Umrühren 5 h bei 5°C im Kühlschrank stehen. Danach filtriert man den Niederschlag durch einen Glasfildertiegel (G 4), wäscht 3–4 mal mit 2–3 ml Methanol durch Dekantation und danach mit 25 ml Methanol im Tiegel. Den ausgewaschenen Niederschlag löst man in Salpetersäure (1:1) und verwendet bei einem Gehalt von Caesium oder Rubidium < 50 mg die gesamte Probelösung zur Titration, sonst nur einen aliquoten Teil. Man fügt tropfenweise Ammoniak zu (so, daß der sich bildende Niederschlag wieder gelöst wird) und füllt mit Wasser auf ein Gesamtvolumen von etwa 50 ml auf. Nach Zugabe von Murexid als Indicator titriert man mit 0,01 M ÄDTA-Lösung bis zum Farbumschlag von gelb nach violett.

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Mgr. Inz. K. Bogusławska
ul. Lniana Nr. 11 m. 37
Łódź, Polen

Gravimetric Determination of Pyrophosphate Using Cis-dichlorobis(ethylenediamine)-cobalt(III) Chloride

Gravimetrische Bestimmung von Pyrophosphat mit Cisdichlor-bis(äthylendiamin)-kobalt(III)-chlorid

C. L. SHARMA

Department of Chemistry, University of Roorkee, India

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Pyrophosphate can be quantitatively precipitated by the addition of cis-dichloro-bis(ethylenediamine)-cobalt(III) chloride [1] in the pH range 10 to 12, adjusted with sodium hydroxide. A golden-yellow precipitate of the empirical formula $\text{Na}[\text{Co}(\text{C}_2\text{H}_8\text{N}_2)_2\text{P}_2\text{O}_7]$ was formed and was dried from 110 to 120°C. A 20% excess of the reagent solution was required for complete precipitation. The precipitate was collected in a No. 4 porosity sintered glass crucible, washed with distilled water until free from chloride. The best results were obtained when the solutions contained 20 to 80 mg of pyrophosphate.

Solutions containing a known quantity of pyrophosphate and varying amounts of diverse ions were prepared and the pyrophosphate was determined. Na^+ , K^+ , Rb^+ , Tl^+ , Zn^{2+} , Pd^{2+} , UO_2^{2+} , Hg^{2+} , La^{3+} , Au^{3+} , Al^{3+} , Ga^{3+} , In^{3+} , Ir^{4+} , Zr^{4+} and Ti^{4+} , CH_3COO^- , NO_2^- , NO_3^- , Cl^- , Br^- , I^- , SCN^- , ClO_4^- , S^{2-} , SO_3^{2-} , $\text{S}_2\text{O}_3^{2-}$, CO_3^{2-} , SiO_2^{2-} , $\text{C}_2\text{O}_4^{2-}$, $\text{S}_2\text{O}_5^{2-}$, SO_4^{2-} , PO_4^{3-} , tartrate, citrate, salicylate, tri and

tetrameta phosphate, triphosphate and EDTA did not interfere in the determination even when present in 10-fold excess.

Interferences caused by the following ions can be eliminated by addition of the reagents given in parenthesis. These reagents keep them in solution. Ba^{2+} , Sr^{2+} , Ca^{2+} , Be^{2+} , Mn^{2+} (EDTA); Ni^{2+} , Sn^{2+} , Sb^{3+} (tartrate); Fe^{2+} , Fe^{3+} , Co^{2+} (citrate); Pb^{2+} , Cd^{2+} (thiosulphate); Cr^{3+} (ascorbic acid).

Ag^+ , Cu^{2+} , VO_2^{2+} , Mg^{2+} , As^{3+} , Bi^{3+} , Ce^{4+} , Th^{4+} were found to interfere. These metals must therefore be removed before the estimation.

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Dr. C. L. Sharma

Department of Chemistry, University of Roorkee
Roorkee, U.P., India

Column-Chromatographic Separation of Metal Ions as Xanthates

Säulen-chromatographische Trennung von Metallionen als Xanthate

A. L. J. RAO and SURINDER SINGH

Department of Chemistry, Punjabi University, Patiala, India

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Some of the common ion xanthates are soluble in ethyl acetate or pyridine/ethyl acetate and are extracted into the organic phase. Preliminary experiments revealed differential adsorption and desorption of these metal xanthates when added to an alumina column making possible the separation of certain groups of elements.

Reagents. Potassium ethyl xanthate was prepared from double-distilled ethanol and CS_2 . All reagents used were of analytical grade. The columns were set with ethyl acetate or pyridine/ethyl acetate. Demineralised water was used throughout the work.

General Technique for Extraction. To an aliquot of metal salt solution (0.05 M), add 1 ml of 4% potassium ethyl xanthate solution, 1 ml of 5 M sodium formate and the remaining of demineralised water to make the volume to 5 ml, extract in 2 batch-extractions with 5 ml of ethyl acetate or ethyl acetate/pyridine (4:1) and make the volume up to 10 ml.

General Technique for the Separation. An aliquot containing 0.1–0.2 mg of metals as xanthates in 1–2 ml of the solvent is added to an alumina column. The adsorbed material is then irrigated to resolve the components into well discern-