The Most Cost-Efficient Synthesis of a Pigment

by J. Gutow 1/06

Introduction

Your team works for a company that makes pigments for paints and coatings. The prices of the raw materials for your flagship pigment recently changed dramatically. Your supervisor wants you to reevaluate the synthesis used for the pigment to make sure that as much of the expensive reagent is converted to product as is possible. Unlike your previous boss, this one does not let the research teams design the experiments. However, she does expect the teams to suggest modifications to the experiments based on their experience in the laboratory. She expects the modified experiments to be carried out only after her approval (pretend your instructor is your supervisor). She expects the research team to decide how many duplicate experiments to do to verify reproducibility and to decide if any data should be thrown out because of experimental errors. She expects written documentation describing the errors which justify exclusion of any data. Below are her instructions for the experiment.

Techniques

Vacuum filtration for quantitative analysis of mass collected. Reading in *Cooperative Chemistry* pp. 70,74-75.

General Information

Table 1: Costs of reagents per gram.

Nickle Chloride hexahydrate $NiCl_2{\bullet}6H_2O$	Dimethylglyoxime DMG	water and ethanol (solvents)
7.38 ¢	19.2 ¢	essentially free

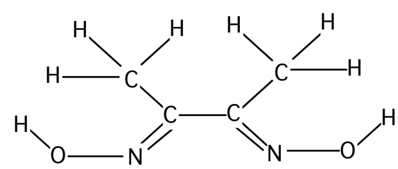

Figure 1: Structure of Dimethylglyoxime. Note that the H's on the O's are acidic and are easily removed.

$$2C_4H_7N_2O_2H(aq) + Ni^{2+}(aq) \longrightarrow Ni(C_4H_7N_2O_2)_2(s) + 2H^+(aq)$$

Figure 2: Net ionic reaction (The Cl^- ions that came with the Ni^{2+} have been left out). Notice that each DMG molecule loses one H^+ in the reaction to become a negative ion.

Handling and Disposal Procedures

•Wear gloves. Ni compounds are suspect carcinogens and DMG is toxic.
•All leftover solutions should be put in the labeled waste bottles for disposal with the wastes from the manufacturing floor.
•All contaminated (used) filter paper should be collected in the labeled waste bin.
•The product powder should be collected for future use in the labeled bin. You will get to use it as a pigment in paint you will make next semester.

Present Synthesis

Stock solutions(available from the manufacturing floor):

1.000 g $NiCl_2{\bullet}6H_2O$ per 100.0 mL of solution in H_2O
1.000 g DMG per 100.0 mL of solution in ethanol.
Concentrated ammonia (about 17.4 Molar)

Solutions are mixed by volume in a ratio of 1 part Ni solution to 2 parts DMG solution and stirred for 2 minutes by hand to mix. Two to three drops of concentrated ammonia are added per 20.00 mL of DMG solution. The mixture is stirred for 15 seconds. Then a moistened piece of red litmus is held just above the solution. If the litmus does not turn blue more ammonia is added in 2-3 drop steps until moist litmus above the solution turns blue. This indicates the solution is basic. The solid is collected by vacuum filtration on paper filter disks and allowed to dry at room temperature for one week. When a customer is anxious it can be dried overnight using an oven set to 100 ºC, but we only have facilities for very small batches.

Experiments to Perform

****No run of any experiment should use more than 0.500 g of the $NiCl_2•6H_2O$ or 0.200 g of the DMG. Previous testing always used 20.00 mL of the DMG solution as that produces reasonable amounts of solid. USE THE SAME AMOUNT OF DMG SOLUTION FOR EACH RUN. Do reactions in 100 mL beakers. Stir with glass stirring rods.******

1) Duplicate the present synthesis. Calculate what should be the limiting reagent and how much product you expect to get. Calculate the percentage yield (what percent of the maximum possible mass of product you actually get). Calculate the fraction of the DMG (the expensive stuff) converted to product.

2) Do the synthesis with a volume ratio of 3-to-2. Calculate what should be the limiting reagent and how much product you expect to get. Calculate the percentage yield (what percent of the maximum possible mass of product you actually get). Calculate the fraction of the DMG (the expensive stuff) converted to product.

3) Do the synthesis with a volume ratio of 2 parts Ni solution to 1 part DMG solution. Calculate what should be the limiting reagent and how much product you expect to get. Calculate the percentage yield (what percent of the maximum possible mass of product you actually get). Calculate the fraction of the DMG (the expensive stuff) converted to product.

Prelab

Prelab calculations marked with a # count as example calculations for data that belongs in the data tables on the data sheets. Copy the results of calculations marked with # to the appropriate box in the data sheet tables. This prelab will be checked off by your instructor, but since you may want it until you finish the lab it should be turned in with the data sheets, your written introduction and discussion/conclusion after the experiment is complete.

1) Show how to calculate the molecular weight* of DMG . You should get: 116.120 g/mol.
*Molecular weight = mass of 1 molecule in amu and is numerically the same as the mass of 1 mole in grams (also called the molar mass, see appendix 1 and text pp 175-177 for example calculations).

2) Show how to calculate the molecular weight of $NiCl_2{\bullet}6H_2O$. You should get: 237.691 g/mol.

3) Show how to calculate the molecular weight of $Ni(C_4H_7N_2O_2)_2$. You should get: 288.896 g/mol.

4) You will be using 20.00 mL of DMG solution. Calculate:

#a) g DMG in solutions (g/20.00 mL):

#b) moles DMG in solutions (mol/20.00 mL, see appendix 2):

5) Experiment 1:

#a) The ratio of solutions is supposed to be 1:2 Ni solution to DMG solution by volume. Since we are using 20.00 mL of DMG solution how many mL of Ni solution do we need?

#b) Calculate g $NiCl_2 \cdot 6H_2O$ to be used in experiment 1:

#c) Calculate moles $NiCl_2 \cdot 6H_2O$ used in experiment 1:

#d) Looking at the reaction:

$2C_4H_7N_2O_2H(aq) + Ni^{2+}(aq) \longrightarrow Ni(C_4H_7N_2O_2)_2(s) + 2H^+(aq)$

we see that 2 DMG are used for each Ni^{2+} used. Given this information and assuming the reaction continues until you run out of one reagent, will the Ni^{2+} or the DMG be used up? How many moles of product will you get?

5) Experiment 2:

#a) The ratio of solutions is supposed to be 3:2 Ni solution to DMG solution by volume. Since we are using 20.00 mL of DMG solution how many mL of Ni solution do we need?

#b) Calculate g $NiCl_2•6H_2O$ to be used in experiment 2:

#c) Calculate moles $NiCl_2•6H_2O$ used in experiment 2:

#d) Assuming the reaction continues until you run out of one reagent, will the Ni^{2+} or the DMG be used up? How many moles of product will you get?

6) Experiment 3:

#a) The ratio of solutions is supposed to be 2:1 Ni solution to DMG solution by volume. Since we are using 20.00 mL of DMG solution how many mL of Ni solution do we need?

#b) Calculate g $NiCl_2•6H_2O$ to be used in experiment 3:

#c) Calculate moles $NiCl_2•6H_2O$ used in experiment 3:

#d) Assuming the reaction continues until you run out of one reagent, will the Ni^{2+} or the DMG be used up? How many moles of product will you get?

Data Sheets

Observations and measurements belong in your lab notebook. The notebook is also a good place to do rough drafts of any calculations you do in lab. Copy appropriate measurements into the tables.

Table 1: 1 part Ni solution to 2 parts DMG solution.

	Run 1	Run 2	Run 3	Average
mL DMG sol'n				
g DMG				
moles DMG				
mL Ni sol'n				
g $NiCl_2 \bullet 6H_2O$				
moles $NiCl_2 \bullet 6H_2O$				
g $Ni(DMG)_2$				
moles $Ni(DMG)_2$				
moles $Ni(DMG)_2$ expected				
% yield (see appendix 3)				
moles DMG used				
Fraction of DMG used				

Example calculation of moles $Ni(DMG)_2$. All other calculations of moles $Ni(DMG)_2$ should be in lab notebook.

Example calculation of % yield. All other calculations of % yield should be in lab notebook.

Example calculation of moles DMG used. All other calculations of moles DMG used should be in lab notebook.

Example calculation of fraction of DMG used. All other calculations of fraction of DMG used should be in lab notebook.

If a run was not used in the average specify why.

Data Sheets

Table 2: 3 part Ni solution to 2 parts DMG solution.

	Run 1	Run 2	Run 3	Average
mL DMG sol'n				
g DMG				
moles DMG				
mL Ni sol'n				
g $NiCl_2 \cdot 6H_2O$				
moles $NiCl_2 \cdot 6H_2O$				
g $Ni(DMG)_2$				
moles $Ni(DMG)_2$				
moles $Ni(DMG)_2$ expected				
% yield (see appendix 3)				
moles DMG used				
Fraction of DMG used				

If a run was not used in the average specify why.

Table 3: 2 part Ni solution to 1 parts DMG solution.

	Run 1	Run 2	Run 3	Average
mL DMG sol'n				
g DMG				
moles DMG				
mL Ni sol'n				
g $NiCl_2 \cdot 6H_2O$				
moles $NiCl_2 \cdot 6H_2O$				
g $Ni(DMG)_2$				
moles $Ni(DMG)_2$				
moles $Ni(DMG)_2$ expected				
% yield (see appendix 3)				
moles DMG used				
Fraction of DMG used				

If a run was not used in the average specify why.

Appendix 1: Primer on Calculating Molecular Weight (Molar Mass).

1) Look at the molecular formula and list the number of each type of atom.

Example $C_2H_5N(C_3H_7)_2$.

\# of C = 2 + 2*3 = 8 # of H = 5 + 2*7 = 19 # of N = 1

2) Look in the periodic table for the average mass of each kind of atom. This is the number at the bottom of the box. It has units of amu (atomic mass units) or g/mole.

C: 12.011 H: 1.0079 N: 14.007

3) Multiply these masses by the number of each type of atom and sum them together to get the final result. Because the number of atoms is a counting number they have an infinite number of significant digits. The significant digits are determined by how well you know the average atomic masses.

8 C = 8*12.011 = 96.088
19 H = 19 *1.0079 = 19.150
1 N = 1*14.007 = 14.007

96.088 g/mol
19.150 g/mol
+ 14.007 g/mol
129.245 g $C_8H_{19}N$/mol $C_8H_{19}N$

4) If you have waters of hydration as in $CoCl_6$•$6H_2O$ you have to include the mass of the 12 H and 6 O in your calculation.

1 Co = 1*58.933 = 58.933
6 Cl = 6* 35.453 = 212.72
12 H = 12*1.0079 = 12.095
2 O = 2*15.999

58.933 g/mol
212.72 g/mol
12.095 g/mol
+ 31.998 g/mol
315.74 g $CoCl_6$•$6H_2O$/mol $CoCl_6$•$6H_2O$

Appendix 2: Primer on Calculating Number of Moles in a Sample.

1) Note that the units on molar mass (molecular weight) are g/mol. This means it is a ratio between grams and moles and thus can be used to perform the unit conversion between the two.

2) Example 1: Given 150.0 g of $CoCl_6$•$6H_2O$ how many moles do you have?

Conversion map: g ——> moles (implies need to divide by g and multiply by moles).

$$(150.0\ g\ CoCl_6 \cdot 6H_2O)\left(\frac{mole\ CoCl_6 \cdot 6H_2O}{315.74\ g\ CoCl_6 \cdot 6H_2O}\right) = 0.4751\ mol\ CoCl_6 \cdot 6H_2O$$

3) Example 2: Given 1.4 moles of $C_8H_{19}N$ how many grams do you have?

Conversion map: moles ——> grams (implies need to divide by moles and multiply by grams).

$$(1.4\ mol\ C_8H_{19}N)\left(\frac{129.245\ g\ C_8H_{19}N}{mol\ C_8H_{19}N}\right) = 1.8 \times 10^2 g\ C_8H_{19}N$$

Appendix 3: % yield

% yield = 100%*(amount collected)/(amount expected)