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A Heterogeneous Precipitate based Sodium Membrane Ion Selective Electrode: Its Preparation and Analytical Application

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Abstract

A heterogeneous precipitates have been as ion carriers for the preparation of Na (I) selective membrane sensor. The electrodes give near–nernstian responses in linear concentration range of 1M to $1x10^{-4}$M with detection limits of the order of 10^{-4}M . The stable potentiometric signals are obtained with in a short time period of 3 minutes. The effect of pH, and the effect of medium have been studied found to give a better response. Selectively coefficient values (log K^{pot}) have been evaluated using fixed interference method. The sensors have also been as an indicator electrode in commercially available products.

Keywords: 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, textile drinking water, soaps detergents and a as a drain cleaner.

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

The field of Ion–selective electrodes (ISEs) bridge fundamental host guest chemistry, membrane science and its specific application. Because of their simplicity, low cost, sufficiently reliable and respectable measurement, ISEs are recognized as novel analytical tools for selective determination of analyte ions[1,2].

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 sensor, effect of pH, effect of medium, response time and selectivity coefficients with respect to different interfering ions have also been studied.

Experimental Process

Preparation of S-Benzyl Isothiouronium 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 isothiouronium chloride reagent (HIMEDIA Labaratories PVT.Ltd).A white precipitate of S-benzyl isothiouroniun chloride was obtained.

Preparation of S-Benzyl Isothiouronium Chloride based Membrane Electrode Using PVC and DOP

About 0.1g of dried precipitate was thoroughly mixed with 0.1g of polyvinyl chloride (KEMPHASOL Bombay) and added Dioctyl Phthalate plasticier, (LOBAL CHEMIE laboratory Reagents & Fine Chemicals). The paste obtained was applied on whatmann filter paper no.42. this was spread uniformly over the filter paper to obtain 0.5 thickness of the electro active materials with matrix. This was left in air to dry for 24 hours to get an electro active membrane. 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 6cm). This tube was filled with saturated solution of $CuSO_4$ and reference copper metal wire was inserted (diameter 0.5mm & length 12cm) through other end of $CuSO_4$ 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

Internal reference Electrode	Internal reference solution(saturated $CuSO_4$ solution)	Ion selective membrane	Sample solution	External reference electrode (Ag/AgCl)

Result and Discussion

Response characteristics of the electrode

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) ion was measured. The electrode gives linear response to Na(I) ion concentration in the range of 1.0M to $1x10^{-4}$M (Table 1). The sodium - ISE revealed near nernstian slopes 27mV/sdecade. Graph-1

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

Table 1: Electrode response

Concentration of Sodium Chloride solution(M)	E.M.F volts
1	-0.018
$1x10^{-1}$	-0.017
$1x10^{-2}$	-0.016
$1x10^{-3}$	-0.015
$1x10^{-4}$	-0.003
$1x10^{-5}$	-0.003

Effect of pH

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 remains unchanged with in the pH range of 4.01-7.

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.

Selectivity

The selectivity, which is an important characteristics of a membrane sensor is measured in terms of the potentiometric selectivity coefficient K^{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 Nicolskii-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 $1x10^{-1}$M concentration in the present case. The potentiometric selectivity coefficient data of sensors for various interfering ions given in Table -2&3. These ions don’t disturb the normal functioning of the sensors and the sensors posses high sodium selectivity and respond weekly to these interfering ions.

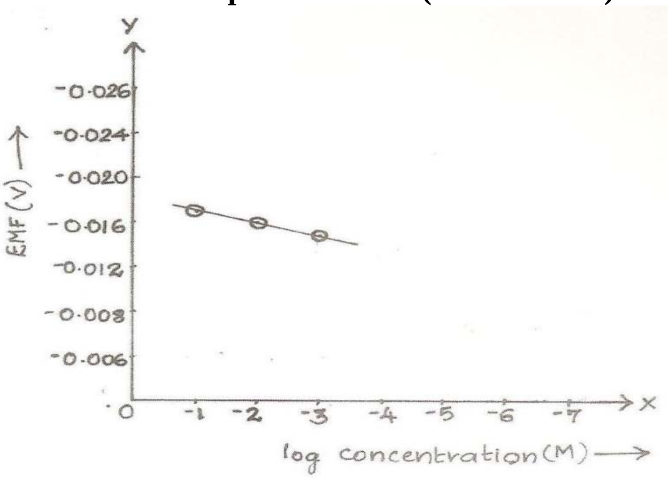
Table 2: Interference by cations

Cation (Interfering ion)	Electrode Log K^{pot}
Mg^{2+}	-0.018
K^+	-0.019
NH_4	-0.019
Na^+	-0.019
H^+	-0.017

Table 3: Interference by anions

Anions(Interfering ion)	Electrode Log K^{pot}
SO_4^{2-}	-0.018
I^-	-0.019
Cl^-	-0.018
Br^-	-0.019
$S_2O_3^{2-}$	-0.019
NH_2-CO-NH^-	-0.017
$C_2O_4^{2-}$	-0.018

Electrode response sodium (electrode-III)



Graph: plot of E.M.F. versus log concentration of sodium (M)

Analytical Application

To asses the applicability of the sensors to real samples an attempt was made to determine sodium ion in real samples like commercially baking powder and ala. The recovery of sodium ion in sample analysis was formed to be quantitative with the maximum recovery of 95%.

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