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Method

Sample Preparation

For samples that are already in solution, such as freshwater, seawater and milk, no further preparation is needed.

For solid samples such as eggshells and limestone, the samples must first be dissolved in acid. Accurately weigh about 0.5 g of the solid into a small beaker or conical flask, add about 20 mL dilute hydrochloric acid and allow the solid to completely dissolve (this may take several minutes). Neutralise the unreacted acid with dilute sodium hydroxide solution until the pH of the solution is almost 7 (according to pH indicator paper). For eggshells, the inner membrane will remain undissolved and may be carefully removed from the solution. Transfer the solution to a 100 mL volumetric flask and make up to the mark with distilled water.

Standard EDTA Solution

1. Pipette a 10 mL sample of the EDTA solution into a conical flask.
2. Add 10 mL of ammonia buffer solution and 1 mL of Eriochrome Black T indicator solution.
3. Titrate the EDTA with the magnesium chloride solution until the endpoint is reached – a permanent colour change from blue to pink.
4. Having determined the average titre of the magnesium chloride solution, determine the number of moles used.
5. Given the Mg^{2+} : EDTA ratio of 1 : 1, calculate the concentration of your EDTA solution.

Figure 1 Colour changes for magnesium chloride back-titration in clear solution using Eriochrome Black T indicator. Left flask: blue colour well before endpoint (all Ca^{2+}/Mg^{2+} ions complexed by excess EDTA, all indicator molecules uncomplexed). Centre flask: last trace of blue/purple colour just before endpoint (excess EDTA almost totally complexed by added Mg^{2+}). Right flask: pink/red colour at endpoint (all EDTA complexed by added Mg^{2+} , indicator also complexed).

Titration of Sample

1. Pipette 10 mL of the sample solution into a conical flask.
2. Add 20 mL of 0.05 mol L⁻¹ EDTA solution.
3. Add 10 mL of ammonia buffer, 50 mL of distilled water and 1 mL of Eriochrome Black T indicator solution.
4. Titrate the sample with the standard 0.025 mol L⁻¹ magnesium chloride solution until a permanent pink colour appears.

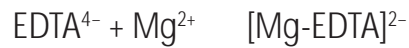
Titration of Standard EDTA Solution

1. Add a 100 mL of the sample solution into a 250 mL conical flask.
2. Prepare a 0.005 mol L⁻¹ EDTA solution by diluting the 0.05 mol L⁻¹ EDTA solution by a factor of 1/10. Add 20 mL of this diluted EDTA to the sample solution.
3. Add 10 mL of the ammonia buffer and 1 mL of Eriochrome Black T indicator solution.
4. Prepare a 0.0025 mol L⁻¹ magnesium chloride solution by diluting the 0.025 mol L⁻¹ magnesium chloride solution by a factor of 1/10.
5. Titrate the sample solution with this 0.0025 mol L⁻¹ magnesium chloride solution until a permanent pink colour appears. Repeat the titration with further samples until concordant results (titres agreeing within 0.1 mL) are obtained.

Figure 2 Same colour changes as in Figure 1, but for a sample containing calcium ions. The colour change is from blue to pink/red.

Result Calculations

1. Calculate the total moles of EDTA added to the sample solution.
2. Calculate the moles of the magnesium chloride solution used in the back titration from your concordant results. From the equation of the titration below, the moles of Mg^{2+} will be equivalent to the moles of excess EDTA.



3. Given the ratio of $\text{Ca}^{2+} + \text{Mg}^{2+} : \text{EDTA} = 1 : 1$, calculate the moles of Ca^{2+} and Mg^{2+} that must have been complexed with EDTA by subtracting the excess EDTA from the total moles of EDTA added to the sample.

This result is the moles of Ca^{2+} and Mg^{2+} in the sample solution.

Additional Notes

1. Ethylenediamine titration.