Use of Cadmium Acetate in Quantitative Separation of Anions

Separation of Arsenate, Selenate, Tellurate, Selenite or Tellurite from: (a) Iodide and (b) Per-Rhenate

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Summary

It has been shown that cadmium acetate can be used for the gravimetric separation of (a) iodide and (b) per-rhenate from arsenate, tellurate, selenate, tellurite or selenite.

In the systematic scheme for the detection of anions, TAIMNI and LAL[1]) recommended the use of zinc, cadmium, lead, calcium and barium ions in slightly alkaline medium. The chief advantage of this new scheme, was the removal of difficulties associated with the simultaneous presence of certain anions resulting in: (i) Complex formation, as in the case of iodide and tellurite or (ii) mutual oxidation and reduction, as in the estimation of iodide in presence of arsenate.

It was considered worthwhile studying this problem from the quantitative point of view and find out whether it was possible to separate and estimate certain anions in such diffucult combinations. The oxidation-reduction and complex formation take place more easily in acid media, so recourse was taken to slightly alkaline medium. A careful study of the reaction of anions with some basic ions revealed that cadmium ions may be of great help, atleast in the separation of some of these anions. The quantitative separations which were successfully attempted using cadmium acetate, are:

(A) Estimation of arsenate, tellurate, selenate, tellurite or selenite in the presence of iodide and

(B) Estimation of arsenate, tellurate, selenate, tellurite or selenite in the presence of per-rhenate.

Though the separations mentioned above are carried out in neutral or slightly alkaline solutions and are therefore of limited utility, still on account of the difficulties referred to above, they can be used with advantage in separation of certain anions under particular circumstances.

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1) I. K. TAIMNI and M. LAL, Anal. Chim. Acta **17**, 367—372 (1957).

Experimental

The procedure which was followed in these separations, briefly, is as follows:

Measured volumes of the solutions containing the two anions to be separated (in the presence of slight excess, 0.5 g, of sodium carbonate) were diluted to about 50 ml and treated with 1 M solution of cadmium acetate, until the precipitation was complete. The presence of sodium carbonate is essential, in all such separations, in order to check the interaction of anions in neutral or acidic medium and also to give a coarse precipitate of cadmium carbonate which facilitates the filtration of the precipitates obtained with cadmium ions. The mixture was then heated to boiling, digested over a hot plate for about 10—15 minutes and allowed to cool to room temperature. The precipitate of the cadmium salt of one of the anions, along with that of cadmium carbonate, was filtered through a gravimetric filter paper and washed thoroughly with water. The filtrate was reserved for the estimation of the second anion, while the precipitate on the filter paper, was dissolved in 3 N hydrochloric acid, the solution concentrated to about 50 ml, made alkaline with caustic soda solution and treated with excess of 2 N sodium sulphide reagent followed by excess of hydrochloric acid. The precipitate of cadmium sulphide was completely dissolved and the thio salt in the solution was decomposed to give a precipitate of the corresponding sulphide, which was filtered off through the sintered glass crucible and weighed after suitable treatment.

Section (A)

Separation of arsenate, tellurate, selenate, tellurite or selenite from iodide

Measured volumes of standard solutions of sodium arsenate, selenous acid, potassium tellurite, potassium selenate or sodium tellurate were made alkaline with sodium carbonate mixed with a standard solution of potassium iodide. All these separations were attempted in a manner described above. The filtrate reserved for the estimation of iodide was concentrated to about 100 ml, allowed to cool to room temperature, acidified with dilute acetic acid and the estimation of the iodide was completed either volumetrically[2]) using eosin as adsorption indicator or gravimetrically[3]) as silver iodide.

31—43 mg of arsenic, 47—59 mg of selenium(IV), 42—55 mg of tellurium(IV), 26—34 mg of selenium(VI) or 47—70 mg of tellurium(VI) were separated from 61—76 mg of iodine.

The metals were recovered with an accuracy of:

As	−0.18 to +0.30%	I	−0.20 to +0.26%
Te(IV)	−0.40 to +0.20%	I	−0.10 to +0.30%
Te(VI)	−0.16 to +0.02%	I	−0.06 to +0.20%
Se(IV)	−0.30 to +0.15%	I	−0.40 to +0.20%
Se(VI)	−0.40 to +0.20%	I	−0.30 to +0.25%

Section (B)

Separation of arsenate, tellurate, selenate, tellurite or selenite from per-rhenate

For these separations measured volumes of standard solutions of sodium arsenate, sodium tellurate, potassium selenate, potassium tellurite or selenious acid were mixed

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[2]) F. P. Treadwell and W. T. Hall, Analytical Chemistry, Vol. II, p. 651; John Wiley and Sins, New York 1942.

[3]) F. P. Treadwell and W. T. Hall, Analytical Chemistry, Vol. II, p. 297; John Wiley and Sons, New York 1942.

with a measured volume of potassium per-rhenate solution, made slightly alkaline with sodium carbonate and treated as above, except that rhenium was estimated in the filtrate as rhenium hepta-sulphide[4]) using 2 N sodium sulphide reagent.

31—43 mg of arsenic, 47—59 mg of selenium(IV), 42—55 mg of tellurium(IV), 26—34 mg of selenium(VI) or 47—70 mg of tellurium(VI) were separated from 33—35 mg of rhenium. The metals were recovered with an accuracy of:

As	−0.30 to +0.30%	Re	0.0 to +1.7%
Te(IV)	−0.17 to +0.67%	Re	0.0 to +0.7%
Te(VI)	−0.25 to +0.25%	Re	0.0 to +1.7%
Se(IV)	−0.33 to +0.82%	Re	0.0 to +1.4%
Se(VI)	−0.38 to +0.68%	Re	0.0 to +1.5%.

Note: The estimation of arsenate, tellurite and selenite was done by the sodium sulphide method[5]) and those of tellurate and selenate by the sodium sulphide method[6]).

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[4]) I. K. Taimni and G. B. S. Salaria, Anal. Chim. Acta **12**, 519—525 (1955).

[5]) I. K. Taimni and R. P. Agarwal, Anal. Chim. Acta **9**, 121—128. (1953).

[6]) G. B. S. Salaria, Anal. Chim. Acta **15**, 514—517 (1956).