

10. CHROMATOGRAPHIC SEPARATION AND PURIFICATION
OF URANIUM FOR MASS SPECTROMETRIC ANALYSIS

The technique employed for this purpose was a modification of the existing methods used in this study to separate and purify uranium (Table 9). Of prime importance was the high purity of the uranium, for it must be free of all organic compounds, cations and anions (with the exception of nitrate). The reason for this was the ultra sensitivity of the mass spectrometer to the presence of interfering species, which results in unstable performance. The end result was a considerable reduction of the quality of the analytical data obtained.

Three stages were required for the purification procedure (Table 12). These are similar to those described earlier.

- (a) Separation of uranium from thorium, alkalis, alkaline earths and rare earths by anion exchange in the chloride form.
- (b) Purification of uranium using tributyl phosphate in reversed phase liquid chromatography.
- (c) Removal of traces of tributyl phosphate by ashing and separation of phosphate using anion exchange in the chloride form.

Three samples were processed in this manner, two of calcrite from the Langer Heinrich and one of uranothorianite from Phalaborwa.

The quality of the spectrum is dependent on two major parameters, the first being the correct functioning of the

mass spectrometer, and the second the quality of the separation. On the assumption that the first was of high quality, any instrumental instability was therefore due to poor analytical separation. A good spectrum capable of giving high precision is shown in Fig. 37.

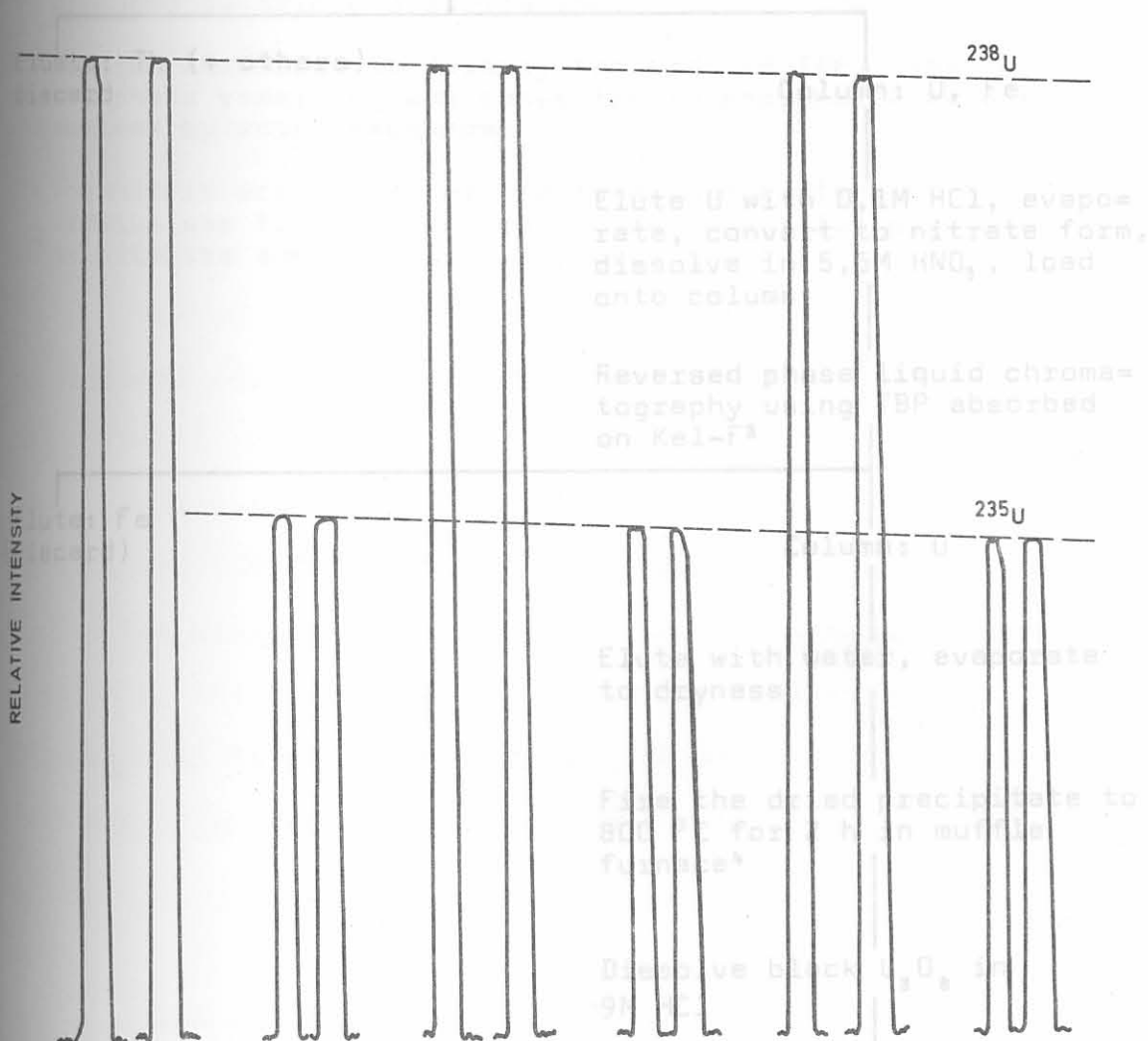
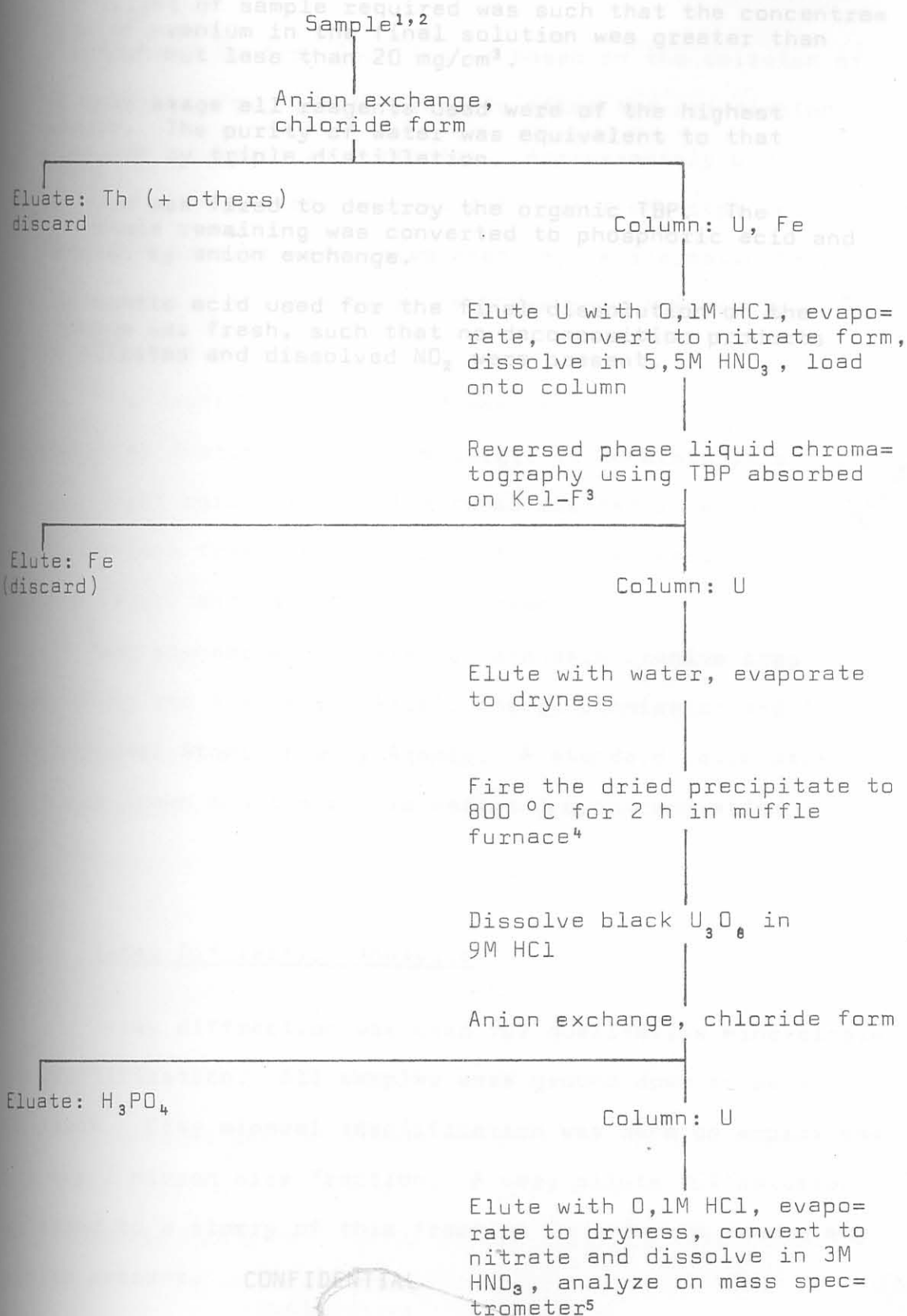


Fig. 37: Spectrum of ^{238}U and ^{235}U obtained from the mass spectrometer.

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TABLE 12: PROCEDURE FOR THE CHROMATOGRAPHIC SEPARATION OF URANIUM FROM CALCRETE, SUITABLE FOR MASS SPECTROMETRIC ANALYSIS. Finally converted to the nitrate form by fuming with concentrated HCl.



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¹ Uranothorianite was dissolved in a mixture of concentrated HF and HClO₄. Fumed with an excess of HClO₄ to ensure the complete oxidation of U⁴⁺ to U⁶⁺. Finally converted to the chloride form by fuming with concentrated HCl.

Delayed Neutron Counting

² The weight of sample required was such that the concentration of uranium in the final solution was greater than 3 mg/cm³ but less than 20 mg/cm³.

³ At this stage all reagents used were of the highest purity. The purity of water was equivalent to that produced by triple distillation. Approximately 0.03 g

⁴ Uranium was fired to destroy the organic TBP. The phosphate remaining was converted to phosphoric acid and removed by anion exchange.

⁵ The nitric acid used for the final dissolution of the uranium was fresh, such that no decomposition products as nitrites and dissolved NO₂ were present.

The counting system used was designed and built by the National Institute for Metallurgy. It consists of a ring of eight boron trifluoride tubes mounted in a tank of water. Pulses from the tubes pass through an amplifier and a discriminator, and are finally counted.

The standards consisted of standard uranium ores supplied by the Australian Atomic Energy Commission and The International Atomic Energy Agency. A standard calibration curve was drawn and the counts versus U₂O₅ concentration plotted.

X-Ray Diffraction Analysis

X-ray diffraction was used for qualitative mineralogical identification. All samples were ground down to pass 325 mesh. Clay mineral identification was done on approximately the 2 micron size fraction. A very dilute HCl solution was added to a slurry of this fraction in order to remove any calcite present.

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