Hard Water Analysis

Problem

How can we determine the total hardness in tap water?

Introduction

If you let tap water boil for long periods of time, it begins to leave a film on the walls of the container. You may have seen this sort of film on beakers and flasks in the lab, or in the pots and pans in your kitchen at home. Most of these whitish deposits are residues of the carbonate and sulfate salts of calcium, usually with small amounts of the sulfates and carbonates of magnesium mixed in as well. The presence of these minerals in water is the condition which we call "hard" water, and the more of them you have, the harder your water is. The tendency of ordinary soaps to form precipitates with the Ca^{2+} and Mg^{2+} in hard water results in a grayish film being left on clothes, and accounts for the widespread use of detergents in today's world.

In this experiment you will explore one type of test which can be run on ordinary tap water to determine its degree of hardness. The analysis involves a technique known as a "titration". The metal ions (Ca^{2+} and Mg^{2+}) in hard water have a particularly strong attraction for a large organic molecule called ethylenediamine tetraacetic acid, or simply EDTA. The metal ions and the EDTA molecules combine to form a large complex ion, known as a "chelate", in which the EDTA surrounds and traps the metal cation. (Chelates are important molecules biologically. Hemoglobin is another example of a chelate; it holds the iron in your blood.)

$$EDTA^{4-} + Ca^{2+} \rightarrow [Ca{\bullet}EDTA]^{2-}$$

In this titration, you will carefully add a solution of EDTA to samples of tap water, stopping when all of the metal ions have been chelated. To help you identify the endpoint, you will add a few drops of an *indicator*--a dye which changes color when all of the metal ions are gone. Since the reaction only works in a moderately alkaline medium, it will be necessary for you to add a solution known as a buffer to your samples; a buffer maintains the pH (acidity-alkalinity) of a system at the desired level. This will ensure that the endpoint is clear and unobscured.

Because there is generally far more calcium than magnesium in water samples, the results are ordinarily reported in parts per million (ppm) of Ca. We will follow that convention here.

Preparation of Solutions:

1. 0.01 M EDTA: Weigh about 4g ethylenediamine tetraacetic acid, EDTA, and about 0.1g of $MgCl_2 \cdot 6H_2O$ into a clean 400 ml beaker. Dissolve the solids in water, transfer the solution to a clean 1 liter container, and dilute to about 1 liter. If the solution is turbid, add a few drops of 0.1 M sodium hydroxide solution until the solution is clear. Mix thoroughly and label the container.

2. calcium carbonate Solution: Weigh accurately 0.4g dry calcium carbonate. Transfer the solid to a 500 ml volumetric flask, using 100 ml distilled water. Add 6 M HCl dropwise until effervescence ceases and the solution is clear. Dilute with water to 500 ml and mix thoroughly.

3. pH 10 Buffer: Dissolve about 6.75g NH_4Cl in 57 ml of concentrated ammonia and dilute to 100 ml.

4. EBT Indicator: Dissolve about 0.5g Eriochrome Black T (EBT) in 100 ml ethanol. This solution will only be good for approximately 6 weeks. Mix thoroughly and label the container.

Procedure for Standardizing EDTA Solution:

1. Set up two burets. Fill one with EDTA solution and the other with calcium carbonate solution.
2. Run 20 ml of the calcium solution into a 125 ml flask. Then add 2.5 ml buffer solution and 3 drops EBT and mix. Be sure to record initial and final readings of your calcium buret.
3. Titrate carefully with the EDTA solution until the wine-red color turns to pure blue. You will need to go slowly near the endpoint. Be sure to record initial and final readings of the EDTA buret.
4. Repeat with two more samples.
5. Calculate the number of moles Ca^{2+} used in the 20 ml sample and calculate the Molarity of the EDTA from an average volume used in the titrations.

Determination of Water Hardness:

1. Set up two burets. Fill one with EDTA solution and the other with a water sample to be analyzed.
2. Run 20 ml of the water sample into a 125 ml flask. (note: the size of the water sample may need to be adjusted.) Then add 1.0 ml buffer solution and 3 drops EBT to the flask and mix. Be sure to record initial and final readings of your water buret.
3. Titrate carefully with the EDTA solution to a color change of wine-red to pure blue. Be sure to record initial and final readings of the EDTA buret.
4. Repeat with two more samples.
5. Calculate the total hardness of the water as parts per million (ppm) of calcium carbonate.

Data:

Mass of $CaCO_3$ in solution = _____g

Moles of Ca^{+2} in solution = _____mole

Volume of solution = _____L

Molarity of Ca^{+2} in solution = _____M

Moles of Ca^{+2} in each 20 ml sample = _____moles

Standardization of EDTA Solution:

Titration:	I	II	III
Initial Buret Reading:	_____ml	_____ml	_____ml
Final Buret Reading:	_____ml	_____ml	_____ml
Volume of EDTA:	_____ml	_____ml	_____ml

Average Volume EDTA: _____ml

Molarity of EDTA: _____M (remember 1:1 mole ratio in reaction)

Determination of Water Hardness:

Titration:	I	II	III
Initial Buret Reading:	_____ml	_____ml	_____ml
Final Buret Reading:	_____ml	_____ml	_____ml
Volume of EDTA:	_____ml	_____ml	_____ml

Average Volume EDTA: _____ml

Average Moles EDTA _____moles

Average Mass Ca^{+2} per sample _____grams (remember 1:1 mole ratio)

Calculate ppm: