



Preparation and Analysis of a Coordination Compound

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The Problem to be Investigated: *A coordination compound containing cobalt, chlorine, and ammonia is to be synthesized and the formula of the compound determined.*

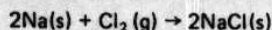
The Nature of This Investigation: *A coordination compound is prepared by the reaction of cobalt(II) chloride 6 hydrate, ammonium chloride, concentrated ammonia water, charcoal, and hydrogen peroxide.*

The formula of the compound is determined by analyzing the compound for percent chloride gravimetrically by precipitation as silver chloride, for percent cobalt spectrophotometrically, and for percent ammonia by difference.

BACKGROUND INFORMATION

Chemical bonds are conveniently classified as ionic or covalent. These two classifications represent the extremes of bond formation. Most chemical bonds fall somewhere between these limits, that is, a bond is not generally purely ionic or covalent but represents some compromise between the two.

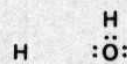
In the formation of an ionic bond between two atoms, a valence electron is transferred from the atom with the greater tendency to lose electrons to the atom with a greater affinity for the electron. This is illustrated by the reaction of sodium with chlorine to form sodium chloride.



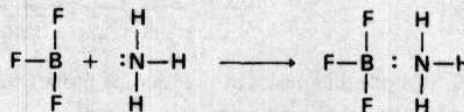
A Lewis formula can be written for this product to illustrate the bonding.



In the formation of a covalent bond between two atoms, valence electrons of these atoms are shared. When two electrons are shared between two atoms, a single covalent bond is formed. This type of bonding is illustrated by use of the Lewis formula to represent the bonding in H_2O .



In some compounds, one atom will donate both the electrons involved in covalent bond formation between two atoms. This type of bond is called a coordinate-covalent bond and can be illustrated by the reaction of boron trifluoride with ammonia.

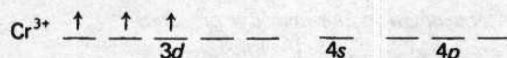


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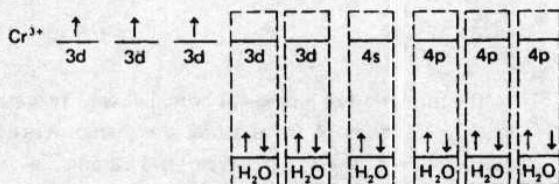
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A characteristic of ionically bonded atoms is their dissociation into ions when placed in water. For example, sodium chloride dissociates into Na^+ ions and Cl^- ions when dissolved in H_2O .

Many elements such as the transition elements have the ability to form ions that consist of more than one element. These ions are called complex ions. Complex ions involving transition metal atoms can be formed by ions or neutral molecules, called ligands, sharing electron pairs with the transition element. The electronic configuration for Cr is $1s^2, 2s^2, 2p^6, 3s^2, 3p^6, 4s^1, 3d^5$. When a transition element forms an ion, electrons are first lost from the outer occupied s orbital followed by the loss of electrons from the d orbitals. Thus, the electronic configuration for Cr^{3+} is $1s^2, 2s^2, 2p^6, 3s^2, 3p^6, 3d^3$. The arrangement of electrons in the outermost orbitals is represented as

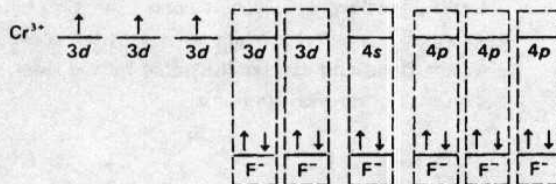


Cr^{3+} ions have empty d , s , and p orbitals which are willing to accept electron pairs to form coordinate-covalent bonds. If H_2O is the ligand, the valence bond representation for the bonding in the complex ion $[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$ is represented as



Because the H_2O molecules are neutral and the chromium ion has a $3+$ charge, the complex ion also has a $3+$ charge.

The valence bond representation for the bonding in the complex ion $[\text{Cr}(\text{F})_6]^{3-}$ is represented as

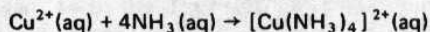


In this complex, each of the fluoride ligands contributes a $1-$ charge, giving the ion a $3-$ charge.

When the complex ions are isolated as a salt, the ions are

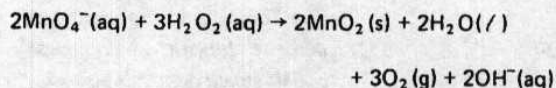
combined through ionic bonding to cations or anions, depending on the charge necessary to neutralize the charge on the complex ion. Thus, the $[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$ complex could be isolated as a nitrate salt with the formula $[\text{Cr}(\text{H}_2\text{O})_6] [\text{NO}_3]_3$. When dissolved in water, the salt dissociates into the complex ion and nitrate ions.

Two types of chemical reactions will be used in the synthesis and analysis of the compound prepared in this experiment. Lewis acid-base reactions are those which involve a substance capable of accepting a pair of electrons, called an acid, and a substance that donates a pair of electrons, called a base. A Lewis acid-base reaction is illustrated in the equation



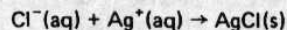
where Cu^{2+} is an acid and the NH_3 is the base.

Oxidation reduction reactions are those which involve a transfer of an electron from one substance to another. This is illustrated in the reaction



where each permanganate ion gains three electrons and each hydrogen peroxide molecule loses two electrons.

In this experiment a coordination compound containing cobalt, ammonia, and chlorine is to be prepared. The analysis of the compound for chloride ion is performed gravimetrically by precipitation of silver chloride. An excess of silver nitrate is added to the chloride solution, which has been acidified with HNO_3 . The reaction is written as

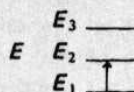


The precipitate is collected in a filter crucible, washed and dried and then weighed as AgCl . Suppose a 1.00 -gram sample when treated with silver nitrate solution yields a precipitate of 0.75 grams of AgCl . The percent chloride ion in the sample would be calculated as

Percent chloride ion = (0.75 g of AgCl)

$$\times \left(\frac{35.5 \text{ g of Cl}}{143.3 \text{ g of AgCl}} \right) \left(\frac{100}{1.00 \text{ g of sample}} \right) = 18.6\%$$

The analysis of cobalt can be conveniently performed spectrophotometrically. The energy of electrons in an atom is quantized; that is, only certain values of energy are allowed. This can be represented by the following diagram:



In order to excite an electron from energy level E_1 to energy level E_2 a quantity of energy equal to the difference between the energy levels ($E_2 - E_1$) must be absorbed by the atom. This energy can be related to the frequency of electromagnetic radiation (light) by Planck's equation $\Delta E = h\nu$, where ν is the frequency of light and h is a constant known as Planck's constant. The relationship between frequency and wavelength of light is formulated as $\nu\lambda = c$, where λ is the wavelength of light and c is the velocity of light.

When continuous light, that is, light which consists of all possible wavelengths within a given range, is passed through a prism, the light is dispersed into its component wavelengths. When the dispersed wavelengths of light are passed through a cell containing a sample of atoms, it is found that at certain wavelengths the emergent light is not as intense as the incident light. Some of the light waves with energy corresponding to that necessary to excite an electron to a higher energy level have interacted with the atoms in the cell. In other words, the atoms have absorbed some of the light. A plot of the intensity of the emitted light versus its wavelength is called a spectrum. An instrument capable of giving these data is called a spectrophotometer. Because each different kind of atomic system has a unique set of energy levels, each system will also have its own characteristic spectrum. A typical spectrum is shown in Figure 1.

The wavelength where the spectrum has a minimum corresponds to the wavelength of absorption of light by

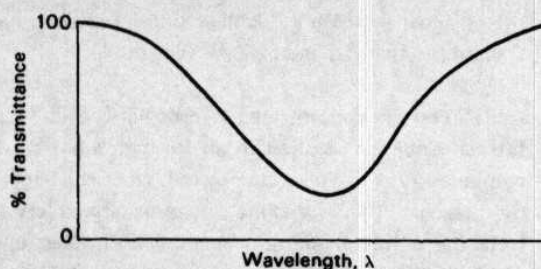


Figure 1. A typical spectrum.

the system. This wavelength of light is that used in the analysis of the sample. Following is a list of definitions used in spectrophotometric determinations.

I_0 = intensity of the original light

I_t = intensity of the transmitted light

Transmittance = I_t/I_0 = fraction of the original light passing through the cell

% Transmittance = $(I_t/I_0) \times 100$

Absorbance = $\log I_0/I_t$

When the concentration of a substance in the cell is increased, the absorbance proportionately increases. If the concentration of the substance in the cell is doubled, the absorbance is doubled. The absorbance is also found to be directly dependent upon the thickness of the cell containing the absorbing species. These relations are known as Beer's law, which stated mathematically is

$$\log \frac{I_0}{I_t} \propto Cl$$

where C is the concentration expressed in mole liter⁻¹ and l is the thickness of the cell in centimeters. The proportionality may be removed by writing

$$\log \frac{I_0}{I_t} = \epsilon Cl$$

where ϵ is a proportionality constant. This equation is in the form of a straight line. By measuring the absorbance for a series of standard solutions and plotting the results, a graph similar to the one given in Figure 2 is obtained.

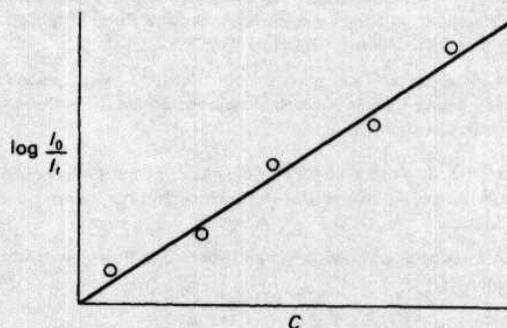


Figure 2. A plot of absorbance versus concentration.

An unknown solution is analyzed by measuring its absorbance and determining its concentration from the graph in Figure 2. The percent of that element in a compound is calculated in a manner similar to that illustrated for chloride.

The relative number of each kind of atom in a molecule may be calculated from the mass of the various atoms in the compound. The number of moles of each kind of atom in the compound is calculated. Each number of moles is divided by the smallest value to give the simplest ratio of the number of each kind of atom in the compound. For example, if a compound was found by analysis to contain 20.0% Ca and 80.0% Br its empirical formula is found by the following calculation:

Consider 100 grams of compound:

$$(80.0 \text{ g Br}) \left(\frac{1 \text{ mole Br}}{79.9 \text{ g Br}} \right) = 1 \text{ mole Br}$$

$$(20.0 \text{ g Ca}) \left(\frac{1 \text{ mole Ca}}{40.1 \text{ g Ca}} \right) = 0.5 \text{ mole Ca}$$

$$\frac{1 \text{ mole Br}}{0.5 \text{ mole Ca}} = \frac{2}{1}$$

Therefore, the formula of the substance is CaBr_2 .

References

1. F. Basolo and R. Johnson, *Coordination Chemistry*, W. A. Benjamin, New York, 1964, pp. 1-34.

An introduction to coordination compounds and a discussion of the coordinate-covalent bond.

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3. O. A. Runquist, C. J. Creswell, and J. T. Head, *Chemical Principles: A Programmed Text*, Burgess Publishing Company, Minneapolis, Minn., 1968, pp. 40-70.

A programmed approach to stoichiometry. Many examples of calculating percent weight, concentrations of solution, and molecular formula.

4. J. S. Fritz and G. H. Schunk, Jr., *Quantitative Analytical Chemistry*, Allyn and Bacon, Boston, 2nd ed., 1969, pp. 39-54, 63-82.

A discussion of gravimetric and spectrophotometric methods of analysis.

PROCEDURE

Preparation and Purification of Coordination Compound

Weigh 10 g of ammonium chloride on a centigram balance. Add the ammonium chloride to 20 ml of

distilled water in a 50-ml beaker. Carefully heat this solution to the boiling point. Add to the boiling solution a 15-g sample of cobalt(II) chloride 6 hydrate weighed to the nearest tenth of a gram on a weighed watch glass. Record all masses on the Data Sheet.

Add the hot cobalt solution to a 250-ml Erlenmeyer flask containing 1 g of powdered charcoal. Cool the solution by running tap water over the outside of the flask. Add 32 ml of concentrated ammonia water to the 50-ml beaker to wash out any remaining cobalt(II) solution. Transfer this solution to the solution in the Erlenmeyer flask.

Chill the flask and contents to below 10°C in an ice bath. By beginning with 1-ml increments, add carefully 40 ml of 30% hydrogen peroxide solution to the basic cobalt(II) solution in the Erlenmeyer flask.

CAUTION: Handle hydrogen peroxide solution with care because of the danger of hydrogen peroxide causing serious burns of the skin.

Clamp the flask and contents in a 600-ml beaker serving as a water bath as shown in Figure 3. Heat the bath to 60°C and maintain the temperature at $60^\circ \pm 5^\circ\text{C}$. Stir the solution in the flask with a glass stirring rod and leave the flask in the bath at 60°C as long as any pink color remains in the solution, which will require 30 to 60 minutes. When the pink color has disappeared, remove the flask and contents from the water bath. Cool the flask and contents to 0°C in an ice bath. Filter the crystals formed in the solution by using a Buchner funnel and filter flask as illustrated in Figure 4. Retain the residue and discard the filtrate.

Place in a 250-ml beaker 125 ml of distilled water and 5 ml concentrated hydrochloric acid. Heat the solution to boiling. By using a spatula, transfer the crude product to the boiling solution. When the compound has completely dissolved, filter the hot solution to remove the charcoal by using a Buchner funnel and filter flask. Discard the charcoal and save the filtrate.

Add 20 ml of concentrated hydrochloric acid to the filtrate. Cool the solution in an ice bath while stirring continuously with a glass stirring rod. When the temperature reaches 10°C , separate the precipitated crystals from the saturated solution by using a Buchner funnel and filter flask. Wash the product twice with 15 ml of

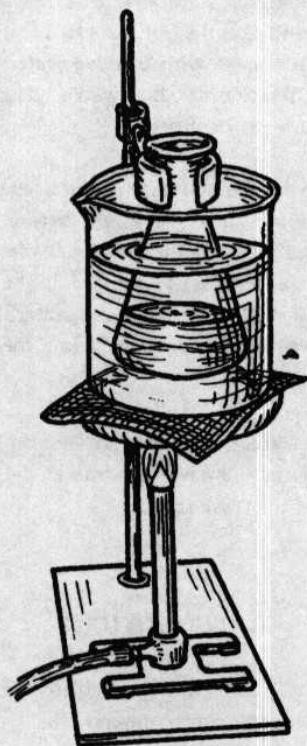


Figure 3. Apparatus for preparing the coordination compound.

60% by volume ethyl alcohol–water mixture followed by washing twice with 15 ml of 95% by volume ethyl alcohol–water mixture. Discard the filtrate and washings. Dry the residue by transferring the crystals from the Buchner funnel, by using a spatula, onto several thicknesses of filter paper on a watch glass.

Weigh a second watch glass to the nearest tenth of a gram. Record the mass on the Data Sheet. When the crystals are completely dry, use a spatula to transfer carefully the crystals to the weighed watch glass. Find the mass of the watch glass and crystals to the nearest tenth of a gram. Record this mass on the Data Sheet. Transfer the compound to a weighing bottle.

Gravimetric Chloride Determination

By using an analytical balance, weigh to the nearest milligram three samples of the unknown compound of 0.45 to 0.55 g. Place each sample in one of three labeled 250-ml beakers, each containing a glass stirring rod and covered with a watch glass. Each sample's mass is

determined by weighing the weighing bottle containing the unknown compound before and after each transfer.

Use the following procedure for each sample of the unknown compound. Add approximately 150-ml of deionized water to the sample in the beaker. Stir the mixture until the sample dissolves. Acidify the solution by adding 1 ml of 6M nitric acid.

Precipitate the chloride as silver chloride by slowly adding, with continuous stirring and at room temperature, 30 ml of 0.25M silver nitrate solution. Carry out the precipitation and succeeding operation in subdued daylight to prevent the decomposition of silver chloride.

After the silver nitrate solution has been added, heat the suspension with frequent stirring until the solution just starts to boil. Stir the mixture for a minute or two with the glass stirring rod to aid coagulation of the precipitate. Remove the beaker and contents from the wire gauze. Cover the beaker with a watch glass. Set the beaker and contents aside in the dark and allow the solution to stand overnight.

Fit a fritted glass filter crucible on a filter flask by using a crucible holder and clean the crucible by sucking through the crucible 10 to 15 ml of 1.0M nitric acid followed by 50 to 75 ml of deionized water. Dry the crucible in an oven at 150°C for 1 hour, cool, and weigh. Collect the silver chloride precipitate in the filter crucible. Care must be taken to make certain that in the transfer none of the precipitate is lost.

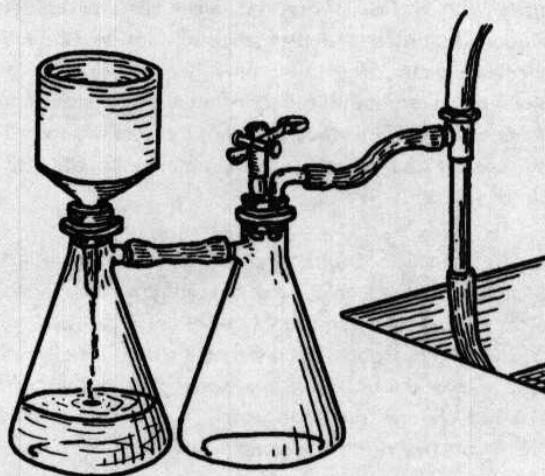


Figure 4. Apparatus for filtering the coordination compound.

Wash the residue with 0.01M nitric acid, added in small portions of 5 to 10 ml, until a few milliliters of washing collected in a test tube give no turbidity or at most only a faint opalescence, when the washing is tested with 1 drop of 3M hydrochloric acid.

Heat the crucible and contents in an oven at 150°C for at least 1 hour. Cool for 30 minutes in a beaker covered with a watch glass and weigh the crucible and contents to the nearest milligram. Record this mass on the Data Sheet.

Spectrophotometric Determination of Cobalt

Part I. Determination of visible spectrum of cobalt(II) ion. Determine the visible spectrum of a standard stock solution of cobalt(II) ion which is 0.075M in cobalt(II) sulfate 7 hydrate by recording the percent transmission of the solution over the visible region of electromagnetic radiation from 350 to 700 mμ. Follow the directions of the laboratory instructor for the use of the spectrophotometer available for this experiment. Record on the Data Sheet the percent transmittance for each specified wavelength for the stock solution of cobalt(II) ion.

Prepare at least five solutions of varying concentration of cobalt(II) by diluting the stock solution with deionized water. Perform these dilutions by use of a 10-ml pipet fitted with a rubber bulb. For example, a 0.037M solution can be prepared from the 0.075M stock solution by pipeting 10 ml of the 0.075M stock solution and 10 ml of deionized water into a 100-ml beaker and mixed with a glass stirring rod. Make the most dilute solution a dilution of 1 part stock solution to 12 parts deionized water. By using only the wavelength of minimum transmittance found from the visible spectrum of the cobalt(II) ion stock solution, measure and record the percent transmittance and absorbance of each diluted solution.

Part II. Analysis of unknown coordination compound for cobalt. Weigh 2 to 2.5 g of the unknown compound to the nearest milligram into a clean, dry 50-ml beaker. The weighing is done by difference using a weighing bottle. Cover the beaker with a watch glass and place it on a hot plate in the hood which is at approximately 350°C and heat until the sample liquefies, foams, and turns blue. Remove the beaker and contents from the hot plate and allow the beaker and contents to cool to room temperature. Add 10 ml of deionized water and

1 ml of concentrated sulfuric acid to the sample. Boil the mixture gently on the hot plate to dissolve the solid. Wash into the beaker with distilled water any sample that has spattered onto the watch glass. Cool the solution to room temperature.

Carefully transfer the contents of the beaker to a clean 100-ml volumetric flask. Rinse the beaker with 10 to 15 ml of deionized water three or four times, adding the washings to the volumetric flask. Fill the volumetric flask up to the mark with deionized water. Stopper the flask and invert the flask 30 times to mix the contents of the flask.

Measure and record the percent transmittance and absorbance of the unknown solution at the same wavelength used for the stock solutions.

CALCULATIONS

- For each determination, calculate the percent of chloride in the unknown compound.

$$\text{Percent chloride} = (\text{g of AgCl}) \left(\frac{\text{gram atomic mass of Cl}}{\text{gram molecular mass of AgCl}} \right) \times \left(\frac{100}{\text{mass of sample}} \right)$$

Find the average of the percent chloride and record on the Data Sheet.

- Prepare a graph of absorbance versus concentration for the standard Co^{2+} solutions. This is done by converting the measured percent transmittance for each solution into absorbance and calculating the concentration from the dilution of the stock solution. Record the absorbance on the Data Sheet.

$$\text{Absorbance} = \log \frac{I_0}{I_t} = -\log \frac{I_t}{I_0} = -\log (\text{transmittance})$$

Draw the best straight line curve through the plotted data.

- Calculate the percent cobalt in the unknown compound. This is done by calculating the absorbance of the unknown solution and by using the graph prepared in number 2 to determine the concentration.

Percent cobalt = (concentration of Co(moles/liter))

$$\times \left(\frac{1 \text{ liter}}{1000 \text{ ml}} \right) (100 \text{ ml}) \left(\frac{\text{gram atomic mass of Co}}{1 \text{ mole of Co}} \right) \left(\frac{100}{\text{mass of sample}} \right)$$

Record the percent cobalt on the Data Sheet.

4. Calculate the percent of ammonia in unknown compound. This percent is determined by difference.

$$\% \text{NH}_3 = 100 - (\% \text{Co} + \% \text{Cl})$$

Record the percent ammonia on Data Sheet.

5. By using the percent composition found in Calculations 1, 3, and 4, determine the empirical formula of the unknown compound. Be sure to allow for experimental error. (For sample calculation see Background Information and Pre-Laboratory Assignment.)

6. Calculate the percent yield for the preparation of the compound.

$$\text{Percent yield} = \left(\frac{\text{mass of unknown compound}}{\text{mass of CoCl}_2 \cdot 6\text{H}_2\text{O}} \right)$$

$$\times \left(\frac{\text{gram molecular mass of CoCl}_2 \cdot 6\text{H}_2\text{O}}{\text{gram molecular mass of unknown compound}} \right) (100)$$

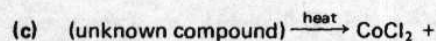
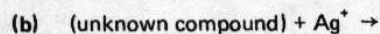
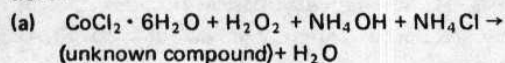
Suggestions for Further Study

1. Instead of finding the percent ammonia by difference of the unknown coordination compound, determine the nitrogen content by using the Kjeldahl method, J. S. Fritz and G. H. Schenk, *Quantitative Analytical Chemistry*, Allyn and Bacon, Boston, 2nd ed., 1969, pp. 187–189, 600–603.
2. Study the magnetic properties of the unknown compound prepared in this experiment. Discuss these data in terms of hybridization of the transition atom. L. J. Brubacher and F. Stafford, *J. Chem. Educ.*, **39**, 574 (1962), and S. Kirschner, M. J. Albinals, and J. G. Bergman, *J. Chem. Educ.*, **39**, 576 (1962).
3. Prepare other complexes of cobalt involving ammonia and chloride ion and analyze each complex. Compare the analysis of the different compounds. R. J. Angelici, *Synthesis and Technique in Inorganic Chemistry*, W. B. Saunders Company, Philadelphia, 1969, pp. 13–17.
4. Study the rate and mechanism of reactions involving complex ions. R. J. Angelici, *Synthesis and Technique in Inorganic Chemistry*, W. B. Saunders Company, Philadelphia, 1969, pp. 25–29.

QUESTIONS

(Use the space provided for the answer and additional paper if necessary.)

1. Write chemical equations for the following reactions:



2. Calculate the average deviation in the percent chlorine determinations. The average deviation is defined by the formula

$$\frac{\sum |\bar{x} - x_i|}{n}$$

where Σ indicates a summation of the terms for all values of i , \bar{x} is the average value of all measurements, x_i is the value of the i th measurement, and n is the total number of measurements.

3. In the calculation of the percent yield for the unknown compound, why were the masses of all materials used in the reaction ignored with the exception of the mass of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$?

4. Evaluate the proportionality constant for Beer's law.

Ans. _____ liters mole⁻¹.

5. In the preparation of the unknown compound 1 g of powdered charcoal was used as a catalyst for the reaction. What is the purpose of a catalyst in a chemical reaction?

Ans. _____

Name _____ Section _____ Date _____

Name _____ Section _____ Date _____

DATA SHEET

Preparation and Analysis of a Coordination Compound

*Preparation*Mass of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and watch glass, g _____

Mass of watch glass, g _____

Mass of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, g _____

Mass of unknown compound and watch glass, g _____

Mass of watch glass, g _____

Mass of unknown compound, g _____

Percent yield of compound _____

Gravimetric Chloride Determination

	<i>Determination</i>		
	<i>First</i>	<i>Second</i>	<i>Third</i>
Mass of weighing bottle and unknown compound, g	_____	_____	_____
Mass of weighing bottle and contents after removing sample of unknown, g	_____	_____	_____
Mass of unknown sample, g	_____	_____	_____
Mass of filter crucible + AgCl, g	_____	_____	_____
Mass of filter crucible, g	_____	_____	_____
Mass of AgCl, g	_____	_____	_____
Percent chloride in unknown	_____	_____	_____
Average percent chloride in unknown		_____	

Name _____ Section _____ Date _____

Spectrophotometric Determination of Cobalt(II) Ion

Concentration of cobalt(II) ion solution _____

<u>Wavelength, mμ</u>	<u>Percent transmission</u>	<u>Wavelength, mμ</u>	<u>Percent Transmission</u>
350	_____	515	_____
375	_____	525	_____
400	_____	550	_____
425	_____	560	_____
450	_____	570	_____
475	_____	580	_____
500	_____	590	_____
510	_____	600	_____

Wavelength of minimum transmittance, m μ _____*Standard Solutions*

<u>Number of solution</u>	<u>Volume of stock solution, ml</u>	<u>Volume of deionized water, ml</u>	<u>Concentration of solution, M</u>	<u>Percent transmittance</u>	<u>Absorbance</u>
1	_____	_____	_____	_____	_____
2	_____	_____	_____	_____	_____
3	_____	_____	_____	_____	_____
4	_____	_____	_____	_____	_____
5	_____	_____	_____	_____	_____

Unknown solution

Mass of weighing bottle and unknown compound, g _____

Mass of weighing bottle and contents after removing sample of unknown, g _____

Mass of unknown sample, g _____

Percent transmittance _____

Absorbance _____

Concentration, M _____

Percent cobalt _____

Percent ammonia _____

Empirical formula of compound _____

Name _____ Section _____ Date _____

PRE-LABORATORY ASSIGNMENT

Preparation and Analysis of a Coordination Compound

1. A substance is composed of one atom of copper (Cu), two atoms of chromium (Cr), and seven atoms of oxygen (O). What is the percent of chromium in the substance?

Ans. _____ %

2. Calculate the number of grams of chromium in 100 ml of a solution which is 0.1M in $[\text{Cr}(\text{H}_2\text{O})_6](\text{NO}_3)_3$.

Ans. _____ %

3. The percent composition of a compound was found to be 23.1% Al, 15.4% C, and 61.5% O. What is the empirical formula of the compound?

Ans. _____

4. The following data were recorded for a solution that is 0.5M in Cr^{3+} ions using a Spectronic 20 spectrophotometer.

<u>% transmittance</u>	<u>wavelength (mμ)</u>
65	700
52	675
24	650
8	625
4	600
3	575
7	550
16	525
32	500
35	475
37	450

On the graph paper provided, prepare a graph of percent transmittance versus wavelength and determine the wavelength of minimum transmittance of chromium(III).

Ans. _____