Potentiometric (ISE) Determination of Fluoride Ion

Theory

Potentiometry is a quick and easy way to determine concentration in a variety of samples over a large range of concentrations. Because potential is related to activity rather than concentration, the ionic strength of all solutions is kept constant, so that the activity will then be proportional to the concentration. In this experiment a total ionic strength adjustment buffer keeps pH as well as ionic strength constant. It also contains a complexing agent. This is because the electrode responds only to fluoride ion. Why would a very acidic pH or metal ions interfere with this experiment? How is concentration related to potential (Think Nernst!)?

Solution Preparation

Prepare a Total Ionic Strength Adjustment Buffer (TISAB) by mixing (with stirring) 57 mL of glacial acetic acid, 58 g NaCl, 4 g of disodium EDTA and 500 mL of distilled water in a 1 L beaker. Cool the contents in a water or ice bath and carefully add 6 M NaOH until a pH of 5.0 to 5.5 is reached. Dilute to 1 L with water and store in a plastic bottle. You do not need one liter of solution so adjust amounts accordingly.

Prepare a 100 ppm F⁻ standard solution from NaF. This solution should have a minimum of four significant figures. The NaF should be dried at 110°C for about two hours before making the solution. WARNING! NaF IS TOXIC. IMMEDIATELY WASH ANY SKIN TOUCHED WITH THIS COMPOUND WITH COPIOUS AMOUNTS OF WATER!!

Standard Preparation

Dilute 25.00 mL of the 100 ppm standard to 500 mL in a volumetric flask to create a 5 ppm solution. Transfer 5, 10, 25 and 50 mL aliquots of the 5 ppm solution to 100 mL volumetric flasks. Add 50 mL of TISAB to each volumetric flask. Dilute to the mark. <u>Sample Preparation</u>

Sample 1: Transfer 50.00 mL portions of drinking water to 100 mL volumetric flask. Dilute to the mark with TISAB.

Sample 2: Accurately weigh 0.2 g of toothpaste into a 250 mL beaker. Add 50 mL of TISAB solution, and boil for 2 minutes with good mixing. Cool and then transfer the suspension quantitatively to a 100 mL volumetric flask, dilute to the mark with deionized water, and mix well.

Potentiometric Measurements

Obtain a fluoride selective electrode and a reference electrode. Connect to a pH meter. Set the pH meter to read mV.

After thorough rinsing and drying with a paper tissue, immerse the electrodes in the most dilute standard. Stir mechanically for 3 minutes; then measure and record the potential. Repeat with remaining standards (low to high concentration) and the samples.

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