MICRO-DETERMINATION OF As(V), V(V), Mo(VI), W(VI) IN THE PRESENCE OF Cu(II), Ni(II) AND Zn(II)

S. SINGH

Department of Chemistry, Birla College Garhwal University, Srinagar, Garhwal (U.P.) (India)

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A rapid procedure is described for the separation and determination of 0.025 mg to 1.0 mg quantities of As(V), V(V), Mo(VI) and W(VI) from small quantities of Cu(II), Ni(II), and Zn(II) using silica gel as the selective sorbent for the cations. The individual anionic components, which remain in the supernatant solution after separation from the cations, are determined by colorimetric methods. The complete recovery of As(V) in supernatant solution has also been tested radiometrically using $^{75}\text{As}$ as the radioactive indicator. The sorbed cations after extraction with dilute hydrochloric acid are determined by EDTA titrations.

Introduction

Separation of small amounts of As(V), V(V), Mo(VI) and W(VI) from Cu(II), Ni(II) and Zn(II) is often encountered in alloys, biological materials and other systems. In addition, the presence of small quantities of the cations, particularly Cu(II) and Ni(II), cause serious interference in spectrophotometric determination of the anions.1-3 The conventional gravimetric methods, if employed to the above separations, are likely to be erroneous and even unsuccessful when the ions are present in microgram quantities.

The problem of separating microgram quantities of the anions has been solved using silica gel, a well known cation exchanger, as a selective adsorbent for the cations.4-5 Investigations revealed that the cations in the aqueous ammoniacal solution were taken up by the gel, while the anions were left in the supernatant liquid. Further, the adsorbed cations could be brought into solution completely by elution with dilute hydrochloric acid. On the basis of these observations, a method has been developed which may be found useful for separating one anion from one or more of the cations.
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Experimental

Reagents and equipments

All reagents, unless otherwise stated, were of analytical grade.

Silica gel. Chromatographic silica gel was kept in contact with conc. HCl for a day, washed free of acid to remove any iron present in the commercial sample. It was then dried at 100–120 °C, and stored in a desiccator.

Radiometric determinations. A liquid G.M. tube (Type M6H) of 20th Century Electronics (U.K.) in conjunction with a decatron scaler (Philips) was used. $^{65}\text{Zn} \ (T = 245 \text{ d})$. Chemical form was ZnCl$_2$ in HCl solution.

$^{76}\text{As} \ (T = 26.5 \text{ h})$. Chemical form was HAsO$_2$ in HCl solution. Both the isotopes were obtained from BARC, Bombay (India). $^{76}\text{As(III)}$ was oxidized to As(V) by H$_2$O$_2$.

Spectrophotometric determinations. Unicam spectrophotometer, SP 600, was used.

Procedure of separation

A typical separation procedure of As(V) and Cu(II) is described. To a 30 ml sample of the mixed ammoniacal solution (pH ~ 11.5) containing the cation (0.25–0.5 mg Cu$^{2+}$) and anion [25–200 µg As(V)], 350 mg of purified silica gel is added, and the mixture allowed to stand for 10 min with occasional shaking. By this treatment the entire amount of the cation is deposited on the silica gel, leaving As(V) in the supernate. The clear supernate is decanted and preserved. The gel containing the adsorbed cation is washed with 5 ml of ammoniacal water (pH ~ 11.5), and the washing is added to the supernatant solution. The deposited cation is brought into solution by 2 × 5 ml of 0.5N HCl.

Procedure for the individual separation of the other cations, namely Ni$^{2+}$ and Zn$^{2+}$ from As(V), is just the same. The other anionic species e.g., V(V), Mo(VI) and W(VI) are isolated from their individual mixtures with the cations by the same method.

Determination of the anions

As(V). As the traces of dissolved silica (from silica gel) accompanies As(V) in the supernatant solution causing interference with the spectrophotometric determination of As(V), the former is removed by adopting a solvent extraction method.\textsuperscript{6} The collected supernate is neutralised with 2N HCl, and diluted to 50 ml. A 2.5 ml aliquot is taken in a 50 cm$^3$ separating funnel with it 2.5 ml of 2N HCl is mixed, 2.5 ml of 5% ammonium molybdate solution is then added, and allowed to stand for 15 min. To the mixture 2.5 ml of 60% HClO$_4$ is added (the pH of