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Direct Estimation of Lead in Solder Alloy Using a Novel Coated Wire Pb(II) ISE

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A new heterogeneous Pb(II) ion-selective coated wire electrode (CWISE) based on lead (II)-violuric acid complex as electroactive material has been described. The working pH range of the electrode is 2–5. The electrode shows a linear response in the concentration range 1×10^{-1} to 5×10^{-6} mol dm^{-3}. The response time of the electrode is 10 seconds. The electrode is selective for Pb(II) ions in the presence of number of cations such as Cu(II), Ni(II), Fe(III), Zn(II) and Ca(II). The electrode was used for direct determination of lead in the sample of solder.

INTRODUCTION

The study of metal present in the alloy is important in metallurgy and industries. Earlier tedious methods like gravimetry were used but with the development and advancement of science, modern sophisticated instrumental techniques[1, 2] are now applied in this field. However, the operating cost of these instruments with latest technology is very high and their availability is restricted to only few laboratories.

Among the instrumental methods of determination for constitutents of alloys, the ion selective electrodes (ISE's) have not been used extensively, only a few works have been documented[3–7], although its approach to trace analysis is advantageous because of speed and ease of ISE procedures in which little is required. Further, ISE possess wide dynamic ranges and are relatively low in cost[8–12]. These characteristics have inevitably led to sensors for several ionic species and the list of available electrodes has grown substantially over the past three decades.

Solder is a non-ferrous alloy containing lead and tin and sometimes very small amounts of other elements as well. Solder is a soft alloy used for soldering, *i.e.*, to join two metal surfaces.

A number of electrodes are known for Pb(II)[13]; they follow the traditional barrel configuration and require large amount of sample. Freiser *et al.*[14] have introduced coated wire electrodes in which an electroactive species is incorporated in a thin polymeric film coated directly on a metallic conductor. These types of devices are capable of such extreme miniaturisation that they find application in biomedical and clinical fields.

In continuation with our work on coated wire electrode[14–21] a new electroactive material, Pb(II) complex of violuric acid, has been used for the development of coated wire lead(II) ion selective electrode. The characteristics of this electrode have been examined and application for the direct determination of Pb(II) has been explored.

EXPERIMENTAL

All the reagents used for the analysis were of analytical grade. They are as follows: Violuric acid (E. Merck), lead nitrate (BDH), PVC (Apex lab), tetrahydrofuran (S.D.'s Lab. Chem.), araldite (Ciba-Geiegy), NaOH (Qualigens), HCl (E. Merck), potassium chromate (K_2CrO_4) (BDH). Solder alloy (No. 11 g) used here is from Bureau of Analysed Samples Ltd., Cleveland.

Fabrication of the electrode: To an ethanolic solution of violuric acid, the solution of lead nitrate was added gradually to obtain a pink colured precipitate of Pb(II)-violuric acid complex[22], which was filtered and subsequently washed and dried at room temperature for 24 h and used as electroactive material.

A coaxial cable was dipped several times in the slurry of electroactive material, PVC in tetrahydrofuran and little amount of araldite, until a bead[23] is formed at the tip. This cable along with the tip worked as the Pb(II) ISE which was dried in air for 48 h.

A Philips pH/mV meter model PR9405M with a saturated calomel electrode as reference electrode was used.

Electrode system: Measurements were made using the following cell:

Metal (wire)	Ion-selective membrane (bead)	Sample solution	External reference electrode (SCE)

RESULTS AND DISCUSSION

After conditioning the electrode (the electrode was dipped in 0.01 mol dm^{-3} $Pb(NO_3)_2$ solution overnight), e.m.f. measuremens were made for a series of standard solutions of $Pb(NO_3)_2$. A linear response was observed for Pb(II) concentration down to 5.0×10^{-6} mol dm^{-3}. The slope was found to be 20 mV/decade change in Pb(II) ion concentration. The above values of slope and linear response limit remained constant for a period of 20 days, after which the electrode showed erratic behaviour.

To find the response time[24, 25], the electrode was dipped in 0.01 mol dm^{-3} $Pb(NO_3)_2$ solution and suddenly the concentration of the solution was changed to 0.001 mol dm^{-3}. The values of potential changes were noted every 5 sec. A constant potential was obtained after 10 sec.

To study the effect of pH,[26] the pH of the 0.01 mol dm^{-3} $Pb(NO_3)_2$ solution was varied by addition of NaOH or HCl. It was found that the potential remained unchanged within the pH range of 2–5. The selectivity coefficients for different cations were determined using mixed solution method[27] (Table-1).

TABLE-1
SELECTIVITY COEFFICIENTS FOR DIFFERENT CATIONS

Ions	Selectivity coefficient
Ni^{2+}	0.0502
Cu^{2+}	0.0090
Zn^{2+}	0.0500
Fe^{3+}	0.0009
Ca^{2+}	0.0300

Application

(A) Titration of $Pb(NO_3)_2$ against K_2CrO_4: The fabricated electrode was used as an indicator electrode in the precipitation titration of $Pb(NO_3)_2$ solution against potassium chromate (K_2CrO_4). For this 5 mL of 0.01 mol dm^{-3} $Pb(NO_3)_2$ solution was pipetted out in a beaker, its volume was raised up to 20 mL by water. Its electrode potential was measured. This solution was titrated against 0.01 mol dm^{-3} potassium chromate solution; electrode potential was noted after each addition of 0.5 mL of the titrant. The value of potential was plotted against the volume of K_2CrO_4 used. It was observed that sharp rise in the titration curve occurs at the equivalence point and the results are highly reproducible.

Estimation of lead in solder alloy: For the estimation, sample solutions were prepared as follows: 0.4 g, 0.5 g, 0.7 g, 0.9 g and 1.0 g of solder sample was weighed separately and each treated with 1 : 1 HNO_3 to bring lead into the solution. The tin in the alloy was treated with nitric acid, a precipitate of hydrated stannic oxide was formed. The solder samples were treated with 10 mL concentrated nitric acid and 10 mL water and evaporated to a volume of 5 mL. This digestion was extended over a period of at least 1 h to ensure quantitative separation of tin.

Then it was diluted with water, heated to 90–100°C. This was filtered hot through a dense filter paper. The precipitate was washed and the washings along with the filtrate were evaporated to a minimal amount. Residual liquid was extracted with distilled water and made up to a volume of 100 mL. Measurements were made using coated wire Pb(II) ISE and the percentage was calculated from calibration curve. The results obtained are summarised in Table-2. There is a fairly good agreement between standard value and observed value.

Conclusion

The attempt to explore the possible application of new electrode in the analysis of solder was quite successful as the values obtained by ISE method and other standard method showed a fairly good agreement thus etablishing the applicability and usefulness of this fabricated electrode.

TABLE-2
ESTIMATION OF LEAD IN SOLDER SAMPLES

S. No.	Weight of solder (g)	Standard value of Pb(g)	Observed value of Pb(g)
A	0.4	0.1600	0.1574
B	0.5	0.2000	0.1989
C	0.7	0.2800	0.2693
D	0.9	0.3600	0.3523
E	1.0	0.4000	0.3936

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