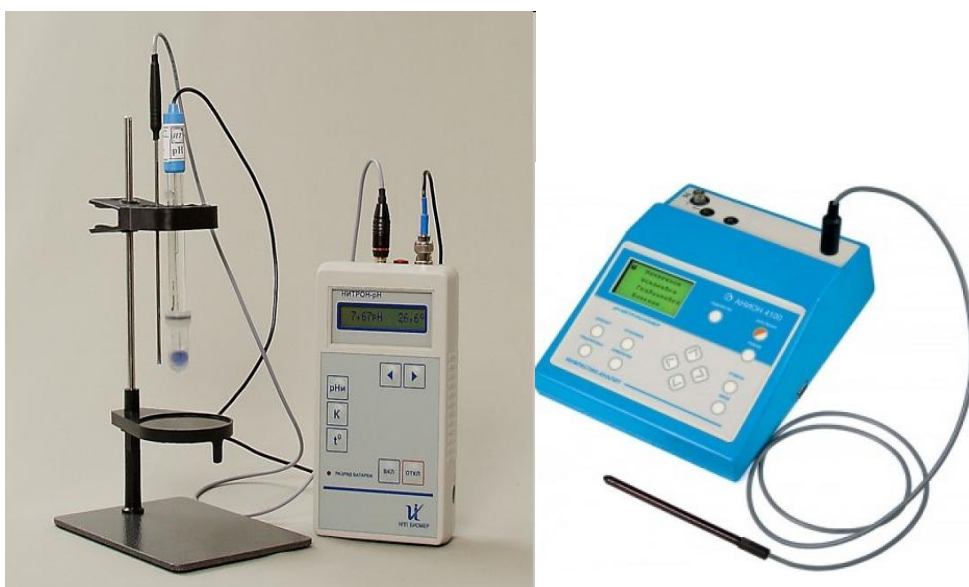


Laboratory work 5

Fluorine Deficiency and Toxicity. Determination of fluoride with ion selective electrodes

Electrochemical methods of analysis are based on the use of ion exchange or electron exchange processes occurring on the surface of the electrode or in the electrode space. Analytical signal is any electrical parameter (potential, current strength, resistance, etc.), functionally related with the composition and concentration of the solution.



Potentiometric analysis is based on measuring the electromotive force of reversible galvanic elements. Galvanic cell consists of the indicator and reference electrodes, immersed in a solution (Electrochemical methods of analysis are based on the use of ion exchange or electron exchange processes occurring on the surface of the electrode or in the electrode space. Analytical signal is any electrical parameter (potential, current strength, resistance, etc.), functionally related with the composition and concentration of the solution. Electrode systems can be divided into different classes depending on the species that determine the electrode potential. The indicator electrodes are either made of noble metals such as Pt or Au, insoluble metal salt/metal electrode (AgCl/Ag) or ion selective electrodes.

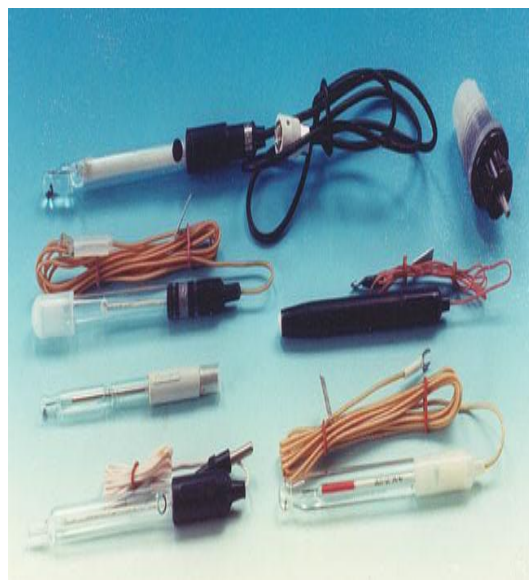
The use of Ion Selective Electrodes in environmental analysis

- Offer several advantages over other methods of analysis. First, the cost of initial setup to make analysis is relatively low.
- Calcium electrode has been used to determine Ca^{2+} ton in milk, sea water, beer, boiler water, sugar, wine etc. As the ton is one of the most important in human physiology, its determination is important.

- Ammonia electrode would help to determine the concentration of NH_3 reducing the complication of Kjeldahl N_2 determination.
- Chloride electrode is used for the analysis of industrial and physiological samples.
- Lead can be measured in blood, urine samples with $\text{PbS}/\text{Ag}_2\text{S}$ electrode.
- These electrodes are found to be easy to use, available in different shapes and sizes and are of nominal cost.

Fluoride are easily and rapidly determined in liquid samples using fluoride selective electrodes. The described method involves only a simple, one-step dilution, and it completely eliminates the need for tedious and complicated methods.

The fluoride selective electrode exhibits linear response to concentrations ranging from 10^{-5} mol/l to saturated and will give analytically useful results down to 10^{-7} mol/l. The electrodes can be used for direct potentiometric determinations, or as end-point indicators for titrations.



Operation Notes:

Electrode preconditioning - The ion selective electrodes (ISE) are ready for use immediately upon removal from the shelf. Soaking prior to use is unnecessary and possibly harmful. The electrodes may be stored dry. The reference electrodes, on the other hand, should be soaked prior to use.

Stirring - The response time of an electrode is generally enhanced by moderate stirring of the sample solution. However, streaming potentials at the reference electrode's liquid junction become apparent when vigorous stirring is employed. The potential observed for stirred samples usually differs slightly from that for unstirred samples.

Electrode handling between samples - The electrode pair (the ISE and the reference electrode) may be rinsed with distilled water and blotted gently to prevent cross contamination between samples. Rinsing the electrode pair with the next solution is preferable, since it avoids the necessity of wiping the electrode, thereby eliminating the possibility of scratching the sensing element surface.

Storage - The halide electrodes can be stored dry without affecting their response. The electrodes should be cleaned before storage to insure that residues of dilute solutions containing interfering ions do not become concentrated by evaporation on the electrode sensing element.

SOLUTIONS

Fluoride standards: Prepare 1l of 200 ppm F^- solution by weighing exactly approximately 0.424 g of NaF into a 1l volumetric flask. Fill to mark with distilled deionized water. Prepare 100 ml solutions containing 10.0, 20.0, 50.0 and 100.0 ppm F^- standards by appropriate dilution of the 200 ppm standard.

Total ionic strength adjustor buffer (TISAB): Place approximately 500 ml of distilled water in a 1-l beaker. Add 57 ml of glacial acetic acid, 58 g of sodium chloride, and 0.30 g of sodium citrate. Stir to dissolve. Place beaker in a water bath (for cooling), insert a calibrated pH electrode and reference electrode into the solution, and slowly add approximately 5 mol/l sodium hydroxide until the pH is between 5.0 and 5.5. Cool to room temperature. Pour into a 1-l volumetric flask and add distilled water to the mark.

PROCEDURE

1. Install a fluoride ISE and a reference electrode to the pH meter. Set the pH meter to read in potential units. For each fluoride standard, pipette 10 ml of TISAB and 50 ml of the fluoride standard into a 100 ml beaker (each standard done separately). Measure the electrode potentials developed in each calibration solution. For the calibration graph, plot electrode potentials in millivolts (linear axis) vs. the log of the fluoride concentration .
3. Record the electrode potentials developed in the unknown solutions and determine the fluoride concentrations from the calibration curve.
4. Determine the concentration of F^- in your unknown (the specific unknown is indicated on the unknown).

Procedure notes:

1. Standards and unknowns should be at the same temperature.
2. Many magnetic stirrers warm the solution being measured and cause a slow drift in electrode reading. A dry cork or asbestos sheet under the beaker minimizes the problem.
3. Perform readings on at least 3 samples of the unknown.

Results