Spectrophotometric Analysis of a Cobalt (II) Chloride Solution

General Chemistry I
(Chemistry 1411)

Professor …….

By

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ABSTRACT:

State the purpose: The aim of this experiment was to determine the absorption spectrum of Cobalt (II) ion using a spectrometer and Beer's Law Plot. State the compound or substance studied: This experiment was performed using pre-diluted Cobalt (II) Chloride solution of known concentration and pre-diluted Cobalt (II) chloride solution of unknown concentration. State the method or specific sections that were studied in the experiment. The absorption spectrum of the cobalt (II) ion was determined by calibrating the spectrometer with a cuvette that contained distilled water, then measured the absorption at different wavelength in the visible light region using a cuvette which contained 0.150M Cobalt (II) Chloride, $CoCl_2$. State the results: The concentration of the Cobalt chloride was determined to be 0.09322M at 500nm on a graph using the Beer's Law Plot and by the method of least squares to get the slope which is k = 2.93.

INTRODUCTION:

Background on the Spectrophotometer and theory of light absorption: In order to investigate either the absorbance or transmittance of light with a series of solutions given of Cobalt (II) 't use personal pronounsChloride ($CoCl_2$) and to determine the unknown solution you will need a device called spectrophotometer. This instrument detects the amount of radiant light energy that is absorbed by the molecules. In order for the spectrophotometer to do this it must have five basic components: a light source, a diffraction grating, a slit, a photoelectric tube, and a digital meter. There are two adjustments before the light-absorbing properties of a solution can be

Comment [AL1]: No personal pronouns throughout the text

measured must be done to the spectrophotometer. First, the diffraction grating must be adjusted for desired wavelength of light passes through slit. This is like adjusting the amount of light you want to come in a room with the venetian blinds. Secondly, the output of the phototube must be calibrated. Here is why it is important to clean your test tubes with lint free wipes to remove any finger prints or water droplets on test tubes. Now you are ready to test your samples given and determine the unknown. Remember to record your results onto a table then graph, zero out before each testing, and do not contaminate your test tube with impurities either in the solution or on the outside glass surface during the experiment.

The Beer-Lambert Law: The theory behind this method of analysis is called the Beer-Lambert Law (1). Since the light that enters the cuvette has a certain strength or Intensity, it will come out with a different Intensity depending on the substance inside the cuvette. If the substance is more concentrated, it absorbs more of the light and allows less to be transmitted. There is a correlation between the amount of light absorbed and the concentration of the solution, all other factors being kept constant as possible.

Formally speaking, the absorbance of light by a solution is proportional to the concentration of the compound in the solution and the thickness of solution that the light must pass through. The relationship is expressed in the general equation of the Beer-Lambert law:

$$A = abc \qquad (1)$$

where A = absorbance, a = a constant for the substance being studied (also known as **€**),

b = the thickness of solution the light passes through, and c = the concentration of the substance.

Since the same instrument will be used throughout the analysis on the same compound, a and b are combined together into one constant: k = ab. The equation is rewritten as:

$$A = kc \qquad (2)$$

Before the analysis, k must be determined for the instrument and the compound that is being analyzed. Absorbance readings (A) are taken for a set of solutions of known concentration (c) and plotted with Absorbance on the y-axis and concentration on the x-axis. This way, k can be calculated from the slope of the graph:

$$k = A/c \qquad (3)$$

Mathematical methods can be used here. It is also important to make sure that the instrument is set to the wavelength at which the compound absorbs highest and therefore gives best results for the readings. This is called the maximum wavelength or lambda max, where lambda is the symbol for wavelength (λ_{max}).

For the same reason, the dilution of the unknown solution is selected to be the one that gives the highest absorbance reading at lambda max.

Alternative methods or calculations if any: Percent transmittance, %T, is sometimes measured instead of absorbance. On instruments with scales rather than digital displays there is less error associated with reading the transmittance. It is also more directly related to the light level being measured. The absorbance and the percent transmittance are related as follows:

$$A = -\log (\%T/100) \qquad (4)$$

Once all of these conditions are met, a reliable analysis can be made. Spectrophotometry is used in many biology and biotechnology applications: for food dyes, protein in milk and protein drinks, rates of photosynthesis, sugar enzyme reactions, and track the growth of bacterial culture (2).

The Purpose of the Experiment in connection to the Theory: Using a set of solutions of Cobalt (II) Chloride ($CoCl_2$) we will investigate the absorption of light. We will be given a solution of this substance whose concentration is unknown but too large for accurate measurement of the absorption of light. The task is to determine the concentration of this solution. Recall that Beer's law states that the absorbance is directly related to the concentration (c) of the substance that absorbs light, or where k is a constant:

$$A = kc \qquad (2)$$

The equation for Beer's law has the same form as the equation for a straight line:

$$Y = mx + b \qquad (2a)$$

A comparison of these two equations, 2 and 2a, above indicates that:

$$y = A,\ x = c$$

$$m = \text{slope} = k$$

$$b = \text{intercept on y axis} = 0$$

A straight line should be obtained when we plot the absorbance at various concentrations against the concentrations. Calculations of concentration will be made from those readings and discussed in detail in the Data and Calculations sections.

PROCEDURE

Explain steps in Preparation: The spectrophotometer was turned on to warm up before the solutions were prepared. 50mL of a stock solution of 0.150M cobalt chloride was added into a 100mL beaker. After that, 50mL of deionized water was placed into another 100mL beaker. By using a 10.00mL pipet, 5.00mL of 0.150M Cobalt Chloride solution was transferred into test tube 5 from the 100mL beaker;

For the rest of the solutions, 4.00mL of 0.150M Cobalt Chloride solution was transferred into test tube 4 from the 100mL beaker; 3.00mL of this solution was transferred into test tube 3; 2.00mL of this solution was transferred into test tube 2 and 1.00mL of this solution was transferred into test tube 1. Also, 5.00mL of deionized water was added into test tube 0 from the 100mL beaker by using another 10.00mL buret. After that, by delivering with the buret, 1.00mL of deionized water into test tube 4, 2.00mL of deionized water into test tube 3, 3.00mL of deionized water into test tube 2, and 4.00mL of deionized water into test tube 1. After the deionized water was added to the 0.150M Cobalt Chloride solution of test tube 1-5, these test tubes were mixed thoroughly.

Explain steps in Instrumental Analysis: Preparing the spectrophotometer: Through following the number order, those mixed solutions of test tube 1-5 were transferred into cuvette 1-5 by using a 10.00mL pipet. The deionized water of test tube 0 was also transferred into cuvette 0 by using a 10.00mL pipet. After all the preparations of solutions were done, the displayed wavelength on the spectrophotometer was set to 430nm and also the cuvette 0 was inserted into the spectrophotometer. Then the blank button was push for setting the 0.00 absorption. The absorption of cuvette 5 in 430 nm was recorded by replacing the cuvette 0 to cuvette 5 inside of

the spectrophotometer. After that, the displayed wavelength on the spectrophotometer was changed to 460nm and also the cuvette 0 was inserted into the spectrophotometer again for setting the 0.00 absorption.

Determining the Wavelength of maximum absorption:The absorption of cuvette 5 in 460 nm was recorded by replacing the cuvette 0 to cuvette 5 inside of the spectrophotometer. By changing the displayed wavelength on the spectrophotometer and repeating above steps, the absorptions of cuvette 5 in 480 nm, 500 nm, 510 nm, 520 nm, 540 nm and 570 nm were recorded one by one. Through compare the absorption of each wavelength, the wavelength of highest absorption was selected for the second part of the experiment---unknown identification.

Explain steps in unknown analysis: The displayed wavelength on the spectrophotometer was set to the selected one which was recorded from previous experiment. And the cuvette 0 was inserted into the spectrophotometer for setting the 0.00 absorption. After this, a Cobalt Chloride solution with unknown concentration was obtained from instructor and the cuvette 5 was washed by deionized water. Then the Cobalt chloride solution with unknown concentration was transferred into this cuvette 5. The absorption of this solution was recorded by replacing the cuvette 0 to the cuvette 5 inside of the spectrophotometer. Then the concentrations of cuvette 1-4 were calculated and recorded. Finally the absorptions of cuvette 1-4 were recorded by replacing the cuvette 5 to all of them.

EXPERIMENTAL DATA AND CALCULATIONS:

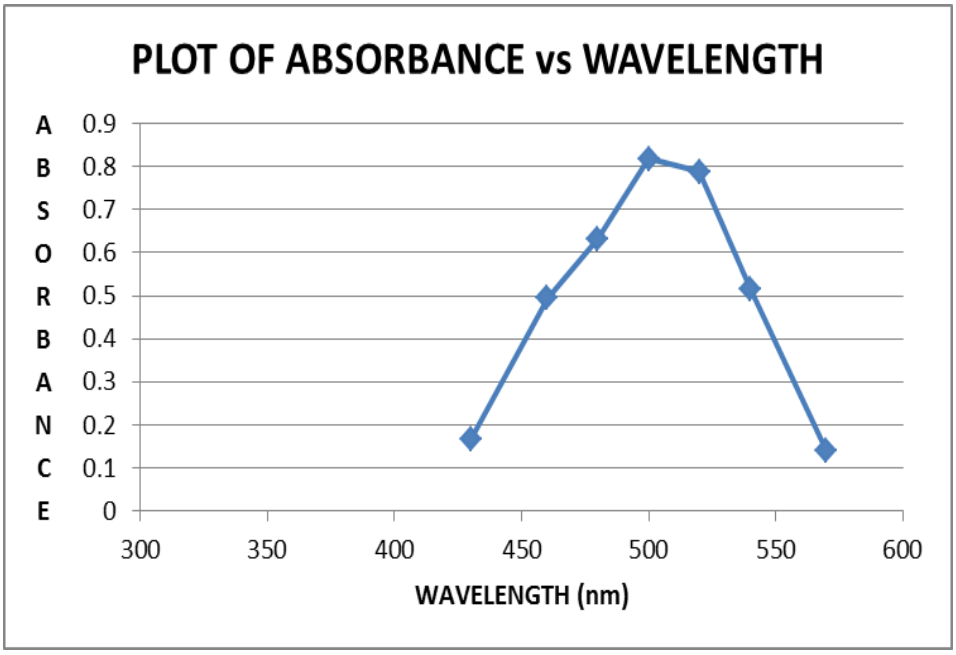
A. Data:

Part 1. Determining Wavelength of Maximum Absorption

Table 1. Absorption spectrum of standard Cobalt ion solution

Wavelength (nm)	absorption	Wavelength (nm)	absorption
430	**0.1662**	**520**	**0.7889**
460	**0.4939**	**540**	**0.5148**
480	**0.6328**	**570**	**0.1396**
500	**0.8180**		

Fig 1. Graph of A (y axis) vs. concentration and determine the optimum wavelength.



Based on this data in the graph , the highest absorption level is 0.8180 when the wavelength is equal to 500nm. Therefore, 500nm would be determined as the optimum wavelength for next step of experiment.

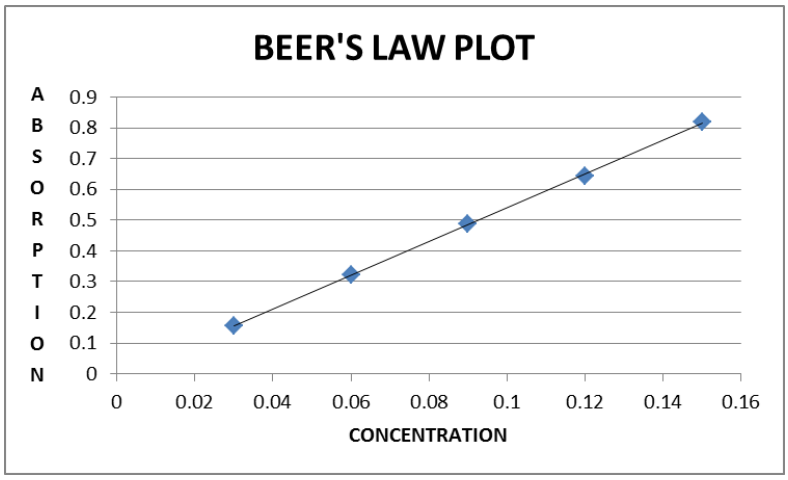
Part 2. Beer's Law Plot for the Standard Solutions of $CoCl_2$

Solutions were prepared by diluting a known volume from the Stock solution that was 0.15M in concentration. Absorbance readings were made at 500nm which was the maximum wavelength, called lambda max (λmax).

Table 2. Concentration and Absorbance Readings for Standard Solutions of $CoCl_2$

Tube #	Concentration (moles/L)	Absorbance (A.U.)
1	**0.0300**	**0.1555**
2	**0.0600**	**0.3208**
3	**0.0900**	**0.4882**
4	**0.1200**	**0.6450**
5	**0.1500**	**0.8181**

Figure 2. Graph A (y axis) vs. c (x axis) and determine the product **€**b from the slope of the line**.** **K = €b= 2.93**



Part 3. Identifying the unknown concentration

Unknown identification number: B

Table 3. Trial solutions were prepared according to the dilution on the table and gave the following Absorbance readings

mL of unknown	**mL of H_2O**	**A**
3.5	**1.5**	**0.192**
3.0	**2.0**	**0.156**
2.5	**2.5**	**0.138**
2.0	**3.0**	**0.113**

Table 4. Concentrations were calculated using k for each of the absorbances:

A	c diluted unknown, M	c undiluted unknown, M
0.192	**0.06552**	**0.09358**
0.156	**0.05324**	**0.08873**
0.138	**0.04709**	**0.09418**
0.113	**0.03856**	**0.09640**

Average: **0.093223 M**

B. Calculations:

1. Concentrations of the Diluted Standard Solutions

The main formula for the calculation of concentrations was based on:

$$M_i V_i = M_f V_f \qquad (3)$$

Sample calculations are:

$0.150M \times 1mL = M_f \times 5mL \quad M_{f1} = 0.300M$

$0.150M \times 2mL = M_f \times 5mL \quad M_{f2} = 0.600M$

$0.150M \times 3mL = M_f \times 5mL \quad M_{f3} = 0.900M$

$0.150M \times 4mL = M_f \times 5mL \quad M_{f4} = 0.120M$

2. These concentrations were used to graph Absorbance vs Concentration (Figure 2) and in calculating the slope by the least squares formula in the next section.

3. Manual Calculation of Slope from Linear Regression

Calculations were made from the formula for the slope and equal to k , the constant:

$$k = \sum cA / \sum c^{2} = M^{-1} \qquad (5)$$

where the slope m from equation (2a) is the constant k which is specific for the type of analysis and the characteristics of the spectrophotometer.

Also, ∑cA is the sum of the products of each Absorbance with its corresponding concentration and $\sum c^2$ is the sum of the squares of the concentration.

$$k = \frac{(0.0300)(0.1555) + (0.0600)(0.3208) + (0.0900)(0.4882) + (0.1200)(0.6450) + (0.1500)(0.8181)}{(0.0300)^2 + (0.0600)^2 + (0.0900)^2 + (0.1200)^2 + (0.1500)^2}$$

$k = 2.93\ M^{-1}$

This value of k will be used to find what the concentrations of the unknown diluted solutions are

4. For the concentrations of the diluted unknown solutions using k from linear regression:

$$c = A / k \qquad (6)$$

Thus:

$c_1 = 0.192 / 2.93 = 0.06857$ M c3 = 0.138/ 2.93 = 0.04710 M

c2 = 0.156 / 2.93 = 0.05324 M c4 = 0.113/ 2.93 = 0.03857 M

5. Concentration of Cobalt (II) chloride calculated

The same formula was used for the calculation of concentration of the undiluted unknown – working backwards to find the concentration of the undiluted original solution, C_1:

$$C_1 V_1 = C_2 V_2 \qquad (7)$$

Calculations were done for the each of the diluted unknown and the final answer averaged.

$$C_1 = C_2 V_2 / V_1 \qquad (7) \text{ rearranged}$$

C_1 = 0.06552 M x 5.0mL / 3.5mL = 0.09358M

C_1 = 0.05324M x 5.0mL / 3.0 mL = 0.08873M

C_1 = 0.04709M x 5.0 mL / 2.5 mL = 0.09418M

C_1 = 0.03856M x 5.0 mL / 2.0 mL = 0.09640M

Average: 0.37289 / 4 = 0.093223 M

Reported average concentration of unknown solution = 0.09322 M

6. Concentration of Cobalt (II) chloride unknown directly from graph on Excel

For comparison:

One method described in the lab manual for determining concentration from absorbance is to read off the value directly from the graph and use this to figure out the original concentration.

Selected Unknown absorbance = 0.192

Matching Concentration of unknown solution on the graph = 0.0412M

To calculate the concentration of the undiluted unknown with this concentration:

C_1V_1 / V_2 = 0.0412M x 5.0mL / 3.5 mL = 0.0588M which is on the low side and shows that a more accurate method would be by linear regression. This makes it clear that our experimental values were not on a perfect straight line.

DISCUSSION

Summarize what you set to do in the experiment: In this experiment, two things had to be measured. First, the relationship between absorption and wavelength of radiation that found the best absorbed wavelength for Cobalt (II) Chloride solution. Second measurement is the relationship between absorption and concentration of solution which proved Beer's law.

Discuss determining lambda max: The best-absorbed wavelength was the first factor that should be figured out in the first part of experiment. Based on data, the 500nm had highest absorption rate by Cobalt (II) chloride. Below 500nm, the absorption kept increasing and it decreased when wavelength reached beyond 500nm. Therefore 500nm was the selected maximum absorbance wavelength for this experiment. It was important because it had to be used in the second part of this experiment.

Discuss the standard curve, your results and how they will be used: The second part of this experiment was looking for the relationship between absorption and concentration of cobalt chloride solution. In fact, the absorption would be increased when the concentration rose. According data read by spectrophotometer, a function had been calculated to give the equation of a straight line and yielded a slope = 2.93 Using this for the relationship between absorbance and concentration: **y = 2.93 x.** In this function, X represents the concentration of solution and Y is the absorption of the solution. It proved the Beer's Law and showed they are directly related between absorption and concentration.

Discussion of errors and limitation of the experiment: There were two main errors happened in the experiment which may impact or affect this experiment. They were the actual concentration of Cobalt (II) ion solution and the spectrophotometer itself.

First, the actual concentration of Cobalt ion solution may be not same as concentration showed on label. The laboratory-made solution should very accurate and pure. However, during reserving and moving those solutions into lab, there have many factors which affect the concentration of solution by environment. Therefore, the error of the solution used may transfer into the experiment and caused error. Fortunately, this error should be really small and will not affect experiment seriously.

Second, the spectrophotometer may have some instrumental errors in this experiment**.** By calibrating the instrument and using a blank, we prevented these errors from becoming too big. This experiment required high accuracy of operation and measurement. Therefore, several procedures were used in this experiment to avoid error.

What was done further to minimize error: First, throughout the experiment the pipets were used to measure the volume of solution and water. Because the accuracy is so important, the experiment should use equipment as accurately as possible. Also, when measuring liquid by pipet, decimal places had to be read according to the marks on the instrument and may have been missed.

Second, the cuvettes had to be rinsed with deionized water, and the Cobalt Chloride solution before they was filled and placed in the spectrophotometer. In addition, the outside of tube was wiped clean to erase any fingerprint oils on the surface of the cuvettes. Finally, the test tubes used to dilute the solutions was dried and not rinsed before being used in order to prevent water droplets from producing an inaccurate result.

Discussion of determining of the unknown: The same formula was used for the calculation of concentration of the undiluted unknown – working backwards to find the concentration of the undiluted original solution, C_1:

$$C_1 V_1 = C_2 V_2 \qquad (7)$$

Calculations were done for the each of the diluted unknown and the final answer averaged. The average concentration calculated for the unknown was 0.09322M. Standard Deviation analysis should have been done on the results of the analysis.

CONCLUSION

Statement of findings: This experiment showed that Cobalt (II) Chloride solutions in dilute concentration follow the Beer-Lambert law at 500nm. The Cobalt (II) Chloride standard solution had highest absorption when the wavelength was 500nm. The method of least squares was selected over direct reading from the graph. The average concentration calculated for the unknown was 0.09322M. Assess the method itself: The results showed good correlation. It also showed that the concentration of an unknown cobalt chloride solution can be determined to a useful degree of accuracy.

REFERENCES: (add lecture and lab book)

(1) Lab Procedure from Ebbing and Gammon given to class

(2) Internet: http://www.hhmi.org/coolscience/resources/SPT--FullRecord.php?ResourceId=34

(3) Houghton Miffin Company, The absorption Spectrum of Cobalt (ll) Chloride, (Experimental requirements were obtain from this source)