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Hardness of Water Determination through Complexometric Titration Visual Abstract Hard water refers to water containing a considerable number of cations that form precipitates with anions like sulfate, carbonate, and soap. The presence of metals such as aluminum, manganese, iron, and zinc in water due to its polarity and hydrogen-bonding properties makes it an excellent solvent. In the Madison area, hard water is a major concern, resulting from mineral deposits formed by ionic reactions between cations in solution, leading to insoluble precipitates. When heated, Ca2+ ions react with bicarbonate ions to form insoluble calcium carbonate, known as scale, which can coat vessels and produce mineral deposits on dishes and in water pipes. High levels of hardness can cause deposits to build up inside pipes, reducing or blocking the flow of gas, and increasing building pressures to dangerous levels. Hardness is measured in ppm CaCO3, despite other metal ions contributing to it, primarily due to tradition and Ca being 95% of the contributor. Soft water is typically defined as containing less than 40 ppm CaCO3, while hard water contains more than 150 ppm CaCO3. To determine the hardness of a water sample using complexometric titration, EDTA (Ethylenediaminetetraacetic acid) and its sodium salts are used as a complexing agent. The procedure is faster and more convenient than gravimetric analysis, but results may be limited to non-specific analysis of metals at fairly high concentrations. The compound's complexation reaction utilizes Y4- in its basic form, necessitating an experiment at a basic pH. This can be challenging since some anions precipitate out of solution at high pHs. You will explore this further in the pre-laboratory exercises. Calmagite indicator solution is used to detect the titration endpoint. It exhibits a wine-red color when bound to calcium and magnesium ions at pH 10 (Figure 2). The procedure involves adding a small amount of Mg-EDTA solution, which sharpens the endpoint by capturing Ca2+ and Mg2+. As EDTA binds metal cations, it competes with calmagite-bound metals. The reaction forming colorless HIn2-(blue) occurs rapidly at the endpoint: In3-(colorless) + H2O ⇄ HIn2-(blue) + OH^- (2). This is detected coloremetrically by the change from wine-red to clear blue. EDTA reactions with metal cations and displacement titration technique. Understanding the chelation reaction and EDTA's interaction with metal cations is crucial for this experiment. The EDTA solution should be prepared well in advance, as it takes time to dissolve. Aliquot preparation should be done before starting the experiment. Data table: | Volume of Aliquot (mL) | Volume of EDTA to titrate CaCO3 Solution (mL) | | --- | --- | | 25.00 | 28.51 | | 25.00 | 22.81 | | 25.00 | 19.96 | Proposed reason for the decrease in EDTA: The slow solubility of EDTA and its hydrophobic properties may cause it to precipitate out of solution over time, affecting the titration results. To dissolve CaCO3, add 60 mL of 1 M NH4OH. Use HCl in moderation to neutralize the acid in step two. Standardize the EDTA solution by mixing it thoroughly before filling the buret. Pipet 25 mL of standard calcium solution into a 250 mL Erlenmeyer flask and add buffer solution, Mg (II) - EDTA reagent, and calmagite indicator. Titrate the sample with EDTA titrant until the last reddish tinge disappears, adding drops at intervals. The endpoint is a crisp blue color with no red or purple hue. Use daylight or fluorescent light to ensure accuracy. To determine the hardness of an unknown sample, quantitatively transfer the sample to a volumetric flask and dilute it with deionized water. Pipet 50 mL of this solution into an Erlenmeyer flask and titrate as before, adjusting the procedure accordingly. Titrate two more 50 mL aliquots and calculate the hardness in ppm (mg/L) of CaCO3. In step five, the sample volume is double that used in step three; how does this change the expected volume of titrant? Fill out the answer sheet completely to ensure accuracy. Challenge questions: Suppose your lab partner misread the instructions and pipetted 10 mL of Mg-EDTA reagent. Must you start over with a new sample? If so, explain the type of error (systematic or random) that will occur. Lab report submission guidelines have been introduced in conjunction with a measurement-based exercise that takes into account potential mistakes and their effects on water hardness readings. To submit the lab report, combine all required documents into one PDF file and upload it to Canvas. This should include completed answer sheets, relevant lab notebook pages with answers to post-lab questions and challenge questions. A grading rubric is available on Canvas for reference. References to Yappert's work (1997) and Harris and Lucy's "Quantitative Chemical Analysis" book (2020 edition) are also noted. Any inconsistencies or errors can be reported through a provided Google form, but please direct specific questions to the instructor or TA as comments cannot elicit responses. A license agreement is in place for ASTM products; by accessing these materials, users acknowledge they have read and agree with its terms. The agreement emphasizes ownership rights, defining individual, single-site, and multi-site licenses, and outlining permitted uses under a limited license. 1. 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