

---

## 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 method.

### APPARATUS AND REAGENTS

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

Fe<sup>3+</sup> 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<sup>2+</sup> complex with o-phenanthroline (figure 1), select the analytical wavelength ( $\lambda$ ).

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<sup>3+</sup> 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<sup>3+</sup>** 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<sup>3+</sup>** standard solution

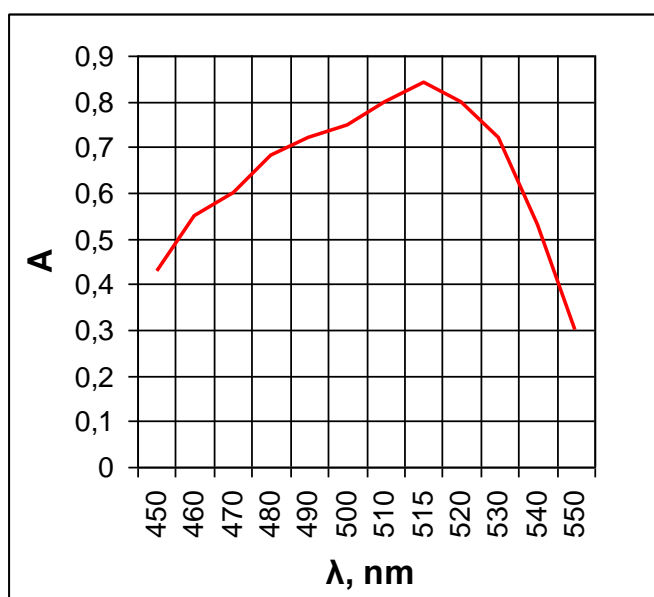
**Blank sample**  $\Rightarrow$  2 mL 10% hydroxylamine solution, 5 mL 10% sodium citrate solution and 5 mL 0.25% o-phenanthroline solution. Make up to the mark with distilled water and mix thoroughly.

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

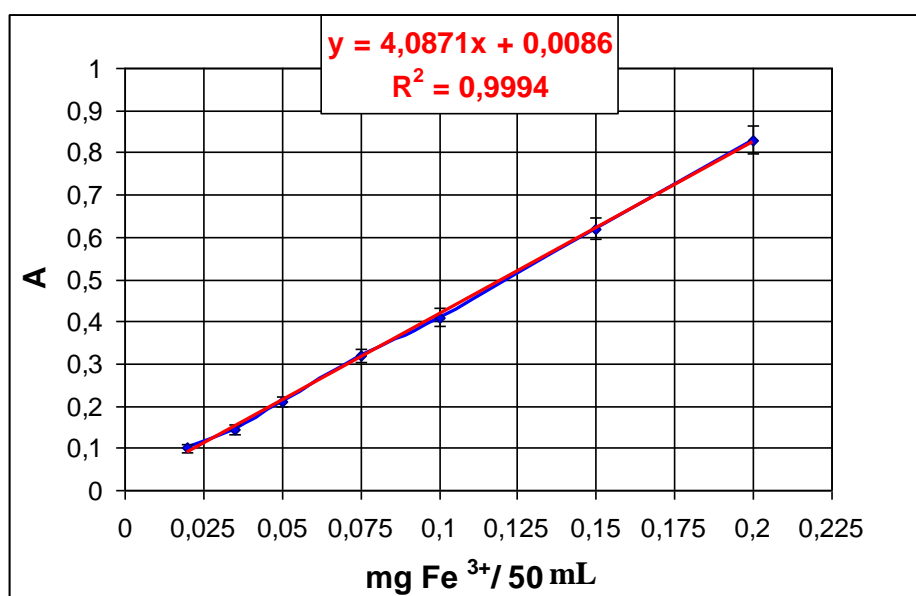
### PROCESSING THE RESULTS

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  $\text{Fe}^{2+}$  complex with o-phenanthroline



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

## LITERATURE

1. D. Kealey, P. J. Haines, *Analytical Chemistry*
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*
5. B. Sivasankar, *Instrumental Methods of Analysis*