

Name: _____ Section _____

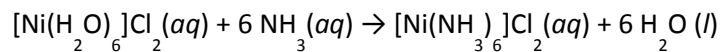
Chemistry 121 Laboratory

Taken in part from the original experiment from the University of Massachusetts Boston

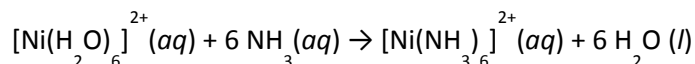
Hexamminenickel(II) Chloride Synthesis and Magnetic Susceptibility

INTRODUCTION – Part I

In aqueous solution nickel ion (Ni^{2+} ; Ni(II)) is surrounded by six water molecules which are actually bonded to the central metal ion. This complex is called the hexaquanickel(II) ion. When aqueous nickel chloride is precipitated from solution, the nickel ions carry their six water molecules into the crystals and so solid nickel(II) chloride is hydrated, has the formula $[\text{Ni}(\text{H}_2\text{O})_6]\text{Cl}_2$, and is more properly called hexaquanickel(II) chloride. If ammonia is added to a solution of this salt, ammonia molecules compete with the water in bonding Ni^{2+} and, because the ammonia forms a stronger bond than water, the ammonia replaces the water according to:



or, as a net ionic equation



In this net ionic equation, the product $[\text{Ni}(\text{NH}_3)_6]^{2+}$ is called hexamminenickel(II) ion.

In this experiment we will synthesize the complex salt hexamminenickel(II) chloride.

IN THE LABORATORY

- 1) Use an analytical balance to tare a weighing dish and then mass out anywhere from 4.0 to 4.5 g of nickel chloride. Record the mass to a tenth of a milligram _____.
- 2) Dissolve your nickel chloride in roughly 10 mL of deionized water in a 250-mL beaker. Mark the beaker with your initials.
- 3) Slowly pour aqueous ammonia into your beaker up to the 50-mL line. Stir with a glass rod. After 15 minutes, cool in an ice bath stirring occasionally.

**Go to a fume hood for the next step.
Do not smell or touch the aqueous ammonia solution.
Do not remove your beaker from the hood until the next step is completed.**

4) Pour about 10 mL of aqueous ammonia into an Erlenmeyer flask and set this in the ice to use in Step 6.

After about 20 minutes, ask your professor to check that you have enough crystals formed.

5) Filter the crystals through filter paper in a Buchner funnel using a suction filtration apparatus.

6) Wash the crystals in two stages, first with ice-cold aqueous ammonia, and then with about 10 mL of ethanol.

7) Air-dry the crystals on the filter paper by continuing to draw air through them with the vacuum for 15 minutes.

8) On a balance, mass an empty petri dish _____g.

9) Transfer the dry crystals to the dish and remass _____g.

10) What is the mass of your experimental sample? _____g.

INTRODUCTION – Part II

One important property of transition metals is their ability to form a large number of coordination compounds. A coordination compound forms when a central metal atom or ion accepts pairs of electrons from Lewis bases to form a coordination sphere. The ions or molecules donating electron pairs are called ligands. The number of electron pairs accepted by the central metal atom or ion is its coordination number.

Many of the compounds and solutions of transition metals are highly colored. Let's consider why this is so. In the first-row transition elements, the five 3d orbitals are not equal in energy. For example, in the presence of atoms which donate electron pairs to form an octahedral complex, a splitting of energy levels (called crystal field splitting) occurs, producing 2 higher energy d orbitals and 3 lower energy d orbitals. When the d subshell is partially filled, an electron can be excited from a lower energy d orbital to a higher one on absorption of light from the visible region. This imparts the color we observe in these compounds. The size of the energy splitting depends on the atoms donating the electron pairs. Examples of colors are the light green Fe(II) in ferrous ammonium sulfate, characteristic of solutions containing $[\text{Fe}(\text{H}_2\text{O})_6]^{2+}$ and yellow Fe(III) solutions, containing a mixture of such ions as $[\text{Fe}(\text{H}_2\text{O})_6]^{3+}$ and $[\text{Fe}(\text{H}_2\text{O})_5(\text{OH})]^{2+}$. On the other hand, phosphate complexes of iron(III) solutions are generally colorless.

Characteristically, iron(III) complexes, like many Ni(II) complexes, have a coordination number of 6 ("take 6 bonds") and are octahedral in structure. The table, below summarizes the various molecular geometry shapes with their coordination number, hybridization and an example of each:

Molecular Geometry	Examples	Orbital Hybridization	Coordination Number
Linear	Acetylene (ethyne)	sp	2
Triangular (planar triangular; trigonal planar)	Ethylene (ethene)	sp ²	3
Tetrahedral	Methane	sp ³	4
Square planar	Pt, Pd, Ni	dsp ²	4
Trigonal bipyramidal	P ⁵⁺	dsp ³	5
Octahedral	S ⁶⁺	d ² sp ³	6

Ni²⁺ always has electrons in the higher 3d orbitals (Ni²⁺ has 8 d electrons) and will always make outer orbital octahedral complexes (utilizes the 4d subshell). The bonds of outer orbital complexes of Ni(II) are more polar, and therefore are more easily broken. Substitution reactions should take place much more readily. Coordinate covalent compounds of this type are said to be labile. We will explore this aspect of the [Ni(NH₃)₆]²⁺ ion in our experimental work.

Magnetic properties of complex ions

Different complexes may have in them a different number of unpaired electrons. Substances that have unpaired electrons in them are said to be paramagnetic. They will be strongly attracted to a magnetic field, primarily according to the number of unpaired electrons. The measure of the attraction to a magnetic field is called magnetic susceptibility.

Substances that do not have unpaired electrons are said to be diamagnetic. These substances don't interact strongly with a magnetic field, and are in fact slightly repelled by a magnetic field. As we discussed in lecture, we can determine the magnetic susceptibility of a compound using a Guoy (or magnetic susceptibility) balance. If the sample is paramagnetic, the sample is attracted to the electromagnet; if the sample is diamagnetic, the sample is repelled from the electromagnet.

From the information obtained from this experimental method, the number of unpaired electrons is determined. This is the way it works: the magnetic moment (μ) of a substance in units of Bohr magnetons is given by:

$$\mu = 2.84 (X_m T)^{1/2}$$

where X_m is the magnetic susceptibility and T is the Kelvin temperature. Another way to measure the magnetic susceptibility is to use a Magnetic Susceptibility Balance (MSB) and is the instrument we'll use in this experiment.

Just as in the case of the Guoy balance, the MSB employs a magnetic field to determine the interaction of the magnetic field with the sample. A sample is placed in a very carefully prepared tube and lowered into a magnetic field. The magnets which apply the field are suspended from a torsion wire. The stress the sample places on the magnets is reflected in the amount of twist placed on the torsion wire. The electronics of the instrument measure this and report out the result in magnetic susceptibility units.

It turns out that the magnetic moment depends on both the orbital and spin angular momentum of the atom and the interactions between them. For first row transition elements, however, the orbital

momentum makes a negligible contribution. That means we can express the magnetic moment, mathematically, in units of Bohr magnetons by

$$\mu = [n(n+2)]^{1/2}$$

where n is the number of unpaired electrons. Therefore, we can create a chart of expected values for a given number of unpaired electrons:

# of Unpaired Electrons	μ in Bohr magnetons
0	0
1	1.73
2	2.83
3	3.87
4	4.90
5	5.92
6	6.93
7	7.94

Magnetic moment of $\text{Ni}[(\text{NH}_3)_6]^{2+}$.

Obtain an already prepared sample of complex and a pestle and mortar. Thoroughly grind a sample of the crystals into a very fine, uniform powder. About 1 mL of solid sample is sufficient.

Obtain a sample tube – they are very expensive (about \$20 apiece), so handle them carefully. Carry it carefully by holding it with a strip of paper towel – do not touch it with your bare hands.

Take the empty tube to the MSB. The instrument should be on. Place the middle knob on the 10X setting. This means that the readout obtained must be multiplied by 10. Adjust the instrument with the zero knob until the readout says “000”. Place the empty tube into the instrument (located on top of the instrument just behind the bubble balance). Notice that the tube has a black band that allows the tube to carefully insert with light pressure only so far. Do not exert much effort to push the tube down into the compartment. When the reading settles down, **record the result as R_0** on your data sheet.

Expect the meter to say something like “-0006”. There is no decimal in the number. Record the results after multiplying by 10. Thus, you would record $R_0 = -60$.

Now mass the empty tube, carrying it with your paper towel strip – no oils from your hands must get on the tube. You can perform this by simply laying it on the pan or by placing it in a tared beaker. Record the empty mass of the tube in your data sheet.

Now introduce your sample into the tube using a semi-micro spatula and a small watch glass. Periodically tamp the sample down by tapping the bottom of the sample tube carefully/gently on the lab bench top. Repeat additions and tappings until the length of the sample in the tube is greater than 1.5 cm. When you have sufficient sample in the tube, and when you are convinced that it is uniformly packed down, remass the tube. Measure the length of the sample and record it on the data form (this is “ l ”).

Now go back to the MSB, readjust the zero if necessary, and place the tube with the sample in the sample holder. After the instrument settles down, record the reading, remembering to multiply the reading by 10. This value is called “R”.

Obtain the room temperature, record it and convert it to Kelvins.

Clean the tube out as carefully as you can by inverting and tapping on the lab bench top. Return the tube to your lab instructor without further cleaning.

Work up your data per the following.

CALCULATIONS

Magnetic Moment of Ni[(NH₃)₆]Cl₂	
Mass of MSB tube with Sample	g
Mass of MSB tube empty	g
Mass of sample (m)	g
Length of sample (ℓ)	cm
MSB reading empty tube, R _o	
MSB reading, tube with sample, R	
Room Temperature (convert to K)	K
Formula weight of Ni[(NH ₃) ₆]Cl ₂	g/mol

Using your data from above, and the formula, below, calculate (in the space below) the magnetic susceptibility (χ_m) of Ni[(NH₃)₆]Cl₂.

$$\chi_m = \frac{0.998(\ell)(R - R_o)(MW)}{10^9 (m)}$$

Your value of χ_m : _____

Using your value of the magnetic susceptibility, solve for the magnetic moment (μ) using the earlier equation and using the temperature in Kelvins that you calculated earlier:

$$\mu = 2.84 (X_m T)^{1/2}$$

Your value of μ : _____

Using the table in the introductory part of this experiment, how many unpaired electrons are in your sample of $\text{Ni}[(\text{NH}_3)_6]\text{Cl}_2$?

_____ unpaired electrons

Is your sample paramagnetic or diamagnetic? Why?

Calculate the theoretical yield of your product, which is the calculated mass of $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$ produced assuming that all of your $[\text{Ni}(\text{H}_2\text{O})_6]\text{Cl}_2$ was converted.

Calculate the percent yield of your product, which is (actual yield/theoretical yield) x 100%.