PURDUE UNIVERSITY INSTRUMENT VAN PROJECT

SPECTROPHOTOMETRY OF Co^{2+}

(Revised: 1-24-93)

INTRODUCTION

In this experiment, the Beer-Lambert Law, A = åcl, will be applied to a series of aqueous solutions of $CoCl_2$. The pink color of the solutions is due to the presence of Co^{2+} ions, not Cl^{1-} ions. The wavelength corresponding the maximum absorbance in the absorption spectrum Co^{2+} will be identified. Then, in order to achieve maximum sensitivity in measurements, that wavelength will be utilized in determining the absorbances of a series of solutions of known concentration. When the measured absorbances are plotted against concentration, a linear relationship should be evident. The slope of the line will correspond to ål. Since the pathlength, l, can be measured, the value of å can then be calculated. As long as the wavelength is not changed, å will be a constant for any solution of $CoCl_2$. Thus, the concentration of Co^{2+} in an unknown solution can be calculated from the measured absorbance of the unknown solution.

PURPOSE

1.To determine the wavelength of maximum absorbance.
2.To validate the Beer-Lambert Law.
3.To use the Beer-Lambert Law to determine the concentration of a solution.

SAFETY CONSIDERATIONS

Safety goggles should be worn at all times. $CoCl_2$ is toxic by ingestion. Gloves should be worn if open wounds are present on the experimenter's hands, in order to prevent blood damage.

PRE-LAB QUESTIONS

1.The absorbances of a series of solutions containing Cr^{3+} were measured at a fixed wavelength and a pathlength of 1.1 cm, and the following data was recorded:

Tube No.	1	2	3	4
Volume of 0.100 M Cr^{3+} solution (mL)	4.0	3.0	2.0	1.0
Volume of water (mL)	1.0	2.0	3.0	4.0
Absorbance (A)	0.807	0.608	0.394	0.193
[Cr^{3+}] (M)	--	--	--	--

A. Complete the above table by calculating the concentration of each solution. To do this, you will need to use the Beer-Lambert Law: A = å l c

B. Plot the absorbances against the concentrations.

C. Determine the slope of the resulting line.

D. Calculate the value of å.

2. The absorbance of a Cr^{3+} solution of unknown concentration was measured at the same wavelength and same pathlength as described in #1. The absorbance was found to be 0.654. Calculate the concentration of Cr^{3+}.

3. When 10.0 mL of a Cr^{3+} solution was diluted to 500 mL, an absorbance of 0.126 was measured. Both the wavelength and pathlength were as described in #1. Calculate the concentration of Cr^{3+} in both the diluted solution and the undiluted solution.

4. The absorbance of a solution of Cr^{3+} is 0.224 at the wavelength and pathlength described in #1. What absorbance would occur if the pathlength were changed to 4.0 cm?

5. The percent transmittance obtained from a Cr^{3+} solution is 20.0% at the wavelength and pathlength described in #1. Calculate the absorbance and $[Cr^{3+}]$. (Hint: Absorbance is defined as the negative logarithm of transmittance; A = -log T)

MATERIALS

0.150 M $CoCl_2$
$CoCl_2$ solution of unknown concentration
6 cuvettes or test tubes
pipet and pipet bulb
stirring rod
ruler
spectrophotometer

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PROCEDURE

PART 1 - OBTAINING THE ABSORPTION SPECTRUM:

Obtain approximately 20 mL of 0.150 M $CoCl_2$. Using a pipet, transfer 5.0 mL of 0.150 M $CoCl_2$ to a cuvette. Prepare a "blank" by placing 5.0 mL of distilled water in another cuvette. Obtain the absorption spectrum of aqueous Co^{2+} by measuring the absorbance of the $CoCl_2$ solution at 25 nm intervals between 400 nm and 600 nm. Remember to "zero" the spectrophotometer at each wavelength. Also remember to adjust the spectrophotometer to 100% transmittance, using the blank, at each wavelength. Record the data.

Graph the absorbance (A) vs. wavelength. Draw a smooth curve to fit the experimental points. Identify the maximum in the absorption curve to the nearest multiple of 25 nm and record.

PART 2 - VALIDATING THE BEER-LAMBERT LAW:

Prepare a series of $CoCl_2$ solutions according to the following table. Using a pipet, add the indicated volume of $CoCl_2$ solution to each cuvette. Rinse the pipet thoroughly, and then add the indicated volume of water. Mix the contents of each tube using a clean, dry stirring rod. **NOTE: Tube 1 was prepared in Part 1.**

Tube No.		1	2	3	4	5
Volume of 0.150 M $CoCl_2$ solution (mL)		5.0	4.0	3.0	2.0	1.0
Volume of water (mL)	0.0	1.0	2.0	3.0	4.0	

Measure and record the absorbance of each of the series of solutions at the wavelength of maximum absorbance (as identified in Part 1). Calculate the % transmittance and the concentration of each of the solutions. If you are using a Spec 20D, measure transmittance and concentration and record.

Graph absorbance vs. $[Co^{2+}]$. Use a straight edge to draw a straight line through the origin and as close as possible to all of the experimental points. Calculate the slope of this line.

Measure the pathlength. Calculate the value of å. Record. Empty all solutions into the waste container. Rinse and dry the cuvettes thoroughly.

PART 3 - DETERMINING THE CONCENTRATION OF THE UNKNOWN:

Obtain about 6 mL of a $CoCl_2$ solution of unknown concentration. Dilution of this solution will be necessary, as its concentration is too large to obtain a direct measurement of the absorbance. Using the previously obtained data and the Beer-Lambert Law, determine [Co^{2+}]. The following guidelines are offered:

1. The absorbance of the diluted solution must lie between 0.3 and 0.5.

2. The minimum volume of solution required for a measurement is 3 mL.

3. The correct dilution should be established by measuring the absorbance after each of a series of successive dilutions. This process will provide only an approximate result, due to the accrual of experimental error after several dilutions.

4. The accumulation of error in the trials must be eliminated by preparing a new sample in which the desired absorbance is obtained by one dilution rather than a series of dilutions. Clearly, this dilution must be equivalent to the overall dilution established in the trials.

DATA TABLES

Part 1 - The Absorption Spectrum of Aqueous Co^{2+}

Wavelength (nm)	Absorbance
400	__________
425	__________
450	__________
475	__________
500	__________
525	__________
550	__________
575	__________
600	__________

The maximum absorbance (to the nearest multiple of 25 nm) occurs at __________nm.

Part 2 - Validating the Beer-Lambert Law

Absorbance as a Function of [Co^{2+}] at __________nm.

Tube No.	Absorbance	% Transmission	[Co^{2+}]
1	__________	_____________	______
2	__________	_____________	______
3	__________	_____________	______
4	__________	_____________	______
5	__________	_____________	______

pathlength = __________cm

Part 3 - Determining the Concentration of the Unknown

Unknown no. = ________

Trial solutions to establish the required dilution

Volume of Unknown (mL)	Volume of Water (mL)	Absorbance	%Transmittance
__________	_________	__________	______________
__________	_________	__________	______________
__________	_________	__________	______________
__________	_________	__________	______________
__________	_________	__________	______________

Required dilution

Volume of Unknown (mL)	Volume of Water (mL)	Absorbance	%Transmittance

DATA ANALYSIS

Part 1 - Attach the graph

Part 2 - Show the calculations for [Co^{2+}].

Show the calculations for %T.

Calculate the value of å.

Attach the graph.

Part 3 -Calculate the concentration of Co^{2+} in the diluted solution.

Calculate the concentration of Co^{2+} in the undiluted solution (the unknown).

Conclusions:

POST-LAB QUESTIONS

1.Calculate the concentration of Cl^{1-} in the undiluted sample of your unknown.

2.In Part 2, why must the straight line pass through the origin?

3.What experimental problems would confront you if you attempted to determine the concentration of the metal ion in solutions of each of the following reagents?

A.Cr^{3+} which has a maximum absorbance at 407 nm with å = 15 M^{-1} cm^{-1} and another at 574 nm with å = 13 M^{-1} cm^{-1}

B.Mn^{2+} which has a maximum absorbance at 530 nm with å = 0.050 M^{-1} cm^{-1}

C.Zn^{2+} which does not absorb light between 400 nm and 600 nm

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TEACHERS' GUIDE
SPECTROPHOTOMETRY OF Co^{2+}

CLASSROOM USAGE

This experiment is suitable for advanced first year chemistry students and/or AP chemistry.

CURRICULUM INTEGRATION

This lab would fit in along with the chapter on solutions and concentrations.

PREPARATION

Prepare a 0.150 M solution of $CoCl_2$. You will need about 30 mL per lab team. Prepare a 0.4 M solution of $CoCl_2$ for the unknown. 30 mL per lab team is ample.

TIME

Instructor will need to make 2 solutions and to set up the lab (about 15 minutes).
Students will need two days to complete the procedure (some will be able to begin calculations during the second lab period.

SAFETY AND DISPOSAL

Cobalt (II) chloride is toxic to ingestion. It can cause blood damage. The LD50 is 766 mg/kg. Use Flinn Catalog Disposal Technique #27f.

VARIATIONS

This lab may be done with the UV/Visible spectrophotometer as well. Parts 1 and 2 of the

PROCEDURE should read as follows:

Part 1

Obtain approximately 20 mL of 0.150 M $CoCl_2$. Using a pipet, transfer 5.0 mL of 0.150 M $CoCl_2$ to a cuvette. Prepare a blank by placing 5.0 mL of distilled water in another cuvette. Obtain the absorption spectrum of aqueous Co^{2+} by measuring the absorbance of the $CoCl_2$ solution from 200 nm to 900 nm. Using the graphed output, identify the wavelength at which maximum absorbance occurs.

Part 2

Prepare a series of $CoCl_2$ solutions according to the following table. Using a pipet, add the indicated volume of $CoCl_2$ solution to each cuvette. Rinse thoroughly, and then add the indicated volume of water. Mix the contents of each tube using a clean, dry stirring rod. **NOTE: Tube 1 was prepared in Part 1.**

Tube No.	1	2	3	4	5
Volume of 0.150 M $CoCl_2$ solution (mL)	5.0	4.0	3.0	2.0	1.0
Volume of water (mL)	0.0	1.0	2.0	3.0	4.0

Calculate the concentration of each of the solutions. Measure both the absorbance and % transmittance of the solutions at the wavelength of maximum absorbance (as identified in Part 1).

Graph absorbance vs. [Co^{2+}]. Use a straight edge to draw a straight line through the origin and as close as possible to all of the experimental points. Calculate the slope of this line.

Measure the pathlength. Calculate the value of å and record. Empty all solutions into the waste container. Rinse and dry the cuvettes.

Neither Part 3 of the PROCEDURE nor any of the remaining sections need to be changed.

REFERENCES

Wentworth, R. A. D. (1990). C125 Laboratory Manual. Prospect Heights: Indiana University Waveland Press.

SAMPLE RESULTS

For 0.150 M $CoCl_2$:

Wavelength (nm)	Absorbance
400	0.056
425	0.136
450	0.357
475	0.578
500	0.740
525	0.692
550	0.359
575	0.126
600	0.063

At 500 nm:

Tube	Molarity	Absorbance	% Transmittance
1	0.150	0.724	18.9
2	0.120	0.582	26.2
3	0.090	0.454	35.2
4	0.060	0.295	50.7
5	0.030	0.149	71.0

For Unknown:

# mL unknown	# mL water	Absorbance
3	2	1.1340
3	3	1.120
3	5	0.830
3	8	0.632
3	13	0.440

(When read off the Spec 20D, c = 0.089 M)

ASSESSMENT

Spectrophotometry of Co^{2+} Checklist

1. Follows safety procedures
2. Displays evidence of prior preparation
3. Acts as a contributing lab partner
4. Handles cuvettes appropriately
5. Uses instrument correctly
6. Exhibits proper pipetting technique
7. Disposes solutions appropriately
8. Constructs appropriate maximum absorbance graph
9. Cleans work area and equipment
10. Uses time efficiently

Assessment Activities

Station 1 - Materials: Spec 20
a variety of samples of different concentrations (numbered)
cuvette

Instruction Card: Using your assigned sample, record its absorbance at 500 nm.

Station 2 - Materials: Spec 20
6 labelled samples of varying color (use food coloring or crepe paper)
cuvette

Instruction Card: Using your assigned solution, identify the wavelength of maximum absorption.

Station 3 - Materials: Spec 20
prepared standard curve for Co^{2+}
sample - unknown concentration (already diluted)
cuvette

Instruction Card: Measure absorbance (at 500 nm) of unknown.
Using standard curve, determine [Co^{2+}]

Portfolio

1. One sentence synthesis
2. Vee diagram
3. Concept map
4. Written summary of teacher-supplied journal article that covers some aspect of spectrometry
5. Research proposal for spectrometry project
6. Report to "research administration" summarizing project
7. Sketch and description of instrument