MODFICATIONS

(Start on Page 29-8, 4th paragraph)

Estimating the sample size for Part 2a. You will use the conductivity reading to estimate a value of the sample size for the titration in part 2a, the next part of the experiment. The reasoning behind the estimate goes like this: $Ca(HCO_3)$ is used as a proxy for the sum of concentrations of Ca^{2+} and Mg^{2+} because most natural waters contain more Ca^{2+} than Mg^{2+} and HCO_3^- is usually the predominant anion. A 0.0010 M solution of $Ca(HCO_3)$ has a conductivity of 208 microSiemens/cm at 25°C. We will aim to use at least 1.5 mL of EDTA titrant—in order to be able to do several titrations from one reagent filled buret— to titrate the Ca^{2+} + Mg^{2+} in the sample. Assuming our EDTA solution is 0.0100 M, we estimate that a sample the size of 15 mL would be about right:

$$\text{mmol EDTA} = \text{mmol } Ca^{2+}$$

$$1.5 \text{ mL EDTA} \times 0.0100 \frac{mol}{L} \text{ EDTA} = 0.015 \text{ mmol EDTA} = 15 \text{ mL} \times 0.001 \frac{mol\ Ca^{2+}}{L}$$

So we can establish an approximate working relationship between the conductivity— proportional to the $Ca(HCO_3)$ concentration—and the sample size:

$$V_{sample} \cong 15 \text{ mL} \times \frac{208\ microSiemens/cm}{sample\ conductivity\ (microSiemens/cm)} \qquad (11)$$

For convenience, you will round the calculated sample volume to the nearest mL (but you must be sure to measure the sample volume for all titrations to an accuracy of ± 0.10 mL).

The estimated volume is approximate because there may be cations beside Ca^{2+} and Mg^{2+} in the tap water or bottled water samples that also contribute to the conductivity. For example, some bottled waters, particularly mineral waters with a high content of dissolved solids, often contain significant amounts of Na^+ or K^+ that contribute to the conductivity. For such waters, the concentration of $Ca(HCO_3)$ would be smaller than estimated from the conductivity. Then our sample size would have to be made larger—but you wouldn't know this until you make a first trial titration.

2a. *EDTA Titration of Ca^{2+} + Mg^{2+} Ions at pH 10 Using Calmagite Indicator*

Procedure:

Fill a 10-mL microburet with 0.0100 M Na_2H_2EDTA. (Read the solution labels carefully so that you do not mistakenly fill the microburet with 0.010 M $Na_2MgEDTA$ solution.) Then put into a 50-mL Erlenmeyer flask a volume of tap water calculated using Equation (11), measured with an accuracy of ± 0.10 mL (after rounding the calculated value to the nearest mL). Add 15.0 mL of 1.5 M NH_3/0.3 M NH_4Cl buffer. Using a 7-mL graduated plastic transfer pipet, add 1.5 mL of 0.010 M $Na_2MgEDTA$. Finally, add 4 drops of 0.1% calmagite indicator.

NO CHANGES IN THE NEXT 4 PARAGRAPHS

2b. *EDTA Titration of Ca^{2+} at pH 12.5 Using Calcon Indicator*

Overview of procedure:

Calcium is titrated with Na_2H_2EDTA at pH 12.5, where magnesium ion is precipitated as $Mg(OH)_2(s)$. The endpoint is easily detected as a color change from pink to pure blue. To avoid using diethylamine, as called for in the procedure originally described by Hildebrand and Reilley (see the Bibliography), we will obtain a solution pH of 12.5 by addition of NaOH.

Procedure:

Use the same 10-mL microburet filled with 0.0100 M Na_2H_2EDTA that was used in part 2a. Put into a 50-mL Erlenmeyer flask a volume of tap water equal to that used in part 2a, measured to an accuracy of ± 0.10 mL. Add 15 mL of 0.10 M NaOH and stir the solution, then add 5 drops of 0.4% calcon indicator. Immediately titrate with 0.0100 M Na_2H_2EDTA contained in the 10-mL microburet. (The indicator is somewhat unstable at high pH.) The color change at the end point is from pink to pure blue. Repeat until you have completed at least two titrations that agree to within 10%.

NO CHANGES IN THE NEXT 4 PARAGRAPHS

3. Determining Alkalinity (HCO_3^- concentration) by Titration with HCl using Bromocresol Green Indicator

Procedure:

Use the sample volumes that were found satisfactory for the EDTA titrations of tap water and bottled water in part 2a. Using a graduated cylinder or graduated pipet, place a sample of tap water in a 50-mL Erlenmeyer flask. (Record the sample volume, accurate to ± 0.10 mL.) Add 2-3 drops of 0.1% bromocresol green indicator. If a magnetic stirrer is available, place the flask on the stirrer over a sheet of white paper to better see the color change. Otherwise swirl the flask by hand while adding the titrant. Using a 10-mL microburet filled with 0.020 M HCl, titrate the sample from blue to a greenish yellow. (The equivalence point comes at approximately pH 4.6). The ratio *mL 0.020 M HCl/mL water sample* should agree within 10% for the two titrations. If it does, move on to the titration of bottled water; if not, titrate a third sample.

Repeat the entire procedure for at least two samples of bottled water, measuring the sample volume to an accuracy of ± 0.10 mL.