

## Separation of americium from rare earths by reversed-phase partition chromatography

Solvent extraction with quaternary ammonium salts of high molecular weight has been proposed recently by MOORE<sup>1</sup> and, independently, by the present authors<sup>2</sup>, as an alternative method of separating trivalent actinides from lanthanides in thiocyanate media.

The same chemical system, basically, has been used by us in a series of reversed-phase partition chromatography experiments to effect laboratory-scale separations of americium (III) from the rare earths. Some separations between single rare earth elements have also been obtained.

### Experimental

Celite (80-120 mesh) obtained from the BDH Ltd. England, made hydrophobic by immersion in a (5%) ether solution of dichlorodimethylsilane was used as the supporting solid. Column beds were prepared by mixing the treated Celite with 0.3 M

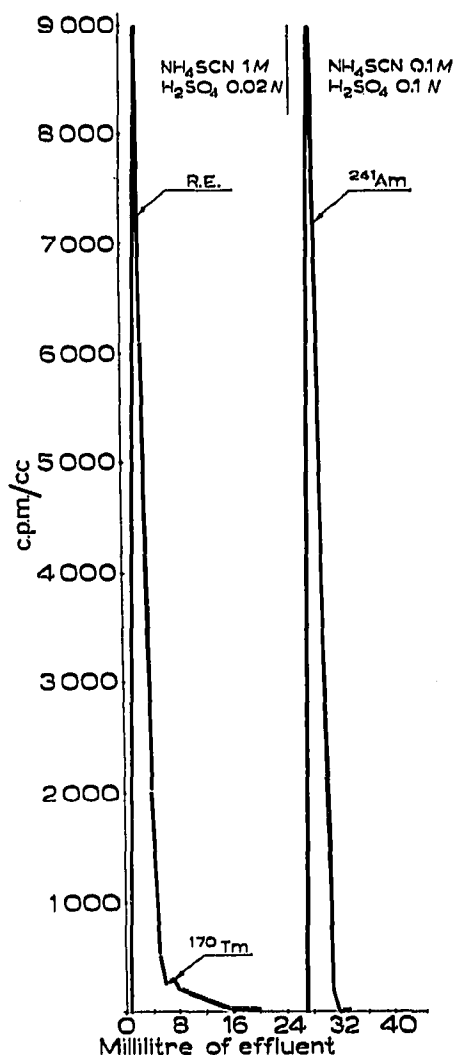


Fig. 1. Separation of americium from rare earths.

solution of Aliquat-336 (methyl-tri(*n*-alkyl)ammonium, principally octyl and decyl, obtained from the General Mills Inc., U.S.A.) in the thiocyanate form and transferring the resulting slurry to a 5.4 mm internal diameter glass column plugged with glass wool. The excess of extractant, not adsorbed on the solid support, was displaced by washing the column with the thiocyanate solution used for the first elution step. The height of the bed was 10 cm and the eluent flux  $0.78 \text{ ml cm}^{-2}\text{min}^{-1}$ . A small aliquot (50  $\lambda$ ) of a thiocyanate solution containing the tracers to be separated was transferred to the top of the resin bed and elution was carried out by the usual chromatographic techniques.

$^{241}\text{Am}$ ,  $^{144}\text{Ce}$ ,  $^{147}\text{Pm}$ , and  $^{152}+^{154}\text{Eu}$  were obtained from the Radiochemical Centre, Amersham, England.  $^{140}\text{La}$  and  $^{170}\text{Tm}$  were prepared by neutron activation of high purity lanthanum and thulium in the nuclear reactor G.Galilei of CAMEN. The samples taken from the elution steps were gamma-counted using a well-type NaI

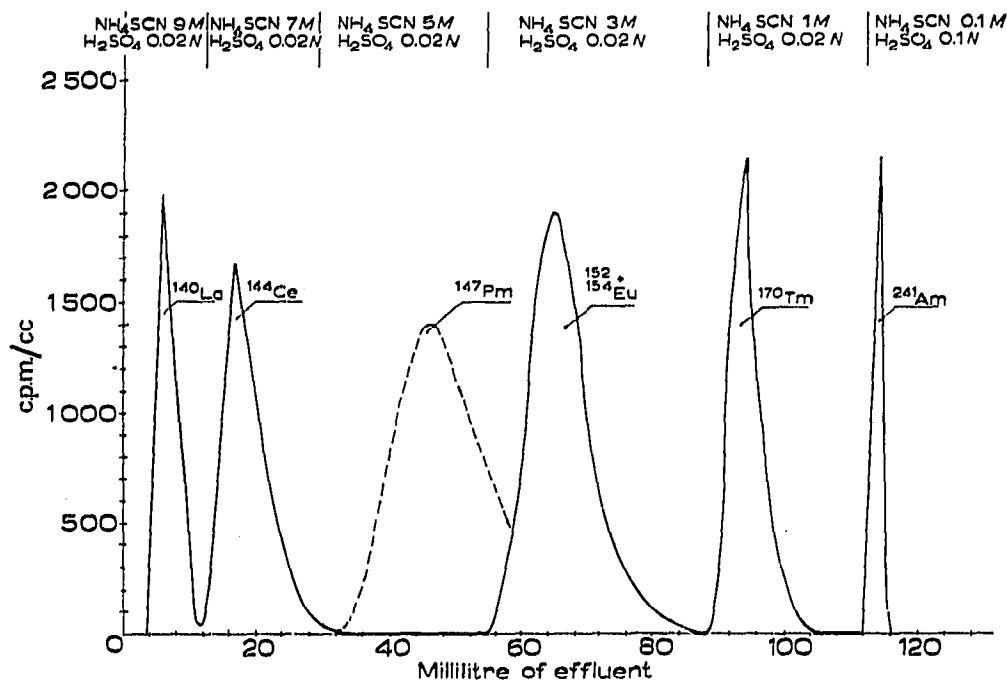


Fig. 2. Separation of  $^{140}\text{La}$ ,  $^{144}\text{Ce}$ ,  $^{147}\text{Pm}$ ,  $^{152}+^{154}\text{Eu}$ ,  $^{170}\text{Tm}$  and  $^{241}\text{Am}$ .

(TI) scintillation counter except for samples of  $^{147}\text{Pm}$  which were deposited on glass discs and beta-counted with a G.M. counter.

### Results

A relatively rapid separation of americium from the rare earth group was obtained using the eluent composition specified in Fig. 1. A small quantity of sulfuric acid was added to the thiocyanate eluent to enhance the separation as suggested by MOORE<sup>1</sup>. In Fig. 2 a selective elution of lanthanum, cerium, promethium, europium, thulium and americium is reported, obtained by gradient elution with thiocyanate solutions of decreasing concentration.

A group separation of americium from the rare earth elements was also obtained using an ascending chromatography technique with paper (Whatman No. 1) strips

treated with Aliquat-336 in the thiocyanate form and a developing solution consisting of 0.2M  $\text{NH}_4\text{SCN}$  + 0.05N  $\text{H}_2\text{SO}_4$  (Am,  $R_F$  0.35; Tm,  $R_F$  0.77; Eu,  $R_F$  0.81).

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### **Application of reversed-phase column chromatography to the determination of tungsten in stainless steels by activation analysis**

As is well known, activation analysis is a very useful tool for the determination of trace elements in various materials, and tungsten is a normal minor component in stainless steels. However, when steel is irradiated, several other radio-isotopes besides tungsten are produced, for example, when samples are irradiated for one hour and then "cooled" for one day, the gamma-activity due to the long-lived nuclides  $^{51}\text{Cr}$ ,  $^{59}\text{Fe}$ ,  $^{58}\text{Co}$ ,  $^{76}\text{As}$ ,  $^{187}\text{W}$  remains. As shown in Fig. 1, the resolution of gamma-spectrometry is not sufficient to resolve the complex spectrum obtained under these conditions, and therefore it is impossible to determine tungsten without previous chemical separation. On the other hand, determination of tungsten by the classical method of tungstic acid precipitation does not give a good quantitative separation because of the co-precipitation of other elements such as As, Mo, and so on<sup>1</sup>. Better results have been obtained by isolating tungsten, as  $\text{WO}_4^-$ , on chromatographic columns of an inert support impregnated with 8-hydroxyquinoline<sup>2</sup> or on anion-exchange resins<sup>3</sup>. In this work, we have used reversed-phase partition chromatography<sup>4-6</sup> to isolate tungsten from the unwanted major components of the steel.

HAMLIN *et al.*<sup>7</sup> used polytrifluorochloroethylene (Kel-F) as an inert support for tri-*n*-octylamine (TNOA); this compound behaves just like a "liquid anion exchanger": the anionic metal complexes are retained on the top of the chromatographic column, while the elements which do not form anionic complexes are quickly eluted. Generally the separative properties of the column depend on the values of the complex formation constants and on the distribution coefficients between the organic and the aqueous phase. The amine extraction technique is particularly convenient because it also works efficiently in strong acid solutions.

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