Sequential Photometric Titration of Chromium(VI) and Vanadium(V) with Isonicotinic Acid Hydrazide

Aufeinanderfolgende photometrische Titration von Chrom(VI) und Vanadium(V) mit Isonicotinsäurehydrazid

Best. von Chrom(VI) und Vanadium(V) mit Isonicotinsäurehydrazid; Volumetrie/Photometrie

P. V. K. Rao, G. B. Bhaskara Rao, and R. S. Rao
Dept. of Chemistry, Andhra University, Waltair, India

Received February 23, 1973; revised May 14, 1973

Isonicotinic acid hydrazide (isoniazid) has been employed for a photometric titration of chromium(VI) and vanadium(V) according to the following procedure:

To a mixture of chromium(VI) and vanadium(V) (2—8 ml of 0.05 N each) taken in the optical cell add 12—20 ml of syrupy orthophosphoric acid and make up the volume to 40 ml with water. Arrange an inlet tube so that it is immersed at one corner of the cell out of light path. Pass carbon dioxide through the solution, to mix it, and stop the passage of the gas 30 sec before taking the absorbance reading. Adjust the dial reading of the instrument to "0". Add small aliquots of isoniazid (0.05 M) from a microburette and note dial readings 2 min after each addition. Plot a graph between dial readings of the instrument and the volume of isoniazid. The experimental curve consists of three straight lines intersecting at two inflexion points, the first corresponding to the volume of isoniazid required for the reduction of chromium(VI) to chromium(III) and the second corresponding to the reduction of vanadium(V) to vanadium(IV) and chromium(VI) to chromium(III). Thus the volume lying between the two inflexion points corresponds to the volume of isoniazid required for the reduction of vanadium(V) to vanadium(IV).

Interferences. Iron(III) (50 mg), cobalt(III) (60 mg), molybdenum(VI) (40 mg), do not interfere. Tungsten(VI) and copper(II) interfere in all proportions.

Amounts of 0.03—0.07 mMol of Cr(VI) and 0.09—0.4 mMol of V(V) have been determined with errors of 0.5% at most.

Acknowledgement. Two of us (G. B. B. R.) and (R. S. R.) are grateful to the Council of Scientific and Industrial Research for the award of respectively a Senior and Junior Research Fellowship'.

Dr. P. V. K. Rao
Dept. of Chemistry
Andhra University
Waltair, India

Simultaneous Determination of Halides and Thioureas in Their Mixture

Simultanbestimmung von Halogeniden und Thioharnstoffen im Gemisch

Best. von Halogeniden und Thioharnstoff; Volumetrie; Reaktion mit $AgNO_3$

Amjed M. Bhatti, S. M. Sondhi, and N. K. Ralhan
Department of Chemistry, Punjabi University, Patiala, India

Received February 5, 1973

In literature a few methods [1, 4, 5] for the determination of a mixture of thiourea and chlorides are mentioned. These methods, however, are laborious and work under rigid experimental conditions.

The proposed method permits the analysis of a mixture of thiourea (substituted or unsubstituted) and halides (Cl^-, Br^-, I^-) with an accuracy of $\pm 1\%$. The method is simple and accurate and has wide applications. It is based on the following chemical reactions where silver nitrate reacts with thiourea and halide in molar ratios of 2 and 1, giving silver sulphide and silver halide, respectively.

$$2\,AgNO_3 + NH_2CSNHR + 2\,NH_3 \rightarrow Ag_2S + CNNHR + 2\,NH_4NO_3$$

$$AgNO_3 + X^- \rightarrow AgX + NO_3^-.$$

Thioureas can also be determined in the presence of Cl^- or Br^- by titration with potassium iodate [7] or potassium periodate [2] in acidic medium satisfactorily. But it was found that these oxidative procedures fail in the presence of I^-. However, presence of iodide does not interfere with our silver nitrate titrations. The mixtures of thioureas with I^- have been as conveniently analyzed as with Cl^- or Br^-.

Experimental

Reagents

0.1 N Silver Nitrate. Silver nitrate (AnalaR, BDH) was dried in vacuum desiccator for a day. Its 0.1 N solution was prepared by dissolving 16.989 g in water and making the volume to 1 l.

0.1 N Ammonium Thiocyanate. Ammnoium thiocyanate pure (S. Merck) was also dried in vacuum desiccator. 8.0 g