



Separation of Rare Earth Elements (REE) by Ion Interaction Chromatography (IIC) Using Diglycolic Acid (ODA) as a Complexing Agent

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Abstract

The possibility of rare earth elements (REE) separation by ion interaction chromatography (IIC) employing their complexes with diglycolic acid (ODA) in anion exchange mode has been studied theoretically and experimentally. Calculations, assuming that only trivalent complex is significantly uptaken by the stationary phase, indicated that at pH 4–6, the retention in the lanthanide series should increase from La to the Tb–Dy–Ho region with yttrium showing apparent atomic number (App.At.No.) of $67\frac{1}{2}$ and then decrease with further increase of atomic number. Chromatographic experiments in the system: Column: Eternity C₁₈—mobile phase 5 mM ODA/8.6 mM TBAOH/0.6 mM HNO₃; pH 4.60 confirmed theoretically predictions. It was found that scandium at pH ≥ 4.0 elutes in front of the whole REE group but at low pH enters the region of light lanthanides. The non-monotonical change of affinity of the REE with the increase of atomic number results in quite unusual order of elution of REE namely: Sc < La < Ce < Lu < Pr < Yb < Nd < Tm < Sm < Eu < Er \approx Y < Gd < Ho < Tb \approx Dy.

Keywords Ion interaction chromatography (IIC) · tetra-*n*-butylammonium hydroxide (TBAOH) · Rare earth elements (REE) · Lanthanides · Diglycolic acid (ODA) · Apparent atomic number (App.At.No.)

Introduction

Scandium, yttrium, lanthanum, and the lanthanides i.e., rare earth elements (REE) have recently gained great significance in high technology applications as catalysts, batteries for hybrid cars, permanent magnets, phosphors for cathode ray tubes, in glass industry and in several military applications [1, 2]. Separation of REE is difficult because of similarity of their chemical properties and therefore only a few techniques like solvent extraction, ion exchange and various forms of chromatography have gained wider acceptance for technological and analytical purposes. Reviews dealing with the separation of REE were recently published [3–5]. Several carboxylic and aminocarboxylic acids have been employed for the column separation of REE by ion exchange chromatography (IEC) or ion interaction chromatography (IIC) in

cation or anion exchange modes. These reagents included: acetic acid, lactic acid, mandelic acid, α -hydroxyisobutyric acid (HIBA), tartaric acid, citric acid, oxalic acid, glycolic acid, iminodiacetic acid (IDA), nitrilotriacetic acid (NTA), ethylenediaminetetraacetic acid (EDTA), *trans*-1,2-diaminecyclohexane-*N,N'*-tetraacetic acid (DCTA), and *N*-hydroxyethylenediaminetriacetic acid (HEDTA) [4, 5]. Diglycolic acid (ODA) was rarely employed for REE separation. Ion Pac CS5 mixed bed column was used together with oxalic acid plus ODA in gradient elution to separate REE by anion exchange mechanism (elution order from La to Lu) [6–9]. ODA was never used so far as a single eluent for chromatographic separation of REE. It has been known that ODA forms negatively charged complexes with rare earth ions and the respective stability constants are available in the literature [10, 11]. Therefore, it seemed tempting to: (1) try to predict what would be the sequence of affinity of REE in the IIC system: RP column (Eternity C₁₈)—mobile phase containing tetra-*n*-butylammonium hydroxide (TBAOH) which dynamically creates anion exchange sites on the column and ODA as a complex forming reagent; (2) check experimentally the possibility of REE separation in

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this system as well as the agreement or disagreement with theoretical predictions.

Experimental

Chemicals

All solutions employed in the studies were prepared using analytical grade reagents in 18 mΩ cm water produced by a Milli-Q Direct System (Millipore, France) and subsequently deoxygenated with nitrogen. The solution of 400 g L⁻¹ TBAOH (Sigma-Aldrich, Switzerland, $M_w = 259.74$ g mol⁻¹) was used as a stock solution of TBAOH. All eluent solutions were prepared by weighing appropriate amount of diglycolic acid (ODA) (Sigma-Aldrich, Japan—98%, $M_w = 134.09$ g mol⁻¹) and pipetting appropriate volume of TBAOH, then dissolving in water in a 1000 mL volumetric flask. Required values of pH of eluent solutions were adjusted generally by TBAOH concentration but additionally also by pipetting appropriate volumes of 1 M stock solutions of HNO₃, or NaOH (POCh, Poland). Mixed standard solutions of REE elements applied in the experiments were prepared by appropriate dilution and mixing of standard stock solutions (1000 mg L⁻¹) of REEs (Chem-Lab NV, Zedelgem, Belgium—99.99%). The post-column color complexing reagent solution contained 0.12 M Arsenazo III and 0.5 M CH₃COOH, pH 2.61. The solution was prepared by dissolving 0.093 g Arsenazo III and 28.5 mL glacial CH₃COOH (all from Fluka, Switzerland) in water in a 1000-mL volumetric flask.

Apparatus and Procedure

The studies were performed using a Dionex 2000i/SP ion chromatograph (Dionex Corporation, Sunnyvale CA, USA), equipped with a Dionex VDM-II, UV-Vis variable wavelength detector and a Dionex AGP gradient pump. Flow rate of all elutions was 1 mL min⁻¹. The individual REE elements were separated by isocratic elution using ODA as the complexing agent in the eluent and TBAOH as IIR. Aqueous solutions of eluent containing appropriate concentrations of ODA and TBAOH were prepared and the required pH of eluent solutions were adjusted finally with TBAOH or also with HNO₃, NaNO₃ or NaOH. Separations of REE elements studied were performed on an HPLC-RP column—Kromasil Eternity XT5—C₁₈, (Akzo Nobel, 250 mm—length and 4.6 mm I.D.), equipped with 13 mm guard column. The samples were injected to the chromatograph manually with a 50 μL of Dionex Tefzel sample loop. During the experiments, the column was immersed in a water bath in which the temperature was controlled within ± 1 °C by a UTU-4 ultrathermostat (Tarnów, Poland). In all experiments, the

chromatograms were recorded by means of photometric detection mode using a Dionex VDM-II UV-Vis detector at λ = 658 nm after the separated REEs had undergone post-column color reaction with Arsenazo III. Data collection and calculation of chromatographic parameters were performed employing the Dionex Peak Net Chromatography Software resident in an IBM P.

Results and Discussion

Theoretical Predictions

Diglycolic acid (ODA, H₂L) is a dibasic acid with pK₁ = 2.79 and pK₂ = 3.93. It forms with REE complexes including the following equilibria [11]:

$$K_1 = K_{LnL} = \frac{[LnL^+]}{[Ln^{3+}][L^{2-}]} \quad (1)$$

$$K_2 = K_{LnL2} = \frac{[LnL2^-]}{[LnL^+][L^{2-}]} \quad (2)$$

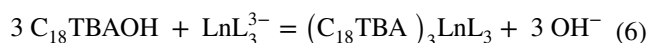
$$K_3 = K_{LnL3} = \frac{[LnL3^{3-}]}{[LnL2^-][L^{2-}]} \quad (3)$$

$$\beta_2 = \beta_{LnL2} = \frac{[LnL2^-]}{[Ln^{3+}][L^{2-}]^2} \quad (4)$$

$$\beta_3 = \beta_{LnL3} = \frac{[LnL3^{3-}]}{[Ln^{3+}][L^{2-}]^3} \quad (5)$$

When the mobile phase containing TBAOH and ODA at appropriate pH is passed through the RP (e.g., C₁₈) column, the TBAOH molecules are adsorbed on hydrocarbon chains creating in a dynamic mode the anion exchange sites. REE which form negatively charged complexes can be in such system retained by the stationary phase and later again desorbed during the flow of mobile phase through the column. This process is the basis for the possible separation of REE by IIC according to anion exchange mechanism, provided the subtle differences in formation constants of REE and column performance are good enough to assure separation of individual lanthanides.

It can be reasonably assumed that only trivalent complex will be effectively retained by the stationary phase. Then, the anion exchange reaction can be written as:



The expression for the bed distribution coefficient of an lanthanide λ'_{Ln} , is then:

$$\lambda'_{Ln} = \frac{[C_{18}TBA]_3LnL3]}{[Ln^{3+}] + [LnL^+] + [LnL2^-][LnL3^{3-}]}, \quad (7)$$

where: $[C_{18}TBA]_3LnL3]$ is concentration of the lanthanide complex in the stationary phase and $[Ln^{3+}]$, $[LnL^+]$, $[LnL2^-]$ and $[LnL3^{3-}]$ are concentrations of the respective species in the solution.

It can be shown that making use of the Eqs. (1–5), this is equivalent to the formula:

$$\lambda'_{Ln} = \lambda'_{LnL3} \left[\frac{\beta_3 [L^{2-}]^3}{1 + K_1 [L^{2-}] + \beta_2 [L^{2-}]^2 + \beta_3 [L^{2-}]^3} \right], \quad (8)$$

where: λ'_{LnL3} is bed distribution coefficient of the complex ion.

The separation factor of the two lanthanides of atomic numbers e.g., (Z) and ($Z - 1$) is thus given by:

$$\alpha^2 = \frac{\lambda'_{LnL3}^{(Z)}}{\lambda'_{LnL3}^{(Z-1)}} \left[\frac{\beta_3^{(Z)} (1 + K_1^{(Z-1)} [L^{2-}] + \beta_2^{(Z-1)} [L^{2-}]^2 + \beta_3^{(Z-1)} [L^{2-}]^3)}{\beta_3^{(Z-1)} (1 + K_1^{(Z)} [L^{2-}] + \beta_2^{(Z)} [L^{2-}]^2 + \beta_3^{(Z)} [L^{2-}]^3)} \right]. \quad (9)$$

As can be seen, the separation factor is a function of the ratio of bed distribution coefficients of the two complex ions and the expression dependent on concentration of the ligand and formation constants of complexes of the two elements. We can assume that distribution coefficients of complex ions of the two lanthanides with “ Z ” and “ $Z - 1$ ” atomic numbers will differ only slightly, i.e., their ratio is close to unity. Then calculations with the use of Eq. (8) treating λ'_{LnL3} as a constant, should show the differentiation of retention factors, k_{ret} (which are proportional to distribution coefficients, λ'_{Ln}) of the lanthanides as a function of atomic number. The calculated retention factors at 5 mM ODA, pH 4.60 are shown in Fig. 1a. Similar affinity pattern with only minor deviations has been observed in similar calculations performed for the pH range 3.0–9.0.

Ion Exchange Behavior of REE in the IIC System: Eternity C_{18} —5 mM ODA/8.6 mM TBAOH/0.6 mM HNO_3 , pH 4.60

Selected elution curves obtained when using Eternity C_{18} column as a stationary phase and 5 mM ODA/8.6 mM TBAOH/0.6 mM HNO_3 , pH 4.60 mobile phase, at 25 °C, flow rate 1 mL min⁻¹, are shown in Figs. 2 and 3. The uncommon order of affinity of the REE to the stationary phase is apparent. The calculated experimental values of retention factors of REE plotted as a function of atomic number are shown in Fig. 1b. Comparison of Fig. 1a, b demonstrates good agreement between the theoretical predictions

and experimental values with minor deviations only, including the Apparent Atomic Number (App.At.No.) of yttrium, which in both cases amounts to 67½. Experimental order of ion exchange affinity of REE in this system is: Dy ≈ Tb ≈ Ho > Gd > Er ≈ Y > Eu > Sm > Tm > Nd > Yb > Pr > Lu > Ce > La, i.e., is quite unusual and different from the popular chromatographic systems employed for REE separation in which most often the affinity changes monotonically with the increase or decrease of At.No. of the lanthanide.

Some Interesting Features of the IIC System: Eternity C_{18} —ODA/TBAOH

When the mobile phase consists of the mixture of ODA + TBAOH (without any other ions) and the C_{TBAOH} is kept constant, the retention factors of REE increase with the increase of C_{ODA} with the simultaneous decrease of pH as shown on the example of yttrium (cf. Fig. 4). Column performance was on average the best at pH 4.60.

pH is only one of the factors which have an influence on the quality of chromatographic separation in the system studied. Comparison of Fig. 5a, b demonstrates how profoundly the choice of concentration of the mobile phase components can affect the separation of REE at nominally the same pH 4.60 of the solution. The numbers of theoretical plates N , for individual peaks and separation factors of adjacent peaks α^2_1 , calculated in the usual way [12] are also quoted in Fig. 5 to better illustrate the problem. High concentrations of ODA and TBAOH in the mobile phase do not favor good separation and result in poor column performance (cf. Fig. 5a). With the mobile phase consisting of 5 mM ODA/8.0 mM TBAOH, the base line separation of all five elements has been achieved. The presence the other electrolyte (e.g., $NaNO_3$) in the mobile phase decreases the retention factors of all REE by usual ion exchange mechanism, but may also affect the values of separation factors. As follows from the comparison of Fig. 5b, c, the presence of nitrate ions caused a significant decrease of the separation factor of the Nd-Y pair.

The Separation of REE at Lower pH Values and the Position of Scandium in the Lanthanide Series

The position of scandium in the lanthanide series is of interest because it can be important e.g., in planning some general REE separation scheme combining if needed different chromatographic systems, (for a given composition of REE mixture). At pH 4.60, scandium is eluted before La, ahead of all other REE, i.e., has the App.At.No. 56½ (cf. Fig. 6a). At pH 2.53, Sc changes its elution position, enters the lanthanide series and elutes after Nd, (cf. Fig. 6b). So, in this case, its App.At.No. equals to 61 or 61½. It can be seen that

a

Theoretically predicted sequence of retention factors in the IIC system
with ODA/TBAOH mobile phase at 5mM ODA, pH=4.60

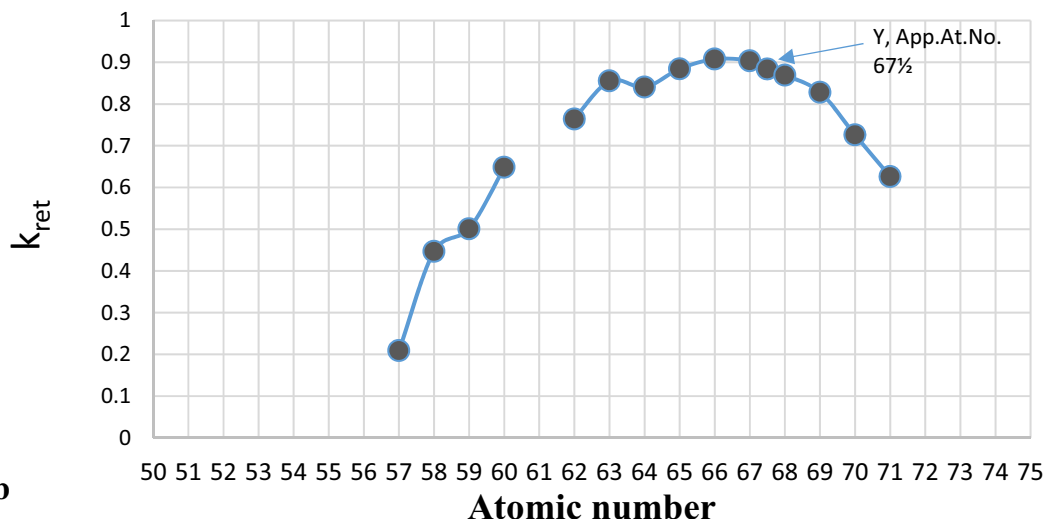
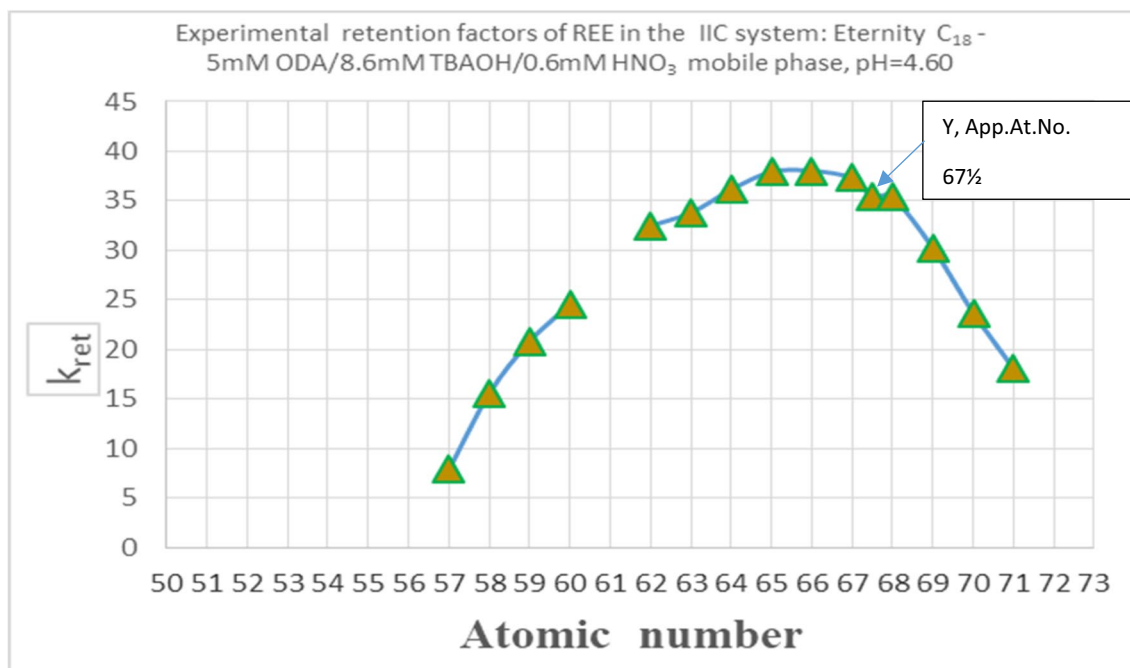
**b**

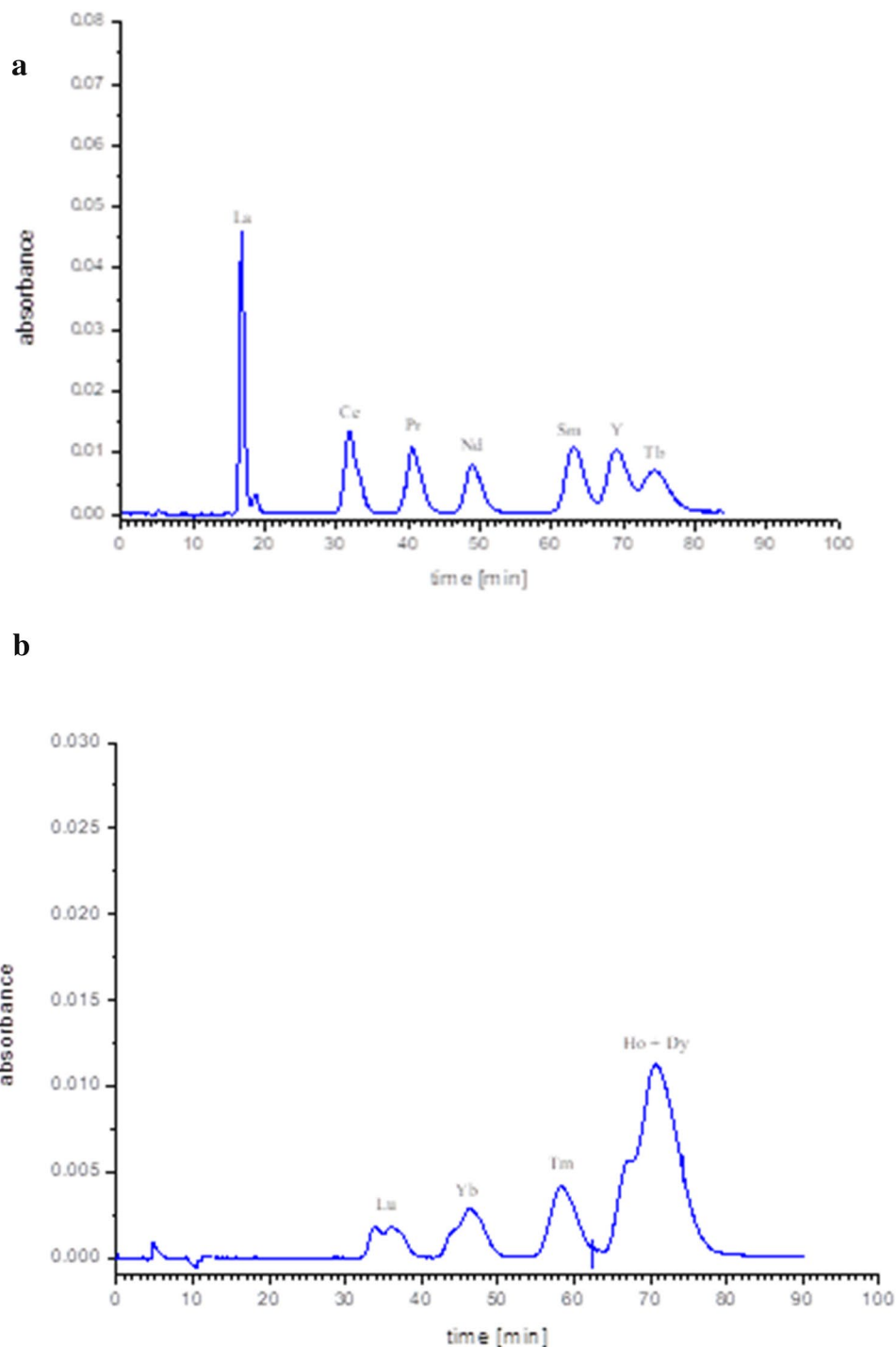
Fig. 1 a Theoretically predicted sequence of retention factors of REE as a function of Atomic Number (At.No.) of the lanthanide in the IIC system: stationary phase: RP column—mobile phase: TBAOH /ODA at pH 4.60; calculations were performed using Eq. 8, for 5 mM ODA, assuming that λ'_{LnL_3} can be treated as constant, i.e., the ratio of dis-

tribution coefficients of complex ions of two adjacent lanthanides is close to unity. **b** Experimentally determined retention factors of REE as a function of At.No. of the lanthanide in the IIC system: Column: Eternity C₁₈, mobile phase: 5 mM ODA/8.6 mM TBAOH/0.6 mM HNO₃; pH 4.60

at pH 2.53, the retention factors i.e., also the distribution coefficients of all REE are low, indicating that the conditions are already not very favorable for the separation of

Ln-ODA complexes. At pH 2.03 (cf. Fig. 6c), all REE are eluted jointly close to the dead volume of the column. At this pH and in more acidic media, the separation based on

Fig. 2 Chromatograms of several REE in the IIC system: Eternity C_{18} —5 mM ODA/8.6 mM TBAOH/0.6 mM HNO_3 ; pH 4.60; temp. 25 °C, injection volume 50 μ L, flow rate 1 mL min^{-1} , detection: visible absorbance at 658 nm after post-column reaction (PCR) with Arsenazo III. **a** La, Ce, Pr, Nd (10 mg L^{-1}), Sm, Y, Tb (20 mg L^{-1}). **b** Lu, Yb, Tm, Er, Ho, Dy (30 mg L^{-1})

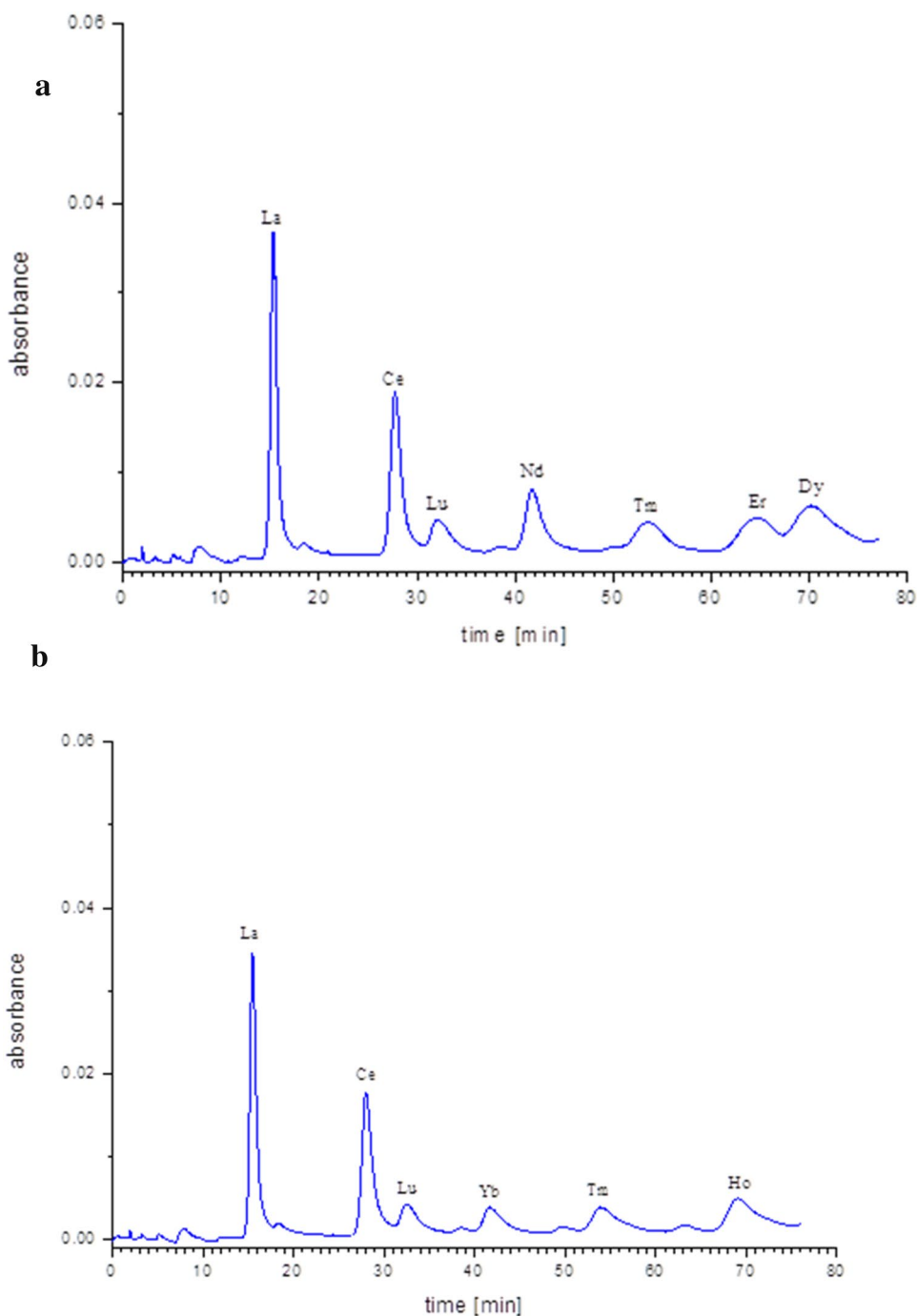


anion exchange mechanism using Ln-ODA complexes is impossible.

It is worth mentioning that scandium similarly as yttrium shows nomadic behavior and depending on the chromatographic system may change its position with respect to lanthanide series [5]. In the case of the system studied in this work, it is the lowering of pH that caused scandium move inwards the light lanthanides group (App.

At.No. 61 or 61½). Similar behavior of scandium but this time caused by the change of temperature was observed when separating REE using ion chromatography with Dionex IonPac CS3 sulfonic acid column and gradient elution with mandelic acid pH 4.0. At the temp. 25 °C, the elution order was: Sc, Lu, Yb, Tm, Er, Ho, Dy + Y, Tb, Gd + Eu, Sm, Nd, Pr, Ce, La, i.e., Sc had the App. At.No. of 71½. At the temp. 70 °C Sc moved inward the

Fig. 3 Chromatograms of several REE in the IIC system: Eternity C₁₈—5 mM ODA/8.6 mM TBAOH/0.6 mM HNO₃; pH 4.60; temp. 25 °C, injection volume 50 µL, flow rate 1 mL min⁻¹, detection: visible absorbance at 658 nm after post-column reaction (PCR) with Arsenazo III. **a** La, Ce, Nd (10 mg L⁻¹), Lu, Tm, Er, Dy (30 mg L⁻¹). **b** La, Ce (10 mg L⁻¹), Lu, Yb, Tm, Ho (30 mg L⁻¹)



lanthanide series and coeluted with yttrium and dysprosium (App.At.No. 66) [13].

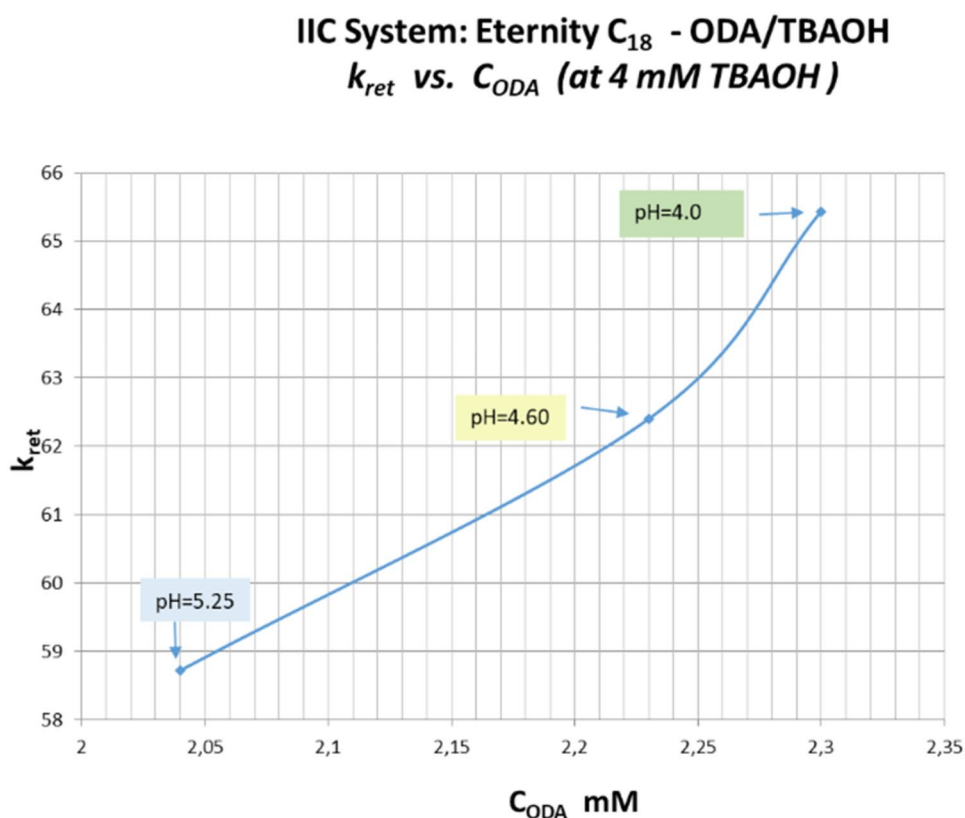
Possible Uses of the New Chromatographic System

Several precious and rare metals (including selected REE) are nowadays being used in high-tech technologies. Y, Eu, Ce, Tb, La are needed for fabrication of cathode ray tubes and fluorescent lamps, Ce, La, Sm, Pr, Nd are used for the production of batteries, La, Tb for light emitting diodes and

Nd, Dy, Pr, and Gd for speakers and magnets [14]. In the last decades, the problem of recovery of precious and rare metals from various forms of electronic wastes gained considerable attention and has been discussed in several papers and reviews [14–16]. Preliminary separation of REE from other elements is usually achieved by solvent extraction and/or ion exchange [3–5].

The process of separation of recovered REE should assure high purity of the individual REE species. Chromatographic methods could perhaps find use in the final act of

Fig. 4 The effect of changing the complexing agent concentration (C_{ODA}) at constant C_{TBAOH} on retention factor of yttrium in the IIC system: Eternity C_{18} —ODA/TBAOH mobile phase (at $C_{\text{TBAOH}}=4$ mM)



separation of REE mixture into individual components as well as for the analytical control of the purity of the final product. The IIC described in this paper, and the other IIC method employing anionic REE complexes with nitrilotriacetic acid (NTA) [17] could possibly also be used for that purpose. The IIC has the advantage over the ion chromatography employing strongly basic anion exchange columns because it can also work at elevated temperatures without the danger of irreversible damaging the column used as a stationary phase [18].

Conclusions

New chromatographic system for REE separation by anion exchange mode employing IIC with ODA as a complex forming agent and TBAOH as a reagent dynamically creating anion exchange sites on RP column was investigated theoretically and experimentally. It was demonstrated that knowing the formation constants of REE anionic complexes with ODA, it was possible to predict the affinity

sequence of individual lanthanides and yttrium to the stationary phase in IIC. The affinity appeared to be a non-monotonic function of atomic number of the lanthanide. The calculated retention factors increased from La to Tb-Dy-Ho region and then decreased down to Lu. Yttrium had an App.At.No. of $67\frac{1}{2}$. Retention factors calculated from chromatograms obtained in the IIC system: Column: Eternity C_{18} —mobile phase: 5 mM ODA/8.6 mM TBAOH/0.6 mM HNO_3 ; pH 4.60 gave very similar affinity pattern as that calculated. This shows the potential possibility of doing such predictions also in the hypothetical case of other complex forming agents which form anionic complexes with REE and for which their complex formation constants are known.

Base line separations for certain groups of RFE, in particular, the light and the heaviest lanthanides, respectively, can be easily achieved. At $\text{pH} > 4.0$, scandium is eluted ahead of the whole REE group. At pH 4–5 in this chromatographic system, the order of elution is very unusual namely: $\text{Sc} < \text{La} < \text{Ce} < \text{Lu} < \text{Pr} < \text{Yb} < \text{Nd} < \text{Tm} < \text{Sm} < \text{Eu} < \text{Er} \approx \text{Y} < \text{Gd} < \text{Ho} < \text{Tb} \approx \text{Dy}$.

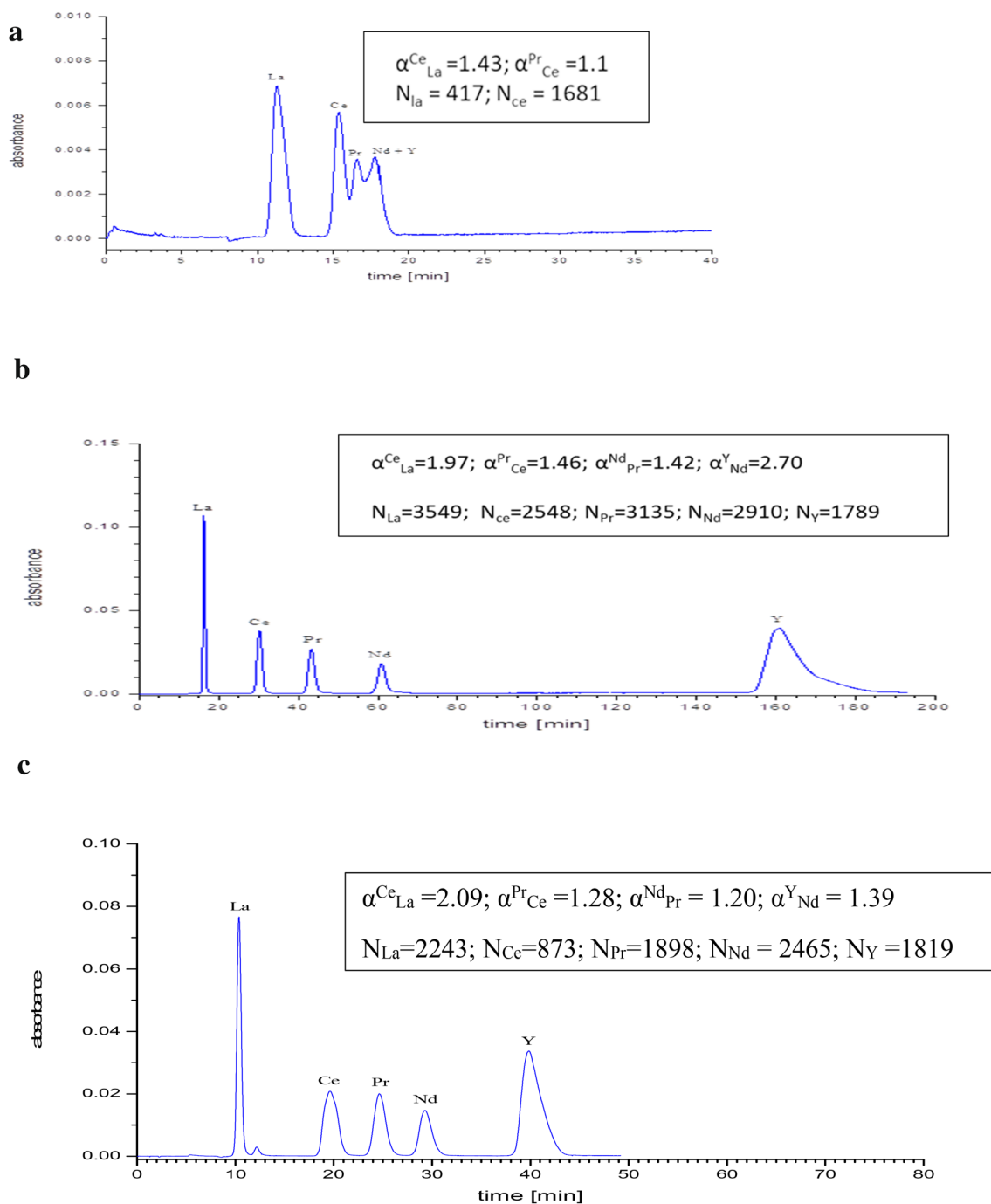
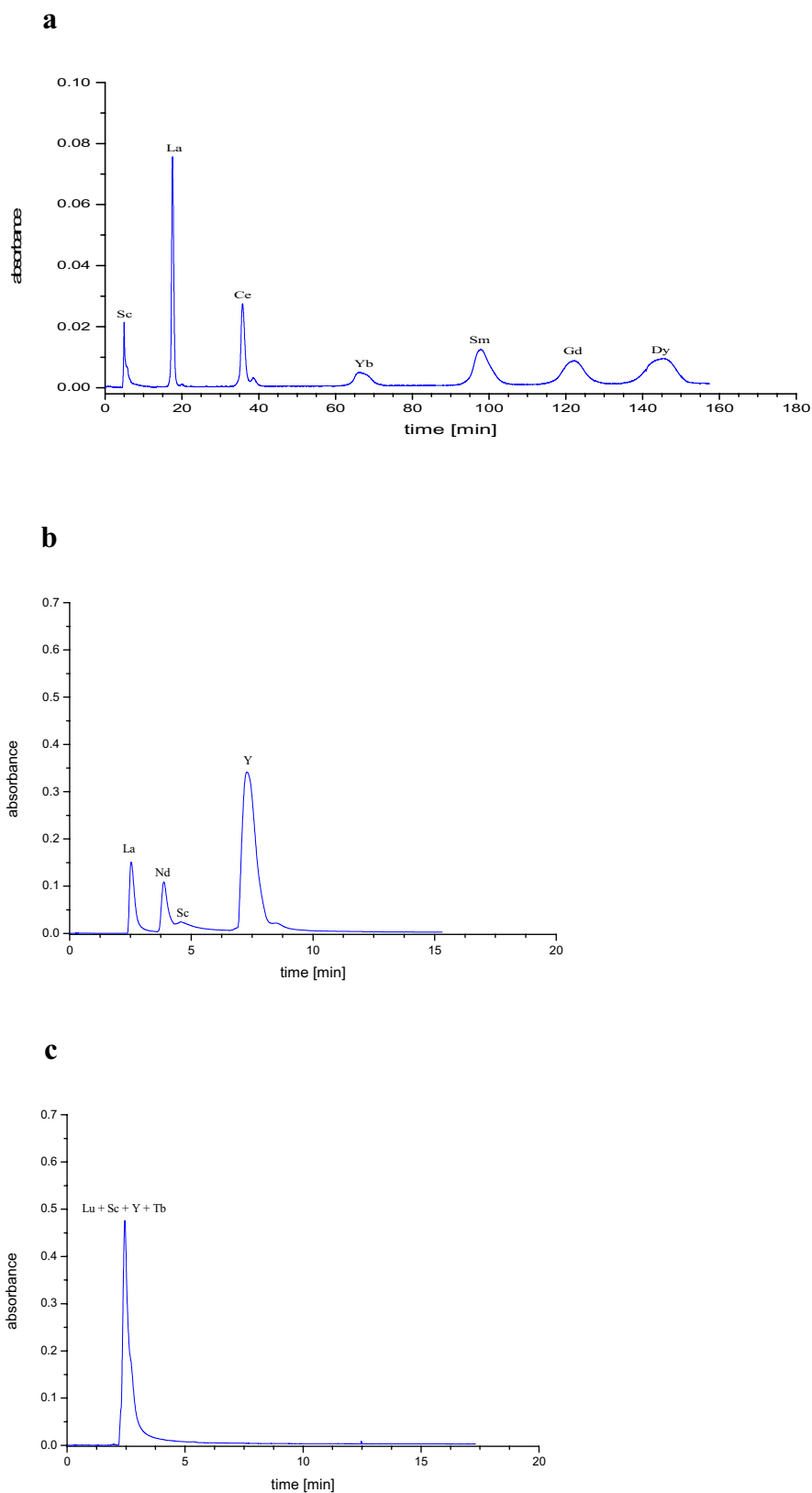


Fig. 5 Effect of mobile phase composition on chromatographic separation of REE. IIC system: Eternity C18—ODA/TBAOH, pH 4.60. temp. 25 °C, injection volume 50 μ L, flow rate 1 mL min^{-1} , detection: visible absorbance at 658 nm after post-column reaction (PCR) with Arsenazo III. Separation factors α^2_1 , and the numbers of theo-

retical plates N , are shown on the figures. **a** 20 mM ODA/34.6 mM TBAOH; La, Ce, Pr, Nd (10 mg L^{-1}), Y (30 mg L^{-1}). **b** 5 mM ODA/8.0 mM TBAOH; La, Ce, Pr, Nd (10 mg L^{-1}), Y (30 mg L^{-1}). **c** 5 mM ODA/8.6 mM TBAOH/7 mM NaNO_3 ; La, Ce, Pr, Nd (10 mg L^{-1}), Y (30 mg L^{-1})

Fig. 6 Effect of pH on REE separation including the changeable position of scandium. IIC system: Eternity C18—ODA/TBAOH, temp. 25 °C, injection volume 50 μL , flow rate 1 mL min^{-1} , detection: visible absorbance at 658 nm after post-column reaction (PCR) with Arsenazo III. **a** 23 mM ODA/4 mM TBAOH; pH 4.60; La, Ce, (10 mg L^{-1}), Yb, Sm, Gd, Dy (20 mg L^{-1}), Sc 30 mg L^{-1} . **b** 2 mM ODA/4 mM TBAOH/6.2 mM HNO_3 ; pH 2.53; La, Nd, (10 mg L^{-1}), Sc, Y (30 mg L^{-1}), c 2 mM ODA/4 mM TBAOH/14.4 mM HNO_3 ; pH 2.03; Lu, Sc, Y, Tb, Sc (30 mg L^{-1})



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Declarations

Conflict of Interest The authors declare that they have no conflict of interests.

Human and Animal Rights This article does not contain any studies with human participants or animals performed by any of the authors.

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