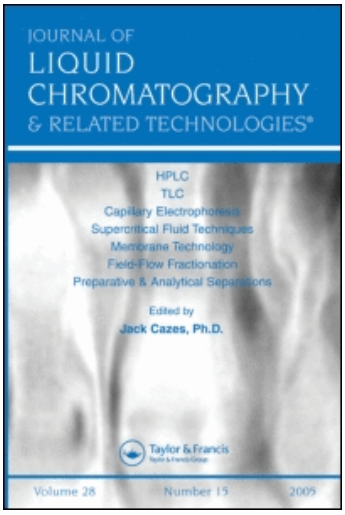
This article was downloaded by:
On: *24 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Separation and Column Performance of Certain Radiotracers Using a Chromatographic Column of Celite Loaded with Adogen-381 and Selected Eluents

M. Raieh[a]; S. M. Khalifa[a]; M. El-dessouky[a]; H. F. Aly[a]
[a] Nuclear Chemistry Department, Nuclear Research Centre, Cairo, Egypt

To cite this Article Raieh, M. , Khalifa, S. M. , El-dessouky, M. and Aly, H. F.(1984) 'Separation and Column Performance of Certain Radiotracers Using a Chromatographic Column of Celite Loaded with Adogen-381 and Selected Eluents', Journal of Liquid Chromatography & Related Technologies, 7: 3, 581 — 590
To link to this Article: DOI: 10.1080/01483918408073987
URL: http://dx.doi.org/10.1080/01483918408073987

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

JOURNAL OF LIQUID CHROMATOGRAPHY, 7(3), 581-590 (1984)

SEPARATION AND COLUMN PERFORMANCE OF CERTAIN RADIOTRACERS USING A CHROMATOGRAPHIC COLUMN OF CELITE LOADED WITH ADOGEN-381 AND SELECTED ELUENTS

M.Raieh, S.M.Khalifa, M.El-Dessouky and H.F.Aly
Nuclear Chemistry Department, Nuclear Research Centre,
Atomic Energy Establishment, Cairo, Egypt.

Downloaded At: 17:17 24 January 2011

ABSTRACT

The radiotracers Mo(VI), Sc(III), Ce(III), Eu(III), Tm(III), Fe(III), Hg(II), Cd(II), Zn(II), Cu(II) and Co(II) could be separated from each others using a hydrophobic celite column coated with Adogen-381 together with the mobile phases hydrochloric acid, nitric acid, potassium thiocyanate, ammonium acetate or water. The elution behaviour was satisfactorilly interpreted. The effect of the flow rate (50-300 ul/min) and temperature (26-70°C) on the column performance of Co(II) was also studied.

INTRODUCTION

Amines and quaternary ammonium salts found many applications in column chromatography (1-6). Different classes of amines were utilized for the separation of many elements using a variety of supporting agents and different mobile phases. In this contribution, the

Copyright © 1984 by Marcel Dekker, Inc. 0148-3919/84/0703-0581$3.50/0

system comprising the commercially available tertiary amine Adogen-381 together with stationary phase, hydrophobic celite and selected mobile phases of HCl, HNO_3, KSCN, CH_3COONH_4 or H_2O was employed to isolate eleven radiotracers from each others. Considering the importance of the flow rate and temperature on the column performance (7, 8) these parameters were also studied.

EXPERIMENTAL

Chemicals and radiotracers

Adogen-381 of average molecular weight 360 was delivered from Archer-Daniels Midland Company, USA. Commercial diatomaceous earth celite No. 560 was supplied from John Manville, USA. The radiotracers : Sc-46, Ce-141, Eu(152+154), Tm-170, Fe-59, Co-60, Cu-64, Zn-65, Cd-115, Hg-203, and Mo-99 were prepared by neutron activation, and their radiochemical purity exceeded 99%. All other chemicals and reagents were of A.R. grade and obtained from B.D.H. or Merck.

Downloaded At: 17:17 24 January 2011

Chromatographic Procedure

The stationary phase, celite, was dried at 105°C over night and made hydrophobic by the addition of 50 cm^3 of 5% silane-ethanol with thorough mixing. The mixture was left in a dessicator under slight vacuum to remove excess silane and ethanol. The hydrophobic celite was washed with distilled water to remove any chloride ions followed by oven drying at 105°C. The Adogen-381 was loaded on the hydrophobic celite by mixing 5.0 g with 100 cm^3 5% Adogen solution in acetone. Excess acetone was removed by evaporation with interrupted stirring at 80°C. A 0.5 cm i.d. x 10 cm

borosilicate glass column with platinum calibrated tip to produce 25 ul drop size was used. This column was loaded with 1.6 celite coated with Adogen-381 according to the procedure previously reported (9), to give a bed height of 9 cm. In all cases, the column was preconditioned with 5.0 cm^3 of the first eluent eomployed and the radiotracers were loaded on the column from a 50 ul syring . Unless otherwise stated, the eluent solutions were allowed to percolate with a flow rate of 75 ul/min. Known volume fractions of the eluent were collected for radiometric assay using an ORTEC-Scintillation counter.

The effects of the flow rate and temperature on column performance were studied using Co-60 and 2M HCl solution as eluent.

From the elution curve, the distribution coefficient, D, was determined using the relation (10) ;

$$D = (V_{max} - v) / w$$

where V_{max} is the eluent volume to the elution maximum, v is the free column and w is the weight of the stationary phase. The number of theoretical plates, N, was calculated by means of the equation (11) :

$$N = 8 (V_{max} / \beta)^2$$

where β is the band width of the elution peak at 1/e of the maximum elution concentration. Dividing the height of the column by N, the height equivalent to theoretical plate, HETP, was obtained.

RESULTS AND DISCUSSION

The radiochemical separation procedures developed in this work are based on the expected extraction behaviour of Adogen-381 and the types of the species of the different radiotracers in the different solutions used

Downloaded At: 17:17 24 January 2011

as eluents. Thus, a- the tertiary amine Adogen-381 is expected to extract both neutral and anionic species(12), b- the lanthanide elements are hardly complexed from hydrochloric or nitric acid solutions up to 8 molar (13), c- in aqueous HCl solution of concentrations higher than 8M, iron(III) is present in neutral or anionic chloride forms (14) and at lower HCl molarities, cationic iron chloride complexes are predominating, d- molybdate is extracted by tertiary amines from all hydrochloric acid concentrations and unextracted from all nitric acid concentrations (15), e- in HCl solutions, copper is present as the chloride complex, while from thiocyanate solutions it is poorly extracted by the tertiary amines due to the competition of SCN^- anion to the amine solution (15,16), f- zinc forms strong chloride and thiocyanate complexes which are extracted by tertiary amines, When zinc is slightly complexed from hydrochloric acid solution, water was found quite suitable to bring it to the free cationic state and thus be eluted. In presence of HCl and KSCN, water was found not enough to elute zinc in a suitable volume. Therefore, 1M NH_4CH_3COO eluted zinc, most probably as the strong cationic ammino complex (16), $[Zn(NH_3)_n]^{2+}$, g-cadmium is highly complexed by chloride ions and extracted by tertiary amine from HCl solution of different concentrations. Cadmium nitrate complexes are hardly present in nitric acid medium and the elution of Cd(II) using nitric acid was found convenient, h- mercury forms chloride and thiocyanate complexes, therefore, its uptake from these solutions is expected (16). Elution from high SCN^- solution is mainly related to the replacement of the SCN^- anion with the mercury complex.

In figures 1, and 2, the elution profiles of the eight radiochemical separation procedures developed are

Downloaded At: 17:17 24 January 2011

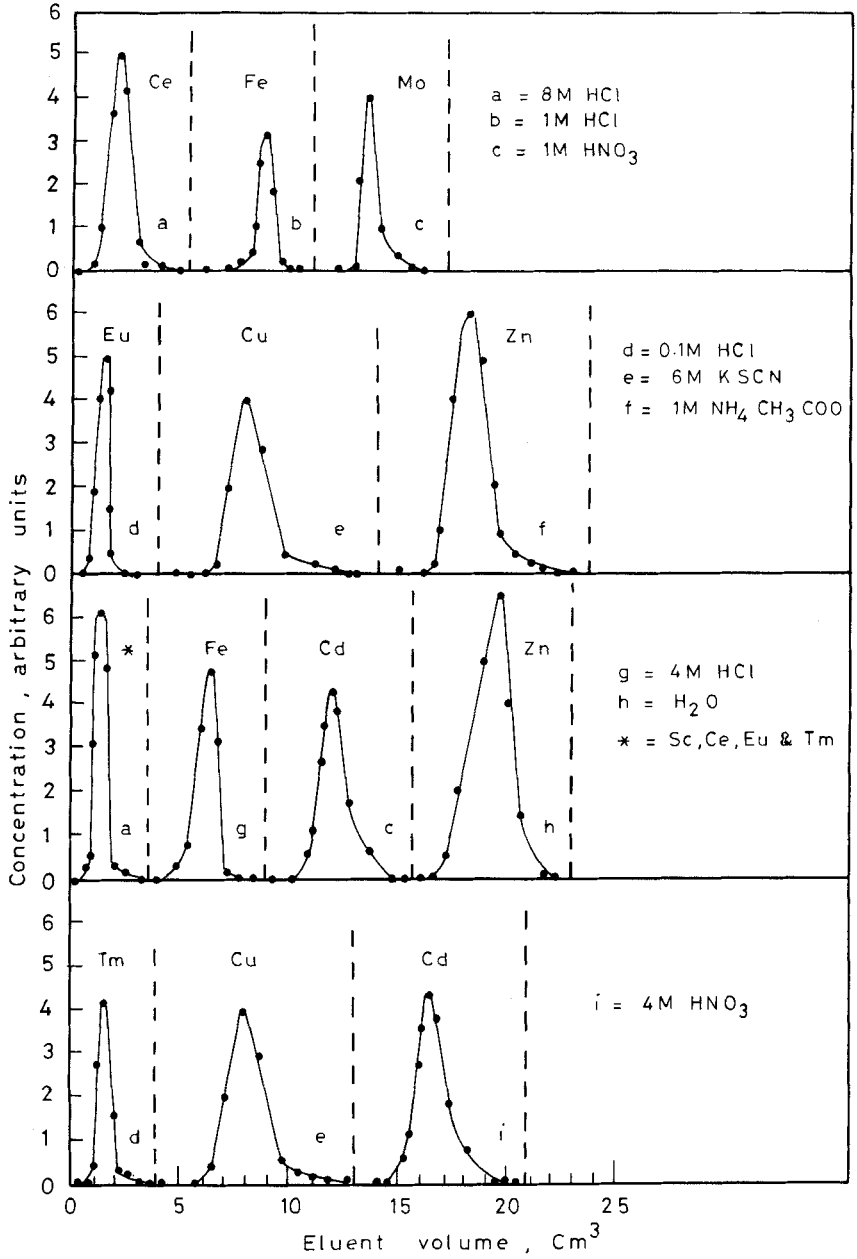


Figure 1 Elution profile : Ce(III), Fe(III), Mo(VI), Eu(III), Cu(II), Zn(II),(Sc(III), Ce(III), Eu(III) & Tm(III)), Tm(III) and Cd(II).

Downloaded At: 17:17 24 January 2011

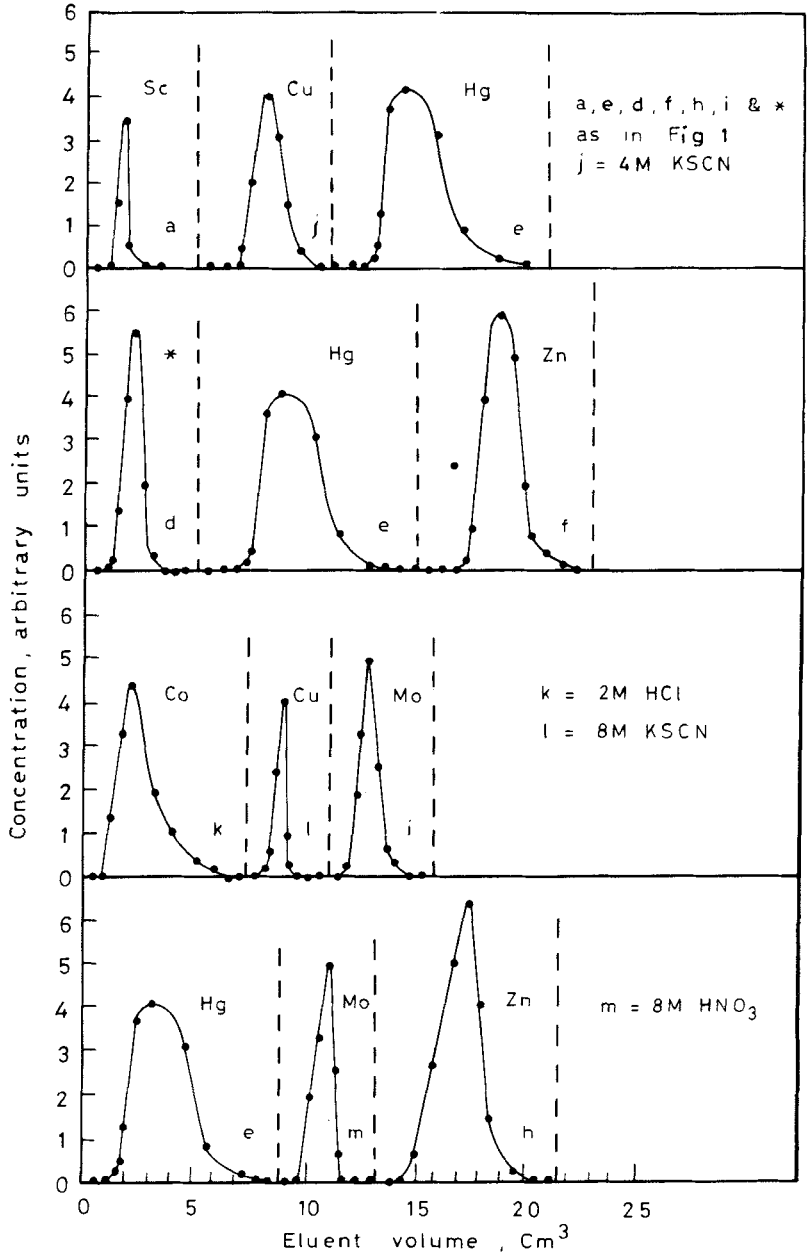


Figure 2 Elution profile : Sc(III), Cu(II), Hg(II), (Sc(III), Ce(III), Eu(III) & Tm(III)), Zn(II), Co(II) and Mo(VI).

Downloaded At: 17:17 24 January 2011

TABLE 1

Effect of Flow Rate on the Elution Profile(Co(II)

Flow rate ul/min.	Distribution coefficient.	Band width cm^3	Number of theoretical plates.	Plate height (HETP) mm
50	0.42	0.58	60.50	1.49
75	0.42	0.89	26.60	3.38
150	0.42	1.00	20.50	4.39
225	0.42	1.30	12.51	7.19
300	0.42	1.45	9.70	9.28

Downloaded At: 17:17 24 January 2011

given. In all cases, the investigations were carried out at 26±1°C. The total concentration of the radiotracers did not exceeded 10^{-4}M (equimolar amounts of each tracer). The radiochemical purity of each separated fraction was more than 98%.

In order to evaluate the column performance utilized, the effects of eluent flow rate and temperature on the elution profile were investigated using Co(II). It was found that, the distribution coefficient does not change with the flow rate (50-300 ul/min) whereby the band width of the eluted cobalt increased by increasing the flow rate (Table 1).

When the theoretical plate height was calculated and plotted against the flow rate, a linear relationship was obtained (Fig. 3). The effect of temperature on the distribution coefficient, elution profile and theoretical plate height of Co(II) is given in Table 2.

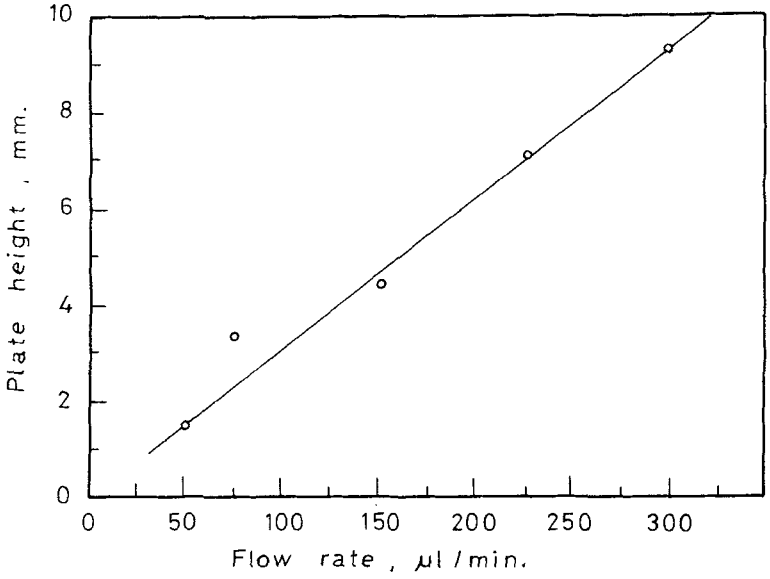


Figure 3 Effect of flow rate on the plate height for the elution of Co(II) by 2M HCl at 26±1°C.

TABLE 2

Effect of Temperature at 75 ul/min.

Temperature °C	Distribution coefficient.	Band width cm^3	Number of theoretical plate.	Plate height (HETP), mm
26	0.42	0.89	26.6	3.38
30	0.42	0.87	28.2	3.19
44	0.42	0.75	37.7	2.39
55	0.42	0.62	55.6	1.62
70	0.42	0.37	155.3	0.58

Downloaded At: 17:17 24 January 2011

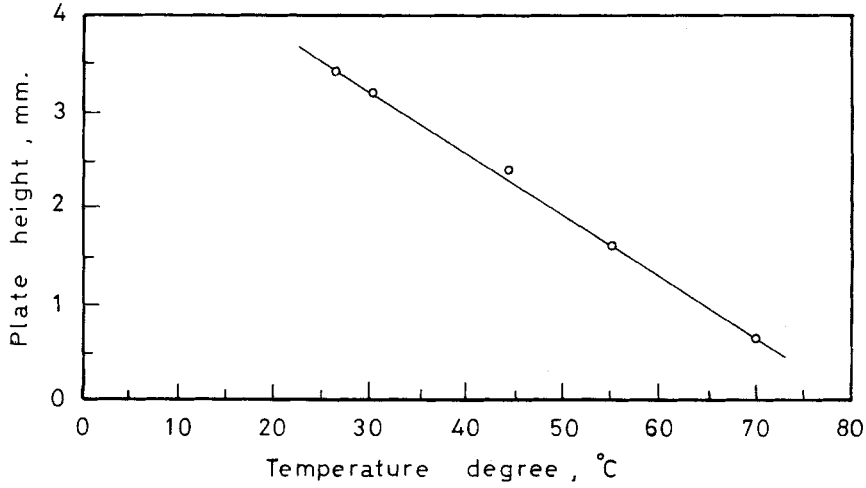


Figure 4 Effect of temperature on the plate height for the elution of Co(II) by 2M HCl at 75 ul/min.

Downloaded At: 17:17 24 January 2011

It is clear that within the temperature range investigated (26-70°C), the D value does not change, however, the band width decreased by increasing temperature. Subsequently, the number of theoretical plates of the column increased. This is illustrated in Fig. 4, where the theoretical plate height decreased linearly by increasing temperature.

REFERENCES

1. Mikulski, J. and Stronski, I., J. Chromatoger., 17, 197, 1965.
2. Akaza, I. and Inamura,J., J. Radioanal. Chem., 54, 27, 1979.
3. Horwitz,E.P., Orlandini,K.A. and Bloomquist,C.A.A., J. Inorg. Nucl. Chem. Letters, 2, 87, 1966.

4. Shimizu,T. and Ikeda,K., J. Chromatoger., 85, 123, 1973.
5. Aly,H.F. and Abdel-Rassoul,A.A., Z. Anorg. Allg. Chem., 387, 252, 1972.
6. Murray,R.W. and Passarelli,R.J., J. Anal. Chem., 39, 282, 1967.
7. Horwitz,E.P. and Bloomquist,C.A.A., J. Inorg. Nucl. Chem., 34, 3851, 1972.
8. Cerrai,E., Testa,C. and Triulizi,C., Energia, Nucleare, 9, 377, 1962.
9. Abdel-Rassoul, A.A., Aly,H.F. and Zakareia,N., Z. Anal. Chem., 27, 272; 1974.
10. Tompkins,E.R. and Mayer, S.W., J. Amer. Chem. Soc., 69, 2866, 1974.
11. Glueckauf,E., Trans. Faraday, Soc., 51,34,1955.
12. McKay,H.A.C., Healy,T.B. Jenkins,I.L. and Naylor,A., Solvent Extraction Chemistry of Metals, MacMillan, London, 1966.
13. Moeller,T., The Chemistry of Lanthanides, Pergamon Press, 1973.
14. Nicholls,D., The Chemistry of Iron, Cobalt and Nickel, Pergamon Press, 1973.
15. Sekine, T. and Hasegawa,Y., Solvent Extraction Chemistry, Marcel Dekker, Inc. 1977.
16. Sillen,L.G. and Marrell, A.E., Stability Constants of Metal-Ion Complexes, The Chemical Society, Special Publication No. 25, 1971.

Downloaded At: 17:17 24 January 2011