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Cation Exchange Chromatographic Separation of Scandium from Other Elements on Dowex 50W-X8

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Abstract

Cation exchange chromatographic studies of scandium on Dowex 50W-X8 are reported. Mineral acids and their salts were tested as eluants. Their efficiency was evaluated in terms of the elution constant and the bed distribution coefficient as $H_2SO_4 > CH_3COONH_4 > NH_4Cl > NaCl > HNO_3 > HCl > NH_4NO_3$.

Scandium was separated from alkali, alkaline earths, iron, zinc, cadmium, mercury, copper, indium, aluminum, vanadium, uranium, and bismuth by the process of selective elution. It was separated from titanium, zirconium, hafnium, and antimony by the process of selective sorption. Separation of cobalt, nickel, manganese, lead, and silver from scandium was accomplished with sodium chloride as an eluant. The separation from thorium was carried out by gradient elution with hydrochloric acid.

INTRODUCTION

Cation exchange separations of scandium from the lanthanide series of elements and trivalent metal ions have been carried out. However, systematic cation exchange chromatographic separations of scandium from common elements are lacking.

Scandium was separated from the lanthanons with 1 *N* sulfuric acid, while the other ions were eluted with 4 *N* hydrochloric acid (*1*). The acid–ammonium sulfate media (*2*) were used for the separation of scandium from many ions. Scandium was separated from gallium, indium, and

thallium (*3*) by gradient elution with hydrochloric acid. Ammonium lactate was used as an eluant (*4*) for its separation from alkaline earths, and an acetone–hydrochloric acid mixture was used (*5*) for its separation from manganese. Scandium was separated from lanthanons by gradient elution with nitric acid (*6*). Similarly, 0.1 *M* oxalic acid (*7*) and trioctylphosphine oxide in tetrahydrofuran (*8*) with hydrochloric acid were also used as eluants in such separations. The separation of scandium and yttrium was achieved (*9*) with EDTA–glycine solution. Recently, a method was developed by us for the anion exchange chromatographic separation of scandium from a large number of elements in malonic acid solutions (*10*).

This paper presents a systematic cation exchange chromatographic study of scandium on Dowex 50W–X8. Various eluants such as mineral acids and their salts were used. A large number of separations of scandium from other elements was also accomplished.

EXPERIMENTAL

Apparatus and Reagents

The ion-exchange column (1.4 × 20 cm) was similar to the one described earlier (*11*). A digital pH meter type pH–822 (Electronic Corporation of India Ltd.) with glass and calomel electrodes and automatic fraction collector (Emnvee Engineering Ltd., India) with a 25-ml siphon was used.

A stock solution of scandium was prepared by dissolving 2.017 g of scandium trioxide (Koch-Light, England) in 20 ml of concentrated nitric acid. Then the solution was diluted with distilled water to 250 ml and standardized complexometrically (*12*). It contained 5.26 mg/ml of scandium.

Dowex 50W–X8 (H^+ form, 20–50 mesh) from Dow Chemical Co., Midland, Michigan, was used.

RESULTS AND DISCUSSION

An aliquot of solution containing scandium (5.26 mg) was sorbed on the column. After washing the column with 50 ml of water, scandium was eluted with various eluants at a flow rate of 1 ml/min. The effluent lot was collected in 12 fractions of 25 ml each. Scandium from each frac-

tion was determined complexometrically (*12*). In the case of mineral acids as the eluants, it was necessary to evaporate the effluent to dryness before the complexometric determination of scandium (Table 1).

The elution constant (E) and volume distribution coefficient (D_v) were calculated (*10*) from the peak elution volume (V_{max}). On the basis of efficiency of eluants, the selectivity scale was $H_2SO_4 > CH_3COONH_4 > NH_4Cl > NaCl > HNO_3 > HCl > NH_4NO_3$.

Sodium chloride and nitrate in low concentrations and citric, oxalic, and ascorbic acids at all concentrations proved to be poor eluants for scandium. However, 1.5 M sulfuric acid proved to be the most suitable eluant for routine work due to its small peak elution volume (Fig. 1).

TABLE 1

Ion Exchange Studies of Scandium (Sc = 5.26 mg, weight of resin = 18.23 g)

Eluant	M	Peak elution volume (V_{max})	Total elution volume (V_t)	Scandium recovery (%)	Elution constant (E)	Volume distribution coefficient (D_v)
HNO_3	3	125	400	94.0	0.287	3.49
	4	75	325	100.0	0.537	1.86
	5	50	200	87.6	0.951	1.05
HCl	3	125	325	92.6	0.287	3.49
	4	125	325	100.0	0.287	3.49
	5	100	325	93.6	0.374	2.67
	6	75	200	80.0	0.537	1.86
H_2SO_4	1	75	250	97.9	0.537	1.86
	1.5	50	200	100.0	0.951	1.05
	2	50	125	87.4	0.951	1.05
NH_4Cl	3	125	450	100.0	0.287	3.49
	5	75	350	89.3	0.537	1.86
	6	50	350	84.5	0.951	1.05
NaCl	1–2	—	200	—	—	—
	4	75	375	69.6	0.537	1.86
	5	50	375	73.5	0.951	1.05
NH_4NO_3	4	100	350	93.1	0.374	2.67
CH_3COONH_4	2	100	225	100.0	0.374	2.67
	3	75	225	100.0	0.537	1.86
Malonic acid 5% (pH 6.0)		150	350	100.0	0.232	4.30

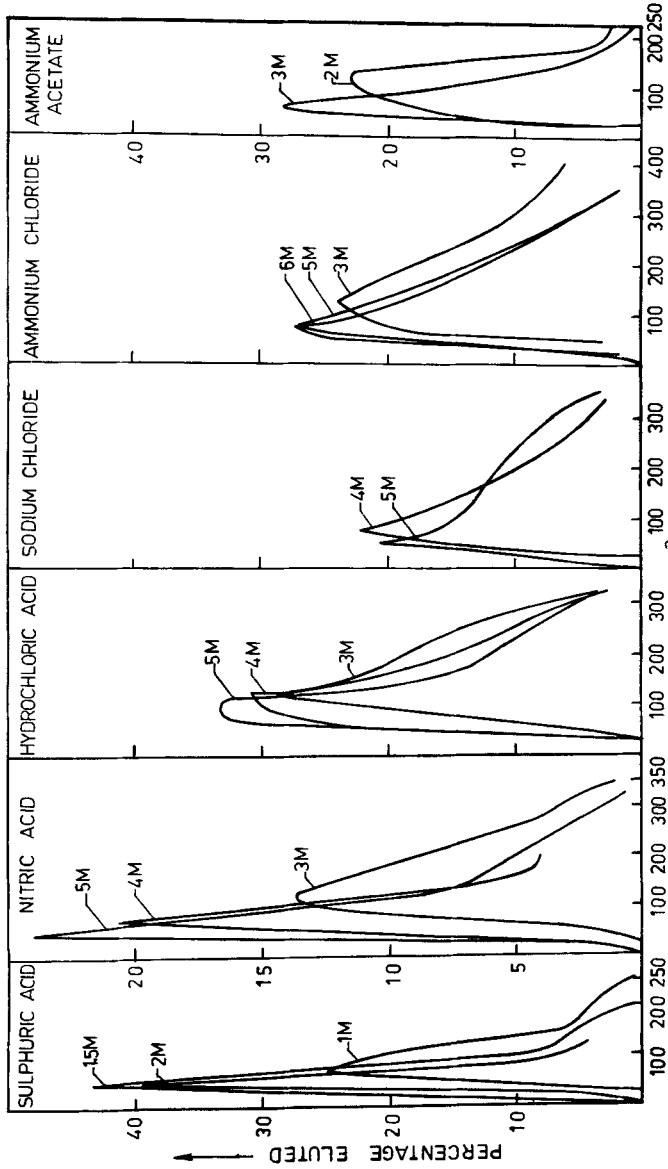

FIG. 1. Elution pattern of scandium(III) on Dowex 50W-X8 with various eluants.

Ion Exchange Separations

Separation from Alkali Metals

Since alkali metals were weakly bound to the resin in comparison with scandium, they were eluted first. Thus in binary mixtures of scandium with lithium, sodium, potassium, rubidium, or calcium, the alkali metals were first eluted with 200 ml of 0.5 *M* hydrochloric acid. Later scandium was eluted with 1.5 *M* sulfuric acid.

Separation from Alkaline Earth Elements

It was seen that 1 *M* nitric acid was the best eluant for alkaline earth metals, but not for scandium. Hence, in mixtures of beryllium, magnesium, calcium, strontium, or barium with scandium, these ions were eluted with 1 *M* nitric acid, followed by the elution of scandium with 1.5 *M* sulfuric acid.

Separation from Yttrium and Lanthanides

Scandium was weakly bound to the resin in comparison with yttrium and lanthanons. Therefore, in a mixture of scandium with yttrium, lanthanum, cerium(III), praseodymium, neodymium, samarium, gadolinium, or dysprosium, scandium was first eluted with 250 ml of 1 *M* sulfuric acid. Subsequently the lanthanons were eluted with 4 *M* hydrochloric acid.

Separation from Iron(III), Zinc, Cadmium, Mercury(II), Copper(II), Indium, and Aluminum

Hydrochloric acid (1 *M*) was a poor eluant for scandium but not for the above-mentioned ions. Hence 200 ml of 1 *M* hydrochloric acid was used for the elution of iron(III), zinc, cadium, mercury(II), copper(II), indium, and aluminum. Scandium was eluted afterwards with 1.5 *M* sulfuric acid.

Separation from Vanadium(IV), Uranium(VI), and Bismuth(III)

Hydrochloric acid (0.5 *M*) was an efficient eluant for vanadium(IV), uranium(VI), and bismuth(III) but not for scandium. Hence all these ions were eluted first with 0.5 *M* hydrochloric acid. Scandium was eluted later with 1.5 *M* sulfuric acid.

Separation from Cobalt(II), Nickel, Manganese(II), Lead, and Silver

Sodium chloride and nitrate, in low concentrations, were poor eluants for scandium but not for these elements. Therefore cobalt(II) and nickel were eluted with 1 *M* sodium chloride, manganese(II) and lead with 2 *M* sodium chloride, and silver with 0.5 *M* sodium nitrate, followed by the elution of scandium with 200 m*l* of 1.5 *M* sulfuric acid.

Separation of Scandium from Thorium by Gradient Elution

Thorium was taken up very strongly in comparison to scandium. Therefore scandium was first eluted with 350 ml of 4 *M* hydrochloric acid. Then thorium was eluted with 300 ml of 8 *M* hydrochloric acid.

Separation from Titanium(IV), Zirconium(IV), Hafnium(IV), and Antimony(III) by Selective Sorption

Citric acid formed negatively charged complexes with titanium(IV), zirconium(IV), hafnium(IV), and antimony(III) around pH 2.2. Scandium was incapable of forming a citrate complex at this pH. Therefore, on passing the mixture of scandium with these ions in 5% citric acid buffered at pH 2.2, cationic scandium was retained by the column while all other anionic complexes passed through unabsorbed. The column was washed with water, and scandium was eluted with 1.5 *M* sulfuric acid as usual.

Separation from Oxy-anion

The separation of scandium from molybdate, arsenite, dichromate, vanadate, tellurite, selenite, and gold was done by selective sorption. All the separations were carried out in the ratio of 1 : 10. The amount of scandium taken in all cases was 5.26 mg.

Separation of scandium from alkali, alkaline earths, lanthanides, iron(III), zinc, cadmium, mercury(II), copper(II), bismuth(III), indium, aluminum, vanadium(IV), uranium(VI), titanium(IV), zirconium(IV), hafnium(IV), and thorium is important because these metals are usually associated with it in several minerals. The overall operation takes about 4 hr. The reproducibility is $\pm 1.25\%$.

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