3—Synthesis of a Coordination Compound

Name: _____

Date: _____

Section: _____

Objectives
- Synthesize *tris*(ethylenediamine)nickel(II) chloride
- Practice writing balanced equations
- Apply stoichiometric principles to a chemical reaction
- Use balanced equations to predict products in chemical reactions
- Learn to identify the limiting reagent in a chemical reaction
- Calculate the theoretical yield and percent yield in a chemical reaction

Pre-Laboratory Requirements
- Read chapter 3.3-3.4 in Silberberg
- Watch the instructional video titled "Vacuum Filtration"
- Pre-lab questions (if required by your instructor)
- Laboratory notebook—prepared before lab (if required by your instructor)

Safety Notes
- Eye protection must be worn at all times.
- Ethylenediamine is a hazardous substance and should not come in contact with your skin or clothing. Do not breathe ethylenediamine vapors. Wear gloves when handling ethylenediamine. Wearing a lab coat or lab apron is recommended whenever you work with hazardous chemicals.
- Tris(ethylenediamine)nickel(II) chloride is a teratogen and an inhalation irritant. Wear gloves when handling this substance and do not inhale particles when transferring your product.

Discussion
Atoms combine with each other to form molecules in exact ratios. For instance, two hydrogen atoms combine with one oxygen atom to form one molecule of water. On the atomic scale, we never see an example of one and a half hydrogen atoms combining with an oxygen atom. This was one of the first observations of the early chemists who explored the properties of chemical elements. This observation is known as the atomic theory of matter.

Chemical equations provide a way to explain these observations. The balanced chemical equation for the formation of water from hydrogen and oxygen is:

$$2\ H_2 + O_2 \rightarrow 2\ H_2O$$
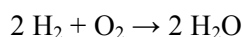
Notice that in this equation it was necessary to include the fact that hydrogen and oxygen are diatomic molecules at room temperature, which means each molecule of hydrogen, and also each molecule of oxygen, consists of two individual atoms. This is called a *balanced* chemical equation because the number of hydrogen atoms on the left

side of the arrow (and also the number of oxygen atoms on the left side of the arrow), equals the number of hydrogen atoms (and likewise the number of oxygen atoms), on the right side of the arrow.

Atoms are the smallest units of matter that retain chemical properties. Atoms are not visible under normal circumstances. Avogadro's hypothesis allows us to relate the number of atoms (or molecules) to the mass of a substance that can be weighed on an analytical balance. One molar mass of a substance contains Avogadro's number of particles (6.02×10^{23}), and would be described as one mole. For instance, the GFW of water, H_2O, is 18.016 ((2×1.008) + 16.00 = 18.016). One mole of water would therefore have a mass of 18.016 g and contain 6.02×10^{23} molecules of water. The mole (abbreviated mol) is the SI unit for the amount of a substance.

The number of moles of a substance is proportional to the number of molecules of that substance, providing a way to relate laboratory quantities to molecules in balanced reactions. You will use the GFW of your starting materials in this experiment to calculate the number of moles of each substance present in the reaction vessel. From the balanced equation you can predict the quantity of *tris*(ethylenediamine)nickel(II) chloride that would be expected if the reaction went to completion. This quantity is called the theoretical yield and may be expressed in either grams or moles. Since most chemical reactions do not go to completion, the amount of product formed is typically less than the theoretical yield. The actual yield divided by the theoretical yield is called the percent yield.

Coordination compounds form when transition metals bond with a small molecule (e.g., H_2O, NH_3 or CO_3^{2-}). When small molecules surround a larger metal atom in this fashion they are called ligands. Transition metals occupy the central portion of the periodic table and share the characteristic of having partially filled d orbitals. There are five different d orbitals, and the degeneracy of the energy levels is removed when transition metals form complexes with ligands. The energy difference between d orbitals in transition metal complexes is similar to the energy of photons in visible light. Many coordination compounds are therefore brightly colored because some of the photons from white light are adsorbed by d electrons, resulting in transmitted light that makes the complex appear colored.

White light is composed of blue, green, and red photons. A spectrum of the $NiCl_2 \cdot 6H_2O$ complex (the starting material for today's experiment) reveals that the nickel adsorbs blue and red photons, and solutions of this nickel material appear green. Replacing the water molecules in $NiCl_2 \cdot 6H_2O$ with ammonia (NH_3) changes the d orbital energy level for the central Ni^{2+} ion, causing solutions of the ammonium complex to adsorb green light, transmitting red and blue light. Ammonium complexes of Ni^{2+} are violet for this reason.

Figure 1. Hexaaquo nickel (II) ion (green) and hexaammonium nickel (II) ion (violet)

Ligands form a coordinate-covalent bond with nickel, where a non-bonded electron pair on the ligand donates two electrons to an empty orbital on the central nickel ion. The non-bonded electron pair comes from oxygen when the ligand is water and the non-bonded electron pair comes from nitrogen when the ligand is ammonia. The ligand used in today's experiment is ethylenediamine which also forms coordinate-covalent bonds with transition metals. One molecule of ethylenediamine contains two nitrogen atoms with non-bonded electron pairs. Therefore, only three ethylenediamine molecules are needed to fill the six available coordination sites on Ni^{2+}. Figure 2 shows the structure of the ammonia and ethylenediamine molecules.
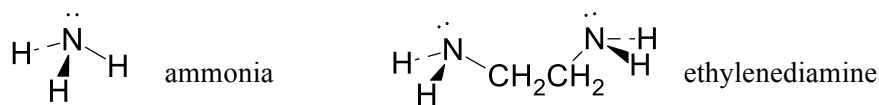

Figure 2.Structures of the ammonia molecule and the ethylenediamine molecule. Note that ethylenediamine contains two nitrogen atoms and will bond twice to a transition metal.

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Procedure

1. Weigh approximately 1.2 g of $NiCl_2 \cdot 6H_2O$ into a 100 mL beaker (Record the exact mass).
2. Add 5 mL of deionized water, and stir to dissolve the $NiCl_2 \cdot 6H_2O$
3. Add approximately 8 mL of 4.0 <u>M</u> ethylenediamine to the beaker (Record the exact volume used) and stir well.
4. Add 30 mL of acetone in 10 mL increments, stirring after each addition (The product should precipitate out of solution).
5. Cool the beaker in an ice bath to maximize precipitation, and try scratching the wall of the beaker to initiate precipitation.
6. Vacuum filter the product. Wash with acetone. **DO NOT USE WATER-**the product is water-soluble and your product will be lost if you add water.
   NOTE: Remember to pre-weigh petri dish <u>AND</u> filter paper.
7. Dry under vacuum filter until product is crystalline (**4-5 minutes).**
8. Transfer the product and filter paper to a pre-weighed petri dish and air dry the product for 5 minutes in the fume hood.
9. Dry in oven at ~60 ^{o}C for 10 minutes or until product is completely dry.
10. Allow the petri dish to cool and then weigh the petri dish, product, and filter paper.
11. Deposit the product in the waste container labeled "Solid *tris*(ethylenediamine)nickel(II) chloride."

Data Table

Mass $NiCl_2 \cdot 6H_2O$ (g)	_____
Color	_____
Volume $NH_2CH_2CH_2NH_2$ (mL)	_____
Concentration of $NH_2CH_2CH_2NH_2$ (mol/L)	_____
Mass $Ni(NH_2CH_2CH_2NH_2)_3Cl_2$ + petri dish + filter paper (g)	_____
Mass petri dish + filter paper (g)	_____
Mass $Ni(NH_2CH_2CH_2NH_2)_3Cl_2$ (g)	_____
Color	_____

Results Table

Moles $NiCl_2 \cdot 6H_2O$ used	_____
Moles $NH_2CH_2CH_2NH_2$ used	_____
Moles $Ni(NH_2CH_2CH_2NH_2)_3Cl_2$ produced	_____
Limiting reagent	_____
Theoretical yield (based on the limiting reagent) (g)	_____
Actual yield (g)	_____
% Yield	_____

Chemical Equations

Calculations