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Letter to the Editors

Chromatographic separation of the heavier lanthanoids on the spherical cation exchanger OSTION with α-hydroxy-α-methylbutyrate

Sir,

The common eluting agents for the chromatographic separation of lanthanoids and transplutonium elements on cation exchangers are α-hydroxycarboxylic acids, with α-hydroxyisobutyric acid (HIB) being mostly used. The first use of α-hydroxy-α-methylbutyrate (HMB) for chromatographic separation of lanthanoids was described by Nishi and Fujiwara[1] and Karol[2]. This ligand was also used for the paper chromatographic separation of lanthanoids (fission products of uranium)[3]. The separation efficiency for Am, Cm and Cf on the selected cation exchanger Ostion LG KS 0800 using HIB and HMB was measured and high values of the separation factors were found for α-hydroxy-α-methylbutyrate[4].

The purpose of this work was to determine the separation factors for heavier lanthanoids on Ostion cation exchanger under the same experimental conditions.

EXPERIMENTAL

Glass columns (15 mm × 1.4 mm I.D. and 40 mm × 2.0 mm I.D.) were packed with a spherical strongly acidic cation exchanger Ostion LG KS 0800 (NH_4^+), particle size 7–11 μm, capacity 5 mequiv. per g, 8% divinylbenzene, supplied by Chemapol, Department of Pure and Special Chemicals, Prague, Czechoslovakia.

The elution rate was 2 drops per minute and the drop volume was 25 and/or 30 μl. The elution rate was controlled by a peristaltic eluent delivery pump S-31 (Domet, Pruszków, Poland), maximum pressure 2 bar.

The eluting agent was a solution of α-hydroxy-α-methylbutyric acid, adjusted to pH 4.0 with ammonia. The acid was prepared by hydrolysis of α-hydroxy-α-methylbutyronitrile with hydrochloric acid and isolated as described earlier for α-hydroxyisobutyric acid[5]; m.p. of pure α-hydroxy-α-methylbutyric acid 72.5°C.

Radioactive indicators of high specific radioactivity, ^{177}Lu, ^{169}Yb, ^{153}Gd and ^{152}Eu, were prepared by irradiation in a nuclear reactor. The carrier-free radionuclide ^{88}Y was used together with the spallogenic carrier-free radionuclides obtained by bombardment of tantalum with protons of energy 680 MeV: ^{170}Lu, ^{171}Lu, ^{166}Yb, ^{165}Tm, ^{168}Tm, ^{160}Er, ^{160}Ho.

The γ radiation from the fractions eluted was measured by a microprocessor controlled system Gamma 8500 (Beckman). The radionuclides were identified by a gamma spectrometer equipped with an HPGe detector full width at half maximum [(FWHM) 1.8 keV, efficiecy 20%].

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RESULTS AND DISCUSSION

The values of the separation factors, α, for most of the heavier lanthanoids on OSTION LG KS 0800 at room temperature with ammonium α-hydroxy-α-methylbutyrate as eluent are given in Table I. For comparison, also shown are published values for HMB at 87°C on Dowex 50-X8[1]. The separation factors of the heavier lanthanoids are not consistent with the values published by Nishi and Fujiwara[1], while the value $\alpha = 1.50$ for the pair Yb–Tm obtained by Karol[2] corresponds to the value determined in this work. The elution peaks obtained by Nishi and Fujiwara[1] are not symmetric; in many cases the resolution of the elements achieved was not complete and the determination of α was performed with different column mass loadings. These seem to be the main reasons for the inconsistencies between the values of α, which is also accompanied by the violation of the relation between the respective α values of individual pairs of the lanthanoid elements. The chemical purity of the eluent must be considered (the published[1] m.p. of α-hydroxy-α-methylbutyric acid is 68°C). Because of the extremely high value of the α reported for the pair Lu–Yb[1], I have used for its determination, besides the radioactive indicators ^{177}Lu and ^{169}Yb of high specific activity, also spallogenic radionuclides in a carrier-free form. In both cases α was 1.3.

From the values of α determined for the heavier lanthanoids given in Table I and from published data for lighter lanthanoids[2], it is evident that for the ligand HMB the values of α are higher the lower is the atomic number.

TABLE I
SEPARATION FACTORS, α, OF HEAVIER LANTHANOIDS

Elements	*This work*[a]	*Nishi and Fujiwara*[b]
Lu–Yb	1.3	2.41
Yb–Tm	1.5	1.35
Tm–Er	1.5	1.70
Er–Ho	1.6	1.30
Y–Tb	1.6	1.95
Tb–Gd	2.3	1.89
Gd–Eu	1.5	1.38

[a] Ostion LG KS 0800, HMB pH 4.0, room temperature.
[b] Dowex 50-X8, 100–200 mesh, HMB pH 4.0, 87°C; ref. 1.

According to a comparison of the separation factors of the individual pairs of lanthanoids (see Table I, refs. 2 and 6), for the separation of heavier lanthanoids HIB is more suitable, while HMB is useful for lighter lanthanoids. This assessment has been made using data obtained under corresponding experimental conditions (quality of cation exchanger).

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