STRONTIUM ION SELECTIVE ELECTRODE-ITS PREPARATION AND ANALYTICAL APPLICATION

BY

S.SUGANYA

(11PCM13)

A DISSERTATION SUBMITTED TO

AVINASHILINGAM INSTITUTE FOR HOME SCIENCE AND HIGHER EDUCATION FOR WOMEN, UNIVERSITY

COIMBATORE-641043

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS

FOR THE DEGREE OF

MASTER OF SCIENCE IN CHEMISTRY

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R. Shyamala

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Head of the department

J. Thamarai Selvi 8.5.13

signature of the guide

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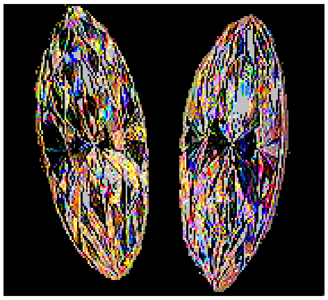
INTRODUCTION

Strontium –Sr

Strontium is a soft, silver-yellow, alkaline-earth metal. It has three allotropic crystalline forms and in its physical and chemical properties it is similar to calcium and barium Strontium reacts vigorously with water and quickly tarnishes in air, so it must bestored out of contact with air and water. Due to its extreme reactivity to air, this element always naturally occurs combined with other elements and compounds. Finely powdered strontium metal will ignite spontaneously in air to produce both strontium oxide and strontium nitride



Strontium is a grey, silvery metal that is softer than calcium and even more reactive toward water, with which it reacts on contact to produce strontium hydroxide and hydrogen gas. It burns in air to produce both strontium oxide and strontium nitride, but since it does not react with nitrogen below 380 °C, at room temperature it will only form the oxide spontaneously. Three allotropes of metallic strontium exist, with transition points at 235 and 540 °C.

Chemical properties

Strontium is so active it must be stored under kerosene or mineral oil. In this way, the metal does not come into contact with air. In a finely divided or powdered form, strontium catches fire spontaneously and burns vigorously. Strontium is active enough to combine even with hydrogen and nitrogen when heated.The compounds formed are strontium hydride (SrH_2) and strontium nitride (Sr_3N_2). Strontium also reacts with cold water and with acids to release hydrogen gas.

PHYSICAL PROPERTIES

Atomic number	38
Atomic mass	87.62 $g.mol^{-1}$
Electronegativity according to Pauling	1.0
Density	2.6 $g.cm^{-3}$ at 20°C
Melting point	769 °C
Boiling point	1384 °C
Vander Waals radius	0.215 nm
Ionic radius	0.113 nm (+2)
Isotopes	14
Electronic shell	[Kr] $5s^2$
Energy of first ionisation	549.2 $kJ.mol^{-1}$
Energy of second ionisation	1064 $kJ.mol^{-1}$
Discovered by	A. Crawford in 1790

Occurrence in nature

Strontium is a relatively abundant element in the Earth's crust. It ranks about 15th among the elements found in the Earth. That makes it about as abundant as fluorineand its alkaline earth partner, barium.

The most common minerals containing strontium are celestine and strontianite. Celestine contains primarily strontium sulfate ($SrSO_4$), while strontianite contains mostly strontium carbonate ($SrCO_3$). Important world sources of strontium are Mexico, Spain, Turkey, and Iran. A small amount of strontium is also obtained from mines in California and Texas.

Isotopes

Four isotopes of strontium occur in nature. They are strontium-84, strontium-86, strontium-87, and strontium-88. Isotopes are two or more forms of an element. Isotopes differ from each other according to their mass number. The number written to the right of the element's name is the mass number. The mass number represents the number of protons plus neutrons in the nucleus of an atom of the element. The number of protons determines the element, but the number of neutrons in the atom of any one element can vary. Each variation is an isotope.

About ten radioactive isotopes of strontium are known also. A radioactive isotope is one that breaks apart and gives off some form of radiation. Radioactive isotopes are produced when very small particles are fired at atoms. These particles stick in the atoms and make them radioactive.

One radioactive isotope of strontium, strontium-90, is of special interest. It is a toxic substance which, at one time, was the cause of great concern because of its connection to atomic bomb testing. Today, strontium-90 has a number of useful applications. For example, it is used to monitor the thickness of materials. Metal sheeting for construction must be the same thickness throughout. The sheeting is carried along on a conveyor belt beneath a small container of strontium-90. The isotope gives off radiation, some of which passes through the metal sheeting. The thicker the sheeting, the less radiation gets through. The thinner the sheeting, the more radiation gets through. A radiation detector is placed below the conveyor belt. The detector measures the amount of radiation passing through the sheeting. An inspector monitors the reading and makes adjustments to the manufacturing equipment to maintain the right thickness.

Strontium-90 is used for a number of other industrial applications, all based on the same principle. For instance, strontium-90 is used to measure the density of silk and tobacco products.

Strontium-90 has medical applications. A recent advance is to use the isotope for the control of pain. People who have cancer of the bone often experience terrible pain. At one time, the only treatment was medication. But those drugs often had unpleasant side-effects, such as nausea, dizziness, or depression.

Strontium in the environment

Strontium is commonly occurs in nature, forming about 0.034% of all igneous rock and in the form of the sulfate mineral celestite ($SrSO_4$) and the carbonate strontianite ($SrCO_3$). Celestite occurs frequently in sedimentary deposits of sufficient size, thus the development of mining facilities attractive. The main mining areas are UK, Mexico, Turkey and Spain. World production of strontium ores is about 140.000 tonnes per year from an unassessed total of reserves.

Foods containing strontium range from very low e.g. in corn (0.4 ppm and oranged (0.5 ppm) to high, e.g. in cabbage (45 ppm), onions (50 ppm) and lattuce (74 ppm).

Health effects of strontium

Strontium compounds that are water-insoluble can become water-soluble, as a result of chemical reactions. The water-soluble compounds are a greater threat to human health than the water-insoluble ones. Therefore, water-soluble forms of strontium have the opportunity to pollute drinking water. Fortunately the concentrations in drinking water are usually quite low.

People can be exposed to small levels of (radioactive) strontium by breathing air or dust, eating food, drinking water, or by contact with soil that contains strontium. We are most likely to come in contact with strontium by eating or drinking.

Strontium concentrations in food contribute to the strontium concentrations in the human body. Foodstuffs that contain significantly high concentrations of strontium are grains, leafy vegetables and dairy products.

For most people, strontium uptake will be moderate. The only strontium compound that is considered a danger to human health, even in small quantities, is strontium chromate. The toxic chromium that it contains mainly causes this. Strontium chromate is known to cause lung cancer, but the risks of exposure have been greatly reduced by safety procedures in companies, so that it is no longer an important health risk.

The uptake of high strontium concentrations is generally not known to be a great danger to human health. In one case someone experienced an allergic reaction to strontium, but there have been no similar cases since. For children exceeded strontium uptake may be a health risk, because it can cause problems with bone growth.

The uptake of high strontium concentrations is generally not known to be a great danger to human health. In one case someone experienced an allergic reaction to strontium, but there have been no similar cases since. For children exceeded strontium uptake may be a health risk, because it can cause problems with bone growth

Strontium salts are not known to cause skin rashes or other skin problems of any kind.When strontium uptake is extremely high, it can cause disruption of bone development. But this effect can only occur when strontium uptake is in the thousands of ppm range. Strontium levels in food and drinking water are not high enough to be able to cause these effects.

Radioactive strontium is much more of a health risk than stable strontium. When the uptake is very high, it may cause anaemia and oxygen shortages, and at extremely high concentrations it is even known to cause cancer as a result of damage to the genetic materials in cells.

Uses and compounds

Strontium and its compounds have relatively few commercial uses. The pure metal is sometimes combined with other metals to form alloys. An alloy is made by melting and mixing two or more metals. The mixture has different properties than the individual metals. Compounds of strontium are sometimes used to color glass and ceramics. They give a beautiful red color to these materials. Strontium compounds also provide the brilliant red color of certain kinds of fireworks.

Consuming 75% of production, the primary use for strontium is in glass for cooler television cathode ray tubes. It prevents X-ray emission. All parts of the CRT tube have to absorb X-rays. In the neck and the funnel of the tube, lead glass is used for this purpose, but this type of glass shows a browning effect due to the interaction of the X-rays with the glass. Therefore, the front panel has to use a different glass mixture, in which strontium and barium are the X-ray-absorbing materials. The average values for the glass mixture determined for a recycling study in 2005 is 8.5% strontium oxide and 10% barium oxide. The amount of strontium used for the production of cathode ray tube is declining because the CRTs are replaced by other display methods. This decline has a significant influence on the mining and refining of strontium Health effects

Most strontium compounds are regarded as harmless to plants and animals. A few, such as strontium chloride ($SrCl_2$) and strontium iodide (SrI_2), are somewhat toxic.

Dental care

$SrCl_2$ is useful in reducing tooth sensitivity by forming a barrier over microscopic tubules in the dentin containing nerve endings that have become exposed by gum recession. Known in the US as Elecol and Sensodyne, these products are called "strontium chloride toothpastes", although most now use potassium nitrate instead which works as a nerve calming agent rather than a barrier.

Biological research

Brief strontium chloride exposure induces parthenogenetic activation of oocytes[6] which is used in developmental biological research

Strontium has a variety of commercial and research uses.

Strontium compounds are used in making ceramics and glass products, paint pigments, fluorescent lights, fireworks, signal flares, optical products, and medicines.

Some forms of strontium are being studied to possibly treat and prevent bone loss.

Strontium chloride hexahydrate is added to toothpaste to reduce pain in sensitive teeth.

Strontium chloride is commonly found in dietary supplements.

Radioactive SR85 has medical and industrial uses.

SR90 has been used as an energy source in research and to produce electricity for weather stations, navigational buoys, and satellites

ION SELECTIVE ELECTRODE

Ion-selective electrodes (ISE) consist of an ion-specific half-cell and a reference half-cell. The ion-specific cell gives a potential against the reference cell depending on the specific ion concentration. When the specific ion concentration (the sample or an ion standard) changes, the potential changes as well.

Ion-Selective Electrodes are part of a group of relatively simple and inexpensive analytical tools which are commonly referred to as Sensors. The pH electrode is the most well known and simplest member of this group and can be used to illustrate the basic principles of ISEs.

As indicated by their name, ion-selective electrodes possess a high degree of selectivity. The selectivity of the ISE is determined by the composition of the membrane. Ideally the membrane allows the uptake of only one specific ion into it. The analyte ion may be a cation or an anion. The three main components of making a measurement at an ISE are an inner reference, or standard, solution and an outer analyte, or sample, solution separated by a thin membrane.

Types of Ion Selective Electrode

The generally accepted classification of membrane electrodes is according to the physical state of the membranes used. The electrodes are classified into four main types as follows: -

- Glass Electrodes
- Solid-state Electrodes
- Liquid ion exchange membrane Electrodes
- Gaseous membrane Electrodes

1. **Glass membrane electrodes**:

The most famous glass electrode determines H^+ activity or pH. The membrane is composed of a silicate glass. Glass electrodes can also be constructed that are sensitive to other cations such as sodium.

2. Crystalline / solid state membrane electrodes:

The membrane is composed of an insoluble inorganic salt. An ion-exchange process leads to the formation of a potential at the membrane. Polycrystalline or mixed crystal membranes such as $AgCl / Ag_2S$ can be used to determine Cl^-. This electrode is used in one of the experiments outlined in the Experimental section.

Single crystal LaF_3 is widely used to determine F^-. The crystal is usually doped with europium to improve the conductivity. At each membrane-solution interface, the following equilibrium takes place:

$$LaF_3\,(s) \rightleftharpoons LaF_2^+\,(s) + F^-\,(aq)$$

3. Liquid membrane electrodes:

An ion-exchanger or ionophore (neutral macrocyclic ion carrier) is dissolved in a viscous organic liquid membrane. Without the exchanger or ionophore the ion of interest is unable to penetrate the membrane. With the exchanger or ionophore present, the processes described in Figures 2 and transport the ion into the membrane.

4. Polymer membrane electrodes:

An alternative to wet liquid membrane electrodes is to use a polymeric membrane, which is composed of a polymer such as polyvinylchloride (PVC), a plasticizer, and the ion carrier or exchanger. The response of these electrodes is highly selective and they have replaced many liquid membrane electrodes. Polymer electrodes have been used to determine ions such as K^+, Ca^{2+}, Cl^- and NO_3^-.

PLASTICIZERS:

Plasticizers represent a category of organic substances that can be added to polymers in order to improve some of their characteristics like elasticity and mechanical properties. Plasticizers are very important for the design of the selective polymeric membrane for potentiometric sensors, an important class of the electrochemical sensors. They fix the dielectric constant of the membrane and improve the selectivity of those devices. Choosing an appropriate plasticizer as well as an adequate electrode component and sometimes an additive is essential for the construction of an ion- selective electrode with high analytical performances such as sensibility, selectivity, fast response and long lifetime.

POTENTIOMETRY

Potentiometry is an analytical technique, much used since 1966. Potentiometry can be described as the measurement of a potential in an electrochemical cell.

Potentiometry finds widespread use by chemists

- For direct and selective measurement of analyte concentration
- For establishing end point in various titrations
- For the determination of several types of equilibrium constant. It is the only electrochemical technique that directly measures a thermodynamic equilibrium potential and in which essentially no net current flows

Direct potentiometric measurements provide a rapid and convenient method for determining the activity of various cations and anions. The technique requires only a comparison of the potential developed in a cell containing the indicator electrode in the analyte solution, with its potential when immersed in one or more standard solution of known analyte concentration.

The most important consideration in using potentiometric technique is the type of indicator electrodes to be used. Depending on the application a variety of electrodes are available, each with inherent advantages and disadvantages.

The use of potentiometry increased with the development of several new types of ion selective electrodes i.e.; electrodes that responds more or less selectivity to various ions.

The potentiometer is rather elementary, yet it is one of the most precise and useful measuring devices. It is fundamental to almost all circuitry and is the basis of one of the most widely used instrumental method in potentiometric titrations.

The basis of potentiometric titration is the Nernst equation

$E_{cell} = E^0_{cell} + (RT/nF) \times \ln Q$;

Which relates the potential of an electrolytic cell to the concentration of the species present in the system.

It is possible to detect endpoint of a titration by measuring the electrode potential of a suitable electrode which is known as indicator electrode.

Potentiometric titrations have become popular as they not only establish equivalence points of many reactions but also provide information about concentrations of one or more of the reactants during the titrations.

REVIEW OF LITERATURE

Membranes of 4-tert-butylcalix(8)arene-octaacetic acid octaethyl ester (I) as an electroactive material, sodium tetraphenyl borate (NaTPB) as an anion excluder, and tri-n-butylphosphate (TBP) as a solvent mediator in poly(vinyl chloride) (PVC) matrix have been tried for a strontium-selective sensor. The best performance was exhibited by the membrane having a composition 5:100:150:2 (I: PVC: TBP: NaTPB*(w/w)*). This sensor exhibits a good potentiometric response to Sr2+ over a wide concentration range (3.2 × 10 –5 –1.0 × 10 –1 M) with a Nernstian slope (30 mV/ decade). The response time of the sensor is 10 s and it has been used for a period of four months without any drift in potentials. The selectivity coefficient values are in the order of 0.01 for mono-, bi-, and trivalent cations which indicate a good selectivity for Sr2+ over a large number of cations. The useful pH range for the sensor was found to be 3-10 and it works well in mixtures with non-aqueous content up to 25 % (v/v). The sensor has been used as an indicator electrode in the potentiometric titration of Sr2+ against EDTA (**Ajay K.Jain et al.,2004).**

A novel PVC membrane sensor for the Sr2+ ion based on 1,10-diaza-5,6-benzo-4,7-dioxacyclohexadecane-2,9-dione has been prepared. The sensor possesses a Nernstian slope of 30.0 ± 0.6 mV decade-1 over a wide linear concentration range of 1.6 ´ 10-6-3.0 ´10-3 M with a detection limit of 6.3 ´10-7 M. It has a fast response time of <15 s and can be used for at least twomonths without any considerable divergence in potential. The potentiometric response is independent of the pH of test solution in the pH range 4.3-9.4. The proposed electrode shows good selectivities over a variety of alkali, alkaline earth, and transition metal ions **[MojtabaShamsipur et al., *2007*]**

The potential response behaviour of a strontium doped poly(dibenzo-18-crown-6) film electrode to strontium prepions in solution has been examined. The effect of polymerization conditions on the characteristics of the response is discussed. The performance of the electrode with respect to electrode reproducibility, response stability and regeneration is also studied. The calibration plots for electrode have an unexpected slope of 58–59 mv per strontium(II) concentration decade at 25[ddot]C and a detection limit of about 2.9 × 10^{-5} M. The selectivity coefficients for this polymeric electrode for most of the potential interferants are in the order of 10^{-3}. This polymeric film electrode has a relatively fast response, is easy to prepare and has a reasonable life time (**Naim et al.,1991**)

Eight different recently synthesized macrocyclicdiamides were studied to characterize their abilities as strontium ion carriers in PVC membrane electrodes. The electrode based on 1,13-diaza-2,3;11,12-dibenzo-4,7,10-trioxacyclopentadecane-14,15-dione exhibits a Nernstian response for Sr^{2+} ions over a wide concentration range (1.0 × 10^{-1}–3.2 × 10^{-5} M) with a limit of detection of 8.0 × 10^{-6} M (0.7 ppm). The response time of the sensor is ~10 s, and the membrane can be used for more than three months without observing any deviation. The electrode revealed comparatively good selectivities with respect to many alkali, alkaline earth, and transition metal ions. It was used as an indicator electrode in potentiometric titration of carbonate ions with a strontium ion solution.(**Mojtaba et al.1999)**

Ion-selective microelectrodes were prepared by coating a matrix containing the crown ether benzo-18-crown-6 or one of five polyether ring-substituted benzo-18-crown-6 derivatives on a preconditioned silver wire in a flow cell. Responses of the electrodes toward lithium, sodium, potassium, magnesium, calcium, strontium, and lead were measured. Selectivity coefficients were obtained by the matched potential, fixed interference, and separate solution methods. Compared with benzo-18-crown-6, the nature of the side arm in the lariat ether derivatives was found to influence the selectivities in different ways. For specific ionophores, high selectivities of potassium over lithium, sodium, magnesium, calcium, and strontium, of strontium over magnesium and calcium, and of lead over magnesium, calcium, and strontium were demonstrated (**Abdulrahmanet al., 1992).**

Poly(vinyl chloride) membrane strontium ion-selective electrodes were developed by using three lipophilic diamides containing pyridine ring as ionophores. The relationship between the structure of the ionophores and the performance of these electrodes, as well as the effects of the plasticizers and additives, were investigated. The Sr^{2+}-electrode based on *N*,*N*,*N′*,*N′*-tetracyclohexyl-2,6-pyridine-bis(methyleneoxyacetamide) as neutral carrier, potassium tetrakis (*p*-chlorophenyl) borate (KTpClPB) as additive, and *o*-nitrophenyloctyl ether (*o*-NPOE) as plasticizing solvent. It exhibits excellent properties with a Nernstian slope of 29 mV/$pSr^{2+}$and a linearity range of 2×10^{-5} to 1×10^{-2} M at 25°C, $K_{Sr,Ba}^{Pot}=2\times10^{-2}$**(Guoying et al.1998).**

A sol-gel electrode, based on 6-(4-nitrophenyl)-2-phenyl-4,4-dipropyl-3,5-diaza-bicyclo [3,1,0] hex-2-ene (NPDBH) as a neutral ionophore, was successfully developed for the detection of Sr^{2+} in aqueous solutions. Theoretical calculations confirmed NPDBH selectivity toward strontium in comparison with some other metal ions. The electrode responds to Sr^{2+} ion with a sensitivity of 29.1±0.4 mV/decade over the range 8.0×10^{-7}–1.0×10^{-1} M. Selectivity coefficients determined by matched potential method (MPM) indicate high selectivity for strontium ions. The electrode has a fast response time of 11 s and a working pH range of 3.0–10.0. The sol-gel electrode shows detection limit of 7.5×10^{-8}M**(Zanjanchi et al.,2009).**

The 2-[(2-mercaptophenylimino)methyl] phenol (MPMP) was used as an excellent ionophore in the construction of a Sr^{2+} PVC-based membrane sensor. The best performance was obtained with a membrane composition of 30%

poly(vinyl chloride), 62% nitrobenzen (NB), 5.5% MPMP and 2.5% sodium tetraphenyl borate (NaTBP). This sensor demonstrates a good selectivity and sensitivity towards the strontium ion for many cations, including alkali, alkaline earth, transition and heavy metal ions. The sensor revealed a great enhancement in selectivity coefficients for strontium ions in comparison with the previously reported strontium sensors. The proposed sensor exhibits a Nernstian behavior (with a slope of 29.6 ± 0.3 mV per decade) for the concentration range of (1.0×10^{-6}–1.0×10^{-1} M) with a detection limit of 5.5×10^{-7} M (48.2 ng/mL). It illustrates a relatively fast response time in the whole concentration range (< 10 s) and it can be used for at least 10 weeks in a pH range of 2.8–9.6. The developed sensor was successfully used as an indicator electrode in the Sr(II) titration with EDTA and the Sr^{2+} ion recovery from binary mixtures(**Hassan et al., 2008).**

The PVC membrane containing 2,3-diphenylquinoxaline-4′,4″-dioxytriethylene glycol (PQTEG) as a suitable ionophore, presents a linear dynamic range of 1.0×10^{-6}–1.0×10^{-2} M with a Nernstian slope of 29.9 mV per decade and a detection limit of 6.7×10^{-7} M. The response time is quick (smaller than 15 s), it can be used in the pH range of 4.1–8.9 and its duration is at least 55 days without noticing any considerable potential divergence. The proposed electrode has very good selectivity with respect to all common alkali, alkaline earth in the range of − 3.28 to − 4.00, and for transition metal ions in the range of − 3.38 to − 4.00. It was successfully applied as an indicator electrode in potentiometric titration of Sr(II) ions with EDTA**(Hassan et al., 2009).**

Strontium tungstoarsenate membrane shows response for S^{r2+} and can be used to determine the activity of these ions. The response time of the membrane is a few seconds and the potentials generated are reproducible. The membrane electrode can be used for one year without any drift in potentials. The membrane was found to be more selective to S^{r2+} ions than to many other of univalent, bivalent and tervalentcations. Anions also show no interference **(Ajay et al., 2007**).

A poly(vinyl chloride)-based membrane of 2,9-dimethyl-4,11-diphenyl-1,5,8,12-tetraazacyclotetradeca-1,4,8,11-tetraene with sodium tetraphenylborate (STB) as an anion excluder and dibutyl phthalate (DBP), *tris*(2-ethylhexyl) phosphate (TEP), dibutylbutylphosphonate (DBBP), and tributyl phosphate (TBP) as plasticizing solvent mediators was prepared and investigated as a Sr(II)-selectiveelectrode. The best performance was observed with the membrane having the ligand-PVC-DBP-STB composition 1:4 : 1:1, which worked well over a wide concentration range (1.4×10^{-6}–0.1 mol L^{-1}) with a Nernstian slope of 28.6 mV per decade of concentration between pH 4.0 and 10.0. This electrode showed a fast response time of 12 s and was used over a period of 100 d with good reproducibility (s= 0.3 mV). The selectivity coefficients for monovalent, divalent and trivalent cations indicate excellent selectivity of Sr(II) ions over a large number of cations including Ca(II) and Ba(II). Anions such as Cl^- and SO_4^2 do not interfere and the electrode also works satisfactorily in partially water-alcohol medium. The sensor has been used as an indicator electrode in the potentiometric titration of Sr(II) with EDTA. The practical utility of the membrane sensor has also been observed in solutions contaminated with detergents i.e., cetyltrimethylammonium bromide and sodium dodecyl sulfate (**Panwar et al.,2001).**

5,11,17,23,29,35-Hexakis(1,1,3,3-tetramethylbutyl)-37,38,39,40,41,42-hexakis(carboxy methoxy)calix[6]arene (I) has been evaluated as an ionophore for the analysis of Sr^{2+}. The influences of the nature of the plasticizers (DBA, CN, DOP, NPOE) and of the anion excluder (NaTPB) on the characteristics of the electrode were discussed. The best electrode was fabricated with a membrane having composition 6:150:170:3 (I:PVC:DBA:NaTPB). The response to Sr^{2+} was Nernstian in the range 1.9×10^{-5} to 1.0×10^{-1} M of Sr^{2+}. The influence of pH has also been studied. The electrode exhibited better potential stability and had an operational lifetime of 4 months. The $K_{A,B}^{Pot}$ values showed that other alkaline earth metal ions are well discriminated. The sensor has also been used as an indicator electrode in the potentiometric titration of sodium carbonate with strontium(II) ions **(Gupta et al., 2005).**

Potential response of strontium(II) ion selective electrode based on dibenzo-30-crown-10 (DBCE) as ion-carrier has been evaluated by varying the amount of ionophore, plasticizer, and anionic additive. The sensitivity, working range, response time, and metal ions interference have shown a significant dependence on the amount of ionophore, kind and amount of additive, and solvent mediator. The electrode prepared with 5% ionophore, 55% benzyl acetate, 10% oleic acid, and 30% PVC has shown a Nernstian slope of 29.20 mV per decade to activities of Sr^{2+}ions with a short response time of 10 s. The sensor has shown an appreciable selectivity for Sr^{2+}ions in the presence of alkali, alkaline earth, transition, and heavy metal ions and could be used in a pH range of 3.0–10.0. The wrap around complex of DBCE with Sr^{2+} contributed significantly to enhance the selectivity of the electrode. The electrode has shown an appreciable average lifetime of four months without any significant drift in the electrode potential. The sensor was applied as an indicator electrode in potentiometric titration of Sr^{2+} with EDTA and for direct determination of strontium in synthetic water samples **(Mohammad et al., 2003).**

A new PVC membrane based strontium(II) ion-selective electrode has been constructed using acetophenonesemicarbazone as a neutral carrier. The sensor exhibits a Nerstian response for strontium(II) ion over a wide concentration range 1.0×10^{-2}–1.0×10^{-7} M with the slope of 29.4 mV/per decade. The limit of detection was 2.7×10^{-8} M. It was relatively fast response time (<10 s for concentration ⍰1.0×10^{-3} and <15 s for concentration of ⍰1.0×10^{-6} M) and can be used for 8 months without any considerable divergence in potentials. The proposed sensor revealed relatively good selectivity and high sensitivity for strontium(II) over a mono, di, trivalent cation and can be used in a pH range of 2.5–10.5. It was also successfully used as an indicator electrode in potentiometer titration and in the analysis of concentration in various real samples **(Sulekh et al.,2011).**

Polyvinyl chloride (PVC) based membranes of dibenzo-24-crown-8(I) and 4-tert-butylcalix[8]arene (II) with and without plasticizers have been fabricated and explored as sensors for Ba2_ and Sr2_, respectively. The membrane incorporating the ingredients in the ratio 8:50:6:36 [I:PVC:NaTPB (sodium tetraphenylborate): CN (1-chloronaphthalene)] works well over the concentration range 1.4_10_5–1.0_10_1 M of Ba2_ with a Nernstian slope of 3091 mV per decade of concentration in the pH range 3.5–8.9 while the membranes of calixarene (11) were tried as Sr2_ selective sensors and it was observed that the membrane incorporating the ingredients in the ratio 9:50:5:36 {II:PVC:NaTPB: TEP [tris-(2-ethylhexyl) phosphoric acid]} showed the best response with a working concentration range of 2.0_10_5–1.0_10_1 M and a near-Nernstian slope of 31.391.0 mV per decade of concentration in the pH range 2.4–4.8. Both sensors exhibit a fast response time of 15 s and work well in partially non-aqueous medium having up to 30% (v:v) alcohol content. The selectivity data showed that the electrodes are sufficiently selective over a number of interfering ions including alkaline earth metals. Anions like Cl_, NO_ 3 also do not interfere in the functioning of the sensors **(Gupta et al.,1999).**

In this study, the comparative study of ZnOnanorods and ZnO thin films were performed regarding the chemical and biosensing properties and also ZnOnanorods based strontium ion sensor is proposed. ZnOnanorods were grown on gold coated glass substrates by the hydrothermal growth method and the ZnO thin films were deposited by electro deposition technique. ZnOnanorods and thin films were characterised by field emission electron microscopy [FESEM] and X-ray diffraction [XRD] techniques and this study has shown that the grown nanostructures are highly dense, uniform and exhibited good crystal quality. Moreover, transmission electron microscopy [TEM] was used to investigate the quality of ZnO thin film and we observed that ZnO thin film was comprised of nano clusters. ZnOnanorods and thin films were functionalised with selective strontium ionophoresalicylaldehydethiosemicarbazone [ST] membrane, galactose oxidase, and lactate oxidase for the detection of strontium ion, galactose and l-lactic acid, respectively. The electrochemical response of both ZnOnanorods and thin films sensor devices was measured by using the potentiometric method. The strontium ion sensor has exhibited good characteristics with a sensitivity of 28.65 ± 0.52 mV/decade, for a wide range of concentrations from 1.00×10^{-6} to 5.00×10^{-2} M, selectivity, reproducibility, stability and fast response time of 10.00 s. The proposed strontium ion sensor was used as indicator electrode in the potentiometric titration of strontium ion versus ethylenediamine tetra acetic acid [EDTA]. This comparative study has shown that ZnOnanorods possessed better performance with high sensitivity and low limit of detection due to high surface area to volume ratio as compared to the flat surface of ZnO thin films **(Khun et al., 2013)**.

Dibenzo-18-crown-6 crown ether is found to exhibit quite promising selectivity for Sr^{2+} ions. It can be used to estimate strontium in the range 5.0×10^{-6} to 1.0×10^{-1} $moldm^{-3}$ (0.28 to 8.76×10^{-3} mg dm^{-3}) with a near-Nernstian slope of 28 mV per decade of concentration. The working pH range of the proposed sensor is 2.0-6.0 with a response time of 25 s. The sensor can be used for more than three months in aqueous as well as in partially non-aqueous media. The practical utility of the membrane sensor has also been observed in solutions contaminated with detergent matter**(Mohammad et al.,2009)**.

Strontium tungsto arsenate membrane shows response for Sr2 plus and can be used to determine the activity of these ions. The response time of the membrane is a few seconds and the potentials generated are reproducible. The membrane electrode can be used for one year without any drift in potentials. The membrane was found to be more selective to Sr**2** plus ions than to many other of univalent, bivalent and tetravalent cations. Anions also show no interference**(Ajay et al., 2007).**

Regenerative Sr(++)-induced Sr++ release from isolated rat liver mitochondria was studied using ion-selective electrode techniques. Mitochondria, when exposed to a pulse of Sr++, demonstrated a reversible and transient increase in inner membrane permeability to K+ and H+ ions. The increase in permeability was an all-or-none process with a threshold dependent on the amplitude of the Sr++ pulse. The threshold concentration of Sr++ was lowered from 120-150 microM to 20-30 microM when mitochondria were preloaded with 100 nmolesSr++/mg protein. Release of matrix-stored divalent cations provided a mechanism for amplification of the extramitochondrialSr++ concentration (regenerative Sr(++)-induced Sr++ release). The mitochondrial inner membrane became refractory, since subsequent cycles of excitation could not be immediately induced. These experiments demonstrate that the inner mitochondrial membrane is an excitable membrane, with threshold-dependent, regenerative and subsequently refractileSr(++)-induced Sr++ release characteristics **(Holmuhamedov et al.,1995)**.

A heterogeneous membrane electrode fabricated with hydrous thorium oxide gel embedded in polystyrene matrix as the sensor material, has been used for the estimation of strontium ions. The electrode responds to strontium ion in a nearly Nernstian manner over the concentration range 10^{-1} to 10^{-4} M. It can be used in the pH range 2.5 to 5.5 as well as in partially non-aqueous system. The membrane electrode shows good selectivity to strotium ions. A membrane treated with cationic surfactant not only becomes immune to detergent interference, but also exhibits a larger validity range. It has also been used as an indicator electrode in potentiometric titrations involving strontium ions **(Srivastava et al., 1984).**

This work introduces a unique ionophore for the selective determination of Sr2þ ions. The ionophore, salicylaldehydethiosemicarbazone (ST), presented a high affinity toward the strontium cations. The demonstrated characteristics of the sensor included a linear dynamic range between 1.0_10_6 and 1.0_10_2M with a near Nernstian slope of 29.4 _ 0.5mV per decade, a detection limit of 4.8_10_7 M, and a very good selectivity for Sr2þ over other cations in a wide pH range (2.8–10.4). The sensor possessed the advantages of short conditioning time, fast response time (<10 s), and, especially, good selectivity toward the transition and heavy metal ions as well as some mono, di, and trivalent cations. Concerning the electrode lifetime, no considerable potential divergence was noticed for at least 10 weeks. The developed sensor was successfully usedas an indicator electrode in the Sr2þ titration with EDTA from binary mixtures, and the Sr2þ monitoring in mixtures of three and five different ions (**Hassan et al.,2008).**

The effects of the addition of ionic liquids (ILs) to the membrane phase on the potentiometric properties of strontium-selective electrodes were investigated. The studies were carried out with plasticized PVC membranes doped with bis[(trifluoromethyl)sulfonyl]amides. The electrode basic analytical parameters, such as detection limit, linear range, slope characteristics, response time and dependence of the electrode potential on pH as well as selectivity coefficients in relation to some inorganic cations were determined. As follows from the studies the addition of ILs to the membrane phase improved the analytical parameters of the studied electrodes, especially the improvement in selectivity was notable (**Wardak et al.,2008).**

In order to preparing strontium ion selective electrode 6-(4- nitrophenyl)-2-phenyl-4, 4- dipropyl-3, 5-diaza-bicyclo [3, 1, 0] hex-2-ene (NPDBH) was used as an ionophore. This electrode responds to Sr (II) ion with a sensitivity of 28.2 ± 0.5 mV/decade over the range 1.0 × 10−6 to 1.0 M at pH 3.0-10.0. The limit of detection was 2.4 × 10−7 M. The influences of membrane composition, the pH of the test solution, the temperature effect and the interfering ions on the electrode performance for the proposed electrode were investigated. In the next step, electrolyte solution system SrCl2, KCl, H2O in the high concentration range were thermodynamically investigated. The thermodynamic investigations were performed by evaluation of potentiometric experimental data, successfully **(Kia et al., 2011).**

A new PVC membrane electrode based on 5,7,12,14-dibenzo-2,3,9,10-tetraoxa-1,4,8,11-tetraazacyclotetradecane (**I**) as an ion carrier, *o*-nitrophenyloctyl ether (*o*-NPOE) as solvent mediator and sodium tetraphenylborate (NaTPB) as lipophilic additive was fabricated and investigated as Sr^{2+}-selective electrode. The best performance was exhibited by the membrane having composition 8:200:4:120 (**I**:*o*-NPOE:NaTPB:PVC). The electrode exhibited a Nernstian response for strontium ion over a wide concentration range 3.98×10^{-6} to 1.0×10^{-1} M with a slope of 29.0 ± 0.1 mV/decade of concentration and a detection limit of 2.82×10^{-6} M. It showed a response time of less than 10 s and could be used for at least 3 months without any divergence in potential. The proposed electrode showed a good discriminating ability towards strontium(II) ion over a wide variety of other metal ions including alkali, alkaline earth, transition, and heavy metal ions. The electrode can be used in the pH range of 2.5–10.5 and in mixtures containing up to 35% (v/v) non-aqueous content. It was used as an indicator electrode in potentiometric titration of strontium ion against EDTA **(Singh et al., 2006).**

A new PVC membrane strontium ion-selective electrode has been constructed using 6-(4-nitrophenyl)-2-phenyl-4,4-dipropyl-3,5-diazabicyclo[3,1,0]hex-2-ene(NPDBH) as a neutral ionophore. The electrode was prepared with 7% NPDBH (as ionophore), 57% acetophenone(as plasticizer), 30% PVC and 6% oleic acid (as lipophilic additive). The electrode responds to Sr2+ ion with a sensitivity of 28.2±0.5 mV/decadeover the range 1.0×10−6–1.0×10−1 mol L−1 and in a pH range of 3.0–10.0. The limit of detection was 2.4×10−7 mol L−1. It has a responsetime of <20 s and can be used for at least three months without any divergence in potentials. The proposed electrode shows good discrimination of Sr2+ ion from several cations. The effect of organic solvents on electrode response was examined. The results show that this electrode can be used in ethanol media up to 15% (v/v) concentration without interference. The isothermal temperature coefficient of this electrode amounted to 0.00019 V/◦C. The electrode was found to work well under laboratory conditions. It was successfully applied to the determination of strontium ions in human urine and bone digests (**Zanjanchi. et al., 2007**)**.**

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A potentiometric strontium-selective electrode based on 18-crown-6 in PVC matrix for measurement of strontium ion is reported. The electrode was prepared by coating the surface of a graphite by a membrane containing dioctylphthalate (DOP), 18-crown-6 and carbon powder in poly(vinyl chloride) (PVC) in tetrahydrofuran (THF). The membrane composition consisting of 18-crown-6, PVC, DOP, carbon, the thickness of coated membrane, effect of pH and possible interfering cations and anions were investigated and optimized electrode were evaluated. The electrode exhibits significantly enhanced response toward strontium ions over the concentration range 1 × 10 -7 to 1 × 10-2 M with slope of 27.35 mV per decade change. Fast and stable response, good reproducibility, long-term stability is demonstrated. The electrode has a response time of 20-25 s and can be used for at least 6 weeks without any considerable divergence in their potential response. The proposed electrode shows fairly good discrimination of strontium from several cations. The electrode was used for direct potentiometric measurements of strontium ions over the concentration range 1 × 10-7–1× 10-2 M. The practical utility of the electrode has been demonstrated by using it successfully as an indicator electrode in the potentiometric titration of Sr2+with EDTA**(Mohammad, et al.,2009).**

AIM AND SCOPE

Several instrumental techniques such as spectrophotometry, chemiluminescence, atomic absorption spectroscopy, polorography, mass spectrometry, gas chromatography etc., have been reported for the determination of individual metals in micro and submicro gram levels. These sophisticated techniques require expensive equipments and skilled personal to obtain reliable results.

The aim of this study is to develop a new novel method for the preparation of strontium ion selective electrode. The use of ion selective electrode in environmental analysis offers several advantages over other methods of analysis. The cost of initial set up to make analysis is relatively low. Time consuming steps such as filtration, weighing and distillation are not required in most cases. ISE determinations are not subject to interference such as colour of the sample. Since electrodes are portable, measurement can be made on a laboratory bench, the bank of a river or on the floor of a manufacturing plant strontium is a trace element, which is toxic if present in higher amount. Considering analytical interest in the environmental analysis, Ion Selective Electrodes offers a simple, rapid, precise and inexpensive potentiometric procedure. It is also a new novel and eco friendly method for the direct determination of strontium ion in vegetables.

EXPERIMENTAL METHOD

Chemicals required:

Polyvinyl chloride, di-n-butyl phthalate, sodium tetra phenyl borate, tetra hydro furan were obtained from E.Merck. the standard stock solutions 1M strontium chloride were prepared using distilled water. Working solutions were made by dilution of the stock solution. A digital potentiometer (EQUIP-TRONICS EQ 602) with Ag/Agcl electrode as a reference electrode was used for this study.

Preparation of ionophore:

About 10 ml of water is added to 3g of semicarbazide hydrochloride followed by 3g of anhydrous sodium acetate and a mixture is warmed gently until a clear solution is obtained. Then a solution of 3 ml of the 4-methoxyacetophenone is added to it and warmed on a water bath. Crytals of semicarbazone separate on cooling

Preparation of membrane Ion selective electrode

The membrane electrode was prepared by 0.3g of ionophore, 0.1g of PVC, predetermined ratio of DOP and NaTPB was dissolved in 3 ml THF and the clear solution was evaporated slowly. Then it was mixed with araldite and spread uniformly over whatmann filter paper No.42 to attain 0.7 mm thicknesses of the electro active membrane. The membrane was air dried for 48 h

A circular piece from each of the membrane was cut and fixed with resin at one end of hollow glass tubes of diameter 2 cm and length 10 cm. The tubes were filled with 1M solution of strontium chloride. Reference copper metal wire of diameter 0.5 mm and length 12 cm was inserted through the other end of tube in such a way, that it remains dipped in the 1 M solution of strontium Chloride. The electrodes were conditioned for 48 hours to attain equilibrium in 1M $SrCl_2$ solution. The EMF measurements were carried out using the following cell assembly,

Internal Reference Electrode (Cu wire)	Internal Reference $SrCl_2$ Solution	Electro Active Membrane	Sample Solution	External Reference Electrode (Ag/AgCl)

Experimental process:

The ion selective electrode was connected to one terminal of a digital potentiometer (Equiptronics MODEL EQ602). The other terminal was connected to the reference electrode (saturated calomel electrode). The ISE was immersed in the sample solution $SrCl_2$ and the reference Ag/AgClis immersed in saturated KCl solution. The solutions are connected using Potassium nitrate agar- agar salt bridge.

- Preparation of stock solution of Lead nitrate was carried out by weighing lead nitrate to its molecular weight (331.20g) for 1 lit.
- Preparation of stock solution of Magnesium sulphate was carried out by weighing Magnesiumsulphate to its molecular weight (246.47g) for 1 lit.
- Preparation of stock solution of NaClwas carried out by weighing NaCl to its molecular weight (331.20g) for 1 lit.
- Preparation of stock solution of $CaCl_2$was carried out by weighing $CaCl_2$ to its molecular weight (100 g) for 1 lit.
- Preparation of stock solution of Zinc chloride was carried out by weighing Zinc chloride to it's equivalent weight (68.145 g) for 1 litre.
- Preparation of stock solution of Barium chloride was carried out by weighing Barium chloride to it's equivalent weight (122.14g) for 1 litre.
- Preparation of stock solution of Potassium iodide was carried out by weighing Potassium iodide to it's equivalent weight (166g) for 1 litre.
- Preparation of stock solution of Potassium nitrate was carried out by weighing Potassium nitrate to it's equivalent weight (101.11 g) for 1 litre.

The electrode was first conditioned in 1M solution of Sr (II) ion till it attained stable equilibrium. After which it can be used for the determination of the characteristic study of the electrode.

The following studies are conducted to ensure the efficiency of the electrode.

The electrode potential of a series of standard solutions of Sr(II) had been measured to determine the electrode response.

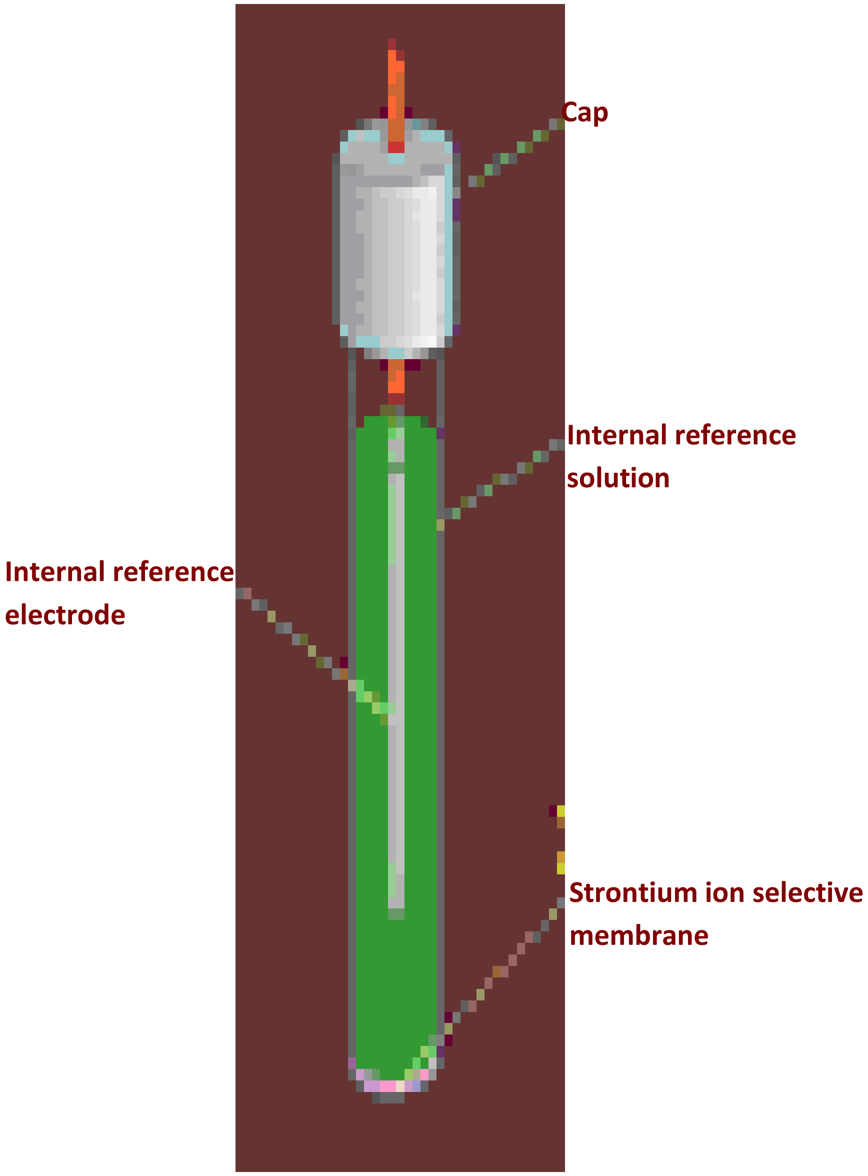
The electrode potential of standard Sr(II) solution in a series of varying concentration of acetone had been measured to determine the effect of solvent.

The electrode potential of standard Sr(II) solution of varying pH had been measured.

The interfering effect of various cations and anions had been determined.

The water used in this study was double distilled deionized water with a conductivity of less than 2 ms/cm. All the reagents used were of analytical grade. The electrode has to be rinsed in deionized water between every measurement to prevent contamination by carry over on the electrode.

Schematic diagram of Strontium ion selective electrode



EXPERIMENTAL PROCESS

Figure 2

RESULTS AND DISCUSSION

CHARACTERIZATION

In order to absorb infrared radiation a molecule must undergo a net change in dipole moment, as a consequence of its vibrational motion. The FT-IR spectrum of semicarbazone of 4- methoxyacetophenone was recorded using FTIR spectrometer (Model: Testscan Shimadzu Prestige-21 series) in the region 4000–500 cm^{-1} and the spectrum is shown in Fig. 3.

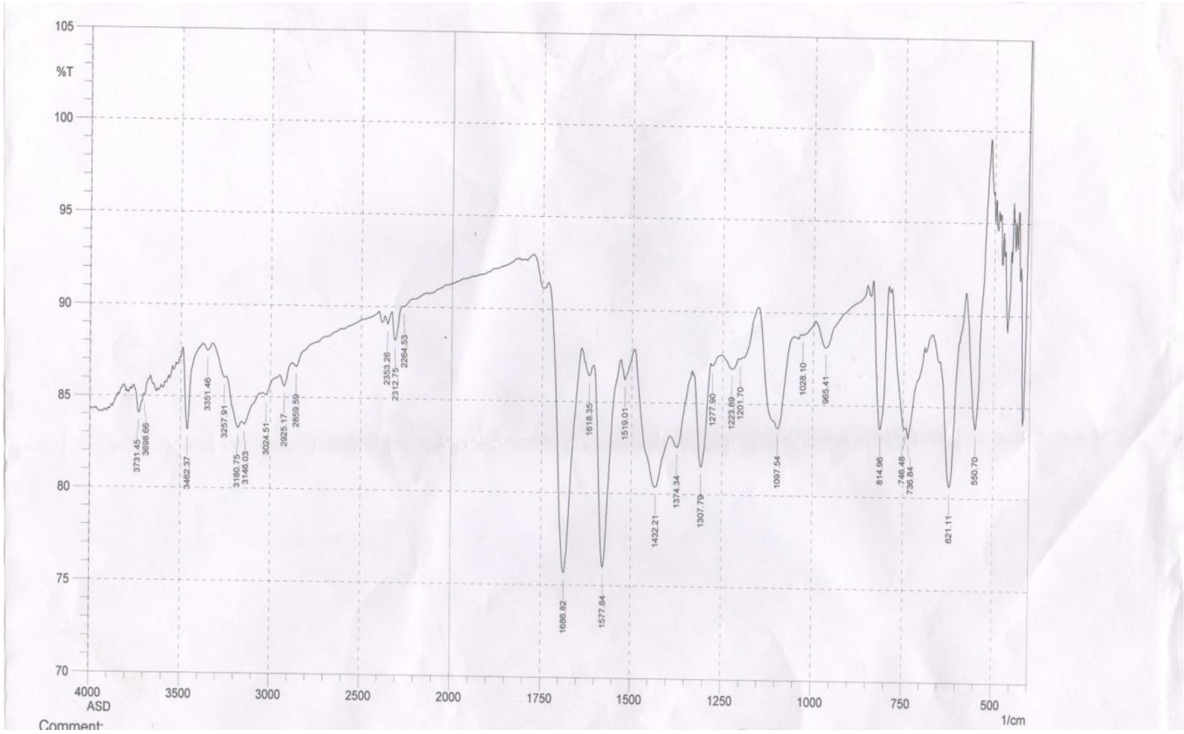


Fig .3 FT- IR Spectrum of semicarbozone of methoxyacetophenone

Below 1400 cm^{-1} the region is called finger print region. There is an intense sharp peak at 3462 cm^{-1}, assigned to the free unhydrogen bonded N–H stretch. The broad envelope between 2859 and 3351.2 cm^{-1} is due to overlapping of peaks of hydrogen bonded N–H and aromatic C–H stretching modes.. The symmetric and asymmetric C–H stretching modes of $-CH_3$ group appear as a shoulder just below 3000 cm^{-1} in the broad envelope. The C =O stretch of semicarbazide moiety is observed at 1686.4 cm^{-1}. The aromatic ring skeletal vibrations are observed at 1577. The $-CH_3$ bending modes are positioned at 1432 and 1307cm^{-1}.

ELECTRODE RESPONSE:

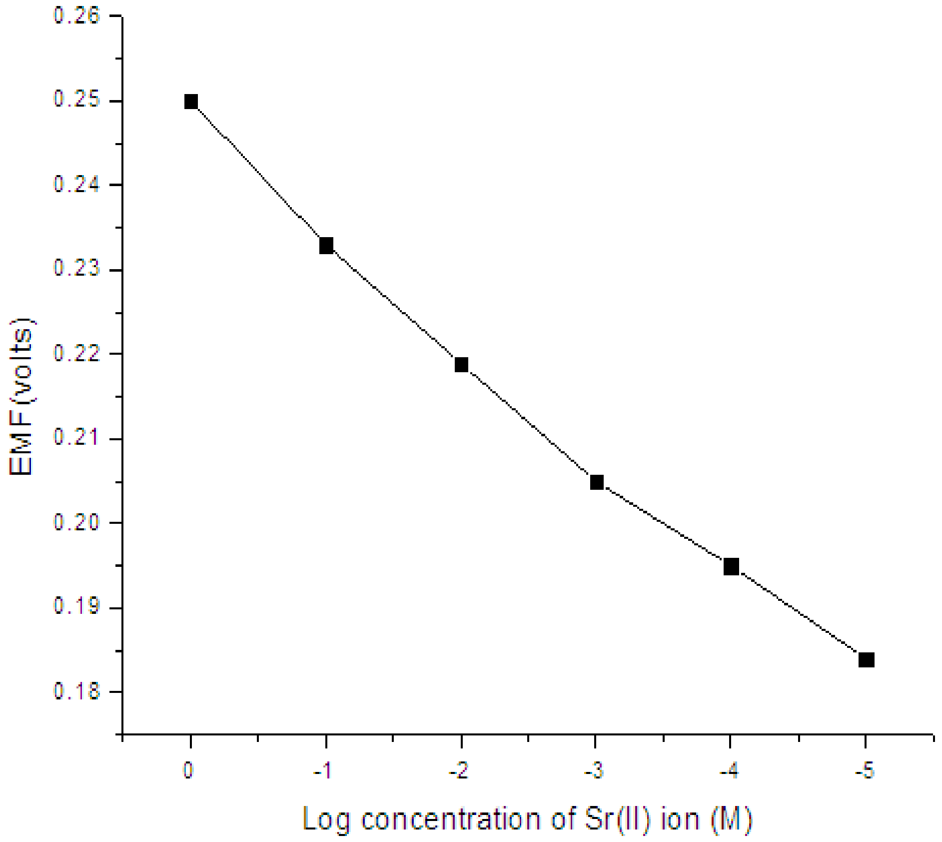
The electrode potential for a series of standard solution of Sr^{2+} was measured. The electrode gave a linear response to Sr^{2+} ion concentration in the range of 1.0 to 1x 10^{-5} M.

TABLE – I

ELECTRODE RESPONSE

Concentration of $SrCl_2$ (M)	**EMF In volts**
1	0.250
$1x10^{-1}$	0.233
$1x10^{-2}$	0.214
$1x10^{-3}$	0.203
$1x10^{-4}$	0.195
$1x10^{-5}$	0.184

Electrode Response



Plot of Log concentration of Sr (II) ion Vs EMF in Volts

Fig . 4

Standard electrode potential of this electrode (E^o) determined by extrapolation method was found to be 0.053V. The slope value was found to be 14. This value shows that the electrode behaves according to Nernst equation. The variation in potential was noted till a constant potential was obtained at about 10 minutes and remains constant. The electrode was used over a period of one month with good reproducibility.

EFFECT OF pH ON ELECTRODE RESPONSE

The effect of pH on the response of electrode was studied in this work. The electrode potential of standard Sr (II) solution of varying pHhad been measured.It was found that the electrode worked well over a wide pH range of 4 to 7.

TABLE – II

EFFECT OF pH ON ELECTRODE RESPONSE

Concentration of $SrCl_2$	pH4	pH7
1M	0.249	0.249
$1x10^{-1}$ M	0.231	0.229
$1x10^{-2}$ M	0.216	0.217
$1x10^{-3}$ M	0.204	0.202
$1x10^{-4}$ M	0.194	0.191
$1x10^{-5}$M	0.183	0.182

EFFECT OF MEDIUM ON ELECTRODE RESPONSE:

To study the effect of medium, a standard solution containing 1M Sr (II) ion in a series of 25%,50%,75% Ethanol, acetone and dimethyl formamide was added. It was found that the potential remains unchanged in the above medium.

Table 2

Effect of medium for Sr^{2+}ions

S .No	Conc. of the $SrCl_2$ Solution (M)	Ethanol			Acetone			DMF		
		25%	**50%**	**75%**	**25%**	**50%**	**75%**	**25%**	**50%**	**75%**
1.	1	0.249	0.248	0.258	0.249	0.248	0.249	0.249	0.250	0.248
2.	$1x10^{-1}$	0.232	0.229	0.228	0.231	0.229	0.229	0.232	0.229	0.227
3.	$1x10^{-2}$	0.216	0.217	0.218	0.217	0.216	0.216	0.216	0.218	0.216
4.	$1x10^{-3}$	0.204	0.202	0.204	0.201	0.201	0.203	0.204	0.203	0.204
5.	$1x10^{-4}$	0.193	0.193	0.193	0.196	0.191	0.192	0.193	0.194	0.193
6.	$1x10^{-5}$	0.182	0.182	0.181	0.183	0.182	0.183	0.182	0.183	0.182

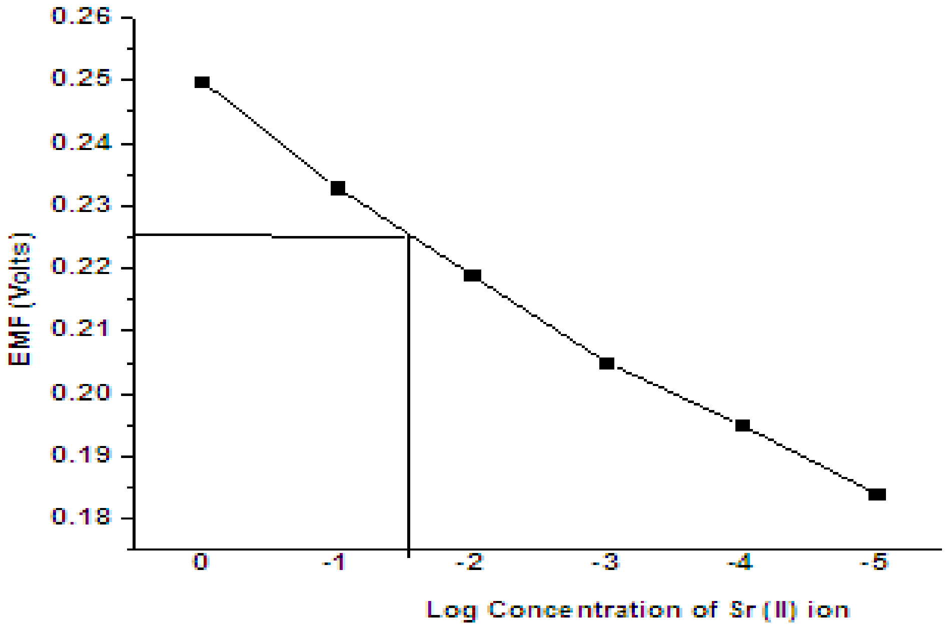
INTERFERENCE STUDY:

Selectivity is one of the most important characteristic of a chemical sensor. The influence of interfering ion and response behaviour of ion selective electrode is usually described in terms of selectivity coefficient. The potential response of the strontium ion selective electrode to different ion have been investigated by determining the selectivity coefficient of the electrode using fixed interference method (FIM) based on semi empirical Nicolskii – Eisenman equation and the concentration of interfering ion was set to 1M. it was found that the potential remains unaffected in the presence of a serious of various cations like Na, Mn, Ca, K, and anions I, Br,NO_3^{2-}, and Cl.

ANALYTICAL APPLICATION

Determination of potassium by direct potentiometric methods

The proposed electrode was found to work well under the laboratory conditions. To asses the applicability of the sensors to real sample an attempt was made to determine strontium ion in vegetable(Beetroot). The recovery of strontium ion in sample analysis was formed to be quantitative with the maximum recovery of 98%.



SUMMARY AND CONCLUSION

- A new simple electrode highly selective and specific for strontium was developed and electrochemically evaluated.
- The electrode exhibited a near Nernstian response in the concentration range of 1M to 1x 10^{-5} M of Sr^{2+}ion.
- The electrode exhibited a response time of 1 minute for Sr^{2+} over wide varieties of other ions.
- The electrode exhibited a near Nernstian response for Sr^{2+} in the acidic pH 4 to 7.
- The potential remaines unchanged in the acetone, ethanol, Dimethyl formamide medium of various concentrations (25%,50%,75%)
- The sensor was successfully applied to determine the Sr-content in vegetable (beetroot) and the electrode was used over a period of one month with good reproducibility.

The Sr ion estimation using this method is very simple. Since this method is done by potentiometry, it is simple, accurate and cost effective. The error involved in this estimation is very less.

- The investigations proved that this study offers an easy, simple and efficient method for determining Sr ion for various concentrations

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