

NOTE

Photometric Determination of Fe(III) After Adsorption of its 1-Hydroxy-1-(*p*-Chlorophenyl)-3-Phenyl-2-Thiourea Complex on Microcrystalline Naphthalene

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The photometric determination of iron(III) with 1-hydroxy-1-(*p*-chlorophenyl)-3-phenyl-2-thiourea is described. A stable water insoluble complex formed with iron and 1-hydroxy-1-(*p*-chlorophenyl)-3-phenyl-2-thiourea is easily adsorbed from aqueous solution on micro-crystalline naphthalene. The solid mass is separated from aqueous solution, dried and dissolved in dimethyl-formamide. The absorbance of the solution is measured at 410 nm. Beer's law is obeyed in the concentration range of 10-170 μg of iron(III) in 10 mL of DMF. The molar absorptivity was found to be $2.11 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$ at 410 nm and the sensitivity being $1.73 \times 10^{-2} \mu\text{g cm}^{-2}$ of Fe(III) for the absorbance of 0.001.

Iron is the second most abundant metal in the earth's crust. Because of excellent mechanical properties of iron and steel, they are our most important engineering materials and are extensively used for structural purposes, bridges, ships, railroads, tools and machineries. The photometric method for the determination of transition and inner transition metals has extensively been employed since last 50 years. Thiourea and its substituted derivatives are of immense importance in the photometric determination of many of them¹⁻⁵. Various photometric reagents have been reported for the determination of iron(III)⁶⁻¹⁰.

The photometric reagent employed for iron(III) is 1-hydroxy-1-(*p*-chlorophenyl)-3-phenyl-2-thiourea which is prepared¹¹ in two stages. In the first stage *N*-(*p*-chlorophenyl) hydroxylamine is formed by reduction of *p*-chloronitrobenzene in neutral medium. This *N*-(*p*-chlorophenyl) hydroxylamine is then reacted with equimolar quantities of phenylisothiocyanate in petroleum ether (b.p. 60-80°C) to form 1-hydroxy-1-(*p*-chlorophenyl)-3-phenyl-2-thiourea.

A 0.1% solution of this reagent was prepared. A stock solution of Fe(III) (1000 ppm) prepared by dissolving required amount of ferric chloride in distilled water, was diluted to obtain a standard solution of iron(III) of 15 ppm.

Buffer solutions of varying pH values were prepared by mixing 1 M acetic acid and 1 M ammonium acetate solution for pH 3-6 and 1 M aqueous ammonia and 1 M ammonium acetate for pH 8-11.

A 20% naphthalene solution was prepared by dissolving 20 g of microcrystalline naphthalene in 100 mL of acetone.

All chemicals used were of analytical reagent grade.

An aliquot of standard solution of iron(III) containing 135 μg of iron was taken in a clean, dry and tightly stoppered Erlenmeyer flask. 3.0 mL of 0.1% solution of 1-hydroxy-1-(*p*-chlorophenyl)-3-phenyl-2-thiourea and 4.0 mL buffer solution of pH 4.0, were added to it. After having been mixed well, the solution was kept standing for about 15 minutes to digest the complex at 50°C. After digestion, 2.0 mL of 20% naphthalene solution was added and it was shaken vigorously for 3 minutes. The complex adsorbed on microcrystalline naphthalene was separated from aqueous solution through a filter paper (Whatman 42), washed with water and dried. The adsorbed complex was dissolved in dimethylformamide and diluted to 10 mL. Absorbance of the solution was then measured.

The absorbance of the solution of the adsorbed complex in DMF was measured at various wavelengths in the range 360 to 650 nm. The absorbance spectra has a single peak at 410 nm. Therefore, 410 nm was chosen as the most suitable wavelength. By studying the effect of different factors it was found that optimum conditions for all measurements were 3.0 mL of 0.1% reagent solution, buffer solution of pH 4.0, 2.0 mL of naphthalene solution, 180 seconds shaking time and 15 minutes digestion time. Effect of diverse metal ions revealed that Ni(II) and Ag(I) interfere if present in large concentrations.

The precision of the proposed method was determined by taking the absorbance of ten sample solutions of iron(III) complex obtained by the given procedure from a series of iron(III) solutions containing 135 μg iron. The standard deviation was calculated as 0.035%.

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