CB101 : Experiment 7 : UV-visible spectroscopy

I- INTRODUCTION

1) What is UV-visible spectrometry?

Electronic transition can occur between orbitals with different energies, making possible the absorption of light, sometimes at visible wavelenghts. The graphical representation of the amount of light absorbed by matter (absorbance) as a function of the wavelenght (λ) is called spectrum. In visible range, it is measured thanks to a UV-visible spectrometer. (Note: $A=\log(I/I_0)$, so it is unitless.)

2) Beer-Lambert law for a single substance solution:

For a solution containing a single colored substance i, Beer-Lambert law states that the Absorbance $A_i(\lambda)$ for a given wavelenght λ is proportional to the molarity M_i of the substance. Note: $A=\log(I/I_0)$, so it is unitless.

$A_i(\lambda)=\varepsilon_i(\lambda)\,l\,M_i$ where $\varepsilon_i(\lambda)$ is the molar absorptivity of the substance at a wavelengh λ, and l the cell path lenght throught the cell containing the solution.

Objectives: Determine the molarities of metal ions in a solution by direct spectrometry method and complexometric spectroscopy method, and compare these methods.

Do do so, for each method, a calibration curve will be plotted measuring the absorbance of solution of known molarity in order to determine the molarity of the unknown solution.

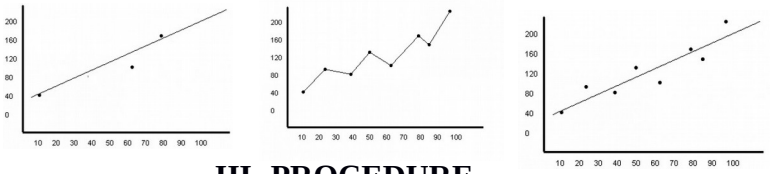
II- PRELIMINARY WORK

1) Read carefully the procedure, and make the necessary preliminary calculations for all the standard solutions.

2) Crystal field splitting diagram of cobalt(II) or copper(II), for water ligands (weak field)

3) What is the unit of molar absorptivity?

4) Which one of the three following graphs is correctly plotted ? Why?



III- PROCEDURE

Chemicals provided:

- Ethyldiamine Tetraacetic acid (EDTA). MW = 292.24

Either (A) or (B)

(A) - Copper sulfate pentahydrated $CuSO_4,5H_2O$. MW=249.685

- Unknown solution of copper sulfate.

(B) - Cobalt nitrate hexahydrated $Co(NO_3)_2,6H_2O$. MW=291.03

- Unknown solution of cobalt nitrate.

Apparatus and laboratory glassware required: See appended.

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How to use UV-visible spectrometer?

ALWAYS USE THE UV-VISIBLE SPECTROMETER IN THE PRESENCE OF A PROFESSOR

- cuvette must be handled carefully : any scratch or fingerprint on the cuvette would lead to incorrect measurements.
- Always hold cuvette by their ridged sides. When inserting the cuvette in the spectrometer, be sure the light source is directed through the clear sides of the cuvette.
- the cuvette used as a reference solution, filled with distilled water, is already inserted in the spectrometer.
- condition a cuvette with the solution to be measured before filling it (about 2/3). Carefully wipe off the outside of the cuvette with a soft tissue paper before placing it in the spectrometer.

1) Direct method

- Visible spectrum of unknown solution

Obtain the spectrum of the unknown solution provided (A or B, depending on your bench number) with the UV-visible spectrometer. Determine the exact wavelenght of the maximum of absorption observed (λ =) and the corresponding absorbances A(λ). Roughtly draw the spectrum obtained. Relate the spectrum to the color of the solution.

- Preparation of solution of known molarity, plot of the calibration curves.

Perform either (A) OR (B), depending on your bench number.

(A)

- Carefully prepare 50.0mL of 0.02M Cu^{2+} standard solution.
- From this solution prepare 10.0mL of 0.001M, 0.002M, 0.003M, 0.004M and 0.005M Cu^{2+} standard solution in 25mL beakers using the pipettes provided.
- Using the UV-visible spectrometer, measure the absorption at wavelenghts of maximum absorption λ for the 5 solutions previously prepared.
- Plot A(λ) VS molarity, and use it to deduce a first estimation of the molarity of the unknown solution of Cu^{2+}.

(B)

- Carefully prepare 50.0mL of 0.05M Co^{2+} standard solution.
- From this solution prepare 10.0mL of 0.005M, 0.010M, 0.015M, 0.020M and 0.025M Co^{2+} standard solution in 25mL beakers using the pipettes provided.
- Using the UV-visible spectrometer, measure the absorption at wavelenghts of maximum absorption λ for the 5 solutions previously prepared.
- Plot A(λ) VS molarity, and use it to deduce a first estimation of the molarity of the unknown solution of Co^{2+}.

2) Complexometric method.

- Spectrum of unknown solution <u>with EDTA</u>

- Prepare 50.0 mL of 0.2M standard solution of EDTA. (If it does not dissolve, leave it for some time, and shake it again later)
- In a 25mL beaker, take 8.0 mL of unknown solution and add 2.0 mL of EDTA solution.
- Obtain the spectrum of this solution, the wavelenght of maximum absorbance and the maximum absorbance. Write your observations.

- Preparation of solution of known molarity <u>in presence of EDTA</u> , plot of the calibration curves

- From the same standard solution prepared in part 1) , prepare 10.0mL of 0.001M, 0.002M, 0.003M, 0.004M and 0.005M Cu^{2+} standard solution in 25mL beakers, but diluting with EDTA solution instead of water.
- Deduce the molarity of the unknown solution. Attention : the spectrum obtained in the previous paragraph is of the unknown solution diluted with 1.0 mL of EDTA.

3) Conclusion:

- Summarize the methods and results obtained
- Compare, discuss the validity and the accuracy of the two methods.
- Draw the EDTACu or EDTACo octahedral complexes, and explain qualitatively the change in wavelenght when adding EDTA using Crystal Field Theory.

<u>APPENDED :</u>

<u>Apparatus and glassware required</u>

- a plastic cuvette (1 for two groups)
- two 50 mL volumetric flask
- five 25 mL (or 50 mL) beakers labelled 1->5
- 500 mL beaker
- watch glass
- funnel
- spatula
- 5 mL volumetric pipette
- 2mL graduated pipettes
- wash bottle with distilled water
- dropper
- pipette pump