



## A Simple Heterogenous Precipitate Based Sodium ion Selective Electrode- Its Preparation and Analytical Application

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### ABSTRACT

A heterogeneous precipitate have been used as ion carriers for the preparation of Na(I) selective membrane sensor. This electrode give near-nernstian responses in the linear concentration range of 1M to  $1 \times 10^{-7}$ M with detection limits of the order of  $10^{-7}$ M. The stable potentiometric signals are obtained with in a short time period of 4 minutes. The effect of pH and the effect of medium have been studied found to give a better response. Selectivity coefficient values ( $\log K_{Na,M}^{pot}$ ) have been evaluated using fixed interference method. The sensor have also been used as indicator electrode in commercial products.

**Key words:** A Heterogeneous precipitate, Na(I)ion membrane sensor, Fixed interference method.

### INTRODUCTION

Sodium hydroxide is a caustic metallic base. It is used in many industries, mostly as a strong chemical base in the manufacture of pulp and paper, textiles, drinking water, soaps and detergents and as a drain cleaner.

Food uses of sodium hydroxide include washing or chemical peeling of fruits and vegetables, chocolate and cocoa processing, caramel coloring production, poultry scalding and soft drink processing.

The field of ion-selective electrodes (ISEs)

bridges fundamental host-guest chemistry, membrane science and its specific applications. Because of their simplicity, low cost, sufficiently reliable and repeatable measurements ISEs are recognized as novel analytical tools for selective determination of analyte ions<sup>1,2</sup>.

Many Na-ISE incorporating a various ion carriers have been reported. In the present study, a simple heterogeneous precipitate based membrane have been prepared along with the potentiometric performances of these sensors, effect of pH, effect of medium, response time and selectivity coefficients with respect to different interfering ions have also been studied.

## EXPERIMENTAL

### Preparation of polyaniline

About 4.6 ml of aniline and 10 ml of aqueous HCl, 0.1 M ammonium per sulphate (FISHER scientific, Qualigens Fine Chemicals, Mumbai) is taken in a 250 ml beaker<sup>3</sup>. A dark green powder of polyaniline precipitates out. The precipitate is washed with ethanol, dried and powdered.

### Preparation of s-benzyl isothiuronium chloride precipitate

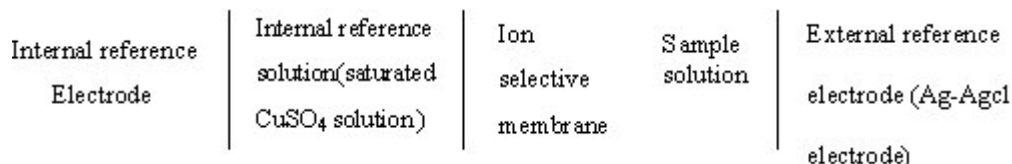
About 0.5g of benzoic acid (HI-PURE, FINE CHEM INDUSTRIES) was added to 6 ml of aqueous NaOH (FINAR REAGENTS, Extra pure) of strength 0.9246 N. Mixed the resulting solution with an equal volume of S-Benzyl isothiuronium chloride reagent (HIMEDIA Laboratories PVT.Ltd). A white precipitate of S-benzyl isothiuronium chloride was obtained.

### Preparation of membrane electrode

About 0.1g of powdered S-benzyl

isothiuronium chloride precipitate was thoroughly mixed with 0.1g of polyaniline and Epoxy resin (Huntsman advanced materials, India Pvt. Ltd) The paste obtained was applied on Whatmann filter paper No.42. This was spread uniformly over the filter paper to obtain 0.9mm thickness of the electro active materials with matrix. This was left in air to dry for 48 hours to get an electro active material with matrix. A circular piece of this membrane was cut and fixed with resin at one end of the hollow glass tube (diameter 0.6cm and length 9cm). This tube was filled with saturated solution of  $\text{CuSO}_4$  and reference copper metal wire was inserted (diameter 0.5mm & length 12cm) through other end of  $\text{CuSO}_4$  solution already filled in this glass tube. This complete assembly will work as an ion selective electrode for sodium ion determination. This ion selective electrode was kept in 1M solution of sodium chloride for one week to attain equilibrium.

The entire electrode system for the measurement can be represented as



## RESULTS AND DISCUSSION

The Electrode was first conditioned in 1M solution of sodium ion till it attained stable equilibrium after which it was used for the determination of characteristic study of the electrode. The electrode potential for a series of standard solution of Na(I) ions was measured. The electrode gave a linear response to Na(I) concentration in the range of 1M to  $1 \times 10^{-7}$ M. The sodium-ISE reveals near Nernstian slopes 27mv/decade graph-1.

To find the response time the electrode was first dipped in 1M of Na(I) solution and suddenly the concentration of the solution was changed to 0.1M. The variation in potential was noted at every 5 seconds till a constant potential was obtained at 3 minutes and remain constant.

### Effect of pH and Effect of medium

To study the effect of pH, a standard solution containing 1M sodium ion were prepared in which a series of buffer solution 4.02 to 9.2 was added. It was found that the potential remained unchanged with in the pH range 7-9.2.

**Table 1: Electrode Response**

Concentration of sodium chloridesolution (M)	E.M.F (in volts)
1	-0.025
$1 \times 10^{-1}$	-0.024
$1 \times 10^{-2}$	-0.022
$1 \times 10^{-3}$	-0.016
$1 \times 10^{-4}$	-0.014
$1 \times 10^{-5}$	-0.011
$1 \times 10^{-6}$	-0.009
$1 \times 10^{-7}$	-0.006

To study the effect of medium, a standard solution containing 1M Na(I) ion in a series of 25%,50%,75% ethanol was added. It was found that the potential remains unaffected in ethanol medium.

**Table 2: Interference by Cations**

Cation (Interfering ion)	Electrode Log $K_{Na,M}^{pot}$
Mg <sup>2+</sup>	-0.024
K <sup>+</sup>	-0.025
NH <sub>4</sub> <sup>+</sup>	-0.024
H <sup>+</sup>	-0.024

**Table 3: Interference by Anions**

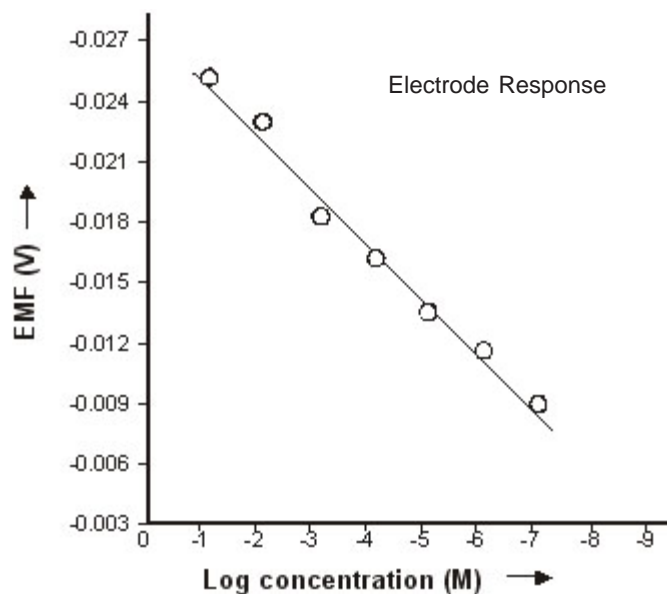
Anions (Interfering ion)	Electrode Log $K_{Na,M}^{pot}$
SO <sub>4</sub> <sup>2-</sup>	-0.024
I <sup>-</sup>	-0.025
Cl <sup>-</sup>	-0.024
Br <sup>-</sup>	-0.024
S <sub>2</sub> O <sub>3</sub> <sup>2-</sup>	-0.025
C <sub>2</sub> O <sub>4</sub> <sup>2-</sup>	-0.025

### Selectivity

The selectivity, which is an important characteristics of a membrane sensor, is measured in terms of potentiometric selectivity coefficient  $K_{Na,M}^{pot}$ . It measures the response of the sensor towards the primary ion in the presence of secondary ion present in the sample solution. The selectivity coefficient has been determined by using fixed interference method (FIM) based on semi empirical Nicolski-Eisenman equation. In this method the concentration of primary ion Na (I) ion is varied, where as the concentration of secondary interfering ion is kept constant in the sample solution which is  $1 \times 10^{-1} M$  concentration of interfering ion in the present case. The potentiometric selectivity data of sensors for various interfering ions is given in table 2 & 3. These ions don't disturb the normal functioning of the sensors and the sensors possess high sodium selectivity and respond weakly to these interfering ions

### Analytical applications

To assess the applicability of the sensor to real samples, an attempt was made to determine sodium ion in real samples like commercial baking powder and ala. The recovery of sodium ion in sample analysis was found to be quantitative with the maximum recovery of 95%.



**Graph 1: Plot of EMF vs log concentration of sodium (M)**

## REFERENCES

1. a) Bakker E, Bahlmann P and Pretsch E, *Chem. Rev.*, **97**: 30383 (1997)  
b) Buhlmann P, Pretsch E and Bakker E, *Chem. Rev.*, **98**: 1593 (1998).
2. a) Chandra S, Agarwal H, Singh CK, Sindhu S.K and Kumar P, *Ind. J. chem.* **44A**: 2060;(6) (2005).  
b) Nanda D, Chouhan HDS, Maiti B, *Ind. J. chem.* **43(A)**: 1685(2004).
3. Asila Al-Musheifri and Graham R.D. Johns, "Glucose interference in direct ion-selective electrode sodium measurement:", *Annals of clinical Biochemistry*, **45**: 530-532 (2008).
4. Bijster and Vink, "Direct potentiometric determination of sodium ion in blood. I. potentiometric response in simple sodium chloride solutions", *Clinical Chemistry*, **30**: 6-10 (1984).
5. Chapman and Goldsmith, "Determination of chloride, sodium and potassium in salted food stuffs using ion-selective electrodes and the dry sample addition method," *Analyst*, **107**: 1014-1018 (1982).