

UV-VIS SPECTROPHOTOMETRY II (SPF II)

THE PURPOSE OF THE EXERCISE

Determination of iron (II) ions as a complex with o-phenanthroline using the standard addition technique.

APPARATUS AND REAGENTS

Spekol 10 (VIS) spectrophotometer, 50 mL volumetric flasks, pipettes (of 1, 2, 5 and 10 mL), 1 cm glass cuvettes.

Fe^{3+} standard solution (0.1 mg/mL), 0.25% 1.10 - phenanthroline solution, 10% hydroxylamine hydrochloride solution, 10% sodium citrate solution

PROCEDURE

1. Based on the absorption spectrum of the Fe^{2+} complex with o-phenanthroline (figure 1), select the analytical wavelength (λ).

2. Prepare solution **no I** and blank sample. After 5 min measure the absorbance of this solution (three repetitions) against the blank sample at the selected wavelength.

Solution I \Rightarrow introduce 10 mL of the sample, 2 mL of a 10% hydroxylamine solution, 5 mL of a 10% sodium citrate solution and 5 mL of a 0.25% o-phenanthroline solution to measuring flask (50 mL). Make up to the mark with distilled water and mix thoroughly.

3. Based on calibration curve attached (figure 2) calculate the Fe^{3+} concentration in Solution I. Prepare two fortified samples (solution II and III) in 50 mL flasks.

Solution II \Rightarrow 10 mL of the sample, **0.25 mL of Fe^{3+}** standard, 2 mL of a 10% hydroxylamine solution, 5 mL of a 10% sodium citrate solution and 5 mL of a 0.25% o-phenanthroline solution make up to the mark with distilled water and mix thoroughly.

Solution III \Rightarrow prepare in the same way as solution II but add **0.50 mL of Fe^{3+}** standard solution

Blank sample \Rightarrow 2 mL 10% hydroxylamine solution, 5 mL 10% sodium citrate solution and 5 mL 0.25% o-phenanthroline solution. Make up to the mark with distilled water and mix thoroughly.

4. After 5 min, measure the absorbance of solutions II and III (three repetitions) against the blank sample at the selected wavelength.

PROCESSING THE RESULTS

1. Plot the standard addition calibration curve.

2. Calculate iron concentration (mg/L) in the sample solution based on the calibration curve.

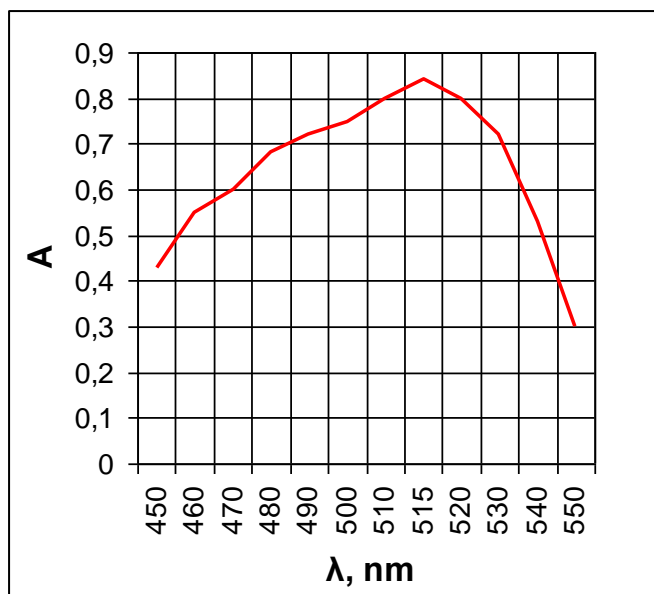


Diagram 1. Absorption spectrum of the Fe^{2+} complex with o-phenanthroline

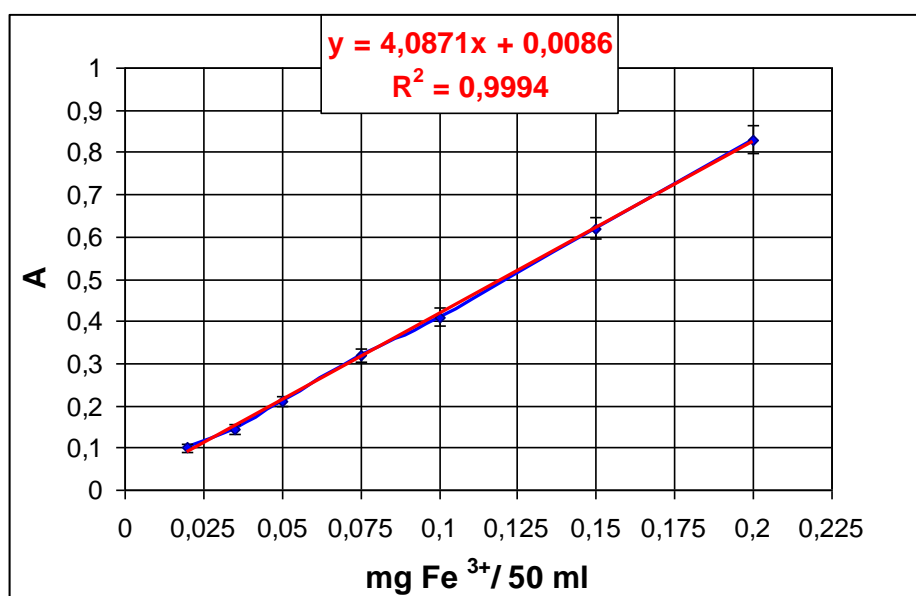


Figure 2. Calibration curve of the Fe^{2+} complex with o-phenanthroline

LITERATURE

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2. D. Harvey, *Modern Analytical Chemistry*
3. Douglas A. Skoog, Donald M. West, F. James Holler, Stanley R. Crouch, *Fundamentals of Analytical Chemistry*
4. Douglas A. Skoog, F. James Holler, Stanley R. Crouch, *Principles of Instrumental Analysis*
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