

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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Ying-Ying Zheng



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National Measurement Laboratory (U.S.), R. A. Velapoldi, 1979 I. Automatic Differential Potentiometric Titrations. Ii. The Determination of Chloride in Titanium Sponge, 1955 **Automation of a Potentiometric Concentration Cell Apparatus and Its Use for the Determination of Chloride in Biological Fluids** Harley Harris Ross, 1957 **A Potentiometric Method for Determination of Sodium Chloride in Food** K. Ishijiki, 1978 **Potentiometry and Potentiometric Titrations** E. P. Serjeant, 1984-06-29 Cells electrodes and basic procedures rudiments of potentiometry classifications of potentiometry and titrimetry classifications and descriptions of electrodes procedures of analytical potentiometry electrodes for potentiometric titrimetry Applications to the determination of solution equilibrium data the determination of ionization constants in aqueous solution acid base equilibria in nonaqueous solvents the determination of stability constants by potentiometric titration Applications to titrimetric analysis the basics of titrimetry acid base titrimetry complexometric titrations oxidation reduction titrations precipitation titrations **Potentiometry and Ion Selective Electrodes (set Price of 34 Books)**
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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 percent precision of analysis

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Table of Contents Determination Of Chloride Using Potentiometry Asdl Home

1. Understanding the eBook Determination Of Chloride Using Potentiometry Asdl Home
 - The Rise of Digital Reading Determination Of Chloride Using Potentiometry Asdl Home
 - Advantages of eBooks Over Traditional Books
2. Identifying Determination Of Chloride Using Potentiometry Asdl Home
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Determination Of Chloride Using Potentiometry Asdl Home
 - User-Friendly Interface
4. Exploring eBook Recommendations from Determination Of Chloride Using Potentiometry Asdl Home
 - Personalized Recommendations
 - Determination Of Chloride Using Potentiometry Asdl Home User Reviews and Ratings
 - Determination Of Chloride Using Potentiometry Asdl Home and Bestseller Lists

5. Accessing Determination Of Chloride Using Potentiometry Asdl Home Free and Paid eBooks
 - Determination Of Chloride Using Potentiometry Asdl Home Public Domain eBooks
 - Determination Of Chloride Using Potentiometry Asdl Home eBook Subscription Services
 - Determination Of Chloride Using Potentiometry Asdl Home Budget-Friendly Options
6. Navigating Determination Of Chloride Using Potentiometry Asdl Home eBook Formats
 - ePub, PDF, MOBI, and More
 - Determination Of Chloride Using Potentiometry Asdl Home Compatibility with Devices
 - Determination Of Chloride Using Potentiometry Asdl Home Enhanced eBook Features
7. Enhancing Your Reading Experience
 - Adjustable Fonts and Text Sizes of Determination Of Chloride Using Potentiometry Asdl Home
 - Highlighting and Note-Taking Determination Of Chloride Using Potentiometry Asdl Home
 - Interactive Elements Determination Of Chloride Using Potentiometry Asdl Home
8. Staying Engaged with Determination Of Chloride Using Potentiometry Asdl Home
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Determination Of Chloride Using Potentiometry Asdl Home
9. Balancing eBooks and Physical Books Determination Of Chloride Using Potentiometry Asdl Home
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Determination Of Chloride Using Potentiometry Asdl Home
10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
11. Cultivating a Reading Routine Determination Of Chloride Using Potentiometry Asdl Home
 - Setting Reading Goals Determination Of Chloride Using Potentiometry Asdl Home
 - Carving Out Dedicated Reading Time
12. Sourcing Reliable Information of Determination Of Chloride Using Potentiometry Asdl Home
 - Fact-Checking eBook Content of Determination Of Chloride Using Potentiometry Asdl Home
 - Distinguishing Credible Sources
13. Promoting Lifelong Learning

- Utilizing eBooks for Skill Development
- Exploring Educational eBooks

14. Embracing eBook Trends

- Integration of Multimedia Elements
- Interactive and Gamified eBooks

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