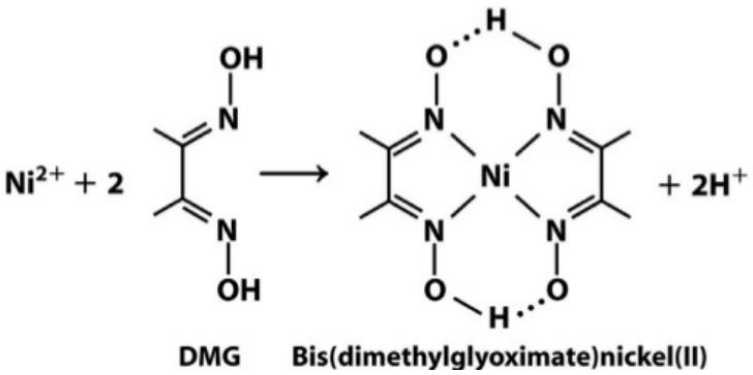
The gravimetric determination of nickel

Introduction

The nickel is precipitated as nickel dimethylglyoximate by adding an alcoholic solution of dimethylglyoxime $C_4H_6(NOH)_2$. When the pH is buffered in the range of 5 to 9, the quantitative formation of the red chelate occurs in a solution according to the reaction:

$$NiSO_4 + 2C_4H_8O_2N_2 + 2NH_4OH \rightarrow (C_4H_7O_2N_2)_2Ni\downarrow + (NH_4)_2SO_4 + 2H_2O$$



DMG Bis(dimethylglyoximate)nickel(II)

The reaction is performed in a solution buffered by an ammonia buffer to avoid falling in pH value below 5. If the pH becomes too low the equilibrium of the above reaction favours the formation of the nickel(II) ion, causing the dissolution of DMG-Ni complex.

A slight excess of the reagent has no action on the precipitate, however, a large excess should be avoided because of the possible precipitation of the reagent itself. It is also important to know that DMG-Ni complex is slightly soluble in alcoholic solutions. Adding an appropriate amount of chelating agent will minimize the errors from this source.

A slow increase in the concentration of ammonia in the solution causes a gradual increase in the pH and results in the precipitation of the complex. As a result, the formation of a denser precipitate is observed. Once the filtrate has been collected and dried, the nickel content of the solution is calculated stoichiometrically from the weight of the precipitate.

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Procedure

A. Preparation of glass crucible

1) Clean and dry glass crucible. Make sure that crucible is marked properly.
2) Weigh a crucible to the nearest 0.1 mg using analytical balance. This will give you a baseline value to determine if heating is removing any fingerprint oils and/or adsorbed water from the crucible. Note the weight of crucible and put it back in the desiccator.
3) Dry crucible in the oven at ~110 °C. After 2 hours of heating, store the glass crucible in a desiccator for cooling to room temperature and weigh to the nearest 0.1 mg.
4) Repeat Steps 1-3 to bring crucible to constant mass (successive weighings agree within ± 0.0004 g).

ATTENTION: Always store crucibles in a desiccator when they are not in use. Wear gloves when working with dried crucibles.

B. Preparation and precipitation of nickel

Take your sample solution placed in a beaker and add 50.0 mL of distilled water using a graduated cylinder. Then, add 2 mL of HCl at a concentration of 6 M (1:1) and heat the solution to 60 °C to 80 °C using a burner in the hood (do not boil!). Remove the beaker from the burner and add 10.0 mL of 1.0% (w/v) alcoholic solution of dimethylglyoxime. Then, with vigorous stirring, introduce a sufficient amount of NH_3. Add NH_3 until a faint smell of NH_3 can be detected in the vapour over the solution. The presence or absence of excess NH_3 is readily established by odour. For this purpose, use a waving motion with your hand to drag the vapour towards your nose. After this treatment, a red precipitate will be formed. Leave a stirring rod in a beaker and carefully cover it with a watch glass. Then, heat the solution to 60 °C for 30 min using a burner in the hood. Later, cool the solution. Do not remove a stirring rod from the beaker. Filter using glass crucible that has been previously brought to constant mass. Wash the precipitate with a distilled water. After filtering, place the crucible in a drying oven and dry at ~110 °C for 1 hour and then, put it in the desiccator. Cool and weigh. Repeat heating, cooling and weighing steps until the mass of a crucible will be constant (successive weighing agree within ± 0.0004 g). Do not heat the precipitate to

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temperatures over 130 °C (Ni complex may decompose). Once a constant mass is reached and noted, rinse the crucible with water and then put it into dilute HCl solution for the cleaning process. Rinse the crucible thoroughly with distilled water and return it to the technical staff.

C. Calculations

Calculate the amount of nickel (g) in your analytical sample using a gravimetric factor (0.2032).

Literature

• **Analytical Chemistry** (7th ed.); G.D. Christian, P.K. (Sandy) Dasgupta, K. A. Schug; John Wiley & Sons, Inc., 2014: Chapter 2: Basic Tools and Operations of Analytical Chemistry; Chapter 10: Gravimetric Analysis and Precipitation Equilibria (10.1-10.4).

• **Modern Analytical Chemistry** (1st ed.); D. Harvey; The McGraw-Hill Companies, 2000: Chapter 2: Basic Tools of Analytical Chemistry (2D); Chapter 8: Gravimetric Methods of Analysis (8A, 8B).

• **Quantitative Chemical Analysis** (7th ed.); D.C. Harris; W.H. Freeman and Company, NY, 2007: 2 Tools of the Trade (2-1÷2-9); 27 Gravimetric and Combustion Analysis (27-1÷27-3).

• **Lecture**: Section 4, part 2; Section 13.

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