Kurze Mitteilungen

Complexometric Determination of Barium with Copper(II)-EDTA-PAR as Indicator

Komplexometrische Bestimmung von Barium mit Kupfer(II)-ÄDTA-PAR als Indicator

Best. von Barium; Volumetrie; Cu(II)-ÄDTA-PAR als Indicator, komplexometrisch

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Received August 29, 1972; revised December 4, 1972

A sensitive method for detecting the end-point of the direct EDTA titration of Ba has been developed using a mixed Cu(II)-EDTA/PAR indicator. The exchange equilibrium between Cu(II)-EDTA and Ba ions was utilized to employ the well detectable end-point of Cu(II) with PAR. The method was applied to the analysis of barite concentrate and to the determination of sulphate. 1—100 mg of Ba were determined with errors of about $\pm$ 0.5—3%. Errors in the sulphate determination were about 0.1—0.5%.

Procedures

1. Ba Titration. To 100 ml of the sample solution containing 1—100 mg of Ba add 10 ml of 6.5 N ammonia, 5—6 drops of 0.01 M solution of Cu(II)-disodium complex of EDTA and 3—5 drops of 0.05% aqueous PAR [4-(2-pyridylazo-resorcinol)] solution. Titrate with 0.01—0.05 M EDTA solution until the red-violet colour fades and continue carefully to a distinct change to pure yellow with no dark shade.

2. Barite Analysis. Fuse 0.3 g of the sample in a platinum crucible with 8 time the weight of Na_2CO_3 and K_2CO_3, treat the melt as usual, collect the slight hydrochloric acid solution in a 250 ml volumetric flask and proceed as above, only add 3 ml of aqueous triethanolamine solution (1:2) before the ammonia as masking agent for Fe and Al.

A turbidity of the solution can be prevented by further addition of tartaric acid, yet results are about 2% higher in this case due to the Ca present as Ba is usually accompanied by Ca and the similarity in their analytical properties is most troublesome in their quantitative determination. A correction for Ca will be dealt with later.

3. Sulphate Determination. Precipitate the slightly acid solution containing 15—30 mg of sulphate with an exactly measured excess of $BaCl_2$ solution, allow to digest and filter. Aliquots of the filtrate are treated as under 1.

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Gravimetric Determination of Thorium with 1-Naphthyl Acetic Acid*

Gravimetrische Bestimmung von Thorium mit 1-Naphthylessigsäure

Best. von Thorium; Gravimetrie; 1-Naphthylessigsäure als Reagens

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Received September 5, 1972; revised December 4, 1972

In an earlier communication [1] the gravimetric determination of thorium with m-phenylenedioxydiacetic acid has been described. We now found that the use of 1-naphthyl acetic acid gives better results and even 1.3 mg of Th can be conveniently determined. 0.01—0.06 g of Th have been determined with errors of about 0.0001 to 0.002 g. For the separation from UO_2^{2+}, Al^{3+} and trivalent cerite earths double precipitation is required. For the separation from Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, Sn^{2+}, Pb^{2+} and Mn^{2+} single precipitation is sufficient. Interference by Ti^{4+} and Fe^{3+} is prevented by masking with chromotropic acid and reducing with hydroxylamine, respectively. Be^{2+} interferes.

Procedure. Add to the boiling sample solution a requisite amount of hot 2% solution of the monopotassium salt of the reagent, stir and adjust the pH to 4.65. Keep the solution warm for some time, filter, wash with 1% reagent solution, dry and ignite to ThO_2.

For the analyses of monazite sand the acid extract was precipitated according to [2], but the extract was made neutral to congo red for this reagent.

The composition of the compound precipitated was found to be $(C_{12}H_9O_3)_2Th(OH)_2$.

References

1. Pande, C. S., Srivastava, T. S.: Z. Anal. Chem. **167**, 332 (1959).
2. Rao, S. V. Raghava, Rao, B. R. Lakshmana: I. Indian Chem. Soc. **27**, 458 (1950).

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* Analytical Aspects of Some Organic Acids. Part IX.