8. Analysis of a Mixture of Carbonate and Bicarbonate

Green Profile
See Section 0

This procedure involves two titrations. First, total alkalinity (= $[HCO_3^-]$ + $2[CO_3^{2-}]$) is measured by titrating the mixture with standard HCl to a bromocresol green end point:

$$HCO_3^- + H^+ \rightarrow H_2CO_3$$
$$CO_3^{2-} + 2H^+ \rightarrow H_2CO_3$$

A separate aliquot of unknown is treated with excess standard NaOH to convert HCO_3^- to CO_3^{2-}:

$$HCO_3^- + OH^- \rightarrow CO_3^{2-} + H_2O$$

Then all the carbonate is precipitated with $BaCl_2$:

$$Ba^{2+} + CO_3^{2-} \rightarrow BaCO_3(s)$$

The excess NaOH is immediately titrated (in a back titration) with standard HCl to determine how much HCO_3^- was present. From the total alkalinity and bicarbonate concentration, you can calculate the original carbonate concentration.

Reagents

Standard 0.1 M NaOH and standard 0.1 M HCl: From Experiment 6.

CO_2-free water: Boil 500 mL of distilled water to expel CO_2 and pour the water into a 500-mL plastic bottle. Screw the cap on tightly and allow the water to cool to room temperature. Keep tightly capped when not in use.

10 wt% aqueous $BaCl_2$: 35 mL/student.

Bromocresol green and phenolphthalein indicators: See Experiment 7 for recipes.

Unknowns: Solid unknowns (2.5 g/student) can be prepared from reagent-grade sodium carbonate or potassium carbonate and bicarbonate. Unknowns should be stored in a desiccator and should not be heated. Heating at 50°–100°C converts $NaHCO_3$ to Na_2CO_3.

Procedure

1. Accurately weigh 2.0–2.5 g of unknown into a 250-mL volumetric flask by weighing the sample in a capped weighing bottle, delivering some to a funnel in the volumetric flask (do not use a narrow stem funnel to deliver the powder), and reweighing the bottle. Continue this process until the desired mass of reagent has been transferred to the funnel. Rinse the funnel repeatedly with small portions of CO_2-free water to dissolve the sample. Remove the funnel, fill volumetric ~ half full, and mix well. Once unknown has dissolved, dilute to the mark, and mix again. **Do not heat to expedite dissolution**.
2. *Total alkalinity:* Pipet a 25.00-mL aliquot of unknown solution into a 250-mL Erlenmeyer flask and titrate with standard 0.1 M HCl, using bromocresol green indicator, 3 drops, as in Experiment 6 for standardizing HCl. Repeat this procedure with two more 25.00-mL aliquots to obtain 3 good trials.
3. *Bicarbonate content:* Pipet 25.00 mL of unknown and 50.00 mL of standard 0.1 M NaOH into a 250-mL Erlenmeyr flask. Swirl and add 10 mL of 10 wt% $BaCl_2$, using a graduated cylinder. Swirl again to precipitate $BaCO_3$, add 2 drops of phenolphthalein indicator, and immediately titrate with standard 0.1 M HCl. Repeat this procedure with two more 25.00-mL samples of unknown to obtain 3 good trials. Note that you are adding excess base and that you are consequently performing a back titration.
4. From the results of step 2, calculate the total alkalinity and its standard deviation. From the results of step 3, calculate the bicarbonate concentration and its standard deviation. Using the standard deviations as estimates of uncertainty, calculate the concentration (and uncertainty) of carbonate in the sample. Express the composition of the solid unknown in a form such as 63.4 (±0.5) wt% K_2CO_3 and 36.6 (±0.2) wt% $NaHCO_3$.