Cooperative Project II: Identification, Properties and Synthesis of an Unknown Ionic Compound

For cooperative project II, we will follow the lab on page 115 of the *Cooperative Chemistry Laboratory Manual, 5th edition*. There will be additional requirements to this lab, which are stated in this document. This lab will be a two part lab. In the first part of the lab, each group will synthesize 0.5 g calcium carbonate. The second part of the lab will require each group to: 1. Investigate the chemical hazards of some of the chemicals you will use in this lab; 2. identify an unknown ionic compound; 3. investigate the chemical and physical properties of the compound; 4. devise two syntheses of the compound and compare them for cost effectiveness, safety and potential yield. This aspect of the project will not be carried out in lab, but will be included in your laboratory report

There will be two prelab quizzes for this lab. The prelab quiz questions will test your overall knowledge of the key concepts of the lab by focusing on questions about the theory of the experiment, experimental procedures, the substances being used and/or the calculations involved in the lab. Before coming to lab, be sure to read all background reading assignments and complete all practice problems. Quiz dates can be found on the course schedule.

Requirements for Part 1:

- As a group, you will need to synthesize about 0.5 g of calcium carbonate by reacting 1 M aqueous solutions of calcium chloride and sodium carbonate.
- Each group will prepare 25.00 mL of 1M aqueous solutions of calcium chloride and sodium carbonate. This is a quantitative experiment, so keep the following in mind:
    1. It is not important that your solutions are exactly 1M, but it is important that you know the exact concentration of the solutions your group prepares. Therefore, you will need to measure the mass of calcium chloride and sodium carbonate to three decimal places and record volumes to the correct number of significant figures.
    2. Be sure to use proper technique (including the proper glassware) for preparing aqueous solutions of known concentration.
    3. Calculate the concentrations of the prepared solutions to the correct number of significant figures.
- Determine how much of each solution is needed to prepare 0.5 g of calcium carbonate. Be sure to use the proper technique for transferring your aqueous solutions to a reaction container.
- Calculate your percent yield and include these calculations in your results and discussion section of your lab report. Report all values to the correct number of significant figures with the correct units.
- Be sure to write the complete balanced chemical equation, the complete ionic equation and the net ionic equation before beginning your lab work.

Requirements for Part 2:

- Consult the appropriate material safety data sheet (MSDS) and find the LD_{50} or LC_{50} value for hydrochloric acid, sulfuric acid and acetic acid and include these values in your lab report.
- Identify the unknown compound. You will only be given 5 grams of the unknown compound.
- Discover as many chemical and physical properties of the compound as you can.
- Devise two syntheses of the compound and compare them for cost effectiveness, safety and potential yield. Be sure to reference all data you obtain from outside sources. The safety analysis must include the LD_{50} for each of the reactants and products in each of your devised reactions.
- Below are guidelines for referencing data from different scientific sources.

Reference Formats:

Reference format for referencing a journal article:

- Author, A. A; Author, B. B; Author, C. C. Title of Article. *Journal Abbreviation (italics)* [Online if online] **Year (boldface)**, *Volume (italics)*, Pagination.
- Example:
    - Slunt, K. M.; Giancarlo, L. C. Student-Centered Learning: A Comparison of Two Different Methods of Instruction. *J. Chem. Educ*. **2004**, *81*, pp 985-988.

- Reference format for referencing a scientific handbook:
    - Editor, A. A., Editor, B. B., Eds. *Handbook Title (italics)*, Edition number [Online if online]; Publisher: Place of Publication, Year; Pagination or other identifying information.
    - Example:
        - Lide, D. R., Ed. *CRC Handbook of Chemistry and Physics*, 84th ed. [Online]; CRC Press: Boca Raton, FL, 2003; p 83.
- Reference format for referencing an MSDS:
    - **Hard copy (paper) MSDS**
        - *Titanium Dioxide*; MSDS No. T3627; Mallinckrodt Baker: Phillipsburg, NJ, November 12, 2003.
    - **MSDS obtained from an Internet search**
        - *Titanium Dioxide*; MSDS No. T3627; Mallinckrodt Baker: Phillipsburg, NJ, November 12, 2003. http://www.jtbaker.com/msds/englishhtml/t3627.htm (accessed 4/15/04).

Requirements for keeping a laboratory notebook:

- Each member of your group is required to keep a scientific notebook. Your notebook is where you will record your day-to-day activities in the lab. You should describe experiments as you do them and note observations as you make them. Record and analyze your data in your notebook.
- The notebook must be a carbonless, duplicate page notebook.
- Be sure to keep the following in mind when recording data in your notebook:
    - Include a descriptive "Table of Contents"
    - Pages must be numbered
    - Include date data was collected
    - Record raw data
    - Record procedures and observations
    - Write in ink
    - Mistakes must be crossed out appropriately (single line through the mistake with your initials)

Requirements for writing weekly summaries:

- List all group members present for the lab
- Give a short overview of the lab
- Present results in tables and/or graphs
- Discuss future work

Requirements for writing your lab report:

- Although you collected data in a group, **the lab report is an individual project.**
- Often scientific data is reported in tabular format. This provides the reader with a quick and easily readable format for obtaining information. If appropriate, you should report your data in a tabular format. (Use your textbook as a reference to see the proper format for a scientific table.)
- In the results and discussion section of your lab report, include the complete balanced equation, the complete ionic equation and the net ionic equation for the reaction between aqueous solutions of calcium chloride and sodium carbonate. Be sure to include the physical state of each of the reactants and products.
- All lab reports must be typed including all tables.

Grading Cooperative Project II:

- Prelab Quizzes (20 pts)
- Notebook (10 pts)
- Two weekly summaries (5 pts each)
- Project Quiz on Projects I and II (20 pts)

- Peer Evaluation (5 pts)
- Lab Report (25 pts)
- Results and Discussion
    - Is data reported in tables?
    - Is data reported in the proper scientific format?
    - Include the complete balanced equation, the complete ionic equation and the net ionic equation for the synthesis of calcium carbonate.
    - Include calculations for determining the percent yield for the calcium carbonate synthesis.
    - Include the complete balanced equation, the complete ionic equation and the net ionic equation for your two devised synthesis reactions.
    - Include the safety analysis and the cost analysis for your devised synthesis reactions.

Safety Precautions:

- **Bunsen Burners:** The Bunsen burner is a common device used for heating. It is limited in usefulness to warming aqueous solutions or other nonflammable substances. Care must be used when using a Bunsen burner. Your laboratory instructor will demonstrate the proper method for adjusting, lighting, and using a Bunsen burner.
- Wear your safety goggles at all times. You will be using several different acid, base and salt solutions. If you get any solution on you, wash immediately with lots of water.
- Dispose of all waste in the labeled hazardous waste container in the common equipment area. Use a wash bottle to rinse glassware into the container.
- You will use several different reagents throughout this experiment. Many of the reagents are located in the common reagent area in the front of the lab. When you need a reagent, take it to your lab bench, use it, put the lid back on it and then put it right back. Do not carry open bottles of solutions to the reagent bench (poor etiquette). Be sure you return it to its proper place.
- Never pour anything back into the stock solution.
- Never lay reagent bottle stoppers or caps on the lab bench. The entire reagent may become contaminated. In addition, the residue on the bench may be hazardous and linger for days or weeks. This could injure someone well after the fact. Hold the stopper in your other hand while you get the material out of the bottle. Replace stoppers immediately and completely.
- **Centrifuge safety:** Do not open the centrifuge until it has completely stopped. Do not try to manually stop the centrifuge from spinning. The blank tube filled with water is necessary to balance the instrument. Do not walk away from a spinning centrifuge.
- Balances are especially sensitive, expensive devices. **Never weigh chemicals directly on the pan**. Use weighing paper or boat or a container such as a beaker or flask. Remove the container from the balance, add the chemical and then replace the container. If you spill anything onto the balance, please notify the instructor **immediately**.

Background Reading and Practice Problems:

Cooperative Chemistry Laboratory Manual

- Safety Rules, Laboratory Etiquette (12-14)
- NFPA Hazard Codes, Waste, MSDS's (15-16)
- Recording and Reporting Results (17-19)
- Writing Lab Reports (19-23)
- Reporting Numerical Results, Significant Figures, Graphs (35-40)
- Measuring Devices (47-49)
- To Make up a Solution of Known Concentration (64)
- Bunsen Burner (53)
- Laboratory Techniques (55-63)
- Filtration (70)
- Identification, Properties and Synthesis of an Unknown Ionic Compound (p. 115)

General Chemistry, 4th Edition by McQuarrie

- Molarity: Read Section 12-2 (395-399); Complete Practice Problem 12-1, End of Chapter Problems 12.2, 12.10
- Precipitation Reactions: Read Section 10-9, (328-332); Complete Practice Problems 10-13, 10-14, 10-15, and End of Chapter Problems 10-52
- Reaction Stoichiometry: Read Section 12-5 (405-408); Complete Practice Problem 12-7, 12-8 and End of Chapter Problems 12.18, 12.22

Additional Background Information: Chemical analysis can be divided according to quantitative or qualitative analysis. In the last experiment, we were asked to quantitatively find the density of various materials. However, sometimes a chemist is interested in what species are present in a sample, as opposed to the amount of species present in a sample. Thus, a qualitative analysis involves the determination of the types (not amounts) of ions present in a solution. A "qualitative analysis scheme" is then a systematic procedure for separating and identifying various ions present in an aqueous solution.

- The procedures in most qualitative analysis experiments involve the use of using **precipitation reactions**. A precipitation reaction is a reaction in which an insoluble solid compound is formed when solutions of two soluble compounds are mixed. The solid compound that forms during a precipitation reaction is referred to as the **precipitate**. An example of a precipitation reaction is the reaction between aqueous solutions of KI and $Pb(NO_3)_2$:

$$2KI\,(aq) + Pb(NO_3)_2\,(aq) \rightarrow PbI_2\,(s) + 2KNO_3\,(aq)$$

- We know that the ionic compounds KI, $Pb(NO_3)_2$, and KNO_3 are soluble in water and PbI_2 is not soluble in water, see "General Solubility Guidelines of Ionic Compounds in Water" on page 60 of the *Cooperative Chemistry Laboratory Manual*.
- There are some general procedures in qualitative analysis that will be used throughout this experiment. One is the use of a centrifuge to aid the separation of a precipitate from a solution in a test tube. There is a centrifuge at the end of each lab bench. To use the centrifuge, make sure the test tube containing the precipitate and solution (sample test tube) is not overly full. Place the sample test tube in one of the centrifuge tubes. Place a blank tube containing the same amount of water as you have in your sample test tube in the centrifuge tube that is opposite your sample test tube. Turn on the centrifuge and allow it spin for about 1 minute. Once you have centrifuged your sample test tube, now you can **decant** the supernatant liquid from the precipitate. Simply pour off (decant) the liquid that is above the precipitate (the supernatant).

Qualitative Procedure for the Analysis of Ca^{2+} and Mg^{2+} Ions

1. Obtain 1 mL (approximately 20 drops) of the solution to be analyzed in a small test tube.

2. Add 2 drops of 6M HNO_3 and then add 1 mL of 6M NH_4OH.

3. Add 1 mL of 0.3 M $(NH_4)_2CO_3$ and stir. If a precipitate forms, Ca^{+2} ions are present. Centrifuge and decant the supernatant, which may contain Mg^{+2}. Save this solution for analysis in step 4. If no precipitate forms upon the addition of the 0.3 M $(NH_4)_2CO_3$, Ca^{2+} are not present and you should continue with step 4.

4. To the solution from step 3, add 1mL of K_2HPO_4 solution, stir and let stand 1 minute. The slow formation of a **white** precipitate indicates **magnesium**.