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Separation of Bismuth(lll) from Other Ions by Cation Exchange Chromatography

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NOTE

Separation of Bismuth(III) from Other Ions by Cation Exchange Chromatography

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Summary

The cation exchange behavior of bismuth was studied on Dowex 50W-X8 with hydrochloric, sulfuric, nitric, and perchloric acids, with ammonium chloride and sodium nitrate as the eluting agents. The efficiency of eluants was evolved in terms of elution constant and volume distribution coefficient. Bismuth was separated from copper, cadmium, tin, aluminum, nickel, manganese, vanadium, magnesium, mercury, iron, zinc, silver, cobalt, barium, and calcium by the gradient elution process. It was separated from lead, uranium, antimony, and strontium by selective elution with specific eluants. The oxyanions were separated from it by the selective sorption process. The method was found to be applicable to analysis of bismuth-based alloys.

Several papers have appeared on the ion exchange selectivity (*1*, *2*) scale of cations, including bismuth, based on equilibrium distribution coefficients from other ions by cation exchange chromatography.

Bismuth was separated (*3*) from copper and lead by selective elution with potassium iodide. It was also separated from zinc, cadmium, and lead (*4*) or polonium (*5*) or thorium (*6*) on cation exchange resin. However, systematic studies on its separation by selective elution techniques are totally lacking. This paper presents such elution and separation studies on Dowex 50W-X8 resin.

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EXPERIMENTAL

Apparatus and Reagents

The ion exchange column (1.4 × 20 cm) was similar to one described earlier (*7*). A Cambridge pH-indicator and an automatic fraction collector (Tower's type model) were used.

A stock solution of bismuth nitrate was prepared by dissolving 5 g of bismuth metal (Johnson Matthey and Co.) in 25 ml of concentrated nitric acid. The solution was then diluted to 1 liter with water. On standardizing volumetrically with EDTA, it was found (*8*) to contain 4.9 mg/ml of bismuth.

Dowex 50W-X8 was from Dow Chemical Co., Midland, Mich.

RESULTS AND DISCUSSION

An aliquot of bismuth nitrate solution containing 24.96 mg of bismuth was sorbed on the column. After washing the column with 15

TABLE 1

Elution Behavior of Bismuth (Bi = 24.96 mgs., wt. of (oven dried) resin = 17.33 gms)

Eluant	M	$\bar{V}$, peak elution volume (ml)	V_t, total volume (ml)	Bismuth recovered (%)	E, elution constant	D_v, volume distribution coefficient
HCl	0.5	50	200	100.8	0.95	0.010
	1	50	200	99.4	0.95	0.010
	2	20	200	99.7	13.98	0.072
	3	20	150	99.8	13.98	0.072
	4	20	50	100.0	13.98	0.072
HNO_3	1	—	200	0.7	—	—
	2	70	200	100.5	0.59	0.017
	3	60	200	99.9	0.73	0.014
	4	30	200	101.5	2.52	0.397
H_2SO_4	2	—	200	33.0	—	—
	4	60	200	99.6	0.73	0.014
	6	30	200	100.7	2.52	0.397
$HClO_4$	4	—	200	0.0	—	—
	6	—	200	2.1	—	—
NH_4Cl	1	—	200	67.7	—	—
	2	—	200	86.8	—	—
	4	20	50	101.0	13.98	0.072
CH_3COONH_4	4	—	200	22.3	—	—
$NaNO_3$	2	—	200	6.4	—	—
	4	—	200	37.8	—	—

ml of water, bismuth was eluted with various eluants (Table 1). The effluent lot was collected in 10 ml fractions, and it was determined volumetrically (*8*) from each fraction with EDTA.

Elution constants and volume distribution coefficients were calculated (*9*) from the peak elution volume ($\bar{V}$). From this information the eluants can be arranged in order of increasing efficiency: $CH_3COONH_4 > NaNO_3 > H_2SO_4 > HNO_3 > NH_4Cl > HCl$. Citric, tartaric, oxalic, and malonic acids were organic acids tried as eluants but were found to be inefficient at lower pH values and at 5% concentration. For routine work 4 *M* sulfuric acid was found to be most suitable because a small volume of eluant was needed for quantitative elution.

Ion Exchange Separation

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Certain ions, such as copper, cadmium, tin, aluminum, nickel, manganese(II), vanadium(V), magnesium, mercury, and iron(III), are weakly bound by the resin and hence can be removed by 1 *M* sulfuric acid (see Table 2), while bismuth, which is strongly bound, is removed from such mixtures with 4 *M* sulfuric acid. Similarly, the gradient elution process was used in the separation of zinc, silver, cobalt, barium, and calcium. These ions, which are weakly sorbed, are removed by 1 *M* nitric acid followed by elution of bismuth with 4 *M* nitric acid.

Some ions, such as zirconium(IV), thorium(IV), and titanium, are, however, strongly sorbed by the resin and are desorbable only by higher concentrations of mineral acids. Therefore, in such mixtures bismuth was first eluted with 1 *M* hydrochloric acid, followed by elution of the aforementioned ions with 8 *M* hydrochloric acid. Thus, by a process of selective elution, it is possible to accomplish clean-cut separations of bismuth either by its removal before or after the other ion in the cation exchange column.

It was further noted that certain eluants are specific for selected metals, e.g., 1 *M* ammonium acetate can elute lead quantitatively, 1 *M* perchloric acid elutes uranium(VI), and 5% tartaric acid (pH $\sim$ 1.0) elutes antimony and strontium completely, but are poor eluants for bismuth. Therefore, in mixtures the former ions can be eluted with specific eluants followed by elution of bismuth with 4 *M* sulfuric acid.

The process of selective sorption was also adopted for the separation of oxyanions such as chromate, tellurite, selenite, arsenite, molybdate, vanadate, and tungstate from bismuth. After rinsing the column with water, bismuth was eluted with 4 *M* sulfuric acid. In all separations

TABLE 2

Ion Exchange Separation of Bismuth (Bi = 4.99 mg)

Foreign ion	Amount added (mg)	Bismuth Found (mg)	Bismuth Recovered (%)
Hg(II)	25.7	5.28	105.2
Cu(II)	24.8	4.89	99.8
Cd	24.7	4.99	100.0
Sn(II)	24.9	5.18	104.0
Fe(III)	24.1	4.69	98.6
Al	24.6	4.99	100.0
V(V)	25.3	5.11	102.2
Mn(II)	25.6	5.27	105.0
Ni	25.3	4.78	99.3
Mg	24.9	4.89	99.8
Ag	25.0	4.99	100.0
Zn	25.1	4.78	99.3
Co(II)	24.5	4.88	99.8
Ca	25.5	4.68	98.5
Ba	25.2	5.07	100.4
Pb(II)	25.3	4.99	100.0
U(VI)	24.6	4.89	99.8
Sb(III)	24.4	5.18	104.0
Sr	24.3	4.78	99.3
Zr(IV)	25.6	4.88	99.8
Th(IV)	25.2	4.68	98.5
Ti(IV)	25.2	4.78	99.3

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200 ml of eluant at a flow rate of 2 ml/min was used for the quantitative separation of these ions.

Application to Bismuth-Based Alloys

A known weight (~0.5 g) of an alloy was dissolved in 10 ml of concentrated nitric acid. The mixture was diluted with water and filtered to remove insoluble tin as metastanic acid (weighed as oxide). The filtrate was made up to 100 ml. A 10-ml aliquot was taken and sorbed on the column. Cadmium was first eluted with 200 ml of 4 *M* perchloric acid (*10*), lead was then eluted with 200 ml of 1 *M* ammonium acetate (*11*), and finally bismuth was desorbed with 4 *M* sulfuric acid and analyzed as described earlier. The results from duplicate analysis showed 49.8 and 49.7% bismuth.

The separation of bismuth from lead, silver, copper, and titanium is important because they are associated with it in several minerals. So also is its separation from cadmium, tin, aluminum, and lead, because they are found together in low melting alloys. The over-all operation takes about 2½ hr. The results are reproducible to ±1.8%.

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