# Isocratic separation of lanthanides and yttrium by high-performance chelation ion chromatography on iminodiacetic acid bonded to silica

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## Abstract

The variation of chromatographic retention of lanthanide ions and yttrium on a column packed with iminodiacetic acid bonded to silica was studied. The influence of nitric acid concentration, ionic strength of the eluent and temperature as well as complexing agents (diglycolic, maleic acid) was investigated. It was shown that with nitric acid as eluent in the presence of $0.5\ M$–$1.0\ M$ potassium nitrate the retention of the lanthanides under increased temperature is defined mainly by the stability of the corresponding surface complexes with iminodiacetic acid functional groups. The combination of increased ionic strength of the eluent ($0.5\ M$ potassium nitrate) with increased temperature of the chromatographic column ($65°C$) allowed, under optimal concentration of nitric acid ($1.6 \cdot 10^{-2}\ M$), the isocratic separation of 14 lanthanide ions and yttrium in 65 min. © 1998 Elsevier Science B.V.

*Keywords:* Mobile phase composition; Silica–iminodiacetic acid; Lanthanides; Yttrium; Metal cations; Rare earth ions

## 1. Introduction

The first chromatographic separations of the lanthanides were performed during the Second World War as a constituent of the Manhattan project and were published a few years later in 1947 in a special issue of the Journal of the American Chemical Society. Kettele and Boyd achieved a separation of 5–7 heavy lanthanides under isocratic conditions on a column packed with Dowex-50 in 2000 min [1]. Since that time a variety of liquid chromatographic methods such as anion, cation ion chromatography and ion-pair reversed-phase liquid chromatography have been successfully used for the separation of the lanthanides. A number of reviews on the separation of the lanthanides have been published [2–4].

There is one special feature of all the developed techniques. Due to the large difference in ionic radii of the lanthanides and hence a drastically changing ion-exchange selectivity for the cations between La(III) and Lu(III), a complete chromatographic separation of all lanthanides can be achieved in a reasonable time only when applying gradient elution. So gradient separation techniques have played a dominant role in the separation and determination of rare earth elements over the last several decades. Thus the isocratic chromatographic separation of the lanthanides remains one of the most challenging tasks in inorganic chromatography.

Several attempts to achieve an efficient isocratic separation of the lanthanides have been published. Sevenich and Fritz used ethylendiamine-tartrate eluent for the separation of eight lanthanides on a column packed with a low capacity polymer based

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strong cation-exchanger [5]. Elchuk and Cassidy used a more efficient ion-pair reversed-phase high-performance liquid chromatography (HPLC) system for the separation of the light lanthanides and determination of La(III) and Nd(III) in uranium dioxide fuel [6] and for determination of several fission products in thorium–uranium dioxide fuel [7]. Walker [8] separated nine heavy lanthanides using a polystyrene based PLRP-S resin (Polymer Laboratories, Amherst, MA, USA) dynamically modified with phenolsulfophthalein dye Phenol-Red with α-hydroxyisobutyric acid as the eluent. So, generally it has not been possible to separate more than eight or nine lanthanides in a single run using ion-exchange chromatographic systems in isocratic mode.

Recently, the possibility of isocratic chelation ion chromatographic separation of 14 lanthanides and yttrium was demonstrated with a chromatographic column (250×4 mm) packed with iminodiacetic acid bonded to silica and with diluted nitric acid solutions in the presence of potassium nitrate as the eluent [9]. The aim of this present paper is to present a more detailed study of the key parameters affecting the regularity of chromatographic retention of lanthanides on this column.

## 2. Experimental

### 2.1. Apparatus

An isocratic ion chromatographic system consisted of a DX-500 HPLC pump (Dionex, Sunnyvale, CA, USA), a Rheodyne 7010 polyether ether ketone liquid six port injection valve (Rheodyne, Cotati, CA, USA) fitted with a 20 μl or 100 μl sample loop and a postcolumn detection system. The latter included a Constametric III HPLC pump (LDC, Riviera Beach, FL, USA) used for delivery of Arsenazo III postcolumn reagent, a zero dead volume poly(tetrafluoroethylene) (PTFE) tee followed by a 1.4 m×0.3 mm ID PTFE reaction coil and Dionex spectral array detector set at 658 nm. The postcolumn reagent was $1.5 \cdot 10^{-4}$ $M$ Arsenazo III in 0.5 $M$ acetic acid. The flow-rate of the postcolumn reagent was 0.5 ml/min unless otherwise stated. A water bath was used for thermostating the stainless steel column (250×4 mm) packed with 6 μm particle size Diasorb IDA silica bonded with iminodiacetic acid purchased from JV BioChemmack (Moscow, Russia). The efficiency of the column calculated from peak benzene retained in pure hexane was equal to $1.2 \cdot 10^{4}$ theoretical plates.

### 2.2. Reagents

All reagents were of analytical grade unless otherwise stated. Nitric acid, potassium nitrate, acetic acid (all AnalaR grade, BDH, Poole, UK), maleic acid (Aldrich, Gillingham, UK) and diglycolic acid (97% purity, Fluka, Glossop, UK) were used for the preparation of eluents and postcolumn reagent. Arsenazo III (65% purity) was obtained from Fluka. ICP (1000 ppm) standard stock solutions of rare earth elements in 5% (v/v) nitric acid as well as a mixture of 100 ppm 16 rare earth elements (13 lanthanides, lanthanum, yttrium and scandium) in 5% (v/v) nitric acid were obtained from Johnson Matthey (Karlsruhe, Germany). All solutions were prepared using deionised water from Milli-Q system (Millipore, Bedford, MA, USA) and stored in poly-(propylene) bottles (BDH).

## 3. Results and discussion

### 3.1. The basic consideration for the choice of chelation ion chromatography as a separation tool for lanthanides

The effective ionic radii of the lanthanides monotonically decreases with atomic number from 1.320 Å for La to 1.194 Å for Lu [10]. Thus, in accordance with the theory of ion-exchange a rather moderate decrease of strength of electrostatic interactions with cation-exchanger must be in the same order. However, trivalent lanthanides ions have a very high affinity for cation-exchangers. Thus, the experimentally found [11] values of coefficients of distribution for the system Dowex 1×4 0.1 $M$ nitric acid–methanol (10:90) have more than two orders of magnitude difference between Lu(III) and Nd(III) (Fig. 1) and in the case of extrapolation of this dependence, using data obtained for the more concentrated solution of nitric acid (7 $M$), the difference in $K_D$ values between Lu(III) and La(III) must be
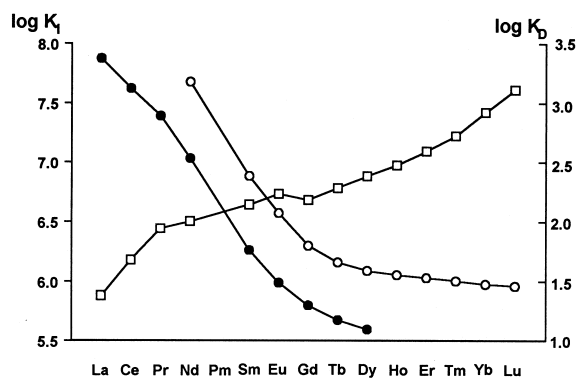
Fig. 1. Formation constants (log $K_1$; □) for chelates of lanthanide ions with iminodiacetic acid (from Ref. [12]) and corresponding distribution coefficients (log $K_D$) for cation-exchanger Dowex 1×4 in 0.1 $M$ (○) or 7 $M$ (●) nitric acid–methanol (10:90) mixture (calculated from the data of Tables LC91 in Ref. [11]).

around three orders of magnitude. For this reason isocratic ion chromatographic separation is impossible without any complexation in the mobile phase or at the surface of a properly selected chelating ion-exchanger. As a rule, addition of a complexing agent such as those commonly used for the separation of lanthanides, i.e., α-hydroxyisobutyric acid [6–8], mandelic [13], diglycolic [14] or nitrilotriacetic acid [15], should decrease the effective charge of separated ions and hypothetically simplify the possibility for isocratic separation. Nevertheless, a concentration gradient elution of at least one order of magnitude range has to be used for the complete elution of all lanthanides in a reasonable time.

A reversed chromatographic system where chelation takes place at the surface of a stationary phase or so called high-performance chelation ion chromatography has attracted attention over the last decade mainly due to the development of new efficient chelating exchange substrates, and has been recently reviewed [16]. The chelating exchangers usually exhibit the opposite effect to cation-exchange affinity for the lanthanides and give rise to the possibility of isocratic separation. Liu et al. [17] achieved isocratic separation of La(III), Sm(III), Eu(III) and Er(III) on a 25 cm length column packed with relatively coarse particles (230–325 mesh, 8% crosslinking) of N-methyl-γ-aminobutyrohydroxamate functionalised

resin. However, the chelating exchangers with aminocarboxylic functional groups were found to be a better option for this purpose. Inoue et al. [18] demonstrated the efficient separation of 13 lanthanides in a slight concentration gradient of nitric acid from 20 to 80 m$M$ over 25 min using a 15 cm length column packed with 10 μm methacrylate based resin with attached lysine-α-N,N′-diacetic acid functional groups. In our opinion, the main reason for the application of the concentration gradient was due to the relatively high stability of the complexes of lanthanides with the attached chelating groups, which was considered by the authors as an analog of nitrilotriacetic acid and hence the stronger retention of these ions in the chromatographic column.

A weaker chelating ion-exchanger is the iminodiacetic acid functionalised chelating silica gel that has been successfully used for efficient separation of alkaline-earth [19,20] and transition metal ions [21–24]. Usually, with low pH eluents and in absence of high ionic strength, iminodiacetic acid–silica works both as a traditional cation-exchanger of moderate strength and a chelation-exchanger. The relative proportion of these two separation mechanisms affecting the retention of metal ions depends on the size and charge of retained ions and on the stability of related complexes formed at the surface of the ion-exchanger. In the case of the lanthanides, the affinity of iminodiacetic functional groups for cations due to electrostatic or traditional ion-exchange forces increases from Lu(III) to La(III) (Fig. 1). There is three orders of magnitude difference in the values of distribution coefficients between Lu(III) and La(III). At the same time the chelation effect is more strongly expressed for heavy lanthanides and hence the chelation affinity of iminodiacetic acid groups should be decreased from Lu(III) to La(III). The log values of the stability constants for complexes of lanthanides with iminodiacetic acid vary from 5.88 for La(III) to 7.61 for Lu(III) (Fig. 1 and Table 1). This complexation is likely to compensate for the strong affinity due to ion-exchange when using iminodiacetic acid functionalised chelating exchanger making the isocratic separation of lanthanides more likely. Our preliminary study [9] demonstrated the possibility of isocratic separation of the lanthanides and confirmed the soundness of this approach.

Table 1
Stability constants of complexes of lanthanides with some agents

| Lanthanide | log $\beta_1$ [12] | | |
|---|---|---|---|
| | Iminodiacetic acid | Diglycolic acid | Maleic acid |
| La(III) | 5.88 | 4.93 | 3.44 |
| Ce(III) | 6.18 | 5.16 | |
| Pr(III) | 6.44 | 5.33 | 3.63 |
| Nd(III) | 6.50 | 5.45 | 3.66 |
| Sm(III) | 6.64 | 5.55 | 3.82 |
| Gd(III) | 6.68 | 5.53 | 3.83 |
| Eu(III) | 6.73 | 5.40 | 3.79 |
| Tb(III) | 6.78 | 5.23 | 3.74 |
| Dy(III) | 6.88 | 5.31 | 3.75 |
| Ho(III) | 6.97 | 5.28 | 3.67 |
| Er(III) | 7.09 | 5.34 | 3.64 |
| Tm(III) | 7.22 | | 3.62 |
| Yb(III) | 7.42 | 5.55 | 3.64 |
| Lu(III) | 7.61 | 5.64 | 3.59 |

### 3.2. The influence of the nature of the eluent

Taking into account the relatively high stability of complexes of lanthanides (Fig. 1) with iminodiacetic acid it was possible to expect that the chelation will be mainly responsible for the retention of lanthanide ions on a iminodiacetate column and the use of a simple acid eluent would provide a selective separation of these ions. However, the initial separation of a standard mixture of 16 rare earth elements performed with $2.3 \cdot 10^{-2}$ *M* solution of nitric acid as eluent showed the reasonable separation of only light lanthanides La(III), Ce(III), Pr(III), Nd(III) and Y(III) (Fig. 2A). All other lanthanide ions were eluted as two broad poor resolved peaks and Sc(III) was strongly retained and could not be eluted from the column. It should be noted that the retention order from La(III) to Lu(III) corresponds mainly to the growth of the stability of related complexes, and not to the cation ion-exchange affinity. However, the poor separation of Sm(III)–Lu(III) ions can be explained by the additional presence of significant cation-exchange effects arising from the intensely charged trivalent lanthanide cations under these conditions. As one carboxylic group from the iminodiacetate functionality is partially dissociated in acidic eluent (p$K_1$ value for iminodiacetic acid is about 2.1 [21]), so the chelating substrate has properties of a cation-exchanger of moderate strength

too, as was noted in previous studies [19,20]. So the occurrence of two mechanisms in the column with different selectivity towards lanthanides (Fig. 1) does not allow the resolution of the heavy lanthanides using only nitric acid eluent.

One possible way to decrease the role of cation-exchange in the considered chromatographic system is to decrease the effective charge of the lanthanide ions through partial complexation in the eluent. For this purpose, maleic acid and diglycolic acid solutions were tested. Maleic acid forms weak complexes of practically the same stability for all lanthanides (log $K_1 = 3.44$ for La(III) and 3.59 for Lu(III); see Table 1) and should have a relatively small effect on the selectivity of separation. The chromatogram obtained with $8 \cdot 10^{-2}$ *M* maleic acid was identical to that obtained with $2.3 \cdot 10^{-2}$ *M* nitric acid as eluent (Fig. 2B). Diglycolic acid forms more stable complexes with lanthanides (log $K_1 = 4.93$ for La(III) and 5.64 for Lu(III); see Table 1). Interestingly its use as an eluent causes the reversal of the elution order of lanthanides from the column which corresponds to cation-exchange separation of positively charged complexes of composition $ML^+$ (Fig. 2C). A good separation was achieved for light lanthanides Nd(III)–La(III) as well as for three heavy lanthanides Lu(III), Yb(III) and Tm(III), but the peaks of seven lanthanides and yttrium were poorly resolved.

### 3.3. The influence of the concentration of the eluent

The change in the concentration of the acid eluent affects the dissociation of the carboxyl ion iminodiacetic acid functional group of the chelating ion-exchanger. For this reason it can lead to a change of chelation ability of iminodiacetic functional groups and thus a change of selectivity. Fig. 3A shows the linear relationship between the retention (log $k'$) and concentration of nitric acid in eluent (log $C_{HNO_3}$). The slope of the plots is very close to 3, that is in accordance with both ion-exchange and chelating exchange for trivalent lanthanide cations. It should be specially noted that small changes in the concentration of nitric acid causes drastic changes in the retention of lanthanides, so fine adjustment of the concentration of the eluent is necessary to achieve
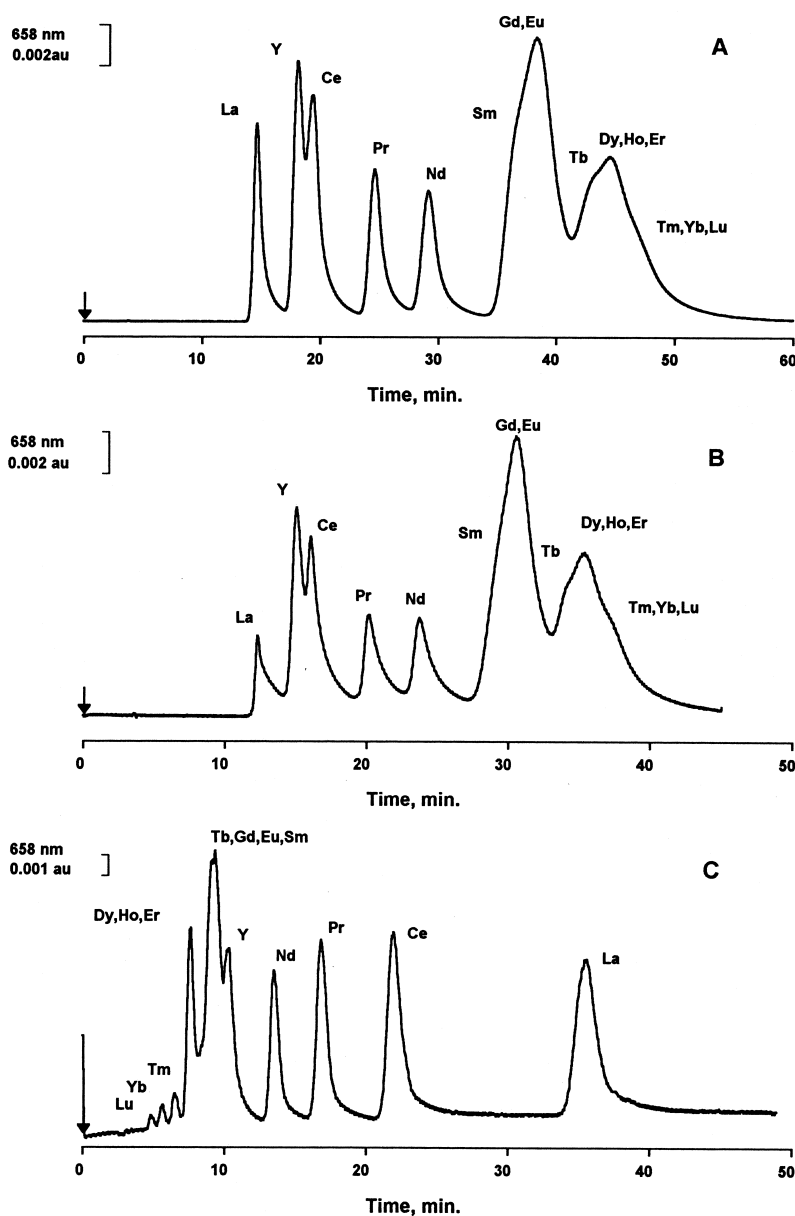
Fig. 2. Chromatogram of standard mixture of rare earth elements. Eluent: $2.3 \cdot 10^{-2}$ $M$ HNO$_3$ (A), $8.0 \cdot 10^{-2}$ $M$ maleic acid (B) and $1.0 \cdot 10^{-2}$ $M$ diglycolic acid (C); flow-rate 0.8 ml/min; 20°C; sample volume 100 μl (A,B) and 20 μl (C); sample concentration of each metal was 4 ppm in 0.2% HNO$_3$.

elution in a reasonable time. However, no particular changes in selectivity of separation were noticed. A deviation from linearity was observed for maleic and diglycolic acid based eluents (Fig. 3B and C). The plots have a concave shape with slopes lower than 3, which reflects the change in degree of dissociation of these acids with dilution and changes in the effective charge of separated cations in the case of diglycolic acid. Again, there was no observed improvement of selectivity of separation.
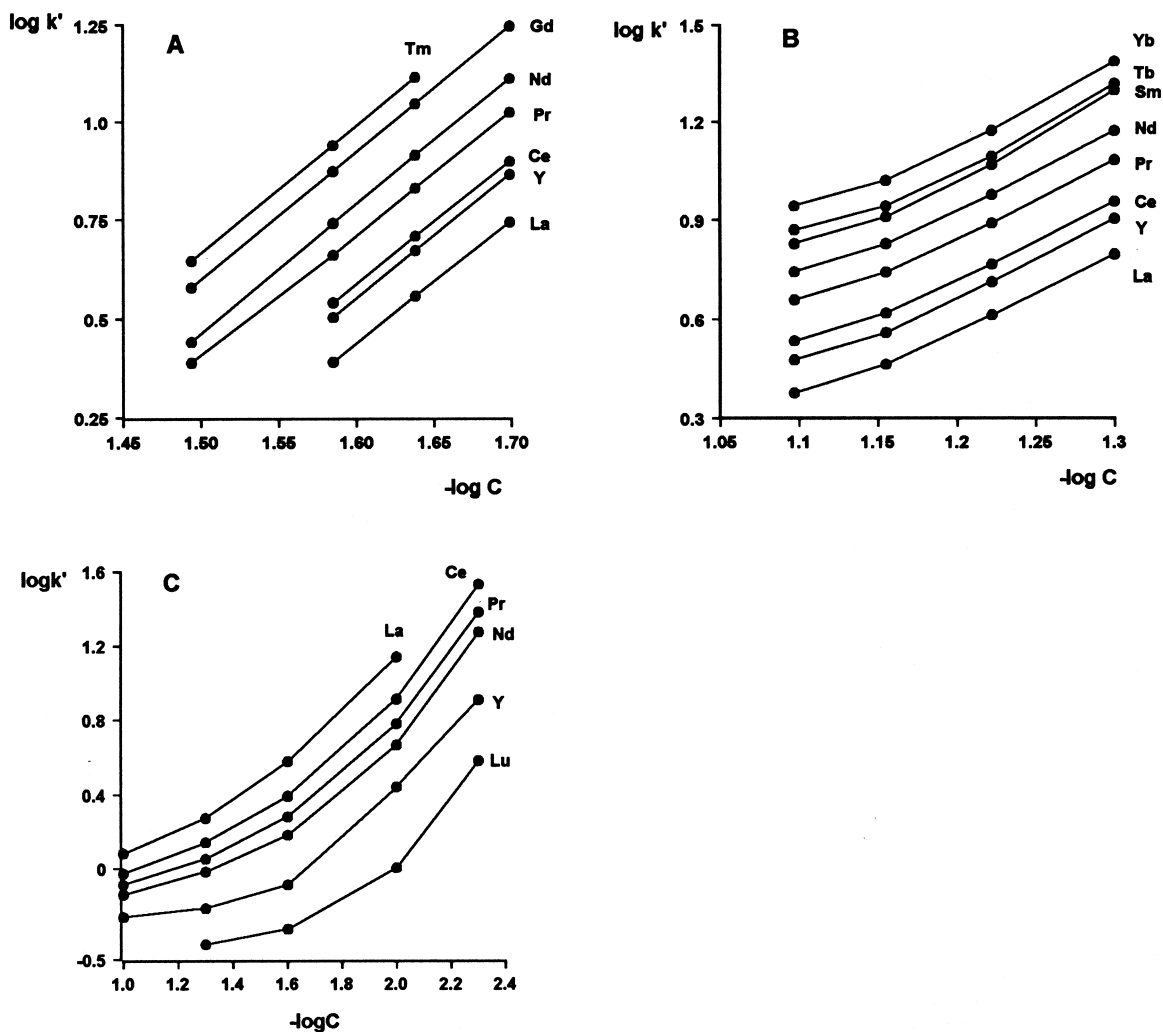
Fig. 3. The dependence of retention (log $k'$) of lanthanide ions as a function of logarithm of concentration of (A) nitric acid; (B) maleic acid and (C) diglycolic acid. $T$=20°C.

## 3.4. The influence of temperature

One more possibility for the improvement of the selectivity and efficiency of chelating exchange is to increase the temperature. Generally, an increase of column temperature improves the kinetics of chelation and changes the impact of chelating exchange on the retention of ions to a greater degree than in the case of simple ion-exchange [25]. In our case the increase of column temperature up to 50°C produced three effects (Fig. 4): (i) increase of retention times of lanthanide ions (the apparent decrease in retention

compared to Fig. 2A is because the flow-rate is faster and the acid concentration is higher); (ii) slight improvement of peak shapes and of separation efficiency and (iii) changes in the selectivity of separation. The last effect included the better resolution of the chromatographic peaks of the heavy lanthanides, Tm(III), Yb(III) and Lu(III) and the peaks of Sm(III), Gd(III), Eu(III) and Tb(III). It is interesting that the elution order of ions remains the same except that Y(III) changed position and eluted now after Ce(III). Nevertheless, neither a column temperature of 50°C nor further increase of column
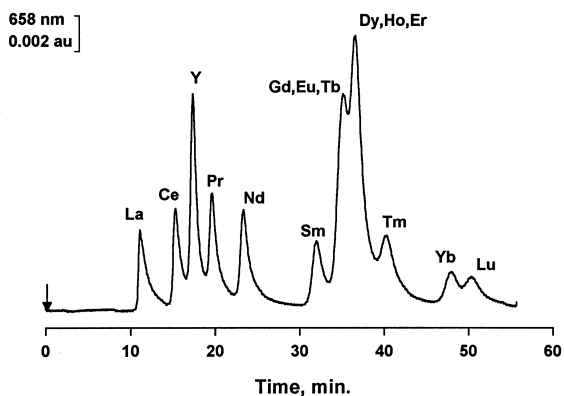
Fig. 4. Chromatogram of standard mixture of rare earth elements. Eluent: $2.6 \cdot 10^{-2}$ $M$ nitric acid; flow-rate 1.0 ml/min; 50°C; sample volume 100 µl; sample concentration of each metal was 4 ppm in 0.2% $HNO_3$.



Fig. 5. Chromatogram of standard mixture of rare earth elements. Eluent: $1.36 \cdot 10^{-2}$ $M$ nitric acid with 0.5 $M$ potassium nitrate; flow-rate 1.5 ml/min; flow-rate for postcolumn reagent 1.0 ml/min; 50°C; sample volume 20 µl; sample concentration of each metal was 4 ppm in 0.2% $HNO_3$.

temperature provided the complete separation of six lanthanide ions between Gd(III) and Er(III). Similar results were obtained with the use of maleic acid as eluent.

## 3.5. The effect of increased ionic strength in the eluent

An extremely useful approach for the suppression of ion-exchange effects was developed by Jones and coworkers [26,27] for a number of separations and determinations of alkaline-earth and transition metal ions in complex matrices. This approach includes the application of an eluent with increased ionic strength (between 0.5 and 1.0) that reduces substantially the electrostatic interactions between negatively charged carboxylic groups in chelating functional groups and the cations in the eluent. So, an increased concentration of the nitrates and perchlorates of alkali, and alkaline-earth metals or ammonia usually makes chelation the most dominating mechanism in the chromatographic separation of transition metal ions. A drastic improvement was observed in our case for the separation of lanthanide ions with $1.36 \cdot 10^{-2}$ $M$ nitric acid containing 0.5 $M$ potassium nitrate (Fig. 5). Obviously, the lower concentration of acid is required for elution of lanthanide ions from the column because of the decreased impact of cation-exchange in their retention. Under these conditions
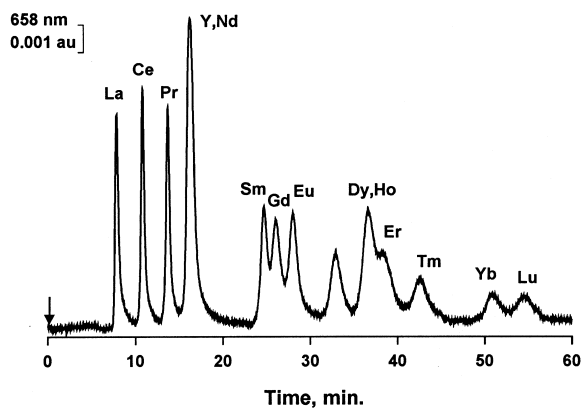
only the pairs Y(III)–Nd(III) and Dy(III)–Ho(III) stayed not quite resolved.

## 3.6. Optimization of separation

From the findings outlined above, it can be seen that both an increase of column temperature and an increase of ionic strength affect the selectivity of separation of lanthanide ions on the column packed with iminodiacetic acid bonded to silica gel. The comparative effect of these parameters on the capacity factors and selectivity coefficients of yttrium and the lanthanides with different concentrations of nitric acid in the eluent is presented in Table 2. The overall decrease in retention between 49°C and 65°C was due to the increase in acid concentration swamping the temperature effect. The effect of increase of column temperature on the selectivity of separation of lanthanides and yttrium under constant elution conditions is best seen in Fig. 6. One can see the significant improvement of selectivity of separation with increase of temperature due to slight differences in slopes of corresponding plots for middle and heavy lanthanide ions. The relatively stronger influence of temperature on the retention of Y(III) causing a major change in the elution order with a large movement of the yttrium peak in the chromatograms should be noted. Due to this behaviour of Y(III) the optimal temperature for the eluent with

Table 2
The capacity factors ($k'$) of lanthanide ions and selectivity of separation of adjacent peaks ($\alpha$) under different ionic strength and temperature of the eluent

| Metal | $1.36\cdot10^{-2}$ M HNO$_3$; 65°C | | | | $1.0\cdot10^{-2}$ M HNO$_3$; 49°C | | | |
|---|---|---|---|---|---|---|---|---|
| | 0.7 M KNO$_3$ | $\alpha$ | 1.0 M KNO$_3$ | $\alpha$ | 0.5 M KNO$_3$ | $\alpha$ | 1 M KNO$_3$ | $\alpha$ |
| La(III) | 2.63 | 1.71 | 1.97 | 1.58 | 5.86 | 1.60 | 3.33 | 1.64 |
| Ce(III) | 4.49 | 1.38 | 3.12 | 1.71 | 9.36 | 1.39 | 5.46 | 1.40 |
| Pr(III) | 6.21 | 1.23 | 5.33 | 1.25 | 13.03 | 1.24 | 7.67 | 1.24 |
| Nd(III) | 7.62 | 1.33 | 6.68 | 1.47 | 16.18 | 1.11 | 9.53 | 1.32 |
| Y(III) | 10.10 | 1.30 | 9.82 | 1.20 | 17.97 | 1.58 | 12.59 | 1.35 |
| Sm(III) | 13.09 | 1.12 | 11.76 | 1.18 | 28.33 | 1.18 | 17.04 | 1.16 |
| Gd(III) | 14.72 | 1.05 | 13.91 | 1.01 | 31.26 | 1.10 | 19.77 | 1.04 |
| Eu(III) | 15.44 | 1.30 | 13.99 | 1.38 | 33.46 | 1.25 | 20.51 | 1.34 |
| Tb(III) | 20.10 | 1.18 | 19.27 | 1.20 | 41.86 | 1.12 | 27.52 | 1.18 |
| Dy(III) | 23.66 | 1.03 | 23.07 | 1.05 | 46.80 | | 32.58 | 1.00 |
| Ho(III) | 24.46 | 1.10 | 24.11 | 1.11 | | | 32.70 | 1.07 |
| Er(III) | 26.97 | 1.16 | 26.82 | 1.21 | | | 35.04 | 1.12 |
| Tm(III) | 31.27 | 1.25 | 32.36 | 1.22 | | | 39.36 | |
| Yb(III) | 39.12 | 1.10 | 39.46 | 1.11 | | | | |
| Lu(III) | 43.17 | | 43.88 | | | | | |

ionic strength 0.7 is 65°C, when this ion is eluted between Nd(III) and Sm(III) taking the possible place of radioactive promethium. Thus, the optimised conditions for the isocratic separation of 14 lanthanides and yttrium were finally achieved (Fig. 7).

There are still two pairs of relatively poorly resolved chromatographic peaks; Gd(III)–Eu(III) and Dy(III)–Ho(III). The isocratic separation of these ions is a possibility, but it was found that any change in the parameters of the chromatographic system that caused enhancement of the selectivity of separation for one pair of ions, definitely caused a worse separation of the other pair of ions.

The retention of lanthanides as well as their elution order is in a good agreement with the values of the stability of lanthanide complexes with iminodiacetic acid. A strong correlation ($r=0.998$) of
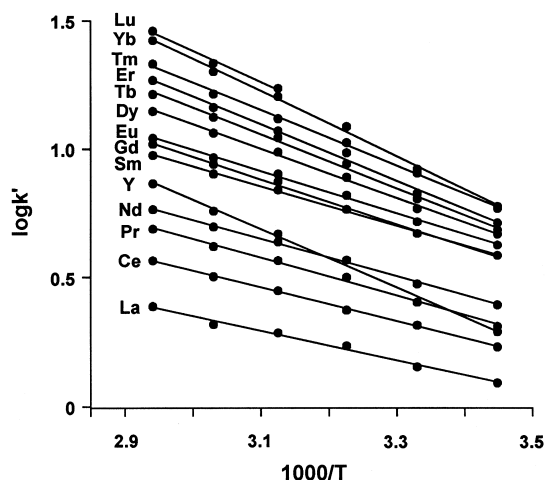


Fig. 6. The dependence of retention of lanthanide ions (log $k'$) as a function of inversed temperature (1000/$T$). Eluent: $1.6\cdot10^{-2}$ M nitric acid–0.7 M potassium nitrate.
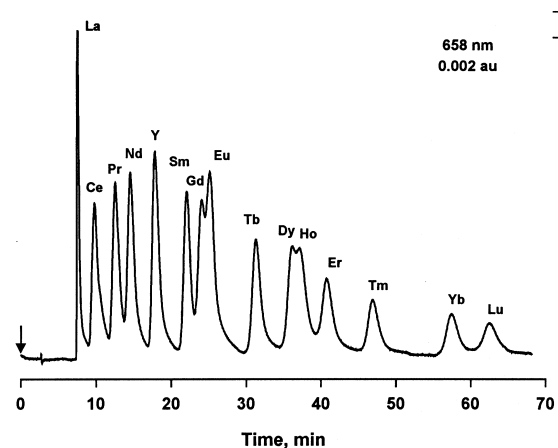


Fig. 7. Isocratic separation of standard mixture of 14 lanthanides and yttrium. Eluent: $1.6\cdot10^{-2}$ M HNO$_3$ with 0.5 M KNO$_3$; flow-rate 1.0 ml/min; column temperature 65°C; sample volume 20 µl; sample concentration of each metal was 4 ppm in 0.2% HNO$_3$.

the retention of lanthanide ions with values of the stability constants at 25°C was found [9]. It was considered that this provided a reasonable proof of the domination of chelating ion-exchange in this chromatographic system. The known effect of the "gadolinium break" for complexes of lanthanides with aminopolycarboxylic acids, where gadolinium has a smaller stability constant than the previously placed europium in the periodic table, was also in good agreement with the observed elution order for iminodiacetate column, with gadolinium eluting before europium.

## 4. Conclusions

The isocratic separation of lanthanides was achieved using the less common separation method known as high-performance chelation ion chromatography. The obvious advantage of this approach includes the possibility of prediction of elution order of metal ions, e.g., lanthanides, that allows the efficient use of the large volume of published values of stability constants [10] of different complexes for selecting a proper chelating exchanger to achieve a reasonable separation. This work also shows that high-performance chelation separations of complex metal mixtures such as the lanthanides and yttrium are definitely possible and dispels the idea that chromatographic systems based on chelating substrates give poor separation efficiencies.

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