The gravimetric determination of iron (Fe^{3+})

Introduction

A sample containing various oxidation states of iron can be analyzed by oxidation to a single oxidation state (Fe^{3+}), precipitation of the hydrated hydroxide from basic solution, and finally, ignition to produce Fe_2O_3 according to the reactions:

$$Fe^{2+}_{(aq)} \xrightarrow{\text{Oxidation with hot } HNO_{3(aq)}} Fe^{3+}_{(aq)}$$

$$Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3\downarrow$$

hydrous ferric oxide
($Fe_2O_3 \cdot nH_2O$)

$$2Fe(OH)_3\downarrow \xrightarrow{\Delta,\ 900°C} Fe_2O_3 + 3H_2O$$

hematite
(red-brown/steel grey)

Procedure

A. Preparation of porcelain crucible

1) Clean and dry porcelain crucible. Make sure that crucible doesn't have any cracks and is marked properly.
2) Weigh a crucible to the nearest 0.1 mg using an 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 store it in the desiccator.
3) Dry crucible in the laboratory furnace. After 30 min of heating, store the porcelain 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).

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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 iron

Take your sample solution placed in a beaker and add 150.0 mL of distilled water using a graduated cylinder. Then, add 5 mL of concentrated HNO_3 and boil for a few minutes using a burner in a fume hood to ensure that all iron is oxidized to Fe(III) (be careful not to boil away all the water!). Remove the beaker from the burner and add, with vigorous stirring, few drops of ammonia (1:1). 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. In this purpose, use a waving motion with your hand to drag the vapour towards your nose. During this treatment, a red/brown precipitate will be formed. Leave a stirring rod in a beaker and carefully cover it with a watch glass. Leave precipitant for few minutes and allow it to settle. Then, set up a filter rack and fit funnel with filter paper into it. Wet the filter paper so that it sticks to the funnel. Do not stir the solution and try to pour the solution without precipitant into the funnel (decantation). Do not pour liquid higher than 1 cm from the top of the funnel. At the end of filtration, stir the residue trying to transfer as much suspended solid as possible with each pouring. The less solid you leave in the beaker the better, but don't spill, or splatter any of the precipitate out of the funnel. Transfer any remaining solid from the beaker to the filter paper with the aid of distilled water and a baguette equipped with a rubber. Wash precipitate with three portions of distilled water. Carefully lift the paper filter out of the funnel containing precipitate using a baguette to free it from the funnel. Fold it and transfer to the crucible. Fold up the filter as much as possible, but don't lose any of the solid. Set up the crucible over the burner. Light the burner and put it under the crucible. Heat with the flame so hot, the filter paper should quickly catch fire. This is important, as it minimizes smoke and also prevents the formation of graphite due to incomplete combustion of the filter paper. After combustion of the filter paper, put the crucible into the desiccator and transfer it to the laboratory furnace for 30 min to complete ignition of the iron oxide. Depending on the conditions under which the ignition takes place, the resulting hematite may be dark red, steel grey, or a combination of these. After this time, put it desiccator. Cool and weigh. Repeat heating, cooling and weighing steps until the mass of a crucible is constant (successive weighing agree within ± 0.0004 g).

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Once a constant mass is reached and noted, rinse the crucible with distilled water and return it to the technical staff.

C. Calculations

Calculate the amount of iron (g) in your analytical sample using a gravimetric factor (0.6994).

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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