

Determination of Chloride using Potentiometry

1. Purpose

This procedure will determine the concentration of chloride ion with a chloride specific ion electrode using potentiometry.

2. Background

Potentiometry is an electrochemical method in which the potential of an electrochemical cell is measured while little to no current is passed through the sample. In titrimetric methods, this measurement can be used to indicate the end point. When an ion selective electrode is used, the measured potential is related to the ion concentration in solution and a quantitative determination can directly be made. The instrumentation used to perform potentiometry is straightforward and relatively inexpensive, consisting of an indicator electrode, a reference electrode, and a potential measuring device. One of the most common applications of potentiometry is in the measurement of pH.

As indicated by the name, ion-selective electrodes (ISE's) possess a high degree of selectivity. These electrodes are routinely used in clinical laboratories to determine various ion concentrations (such as calcium ion) in blood samples. In this laboratory, the electrode used is specific for chloride ion. Because specific ion electrodes measure activity and not concentration, a large amount of an inert strong electrolyte (e.g. nitrate ion) can be added to fix the ionic strength to a constant value. When the ionic strength is constant, the activity is constant and concentration can be accurately measured. In this laboratory you will fix the ionic strength with an ionic strength adjustment buffer (ISAB). You will be asked to determine the concentration of chloride in an unknown sample at the ppm level.

The chloride ion selective electrode you will use is a crystalline solid-state electrode that contains a membrane, as shown in the diagram on the next page (Figure 1). The membrane consists of a solid salt of silver sulfide / silver chloride ($\text{Ag}_2\text{S} / \text{AgCl}$). The membrane must be insoluble in the analyte solution and contain the analyte ion of interest. The membrane is placed at the end of a solid plastic tube. This membrane is in contact with the analyte solution during the measurement. Inside of the tube is a reference solution, which contains a known and fixed concentration of analyte (Cl^-) solution. The concentration difference between this inner solution and analyte solution causes the migration of charged species across the membrane. This ion exchange process at the surface of the membrane causes a potential to develop. Since the potential of both the reference electrode and the inner reference (immersed in the standard solution) are constant, any change in measured potential is caused only by a change in potential across the membrane and is a function of the analyte chloride ion activity (or concentration).

It is also important to note that Figure 1 depicts a reference electrode external to the ISE (indicator electrode). However, a reference electrode can also be placed inside of the tube containing the ISE. This is called a "combination electrode" and is the type that you will be using in this laboratory experiment.

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Derek Midgley, Kenneth Torrance



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Analysis of the Chloride Ion Concentration in Aqueous Solutions by Potentiometric Titration Lyndsie Selwyn, Canadian Conservation Institute, 2001-01-01 **Determination of Chloride by Precision Null-point Potentiometry** James Dudley Winefordner, 1958 **DETERMINATION OF CHLORIDE BY PRECISION NULL-POINT POTENTIOMETRY**, 1959

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Potentiometric Water Analysis Derek Midgley, Kenneth Torrance, 1978 **Determination of Chloride in TATB. Progress Report, October--December 1975. [Potentiometric Analysis].**, 1975 To determine total chloride content in TATB the sample was burned in a Parr Bomb and the chlorides formed were titrated potentiometrically using silver nitrate The electrode system is a chloride specific ion sensing element and a single junction reference Results on seven test samples agree generally within 10 percent with previous analyses by the Development Division Replicate analyses show precision of the method to be 10 relative percent Inorganic chloride is assayed by dissolving the TATB in concentrated sulfuric acid The HCl which is liberated is swept into dilute base by a nitrogen sweep and analyzed potentiometrically with the chloride specific electrode Results on three test samples agree within 5 percent with Development Division analyses with a 2 relative

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