

# Separation of Ba(II) from Other Metal Ions on Dowex 50W-X8 Column in NH<sub>4</sub>F-HNO<sub>3</sub> Media

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Distribution coefficients ( $K$ ) determined by batch technique in fixed (0.80  $M$ ) NH<sub>4</sub>F-varying (1.0-4.0  $M$ ) HNO<sub>3</sub> media indicated the separation of Ba(II) from Mg(II), Ca(II), Sr(II), Hg(II), Bi(III), Cr(III), Mn(II), Co(II), Ni(II), Co(II), Zn(II) and Cd(II). Actual separation was obtained in about 7-8 cm column of Dowex 50W-X8 (H<sup>+</sup>-form; 100-200 mesh) in 0.80  $M$  NH<sub>4</sub>F-1.0  $M$  HNO<sub>3</sub> mixture. Distribution coefficients, elution curves and the results of various separations along with standard deviations are presented.

A systematic study to explore the analytical applications of NH<sub>4</sub>F-HNO<sub>3</sub> medium in the separation of metal ions by cation exchange Dowex 50W-X8 resin has been undertaken by the authors. The earlier studies, made at fixed (0.70  $M$ ) HNO<sub>3</sub>-varying (0.20-1.0  $M$ ) NH<sub>4</sub>F media produced some very good separations of Al(III), Fe(III) and Hg(II) from other cations<sup>1</sup>. The work presented in this paper is an extension of the work to fixed (0.80  $M$ ) NH<sub>4</sub>F and varying (1.0-4.0  $M$ ) HNO<sub>3</sub> concentrations and reports the separation of Ba(II) from other metal ions.

## Experimental

Dowex 50W-X8 of 100-200 mesh size in H<sup>+</sup>-form was used for both batch as well as columnar studies. Aqueous solutions (0.06-0.20  $M$ ) of Mg(II), Ca(II), Mn(II), Fe(III), Co(II), Zn(II), Cd(II), Sr(II), Ba(II), Hg(II) and Bi(III) were prepared from their reagent grade nitrate samples. The metal ion concentration was determined complexometrically<sup>2,3</sup>. 4.0  $M$  stock solution of NH<sub>4</sub>F and 8.0  $M$  stock solution of HNO<sub>3</sub> (B.D.H., AnalaR) were prepared as usual and suitably diluted to obtain the working solutions. 0.80  $M$  NH<sub>4</sub>F-1.0  $M$  HNO<sub>3</sub> was used as the wash solution and the elutant was either 0.80  $M$  NH<sub>4</sub>F-1.0  $M$  HNO<sub>3</sub> or 4.0  $M$  HCl.

### Procedure :

**Distribution studies :** Distribution coefficient ( $K$ ) was determined by batch technique. 1 g air dried Dowex 50W-X8 resin was added to the solution containing metal ion, HNO<sub>3</sub> of desired concentration (1.0-4.0  $M$ ) and NH<sub>4</sub>F (0.80  $M$ ). After shaking for 1 hr the solution was filtered and metal contents of the solution were determined. Distribution coefficients were determined by employing the following relationship :

$$K = \frac{\text{mg metal ion per g resin}}{\text{mg metal ion per ml solution}}$$

All experiments were performed in duplicate and the results are recorded in Table 1. The relative experimental error in the determination of  $K$  was less than 5%.

TABLE 1—DISTRIBUTION COEFFICIENTS OF METAL IONS ON DOWEX 50W-X8 IN FIXED (0.8  $M$ ) NH<sub>4</sub>F-VARYING (1.0-4.0  $M$ ) HNO<sub>3</sub>

| Metal ion | Distribution coefficients ( $K$ )         |     |     |     |
|-----------|---|-----|-----|-----|
|           | Concentration of HNO <sub>3</sub> ( $M$ ) |     |     |     |
|           | 1.0                                       | 2.0 | 3.0 | 4.0 |
| Mg(II)    | 11  | 8   | 6   | 1.3 |
| Ca(II)    | 8   | 3   | 0.5 | 0.0 |
| Mn(II)    | 5   | 4.5 | 4   | 2.5 |
| Fe(II)    | 0.0                                       | 0.0 | 0.0 | 0.0 |
| Co(II)    | 4   | 3   | 2   | 0.0 |
| Zn(II)    | 13  | 10  | 8   | 7   |
| Cd(II)    | 7   | 5   | 3   | 2   |
| Sr(II)    | 6   | 4   | 3.7 | 0.0 |
| Ba(II)    | 90  | 80  | 75  | 70  |
| Hg(II)    | 7   | 5   | 3   | 0.0 |
| Bi(III)   | 8   | 3   | 1   | 0.0 |

## Results

Table 1 shows that with increase in concentration of HNO<sub>3</sub> at fixed (0.80  $M$ ) NH<sub>4</sub>F, the  $K$  values, decrease in general. This decrease is nominal and not so pronounced. The metal ions La(III), Ce(III), In(III), Y(III), Pr(III) and Sc(III) were precipitated and therefore not included.

Since Ba(II) has the highest  $K$  values amongst the elements studied, it can be separated from the other metal ions. The following separation procedure was adopted :

**Preparation and pretreatment of resin column :** A slurry of fully water-swollen cation exchanger Dowex 50W-X8 (100-200 mesh, H<sup>+</sup>-form) was transferred to a graduated ion exchange column and the height of the resin bed was adjusted to about 7.5±0.5 cm. It was then washed with wash solution.

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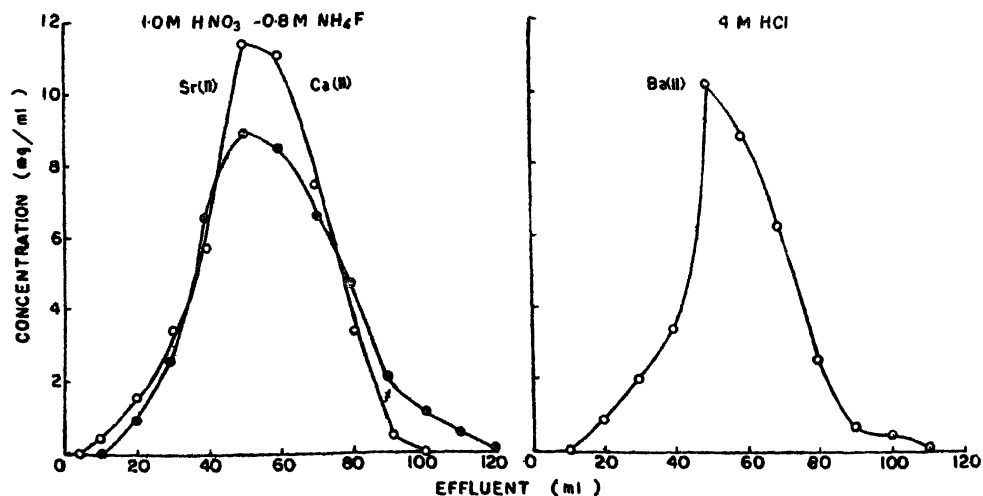


Fig. 1. Elution curves of Ca(II)/Sr(II) and Ba(II).

**Sorption :** The feed, 25 ml in 1.0 M HNO<sub>3</sub>-0.8 M NH<sub>4</sub>F was prepared in duplicate by mixing an aliquot of standard solutions of Ba(II) and one of the following metal ions: Mg(II), Ca(II), Sr(II), Fe(III), Mn(II), Co(II), Zn(II), Cd(II), Hg(II) or Bi(III). The feed was transferred to the column and passed through at a flow rate of 1.0 ± 0.3 ml/min.

**Elution :** The weakly sorbed metal ions Mg(II), Ca(II), Sr(II), Fe(III), Mn(II), Co(II), Zn(II), Cd(II), Hg(II) and Bi(III) accompanied the feed effluent and were quantitatively recovered with 1.0 M HNO<sub>3</sub>-0.80 M NH<sub>4</sub>F eluant. The strongly sorbed Ba(II) was eluted last with about 110 ml 4.0 M HCl. The results of these separations are presented in Table 2 along with standard deviations. The separation of Ca(II) and Sr(II) from Ba(II) are shown in Fig. 1. Ca(II) and Sr(II) were stripped off with about 120 and 100 ml 1.0 M HNO<sub>3</sub>-0.8 M NH<sub>4</sub>F mixture eluant, respectively and then Ba(II) was eluted with 110 ml 4.0 M HCl.

All the separations achieved were quantitative and the column kinetics was found to be favourable.

TABLE 2—RESULTS OF SEPARATION OF Ba(II) FROM OTHER METAL IONS ON DOWEX 50W-X8 IN 0.8 M NH<sub>4</sub>F-VARYING (1.0-4.0 M) HNO<sub>3</sub> MIXTURE

| Metal ion                     | Mean amount taken mg | No. of determinations | Mean amount found mg | Standard deviation mg |
|-------------------------------|----------------------|-----------------------|----------------------|-----------------------|
| <b>First component ion :</b>  |                      |                       |                      |                       |
| Mg(II)                        | 12.25                | 2                     | 12.35                | 0.18                  |
| Ca(II)                        | 17.00                | 2                     | 16.90                | 0.12                  |
| Sr(II)                        | 38.28                | 2                     | 38.40                | 0.24                  |
| Fe(III)                       | 25.00                | 2                     | 25.16                | 0.10                  |
| Co(II)                        | 21.75                | 2                     | 21.60                | 0.26                  |
| Ni(II)                        | 21.84                | 2                     | 21.90                | 0.20                  |
| <b>Second component ion :</b> |                      |                       |                      |                       |
| Ba(II)                        | 48.63                | 12                    | 48.60                | 0.30                  |

**References**

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