Kurze Mitteilungen

Gravimetric Estimation of Silver as Silver Tetraisoithiocyanatodanilinechromate(III)

Gravimetrische Silberbestimmung als Silbertetraisoithiocyanato-danilinchromat(III)

Best. von Silber; Gravimetrie; als Silbertetraisoithiocyanato-danilinchromat(III)

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A systematic investigation indicated that anilinium tetraisoithiocyanatodanilinechromate(III) can be used as a selective reagent for the gravimetric estimation of silver. The reagent gives a red precipitate with silver nitrate solution, insoluble in common organic solvents and easy to wash. The exceedingly high molecular weight of the precipitated compound rendered it possible to estimate silver in minute quantities. The ratio of silver and the precipitated compound is 1:5.35. Repeated attempts were made to confirm the results of this new technique through a number of experiments involving determination of silver in various silver solutions of varying strengths.

Experimental

Reagent Solution. (Aniline)H[Cr(SCN)₄(aniline)₂] was prepared by the method of Ganesu [1]. A saturated solution of the reagent in alcohol was used for all precipitations.

Procedure. Add freshly prepared saturated solution of the reagent in alcohol to the silver solution (pH ≈ 2) in slight excess to precipitate out red coloured Ag[Cr(SCN)₄(aniline)₂]. Thereafter, keep in an ice-bath for 30 min with stirring, filter off on a weighed sintered crucible, wash with cold water, alcohol then ether and dry at 110–120°C.

Table 1. Separation of silver from foreign ions. Ag taken 19.12 mg

Expt. No.	Metal ions		Wt. of ppt. mg	Ag found mg	Error mg
	added	mg			
1	Li ⁺	18.71	102.4	19.13	+ 0.01
2	Be ²⁺	14.72	103.0	19.25	+ 0.13
3	Mg ²⁺	15.36	103.2	19.28	+ 0.16
4	Zn ²⁺	23.41	102.4	19.13	+ 0.01
5	Cd ²⁺	18.88	103.2	19.28	+ 0.16
6	Sr ²⁺	19.44	103.6	19.25	+ 0.13
7	Al ³⁺	20.46	103.4	19.32	+ 0.20
8	Pb ²⁺	22.52	102.4	19.13	+ 0.01

Amounts of 19–32 mg of Ag (corresponding to a weight of precipitate of 102–171 mg) have been successfully determined with errors of – 0.02 to + 0.09 mg. Many cations do not interfere, e. g., Li⁺, Be²⁺, Mg²⁺, Zn²⁺, Cd²⁺, Sr²⁺, Al³⁺ and Pb²⁺. Results are shown in Table 1. However, the estimation cannot be carried out in presence of Cl[–], Br[–], I[–] and SCN[–] ions. These ions precipitate with silver ions.

In the analysis of Ag[Cr(SCN)₄(aniline)₂], silver was estimated as AgCl volumetrically, chromium also volumetrically and nitrogen by Kjeldahl method. The following values have been found: Ag, 18.53 (18.67); Cr, 8.89 (9.00); N, 14.41 (14.53); calculated values in parenthesis.

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Reference

- Ganesu, I.: Chem. Abstr. 68, 45740m (1968).

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Massenspektroskopische Fragmentierung der Trimethylsilyl-Derivate von 6-Sulfonsäure- und Phosphorsäure-substituierten Pteridinen

Mass Spectral Fragmentation of Trimethylsilyl Derivatives of 6-Sulphonic and Phosphoric Acid Substituted Pteridines

Untersuchung von Pteridinen; Massenspektrometrie; Fragmentierung der Trimethylsilylderivate von 6-Sulfon- und Phosphorsäure-subst. Pteridinen

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Wir haben die Massenspektren von 4 substituierten Pteridinen mit einer Säurefunktion in 6-Stellung bzw. in 6-Stellung in einer Seitenkette untersucht. Von diesen Verbindungen wird angenommen, daß sie