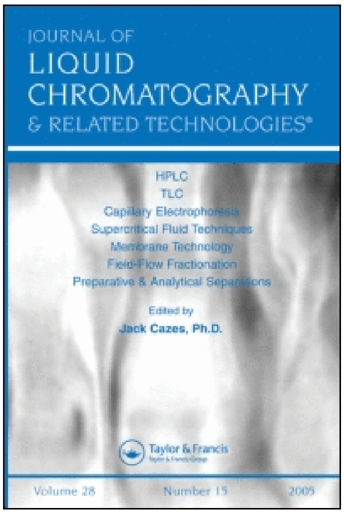

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Extraction Chromatograph Ic Separation of Tellurium(Iv) from Selenium and Associated Elements with Tri-Butyl-Phosphate

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EXTRACTION CHROMATOGRAPHIC SEPARATION OF TELLURIUM(IV) FROM SELENIUM AND ASSOCIATED ELEMENTS WITH TRI-BUTYL-PHOSPHATE

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ABSTRACT

A novel methods presented for the extraction chromatographic separation of tellurium(IV) from selenium(IV) and associated elements. Tellurium(IV) was quantitatively extracted from 2.5 M hydrochloric acid on silica gel coated tributylphosphate. It was stripped from the organic phase with 0.1 M hydrochloric acid and determined spectrophotometrically after reduction with stanous chloride at 400 nm. It was possible to separate tellurium(IV) from as many as forty elements from binary mixture. It was separated from multicomponent mixtures containing as many as five elements by exploiting the difference in acidity at which various metals are solvated and hence extracted. The method was extended for the analysis of tellurium from pyrite and acid sludge.

INTRODUCTION

Although some methods for the solvent extraction separation of tellurium from selenium are known, reverse phase extraction chromatographic methods in comparison

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offers better separation with judicious choice of stationary and mobile phase.

Selenium(IV) was separated from tellurium by reverse phase paper chromatography with tributyl phosphate as stationary phase with 2-10 M of hydrochloric acid as the mobile phase(1). In the paper chromatographic separation from bromide media(2), the R_f values decreased with increase in bromide concentration. Tellurium was separated from antimony on polychloroprifluroethylene (PTFCE) column coated with bis (2-diethylhexyl) phosphoric acid. However, such separation was possible provided if the temperature was controlled during elution(3). The separation of antimony, tellurium and tin on silica coated with tributylphosphate was not unsuccessful(4). The systematic investigations on the extraction chromatographic separation of tellurium(IV) is lacking. This paper describes such studies, with tributylphosphate as an extractant. The proposed method provides not only the separation of tellurium(IV) from selenium(IV) but also from the other elements such as copper(II), gold, thallium(III), the method is extended for the analysis of tellurium from minerals and sludge.

EXPERIMENTAL

Apparatus and reagents

The apparatus and chromatographic column was similar to the one described earlier(5).

The stock solution of tellurium(IV) was prepared by dissolving 0.687 gm. of sodiumtelluride (BDH Anala R) in 10 ml of concentrated hydrochloric acid. The solution was made upto 100 ml. The solution was standardise complexometrically with potassium dichromate(6). It contained 4.5 mg/ml of tellurium(IV). The diluted solution containing 50 µg/ml of tellurium was prepared by appropriate dilution.

The silica gel (100-200) was hydrophobized as per procedure described earlier(7). The gel was coated with tributylphosphate as usual. The chromatographic column of the dimension of (0.8 x 20 cms.) with the bed height of the column of 6 cms was than used.

General procedure

An aliquot of solution containing 50 µg of tellurium(IV) was made upto 2.5 to 6 M in hydrochloric acid in total volume of 4 ml. The solution was then passed through the column at the flow rate of 1 ml/min. Tellurium(IV) which was extracted by tributylphosphate was stripped with various mineral acids and salts as the eluants. Twenty fractions each of two ml were collected. They were analysed for tellurium spectrophotometrically at 400 nm by reduction with stanous chloride(8).

RESULTS AND DISCUSSION

The optimum condition for quantitative extraction of tellurium(IV) was ascertained by extracting it from varing concentration of hydrochloric acid (1 - 8 M). No extraction was observed from 0.1 - 1.5 M hydrochloric acid. The extraction started at 2 M hydrochloric acid and it was quantitative from 2.5 - 8 M hydrochloric acid.

The systematic studies on the stripping of tellurium after extraction were carried out with various eluants, like 0.1 - 1.5 M hydrochloric, 0.1 - 4 M nitric, 0.1 - 4 M sulphuric acid and water. Tellurium was quantitatively stripped with all the mineral acids and also with 0.5 - 2 M sodium acetate, 0.1 - 0.5 M sodium citrate, 0.5 - 4 M ammonium thiocynate, 1 - 4 M lithium chloride. Although nitric and sulphuric acid were not prefered on account of the problems in spectrophotometric determination of tellurium. 0.1 M Hydrochloric acid was prefered as the stripping agent(Table-1).

Separation from binary mixture

When tellurium(IV) was extracted at 2.5 M hydrochloric acid foreign ions like transition and main group elements were not extracted at this acidity and they passed through the column. The extracted tellurium(IV) was stripped with 0.1 M hydrochloric acid. It was thus possible to separate tellurium(IV) from commonly associated elements such as alkali alkaline earths, manganese, copper lead, solenium(IV) antimony(III) etc. in a binary mixture in the ratio of 1:50 (Table 2).

Iron(III), gold, chromium(VI), thallium(III) were coextracted with tellurium. Since tellurium(IV) was first stripped with 1.5 M hydrochloric acid followed by stripping of iron(III), chromium(VI) and gallium with 0.1 M hydrochloric acid, thallium with 0.5 in sodium acetate, gold with 0.5 M hydrochloric acid containing 70% acetone.

Separation of tellurium from multicomponent mixtures

It was interesting to note that various metals were extracted at various acidities(9). Thus it was possible to separate tellurium(IV) from various elements by the use of selective extraction and selective stripping of the extracted species on the column.

The mixture of lead/tin(II), antimony(III) and tellurium(IV) was passed on the column from 6 M hydrochloric acid. When lead and tin(II) were not extracted and therefore they passed through the column. The extracted antimony and tellurium(IV) which were stripped with 2.5 M hydrochloric acid and 0.1 M hydrochloric acid respectively.

The mixture containing selenium(IV), tellurium(IV) and mercury was passed through the column with 2.5 M hydrochloric acid, when selenium(IV) was not extracted and it passed through. The extracted tellurium(IV) was stripped with 0.5 M hydrochloric acid and finally mercury with 0.1 M sodium citrate buffered at pH 5(10).

The mixture of zirconium, uranium and tellurium(IV) was passed through the column from 4 M hydrochloric acid when zirconium was not extracted. The extracted uranium

TABLE-1

Studies on stripping of Tellurium(IV)

Eluant	Molarity (M)	V_{max} (ml)	V_t (ml)	% recovery
HCl	0.1-0.5	4	6	100
	1.0	4	8	99.9
	1.5	6	12	99.8
HNO_3	0.1-1.0	4	6	100
	2.0-4.0	4	10	99.8
H_2SO_4	0.1-4.0	4	6	100.0
H_2O	--	4	6	99.8
CH_3COONa	0.5-2.0	4	6	99.5
Sod.citrate	0.1	6	10	99.5
	0.5	4	6	99.5
NH_4SCN	0.5-4	4	10	99.5
HCl	1 - 4	4	10	100.0

was stripped with 2.5 M hydrochloric acid and finally tellurium with 0.1 M hydrochloric acid.

The separation of aluminium, germanium tellurium(IV) and gallium was accomplished by passing the mixture from 6 M hydrochloric acid when aluminum was not extracted. Then germanium was stripped with 2.5 M hydrochloric acid, tellurium with 1.5 M hydrochloric acid and gallium with 0.1 M hydrochloric acid.

TABLE-2

Separation of Tellurium(IV) from binary mixture
Te(V) - 50 μg

Metal ion	Taken mg	Te found μg	% recovery
Li	2.50	49.3	98.7
Na	2.50	50.0	100.0
K	2.50	49.6	99.3
Rb	2.50	50.0	100.0
Cs	2.50	49.3	98.7
Be	4.24	50.0	100.0
Mg	2.50	50.0	100.0
Ca	2.50	50.5	101.0
Sr	2.50	49.6	99.3
Ba	2.50	50.0	100.0
Sc	2.00	50.0	100.0
y	2.50	49.6	99.3
Ti	4.68	49.3	98.7
Zr	2.50	50.0	100.0
Ce(III)	2.50	49.6	99.3
V(V)	2.00	49.3	98.7
Mn(II)	2.50	51.0	102.0
CO(II)	4.36	49.0	98.0
Ni	2.50	49.6	99.3
Cu(II)	2.54	49.3	95.7
Ge	2.00	50.0	100.0
Se(IV)	2.50	49.6	99.3
Al	2.50	50.0	100.0
Pb II	3.11	50.0	100.0
Sb(III)	2.50	49.6	99.3
Bi	2.00	49.6	99.3
As(II)	2.00	50.0	100.0
Fe(III)	3.41	49.6	99.3
Cr(VI)	2.00	49.0	98.0
T1(III)	1.00	49.6	99.3
Au	1.00	50.0	100.0
Ga	1.1	49.6	99.6

Selenium(IV), tellurium(IV), gallium, chromium(VI) and thallium(III) were separated by extracting the mixture from 2.5 M hydrochloric acid when selenium(IV) was not extracted. The extracted tellurium(IV) was stripped with 1.5 M hydrochloric, gallium with 0.5 M hydrochloric, chromium(VI) with 0.02 M hydrochloric acid and finally thallium(III) with 0.5 M sodium acetate.

The separation of copper(II), vanadium(V), tellurium(IV), iron(III), chromium(VI), was effected by passing mixture from 6 M hydrochloric acid when copper was not extracted. The extracted vanadium(V) was stripped with 2.5 M hydrochloric acid, tellurium with 1.5 M, iron(III) with 0.5 M hydrochloric acid and finally chromium(VI) with water.

The separation of lead, antimony(III), tellurium(IV), iron(III) and gold was carried out by extracting these elements with 6 M hydrochloric acid, when lead was not extracted, extracted antimony(III) was stripped with 2.5 M hydrochloric acid, tellurium(IV) with 1.5 M hydrochloric acid iron with 0.5 M hydrochloric acid and finally gold with 0.5 M hydrochloric acid containing 70% acetone.

Finally the separation of bismuth, germanium, tellurium(IV), gallium and thallium was carried out by extracting mixture from 6 M hydrochloric acid when bismuth was not extracted. While extracted germanium was stripped with 2.5 M hydrochloric acid, tellurium with 1.5 M hydrochloric gallium with 0.1 M hydrochloric acid, and thallium with 0.5 M sodium acetate (Table 3).

Through out the above separations flow rate was maintained at 1 ml/min. All the elements after the separation were determined spectrophotometrically with appropriate chromogenic ligands(11).

Separation of selenium(IV) from tellurium(IV)

The most interesting aspect of the proposed method was the separation of selenium and tellurium. When the mixture of selenium(IV) and tellurium(IV) were extracted from 2.5 M hydrochloric acid, selenium was not extracted in the column and passed through while extracted tellurium(IV) was stripped with 0.1 M hydrochloric acid. Thus it was possible to separate tellurium(IV) from selenium(IV) in any ratios from 1:12 to 10:1 of tellurium:selenium.

The method was extended for the analysis of tellurium(IV) from pyrite ore and sludge.

Separation of pyrite ore and acid sludge

The sample of 0.5 gm was brought into the solution as usual(12). An aliquot of 10 ml contained copper, iron, lead, tellurium was passed on the column from 3 M hydrochloric acid when lead and copper were not extracted and they passed through. The extracted tellurium(IV) was stripped with 1.5 M hydrochloric acid finally iron(III) with 0.1 M hydrochloric acid. The amount of tellurium

TABLE-3

Separation of Tellurium(IV) from Multicomponent mixtures

Element	Taken µg	Found µg	% recovery	Eluant	Volume of Eluant
1. Pb/Sn(II)	250	249	99.6	6 MHCl	15
Sb	100	99.5	99.5	2.5 MHCl	15
Te(IV)	50	49.6	99.2	0.1 M HCl	10
2. Se(IV)	68	67.5	99.2	2.5 M HCl	15
Te(IV)	50	49.3	98.7	0.5 M HCl	10
Hg(II)	100	98.5	98.5	0.1 M CH_3COONa PH5	25
3. Zr	50	48.2	96.4	4.0 M HCl	15
U	50	50.0	100.0	2.5 M HCl	15
Te	50	50.0	100.0	0.1 M HCl	6
4. Al	50	49.0	98.0	6.0 M HCl	15
Ge	50	50.0	100.0	2.5 M HCl	15
Te	50	49.6	99.3	1.5 M HCl	10
Ga	50	50.0	100.0	0.1 M HCl	12
5. Se	50	50.0	100.0	2.5 M HCl	15
Te	50	49.6	99.3	1.5 M HCl	10
Ga	50	50.0	100.0	0.5 M HCl	10
Cr	25	24.5	98.0	0.02 M	10
Tl	50	49.0	98.0	0.5 M Sod. acetate	10
6. Cu(I)	100	100	100	6.0 M HCl	15
V(V)	50	49.5	99.0	2.5 M HCl	12
Te(IV)	50	49.6	99.3	1.5 M HCl	10
Fe	50	50	100	0.5 M HCl	10
Cr (VI)	25	25	100	Water	10

Contd..

TABLE-3 (Contd..)

Element	Token µg	Found µg	% recovery	Eluant	Volume of Eluant
7. Pb	50	49.5	99.0	6.0 M HCl	15
Sb(III)	100	98.7	98.7	2.5 M HCl	15
Te(II)	50	49.6	99.3	1.5 M HCl	10
Fe	50	50.5	101.0	0.1 M HCl	10
Au	40	40.0	100.0	0.5 M HCl + 70% acetone	20
8. Bi	50	50	100	6 M HCl	15
Ge	50	49.5	99.0	2.5 M HCl	15
Te(IV)	50	49.3	98.7	1.5 M HCl	10
Ga	50	50.5	101.5	0.1 M HCl	10
Tl (III)	50	49.5	99.0	0.5 M CH_3COO Na	15

was determined spectrophotometrically. It was 48.5 µg against a standard value of 50 µg in given sample.

The method was extended for analysis of tellurium(IV) from acid sludge. The sludge was brought into the solution as per usual procedure described(13). An aliquot of (5 ml) was passed through the column when selenium(IV) and arsanic were not extracted and passed through the column, but tellurium and iron were extracted. Then tellurium was stripped with 1.5 M hydrochloric acid while iron(III) was back washed with 0.1 M hydrochloric acid and determined as usual. The amount of tellurium found was 37 µg against a standard value of 37.5 µg.

The separation of tellurium from lead antimony, bismuth, selenium was important as they were associated with it in minerals. A new method for separation of tellurium(IV) and selenium(IV) from the ratio of 1:10 to 10:1 is available. The total time required for separation and determination is 2 hrs. The results are reproducible. The method is simple, rapid and selective.

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