g), or equal to 1 (unit less) if radiochemical sample total activity to be calculated} \]
The units are determined by the ‘Tissue Type’ and the ‘Method’ columns. For bone tissue analyzed via radioanalysis, the units are \( \frac{\text{Bq}}{\text{kg of ashed weight}} \), and for ‘soft’ tissue, \( \frac{\text{Bq}}{\text{kg of wet weight}} \), or \( \frac{\text{Bq}}{\text{Radiochemical Sample}} \) for total isotopic activity in the radiochemical sample.

5.8. Calculation of uncertainty associated with the sample result \( \sigma_{\text{As}} \)

\[
\sigma_{\text{As}}(\text{Bq / kg}) = \frac{W_s \cdot \sigma_{\text{Ac}}}{W_a \cdot W_s} \cdot \frac{1000 \text{ g}}{1 \text{ kg}} \cdot \frac{1\text{Bq}}{60 \text{ dpm}}, \text{ where}
\]

\( \sigma_{\text{Ac}} \) = Uncertainty associated with the blank corrected activity of the aliquot (dpm)

5.9. Calculation of concentration equivalent \( (C_E) \) for sample result

\[
C_E(\text{Bq / kg or Bq / sample}) = \frac{W_s \cdot L_D}{W_a \cdot W_s} \cdot \frac{1\text{Bq}}{60\text{dpm}} \cdot 1000 \frac{\text{g}}{\text{kg}}
\]

6. Sample calculations for absolute analysis

6.1. Calculation of sample activity \( (A_s) \)

\[
A_s(\text{dpm}) = \frac{\left( \frac{C_s - C_b}{t_s} \right) \cdot 60(\text{S / min})}{B_s \cdot E}
\]

6.2. Calculation of uncertainty in sample activity \( \sigma_{A_s} \)

\[
\sigma_{A_s}(\text{dpm}) = \left[ \frac{3600}{B_s E^2} \left( \frac{c_s}{t_s^2} + \frac{c_b}{t_b^2} \right) + \frac{A_s^2}{E^2 \sigma_E^2} \right]^{1/2}
\]

6.3. Calculation of the limit of detection for sample activity \( L_{D_s} \)

\[
L_{D_s}(\text{dpm}) = \frac{3.29 \sqrt{\frac{c_s}{t_s} \left( 1 + \frac{t_s}{t_b} \right) + 3}}{B_s E t_s} \left( \frac{60 \text{ dpm}}{\text{Bq}} \right)
\]

7. Literature Sources


Table 2
Primary α Emissions for Selected Radionuclides

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<th>Isotope</th>
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<th>α1 (%)</th>
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Reference: Browne 1986
Only principal emissions shown.
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<th>Expected Primary α Peak Channel</th>
<th>FWHM (keV)</th>
<th>Left (channels)</th>
<th>Right (Channels)</th>
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<td>4395</td>
<td>180</td>
<td>50</td>
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<td>36</td>
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<td>87.75</td>
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<td>$3.763 \times 10^{5}$</td>
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<td>397</td>
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<td>4788</td>
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</table>

*Half life information from Browne 1986.

Expected channel based on $E (\text{keV}) = 3500 + 5 (\text{keV/channel}) \times (\text{Channel} \#)$

$^{229}$Th provides a spectral interference for $^{230}$Th at a rate of $2.7E-3/\text{count}^{229}$Th
Figure 1

Alpha Spectra of Typical Radionuclides

Amercium

![Amercium Spectrum](image)

Plutonium

![Plutonium Spectrum](image)

Uranium

![Uranium Spectrum](image)

Thorium

![Thorium Spectrum](image)
Figure 2
L_d as a Function of Time

Plot of L_d (dpm/ aliquot) as a Function of Time

Assumptions:
100% Recovery
3.3E-3 cts background (20 counts/600,000 s)
20% Efficient
Figure 3
Relative Precision for 1 dpm Aliquot for Variable Recovery and Count Time

Assumptions:
- Variable Recovery
- 3.3E-5 c/s background (20 counts/600,000 s)
- 25% Efficient

Relative Precision for 1 dpm Aliquot (%)

Sample Count Time (seconds)
Figure 4
Relative Precision for 0.1 dpm Aliquot for Variable Recovery and Count Time

Assumptions:
Variable Recovery
3.3E-6 c/s background (20 counts/600,000 s)
25% Efficient
Figure 5
Relative Precision for 70% Recovery as a Function of Count Time and Aliquot Activity

Assumptions:
70% Recovery
3.3E-5 c/s background (20 counts/600,000 s)
25% Efficient