Spectrophotometric determination of iron in medicine tablets

1 Scope

This task includes spectrophotometric determination of the amount of iron in the iron tablets "Hematopan 100", in which the declared amount of iron is 100 mg per tablet.

2 Principle

An iron tablet is dissolved in an acid, iron is reduced to Fe^{2+} with hydroquinone and complexed with o-phenanthroline to form an intensely colored complex: $Fe(C_{12}H_8N_2)_3^{2+}$.
Ortho-phenanthroline is a weak base; in acid solution the principal species is the phenanthrolinium ion: PhH^+. The complex formation can thus be described by the equation:

$$Fe^{2+}(aq) + 3\ PhH^+(aq) == Fe(Ph)_3^{2+} + 3\ H^+(aq)$$

Quantitative formation of the complex is observed in the pH region 2 to 9. Usually the pH of about 3,5 is recommended to prevent precipitation of various iron salts. Careful control of pH is not required. When the complex is formed, the colour of the solution is stable for a long time.

To determine the mass of iron in the sample, a calibration curve with three points will be made. Concentrations of calibration standard solutions should be in the limits ±10 % of the mass concentration in the sample solution which is prepared from the tablet.
For the preparation of standard solutions, a stock solution of amonium iron(II) sulfate will be used.
Suitable measuring wavelength will be selected on the basis of the absorption spectrum of the iron(II)-phenanthroline complex provided. The concentration of iron in the sample will be determined by measuring absorbance of the sample solution and use of the calibration curve.

3 Equipment

Visible spectrophotometer**.**
Laboratory glassware

beaker	100 mL,	2 piece
watch glass (ϕ = 6 cm)		2 piece
graduated cylinder	50 mL,	1 piece
volumetric flask	250 mL,	2 piece
volumetric flask	100 mL,	8 pieces
transfer pipette	10 mL,	1 piece,
transfer pipette	5 mL,	1 piece
dispensette		3 pieces
electronic burette		1 piece
funnel (ϕ = 6 cm)		1 piece
wash bottle,		1 piece

- fume hood
- electric hot plate
- ring stand with two rings
- filter paper
- pipette filler

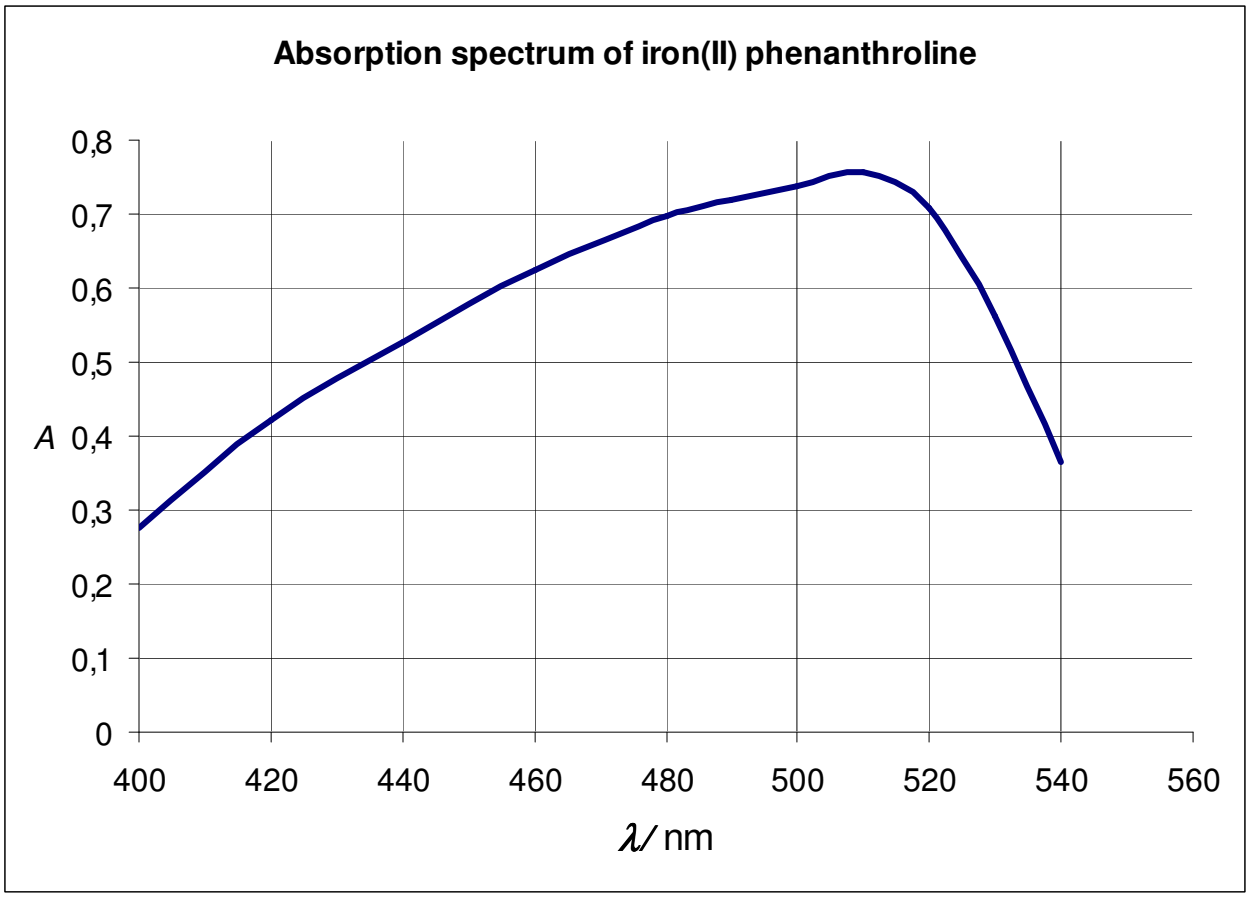
3.4 Materials and their safety codes

Name and formula	. CAS No.	R/S code
Hydroquinone $C_6H_6O_2$	123-31-9	R: 22-40-41-43 -50 S: 26-36/37/39-61
Trisodium citrate Dihydrate $C_6H_5Na_3O_7 \cdot 2H_2O$	6132-04-3	S: 22-24/25
Orto-phenanthroline monohydrate $C_{12}H_8N_2.H_2O$	5144-89-8	S: 22-24/25
Ammonium iron(II) sulfate hexahydrate $Fe(NH_4)_2(SO_4)_2 \cdot 6\ H_2O$	778-85-9	R: 36/37-38 S:26-36
Hydrochloric acid (24 %) 6 mol L^{-1}	7647-01-0	R: 34-37 S:26-45
Sulfuric acid $w(H_2SO_4) =$ 91 %	7664-93-9	R: 35 S: 26-30-45
Ethanol C_2H_5OH w = 96%	64 -17 -5	R 11 S 7-16

3.4.1 *Reagents solutions*

All reagent solution are ready for use.

SOLUTION	LOCATION
Water solution of hydroquinone γ = 10 g L^{-1} (in an amber bottle).	On the separately table.
Water solution of trisodium citrate γ = 25 g L^{-1}.	On the separately table
Solution of o-phenanthroline γ = 2,5 g L^{-1}	On the separately table.
Hydrochloric acid c(HCl) = 6 mol L^{-1}	In fume hood
Sulfuric acid solution: 1mL conc. H_2SO_4 in 1 L solution	On the separately table.
Stock solution of Fe(II) ions γ(Fe) = 40 mg L^{-1}	On the separately table.



4 Procedure

4.1 Preparation of the sample

Place the iron tablet in a 100 mL beaker and heat it gently with 25 mL hydrochloric acid (c= 6 mol L^{-1}) for 15 min, with glass beads added to the beaker. This step should be carried out in a fume hood.
Filter the warm solution directly into a 250 mL volumetric flask containing 100 mL of water. Wash the filter paper into the flask and cool to room temperature. Dilute the prepared sample to the mark (solution A).

Pipet 10,00 mL of the solution A, transfer it into a 100 mL volumetric flask and dilute it to the mark (solution B).

5,00 mL of the resulting solution B transfer into another 100 mL volumetric flask. Add the reagents for developing of the color. The reagents are:
- 3,8 mL of trisodium citrate solution
- 2 mL solution of hydoquinone,
- 3 mL solution of o-phenanthroline.

Upon the addition of reagents, colour develops. Dilute the content of volumetric flask to the mark (solution C) and measure the absorbance at wavelength, which you have selected on the basis of the absorption spectrum.
Measurement should be done in a presence of supervisor.

4.2 Preparation of standard

The mass concentration of iron in the stock solution is $\gamma(Fe) = 40$ mg L^{-1}.
In three 100-mL volumetric flasks, prepare three standard solutions, which concentrations are in the range ± 10 % of the mass concentration in the sample solution C (which is prepared from the tablet).

The exact volume of stock solution is delivered by electronic burette.
Place the calculated volume of the stock solution into the volumetric flask and add the reagents for developing the colour as follows:
- 0,25 mL of trisodium citrate solution per each millilitre of stock solution,
- 2 mL solution of hydroquionone,
- 3 mL of o-phenantroline.

Dilute the solution to the mark and measure the absorbance at the selected wavelength and in the presence of the supervisor.

4.3 Blank sample

In a 100 mL volumetric flask place: 5 mL of sulfuric acid solution, 1 mL of citrate solution, 2 mL solution of hydroquionone and 3 mL of o-phenantroline. Then dilute the solution to the mark.
This is a reference solution for spectrophotometric measurements.

4.4 Data handling

Draw the calibration curve and read the concentration of iron from the graph. Calculate the mass of iron in the tablet (in mg).