Note

Determination of Mercury by Radiochemical Displacement Method†

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A radiochemical displacement method for the determination of micro amounts of Hg(II) has been developed. ^{65}Zn was displaced from the Zn-1-(2-pyridylazo-2-naphthol) complex at pH 5 in borate buffers by Hg(II). 10–80 μg of mercury could be determined. Interference of various metal ions and methods for suppression have also been carried out.

Key Words: radiochemical displacement, mercury, zinc-1-(2-pyridylazo-2-naphthol)

1. Introduction

1-(2-Pyridylazo-2-naphthol), PAN, has been introduced and extensively employed as a reagent in radioanalytical chemistry in our laboratories. An earlier communication dealt with the determination of micro amounts of Cu(II) by displacement of labelled zinc from Zn-PAN complex¹⁾. The present work is intended to develop a similar method employing Zn-PAN complex for the determination of mercury(II). Earlier methods for the determination of mercury by displacement technique employed solids, especially sulphides of thallium and silver^{2)–4)}. The present method is superior as the complex in chloroform from which zinc is being displaced offers flexibility regarding the selection of concentration of the complex, the specific activity and the conditions such as pH, for the displacement reaction. Further, the equilibrium attainment will be faster.

2. Solutions and Reagents

Hg(II) stock solution (1 mgHg/ml) was pre-

pared in double distilled water by dissolving appropriate amount of mercuric chloride (Analar, BDH). The solution was standardised by a standard method⁵⁾. Stock solution of zinc was prepared by dissolving 448 mg of $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ (Merck, ProAnalyse) in 100 ml of distilled water and its zinc content was determined⁶⁾. ^{65}Zn was obtained from M/s Isotope Group, B.A.R. C., Bombay with a specific activity of 1.85 GBq/g (50 mCi/g) Zn. $2.007 \times 10^4 \text{ M}$ PAN (Reanal) was prepared in methanol. Chloroform was purified and distilled before use. Borate buffers of pH 5.0–9.0 were prepared. Preparation of ^{65}Zn labelled, Zn-PAN complex in chloroform free from reagent has been described elsewhere¹⁾.

3. Development of the Method

Preliminary experiments were conducted using borate buffers of varying pH. pH 5 was found to be suitable for quantitative displacement of zinc by Hg(II). Hence further investigations were carried out at pH 5.

Solutions of Hg(II) in increasing amounts in the range 10–80 μg were taken in a series of stoppered centrifuge tubes. pH of the solutions were adjusted to 5 with borate buffer. The total volume was kept at 5 ml. Three ml of labelled zinc complex in chloroform was added and equilibrated for 10 minutes. One ml of the

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aqueous layer was withdrawn and its γ -activity was measured. The activities obtained were corrected for blank value. The effect of 80 μg of diverse metal ions on the determination of 40 μg of mercury was also studied.

4. Results and Discussion

PAN forms a 1:2 complex with zinc as well as Hg(II) . The stability constant of mercury complex being greater than that of zinc, mercury is expected to replace zinc from its PAN complex. The displacement reactions was quantitative at pH 5.0 in borate buffers. Further, the blank value was also negligible (5%) at this pH. The amount of Zn-PAN complex used in a single determination decides the upper limit and specific activity and blank value considerations limit the lower value. Under present set of conditions the range of determination was 10-80 μg of mercury (Fig. 1).

The metals, whose complexes of PAN have higher stability constants than mercury are expected to interfere with the determination. Hence, use of masking agents is definitely necessary for better selectivity. In practice, it was observed that Cd(II) , Pb(II) , Ni(II) , Ag(II) , Cu(II) , As(V) , Se(VI) , Mo(V) and Te(VI) do not interfere. Bi(III) , Sb(V) , Co(II) and Mn(II) interfere. Among the commonly used masking agents only citrate was useful. The interference of cobalt can be masked by citrate. Among

Fig. 1 Calibration plot.

the anions sulphate, nitrate, chloride, acetate and borate do not interfere, whereas nitrite, bromide and iodide interfere with the determination.

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