Lab 3: Limiting and Excess Reagents

Purpose:

- Understand, and apply, the concepts of limiting and excess reagents using the following reaction:

$$Cu(NO_3)_{2(aq)} + KI_{(aq)} \rightarrow CuI_{(s)} + I_{2(aq)} + KNO_{3(aq)}$$

Focus on:

- Stoichiometry, limiting and excess reagents
- Precipitate and Supernatant
- Percent Yield and Percent Atom Economy
- Centrifuge and Desiccator

Pre-lab work:

- Prepare your lab notebook as usual.
- Answer the prelab questions on Moodle.

Materials:

- 6 centrifuge test tubes
- Test tube rack
- Clamps or tongs
- Kimwipes
- 2 x 10 mL labelled graduated cylinders
- 1x 100 mL beaker
- Disposable transfer pipettes
- Drying oven (set at 90°C)
- Centrifuge
- Dessicator
- Permanent marker (provided by student)
- 0.001 g scale balance

Chemicals:

- Dropper bottle of 0.50 M Copper (II) nitrate
- Dropper bottle of 0.50 M Potassium iodide
- Methanol

Procedure:

Before starting this lab, identify a 100 mL beaker with your name using your own permanent marker. Place the empty beaker into the drying oven (to be used later).

Part 1: Precipitation Reaction

1. You will be assigned volumes of $Cu(NO_3)_2$ and KI solutions.
2. Label six centrifuge test tubes:
    - 1, 1a, 1b
    - 2, 2a, 2b
3. Wearing gloves and using a Kimwipe, wipe off any fingerprints, or other smudges, from test tubes labelled 1 and 2. Weigh them carefully to the nearest 0.001 g. This does not have to be done for the other test tubes because they will not be weighed.
4. Get the 10 mL graduated cylinders labelled: $Cu(NO_3)_2$ and KI.
5. Measure the assigned volume of 0.50M $Cu(NO_3)_2$ in the appropriately labeled graduated cylinder. Measure the assigned volume of 0.50M KI in the other appropriately labeled graduated cylinder.
6. Add these assigned volumes to test tube 1.
7. Repeat steps 5 and 6 for test tube 2 because it will be used as a duplicate.
8. Record any observations.
9. To thoroughly mix these reactants, hold the top of the test tube gently but firmly between the thumb and forefinger of one hand. Also, gently tap the bottom of the test tube.
10. Set these test tubes in the test tube rack for 5 minutes to allow the reaction to go to completion.
11. Centrifuge these test tubes to collect the solid. (The test tubes do not need a cap). Proper use of a centrifuge will be demonstrated. Here are the steps:
    a. Each centrifuge can accommodate more than two test tubes. The experiment will go faster if several groups use the centrifuge at the same time.
    b. Label your test tubes with your initials and properly place them directly opposite each other in the holders of the centrifuge.

c. Turn on the machine and let the test tubes spin for 5 min. NOTE: *Turn off the centrifuge immediately if there is an unusually loud noise or movement of the centrifuge. This may indicate that the tubes are off balance. Reset the test tubes.*
d. After 5 minutes, turn off the centrifuge and wait until the rotor has completely stopped.
e. Remove the test tubes and put them in the test tube rack. They should contain a clear brown-green colored liquid - called the **supernatant** - and a gray solid at the bottom, which is the **precipitate**.

12. Use two disposable pipettes to draw off the supernatant. Squeeze the air out of the pipette before putting it into the liquid and then carefully release to draw up the supernatant. It is not essential that every drop of the supernatant is removed. However, do not disturb the precipitate.
13. Split the supernatant from test tube 1 - as equally as possible - between the test tubes labeled 1a and 1b. Discard the pipette as instructed.
14. With the second disposable pipette, repeat steps 11 and 12 using test tubes 2, 2a, and 2b.
15. NOTE: Test tubes 1a, 1b, 2a, and 2b contain the supernatant, which is either $Cu(NO_3)_2$ *or* KI. They will be tested later in this lab, so do NOT throw them away.
16. Test tubes 1 and 2 contain the precipitate. To remove any unreacted reagent, the precipitate needs to be washed with methanol. Methanol is used because it is more **volatile** than water, which means it evaporates quickly and allows the precipitate to dry faster.

    Follow these steps:

    a. Add enough methanol to fill each test tube half-way. Use the same amount for each test tube.
    b. Using a disposable pipette, squeeze the bulb and lower the tip into the liquid. Suck up a small amount of methanol and squirt it against the precipitate so that the precipitate goes back into suspension. Repeat as many times as needed so that no precipitate remains on the sides of the tube.

17. Spin the test tubes again in the centrifuge for 5 minutes. Remove the supernatant with a disposable pipette without disturbing the precipitate. Since this supernatant will not be tested, you can use the same pipette for both test tubes. Properly discard the supernatant.
18. To dry the precipitate, place test tubes 1 and 2 into the 100 mL beaker that was in the oven. Put this beaker back into the oven for at least 15 minutes. Allow the test tubes to cool before weighing.

Part 2: Testing the Supernatant

19. While test tubes 1 and 2 are in the oven,
    a. Add 1 mL (~20 drops) of $Cu(NO_3)_2$ to test tubes 1a and 2a. Record observations.
    b. Add 1 mL (~20 drops) of KI to test tubes 1b and 2b. Record observations.

Part 3: Weigh the Precipitate

20. Remove test tubes 1 and 2 after they have been in the oven.
21. If necessary, place them in a desiccator to cool for at least 10 minutes.
22. Weigh each test tube to the nearest 0.001 g. Record.
23. Calculate the mass of precipitate for each test tube.
24. Clean up as instructed.

Follow-Up Work:

- Each student is to complete and submit the follow-up work *individually*.
- The following is an **overview** of what is required. Evaluation will be based on the detailed "Follow-Up Guidelines" available on Moodle.
- Complete the following:
    - Title page
        - Include the letter of your assigned volumes (ex. A, B, etc.)
    - Purpose
    - Results
    - Analysis
        - Include the balanced chemical equation for this experiment.
        - State which reactant was - *theoretically* and *experimentally* – limiting. Support your answer.
        - State the amount of excess reagent that remains after the reaction is complete.
        - What is the Percent Yield of the product?
        - Percent yield is usually less than 100%. Give at least two reasons why and include proper citations.
        - Calculate the Percent Atom Economy for this reaction.
        - Compare and analyze the results of Percent Atom Economy and Percent Yield for this reaction.
    - Reference
        - Use and include at least 2 proper references (excluding class notes and the etext).
    - Appendix

Due date: Submit your Follow-Up as a SINGLE PDF in the appropriate folder on Moodle by the due date and time indicated by your instructor.