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Extraction-Spectrophotometric Determination of Bismuth(III) in Wood's Alloy through Complexation of Hexa-iodobismuthate and Prochlorperazine Mesylate#

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A new modified spectrophotometric method for the determination of trace quantities of bismuth has been developed based on the complexation between prochlorperazine mesylate and hexaiodobismuthate. The orange-red complex, which is stable for 48 h has an absorption maxima at 485 nm with a molar absorptivity of 3.53×10^3 L per mol per cm and Sandall's sensitivity 0.16 μg cm^{-2}. Calibration graph is rectilinear for 0.6 to 18 ppm of bismuth. Effect of time, temperature, reagent concentration and order of addition of reagents have been studied. The proposed method had been used for the determination of bismuth in Wood's alloy as it offers the advantages of simplicity, rapidity and accurate procedure.

INTRODUCTION

Spectrophotometric methods for bismuth are relatively rare compared with transition metals because its ions are lacking in characteristic colour reactions. Literature survey reveals that there are very few methods for the spectrophotometric determination of bismuth[1–3]. Prochlorperazine mesylate (PPM), an antihistaminic drug[4], is 2-chloro-10-[3-(4-methyl-1-piperzinyl)propyl]-phenothiazine mesylate forms a mixed ligand orange-red coloured complex in dilute hydrochloric acid medium in the presence of hexa-iodobismuthate. The orange-red coloured complex can be quantitatively extracted into chloroform. PPM has been found to form complex with transition metals[5]. In the present communication the author reports a new simple extractive spectrophotometric method for the determination of bismuth in Wood's alloy based on the reaction between PPM and hexa-iodobismuthate.

EXPERIMENTAL

A stock solution of bismuth(III) (1000 ppm) was prepared from bismuth(III) nitrate pentahydrate(AnalaR) in doubly distilled water and standardised by volumetric method[6]. Dilute solutions were prepared as and when required. A 2%

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(m/V) solution of prochloroperazine mesylate (May and Baker (India) Pvt. Ltd., Bombay) and potassium iodide (BDH) were prepared in doubly distilled water stored in amber bottle in a refrigerator.

Beckman Model DB and JASCO UVI-DEC 610 spectrophotometers with 1 cm quartz cells were used for absorbance measurements.

Procedure: An aliquot of the stock solution 0.6 to 18 ppm of bismut(III) was transferred into a 50 mL separatory funnel. A 2.5 mL of 2% potassium iodide solution, 5 mL of 2% PPM and 10 mL chloroform were added. The contents were shaken vigorously and left at room temperature for 10 min. Orange-red chloroform layer was separated and dried over anhydrous sodium sulphate. The chloroform extract was transferred to 10 mL graduated flask, diluted to volume with chloroform and the absorbance was measured at 485 nm against a reagent blank prepared identically without bismuth. The amount of bismuth was then deduced from the standard calibration curves.

Determination of Bismuth in Wood's Alloy: 0.1 g of dried Wood's alloy was accurately weighed out into a beaker and dissolved in hot nitric acid (1 : 1). The precipitated tin(IV) oxide was removed by filtration. The filtrate was diluted to 250 mL with doubly distilled water in a gruduated flask. 5 mL of this stock solution was transferred to a 50 mL separatory funnel and the bismuth concentration was determined by the procedure described above.

RESULTS AND DISCUSSION

PPM reacts with hexa-iodobismuthate at room temperature to form orange-red mixed ligand complex which can be extracted into chloroform layer. The effect of acid concentration on the formation and extraction of the complex into organic phase was investigated in hydrochloric, sulphuric, nitric and phosphoric acids. The maximum intensity of orange-red colour was achieved in 1–3 M hydrochloric acid, 1–4 M sulphuric acid, 1–3 M nitric acid and 2–5 M phosphoric acid. However, hydrochloric acid is recommended for the determination because of the higher sensitivity and stability of the orange-red complex.

The effect of reagent concentration was studied by varying the concentrations of potassium iodide and PPM. For maximum colour development 2.0 to 4.0 mL of 2% potassium iodide and 3.5 to 6.5 mL of PPM solution were required for 0.6 to 20 ppm of bismuth. The orange-red complex exhibits maximum absorbance at 485 nm.

A calibration graph was constructed for chloroform extract of the mixed ligand complex. Beer's law is valid over the concentration range 0.6 to 18 ppm of bismuth and the optimum concentration range evaluated by Ringbom's method[7,8] is 1.3 to 14.5 ppm. The Sandall's sensitivity is 0.16 $\mu g\ cm^{-2}$ and the molar absorptivity is 3.53×10^3 L per mol per cm. The precision and accuracy of the method were studied by analysing solutions containing known amounts of bismuth and the relative error is within ±1.3%.

The extent of interference by other ions usually associated with bismuth was determined by measuring the absorbance of solutions containing 20 μg per mL

of bismuth and various amounts of foreign ions. An error of 2% in the absorbance reading was considered tolerable.

In order to confirm the usefulness of the proposed method, it was applied to the determination of bismuth in Wood's alloy. The results of the assay gave a value of 48.15% bismuth compared to the value of 48.37% bismuth obtained by standard method[5].

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