

Fabrication and Characterization of Novel Pb(II) and Cr(III) Ion Selective and Sensitive Electrodes

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ABSTRACT

The Lead - Ion Selective Electrode (Pb-ISE) and Chromium-Ion electrode (Cr-ISE) is a method for the analytical determination of Pb(II) and Cr(III) in natural matrices. The ISEs are very sensitive, selective, and inexpensive analytical tools that can be applied to determine Pb(II) and Cr(III) in soil samples due to relatively high limits of detection. In the present work, we examined the possibility of using solid contact ISEs in soil analysis, combined with a simplified extraction method based on dilute HNO₃ that can be carried out at the field site. The Ionic strength of lead and Chromium is measured by using The Pb-ISE and Cr-ISE were fabricated and their characteristics were studied separately and the Ionic strength of Pb(II) and Cr(III) was measured by using them, the results were found to be quite satisfactory.

Keywords: Analytical Technique, Cr(III) ISE, Pb(II) ISE, Soil Samples.

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I. INTRODUCTION

An ion-selective electrode (ISE) also known as a specific ion electrode is a sensor that converts the activity of a specific ion dissolved in a solution into an electrical potential. Ions selective electrodes are used in analytical chemistry and biochemical/biophysical research where measurements of ionic concentration in an aqueous solution are required.

Ion-selective electrodes have a wide concentration range. It is highly selective to a particular ion. Ion-selective electrodes are unaffected by the color or turbidity of the solution. ISE has many advantages compared to other techniques, including its relatively inexpensive and easy-to-operate, wide concentration measurement range, and measures activity instead of concentration, it is particularly useful in biological, medical, and agricultural applications¹⁻².

The fabrication of novel Ion selective electrodes has caused the fundamental development in potentiometry over the past few decades; they have been applied for direct determination in samples. The pioneers Cremer and Haber explained how the potential developed across the membrane in the solution on both surfaces. The essential component is a semi-permeable membrane, in this present paper novel Pb(II) and Cr(III) coated wire selective and sensitive electrodes were fabricated to analyze sample³⁻⁸.

II. PROCEDURE

A. Preparation of Electroactive Material

Lead chromate [PbCrO₄] was prepared by adding 50 cm³ of 0.1 mol dm⁻³ of lead nitrate solution, NaOH until a precipitate formed 10 cm³ acetate buffer solution, 10 cm³ chromium chloride solution and 10 cm³ potassium bromated solutions and were added followed by heating. At 90-95°C precipitate was obtained. The precipitate was filtered and washed with water, followed by 1% nitric acid. The precipitate was dried under an IR lamp and crushed into powder form.

Chromium (α -nitroso- β -naphthol [Cr(C₁₀H₇O₂N)] prepared by adding the minimum quantity of α -nitroso- β -naphthol, a mixture of 20 cm³ of glacial acetic acid and 20 cm³ hot distilled water was added. This mixture was added to 50 cm³ of 0.1 moldm⁻³ chromium chloride solution dropwise by constant stirring. Further, the precipitate was filtered, washed with water, and dried under an IR lamp. The dried compound was crushed into powder form.

B. Preparation of Coated Wire Electrodes

This compound was mixed with an inert binder to obtain a homogeneous viscous mixture; the performance of the electrode depends on the kind of which polymeric material used as a matrix. The precipitate was mixed with Araldite and coated on a copper wire and used as coated wire ion selective electrode. The Coated wire Ion Selective Electrode was prepared by the electroactive material of Lead and Chromium with Araldite in 1:4 proportions separately and dispersed uniformly with a glass rod on two different copper wires and kept for drying for 24 hrs. Dried coated wires were dipped in relevant 0.1 mol dm^{-3} solutions of $\text{Pb}(\text{NO}_3)_2$ and CrCl_3 respectively for 24 hrs.

III. RESULTS AND DISCUSSION

The pH was measured by using a pH meter (Equiptronics EQ 615). Electrode potential was measured by the potentiometer (EQ 555). Calomel Electrode was used as a reference electrode. A magnetic stirrer was used for the stirring purpose. All physiochemical measurements were performed at room temperature.

Solutions were obtained by dissolving Copper sulfate, Aluminum Nitrate, Lead Nitrate, Manganese Sulphate, Nickel Sulphate, Chromium chloride, Aluminum Nitrate, and Calcium Chloride in water and adding acid to prevent hydrolysis wherever necessary.

A. Characteristics of ISE

1) Effect of pH

A plot was made between pH and electrode potential. For $\text{Pb}(\text{II})$ there is an increase in electrode potential up to pH 5.4 After pH 5.4 the potential remains constant. The pH range of the $\text{Pb}(\text{II})$ ion was between 5.4 to 7.7 pH.

In the case of the $\text{Cr}(\text{III})$ electrode the working pH range of the electrode was 4.1 to 6.6 Up to pH 4.1, there was an increase in electrode potential but after that, the electrode potential remained constant. After pH 6.6 there was a decrease in potential was found.

2) Response Time

To determine the response time of the electrodes each was subjected to rapid change in metal ion concentration. The electrode was dipped in $1 \times 10^{-3} \text{ mol dm}^{-3}$ solution of a relevant metal ion, taken out, and immediately dipped in $1 \times 10^{-4} \text{ mol dm}^{-3}$ solution of the metal ion. The corresponding change in potential was recorded at intervals of 10 sec. The period required to attain a steady potential was read from plots⁹⁻¹⁰ (Fig. 1a and 1b).

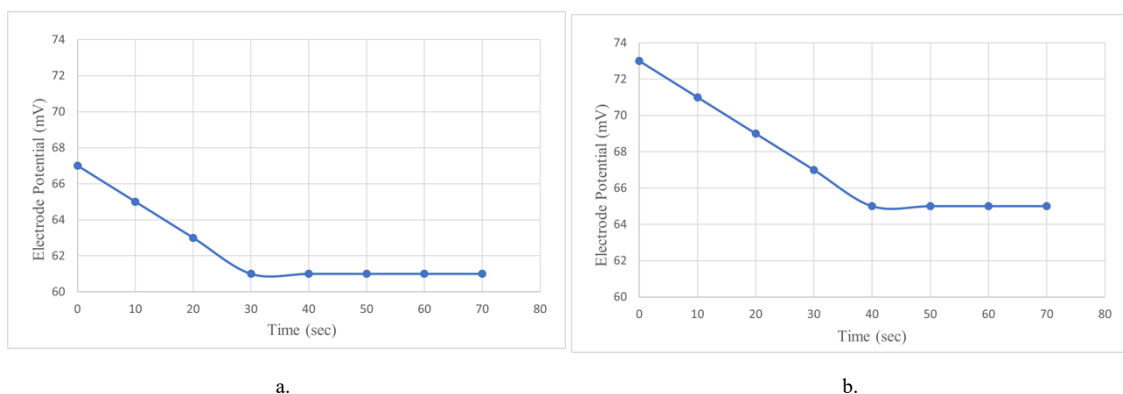


Fig. 1. a) Dynamic response time of $\text{Pb}(\text{II})$ -ISE; b) Dynamic response time of $\text{Cr}(\text{III})$ - ISE.

B. The linear Response of $\text{Pb}(\text{II})$ and $\text{Cr}(\text{III})$ Electrode

The linear responses of different electrodes were tested at different concentrations of the metal solution. Solution of various concentrations 1×10^{-2} to $1 \times 10^{-8} \text{ mol dm}^{-3}$ prepared and their electrode potential was measured. The curve furnishes information on the lower detection limit and the slope was determined to know the change of potential per electrode.

The $\text{Pb}(\text{II})$ electrode shows a linear response to PbNO_3 solution. It shows a response range of 1×10^{-7} to $1 \times 10^{-8} \text{ mol dm}^{-3}$ and the slope is 9 mV. A plot was made between electrode potential and the negative logarithm of concentration the $\text{Cr}(\text{III})$ electrode (Table I, Fig. 2a and 2b) shows a linear response to CrCl_3 solution in the range of 1×10^{-7} to 1×10^{-8} and slope is 14 mV¹¹⁻¹⁵.

TABLE I: CHARACTERISTICS OF NOVEL Pb(II) ISE AND Cr(III) ISE

Novel ISE	Linear working range	Slope	Working PH range	Response Time	Shelf life
Pb(II) ISE	1×10^{-7} - 1×10^{-8}	9.0 mV	5.4-7.7	30 sec.	20 weeks
Cr(III) ISE	1×10^{-7} - 1×10^{-8}	14.0 mV	4.1-6.6	40 sec.	28 weeks

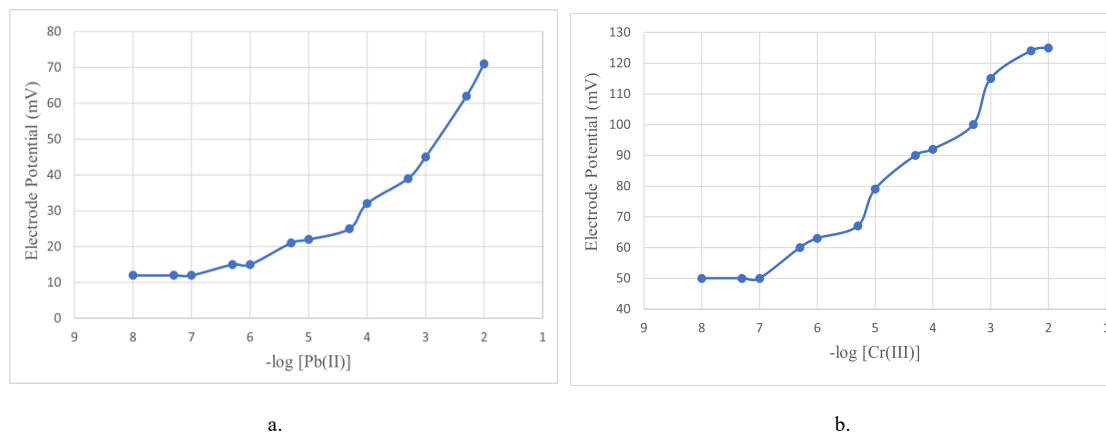


Fig. 2. a) Calibration curve of Pb(II)-ISE; b) Calibration curve of Cr(III)-ISE.

C. Interference

Potentiometric selectivity coefficient values were determined by mixed solution method using (1) (as shown in Table II).

$$k_{ij} = a_i / (a_j)^{z_i/z_j} \quad (1)$$

TABLE II: SELECTIVE COEFFICIENT		
	Selective Ion	K^{Pot}
		Pb(II) M
Pb(II) ISE	Ni (II)	5×10^{-3}
	Mn (II)	1×10^{-3}
	Ca (II)	5×10^{-1}
	Al (III)	5×10^{-5}
	Selective Ion	K^{Pot}
		Cr(III) M
Cr(III) ISE	Ni (II)	0.0316
	Mn (II)	0.0316
	Ca (II)	0.0158
	Al (III)	5×10^{-4}

The obtained selectivity coefficients indicate that the functions of Pb(II)-ISE and Cr(III)-ISE cannot be disturbed by the presence of these cations.

IV. APPLICATION

The Pb(II)-ISE and Cr(III)-ISE was used for the determination of Pb(II) ions and Cr(III) ions respectively in the soil samples under the laboratory conditions. The results obtained were very satisfactory and accurate. They were compared by other techniques also and found to be quite similar.

V. CONCLUSION

It can be concluded that the ISE can be employed as a convenient and useful tool for the concentration of ion in the sample solution.

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