PRACTICAL PROBLEMS

PROBLEM 1 (Practical)

Effects of CO_2 on Solubility

Calcium carbonate is a partially soluble material commonly known as limestone, marble, chalk, or calcite. In the presence of CO_2-rich groundwater, calcium carbonate dissolves to form cavities and caves in limestone formations.

In this experiment you will determine the solubility of calcium carbonate in an aqueuos solution saturated with carbon dioxide and in solution free of carbon dioxide. The $[Ca^{2+}]$ will be determined by complexometric titration with EDTA (ethylenediamine-tetraacetic acid).

a) Procedure for the determination of $[Ca^{2+}]$

1. Calibrate your *pH* meter.
2. Measure and record the *pH* of the sample equilibrated with solid $CaCO_3$ and CO_2 gas.
3. Filter all your sample into a second 250 cm^3 plastic bottle to remove any suspended $CaCO_3$. Quickly cap the bottle when the filtering is complete.
4. Uncap the bottle long enough to measure and record the pH of the filtered solution and recap the bottle until you are ready for the next step.
5. As rapidly as possible, transfer three 25 cm^3 aliquots of this sample to three Erlenmeyer flasks. Recap the sample bottle after removing each 25 cm^3 aliquot.
6. Add 15 drops of 6 M HCl to each of the three flasks with stirring. Any $CaCO_3$ that may have formed should dissolve. Before proceeding with the next step, make sure there is no solid $CaCO_3$ in the Erlenmeyer flasks. If a flask has some solid present, then more stirring is necessary.
7. To one, and only one flask, add 5 cm^3 of a pH 10 NH_3 buffer. Proceed immediately with steps 8 - 10.
8. Add 20 drops of 0.001 M $Mg^{2+}/EDTA^{4-}$ solution to the flask in order for the indicator to function properly.
9. Add 5 drops of Calmagite indicator to the sample.

10. Titrate the sample with an approximately 0.01 M EDTA solution provided (to be standardized as directed below) to a colour change from red to blue. You may wish to make a reference for colour with water, buffer, two drops EDTA and indicator.
11. Quickly repeat steps 7 - 10 for each remaining flask, one at a time.
12. After steps 1 - 11 have been completed to your satisfaction, transfer the remaining filtered, saturated solution from step 3 to a suitable beaker. Heat this solution to 96 – 99 ^{o}C and allow it to remain at that temperature, with magnetic stirring, for 5 minutes. A stirrer setting on 6 is adequate, and an initial high setting of heat will be required. You should see CO_2 being evolved and some $CaCO_3$ may precipitate.
13. At the end of the five minutes heating period, use the beaker tongs to remove the beaker from the hot plate and place it in an ice bath. Allow the solution to cool to room temperature.
14. Measure and record the *pH* of the cooled solution.
15. Filter the solution to remove any suspended $CaCO_3$.
16. Transfer three 25 cm^3 aliquots of this filtered solution to three Erlenmeyer flasks. Add about 25 cm^3 of deionized water to each flask, followed by 15 drops of 6 M HCl.
17. Titrate each sample according to steps 7 - 11.

b) <u>Procedure for standardization of EDTA</u>

1. Weigh approximately 0.35 g of dried primary standard $CaCO_3$, (molar mass 100.09 g mol^{-1}) which will be found in the weighing bottle in the desiccator. Note: $CaCO_3$ is hygroscopic.
2. Add 25 cm^3 of deionized water to the $CaCO_3$, then carefully add 5 cm^3 of 6 M HCl. Quickly cover with a watch glass.
3. When the $CaCO_3$ has dissolved, quantitatively transfer the solution to a 250 cm^3 volumetric flask and dilute to the mark.
4. Transfer three 25 cm^3 aliquots of the standard Ca^{2+} solution to three Erlenmeyer flasks.
5. Proceed to titrate each sample according to steps 7 - 11, following each step in sequence.

Tasks:

1.1 Calculate the solubility of calcium carbonate (expressed in mol dm^{-3}) in a solution saturated with both $CaCO_3$ and CO_2 and in solution free of CO_2.

1.2 List all the ionic species that increase in concentration as a result of the dissolving of $CaCO_3$ in water.

1.3 Given below are brief descriptions of two solutions. Circle the one in which the concentration of CO_3^{2-} would be highest:

a) a solution produced by dissolving $CaCO_3$(s) in CO_2 -saturated water

b) a solution produced by dissolving $CaCO_3$(s) in CO_2 -free water

1.4 Will the solubility of $CaCO_3$ increase or decrease in a solution from which the CO_2 has been removed?

SOLUTION

1.1 The results of the titration (volumes of titrants, mass of $CaCO_3$) and calculation of the concentration of the standardized EDTA solution were required to be written on the Answer Sheet.

Moreover, it was necessary to measure and record *pH* values of the sample at steps 2, 4, and 14.

It was expected to show the calculation of the solubility of $CaCO_3$ in its saturated solutions containing CO_2 and in those without CO_2.

The other tasks to be solved:

1.2 The ionic species mentioned above are as follows: Ca^{2+}, HCO_3^-, OH^-.

1.3 Correct answer: (b) A solution produced by dissolving $CaCO_3$(s) in CO_2-free water.

1.4 The solubility of $CaCO_3$ will decrease.