Determination of the Chemical Formula of a Compound

Last revised: 2024/09/08

Preparation

- Put on your lab coat and safety goggles
- Turn off your mobile phone
- Place your backpack in the drawer or the cabinet
- Put your prelab on the lab bench for ATA to sign
- Hand in your *Lab Safety Certification*

Collect the following items

- ❑ Put one large test tube + one drying tube into the oven
- ❑ 250 mL Erlenmeyer flask, thistle tube, rubber tube, and alcohol burner
- ❑ Matches, windshield

Objective and Principles

- **Objective:** Determine the empirical formula of copper oxide (CuO_x) by the elemental analysis method

- **Lab techniques:**
  - Using an analytical balance to weigh chemicals
  - Producing and collecting hydrogen gas over water
  - Using an alcohol burner

H_2 is explosive when mixed with O_2, so controlling the reaction atmosphere is very crucial

- **Chemical reactions**

$$Zn(s) + 2\ HCl(aq) \rightarrow ZnCl_2(aq) + \mathbf{H_2(g)}$$

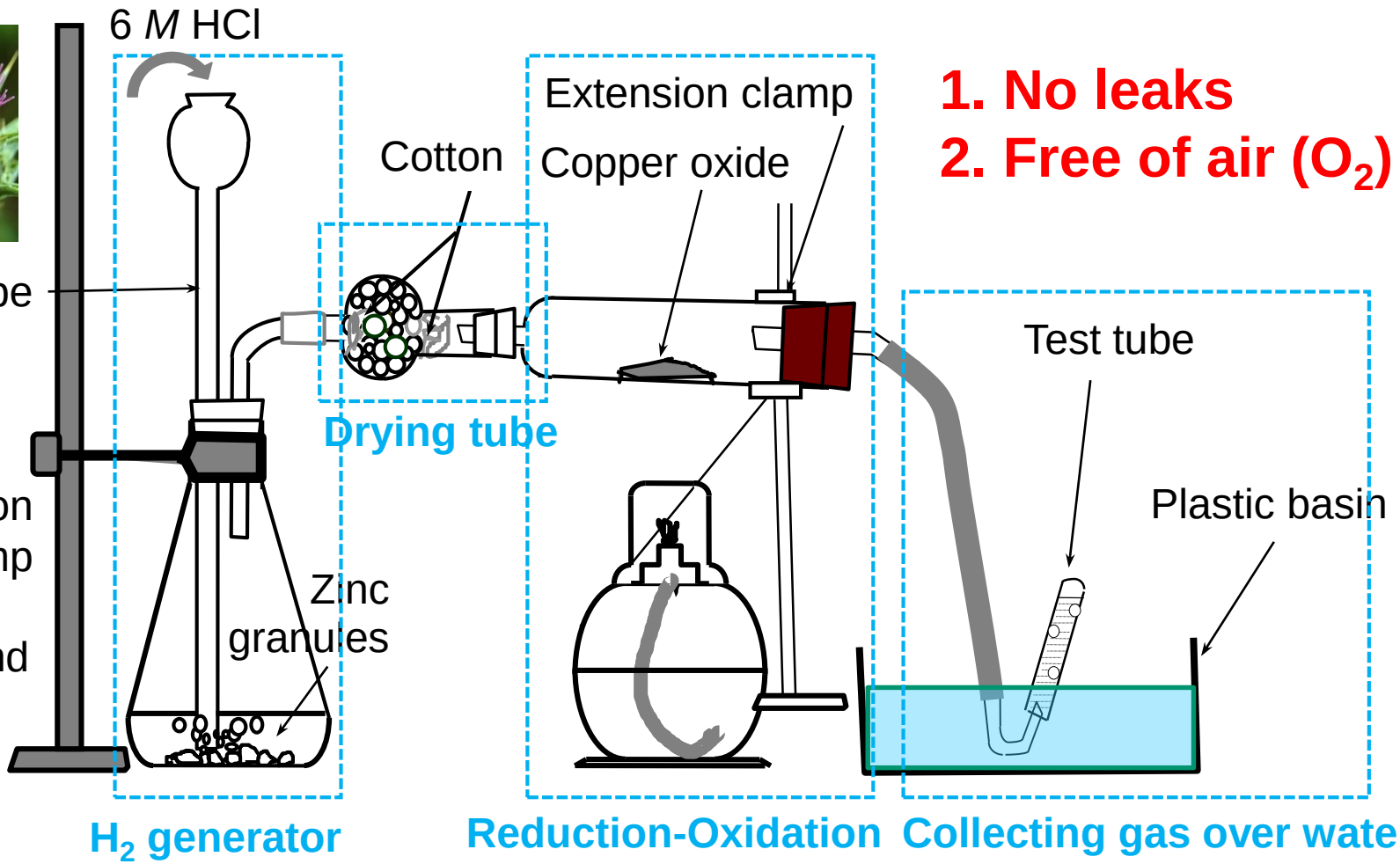
$$CuO_x(s) + x\ \mathbf{H_2(g)} \xrightarrow{\Delta} Cu(s) + x\ \mathbf{H_2O(g)}$$

x = ?

Reactant (black powder)

Product (red powder)

Experimental Setup



1. No leaks

2. Free of air (O_2)

Do not remove the extension clamp from the stand on your lab bench

Step 1/9: Fill the Drying Tube

- Use an iron wire to place small pieces of cotton wool on both ends of the drying tube to keep $CaCl_2$ from falling out
- Fill $CaCl_2$ into the drying tube above a plastic bin (use the provided plastic funnel and work neatly)
- Do not pack cotton wool and $CaCl_2$ too tightly, or the gas flow may be obstructed
- Cap the $CaCl_2$ bottle immediately after use



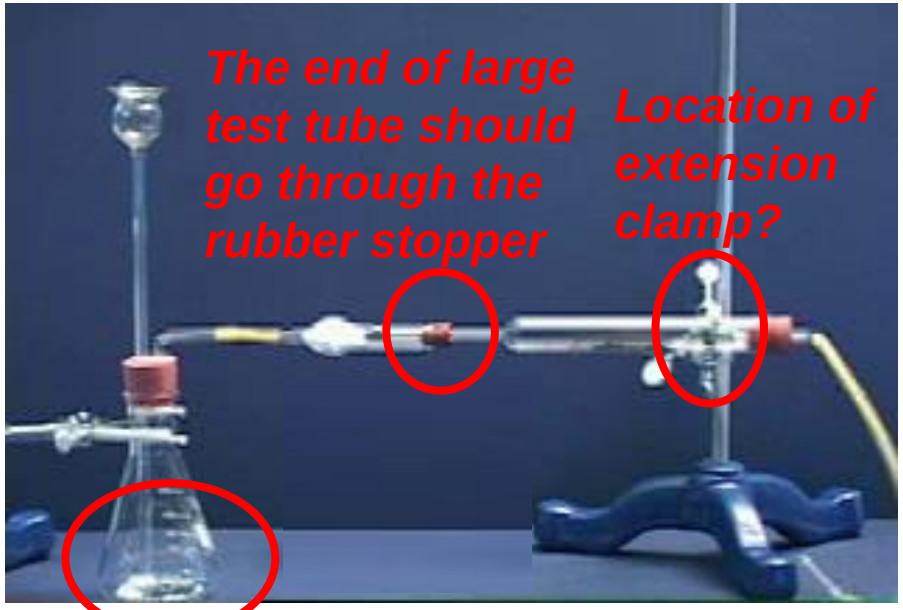

Step 2/9: Prepare Copper Oxide

- The large test tube should be clean and dry
- Record the accurate weight of the test tube **(W_1)** using an analytical balance
- Use the skinny end of a spatula to put ~1 g of copper oxide in the middle part of the test tube (do not disperse powders)
- Record the weight again **(W_2)**

- ✓ Use the same analytical balance throughout the experiment
- ✓ Don't let the test tube touch the wind shield of the balance

Step 3/9: Set up the Apparatus

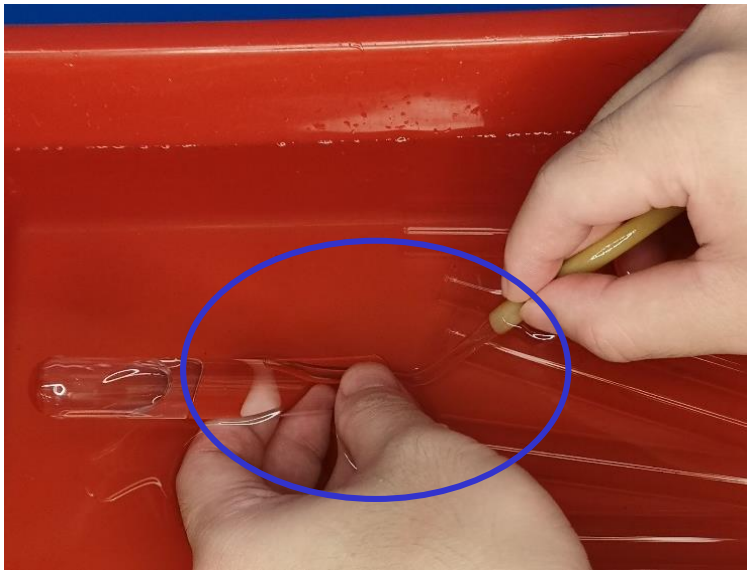
- Measure 15 g zinc granules into Erlenmeyer flask
- Use separate utility clamps to fix the Erlenmeyer flask and the test tube
- Do not clamp over the area where copper oxide is placed
- The thistle tube should nearly touch the bottom of Erlenmeyer flask



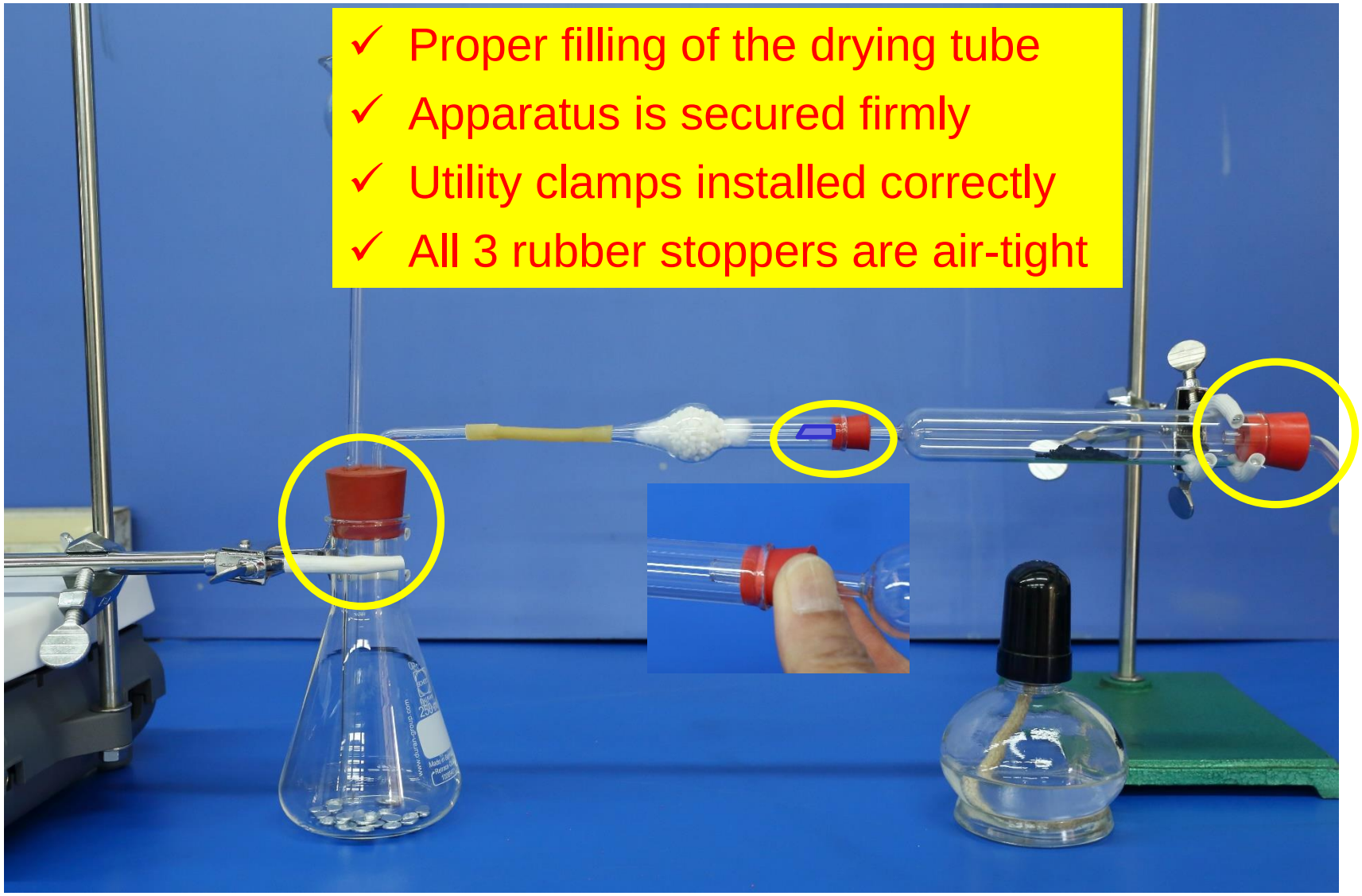
✓ Use a rag to cover the thistle tube and adjust its height by rotating slowly to avoid shattering and getting injured

Step 4/9: Prepare Small Test Tubes

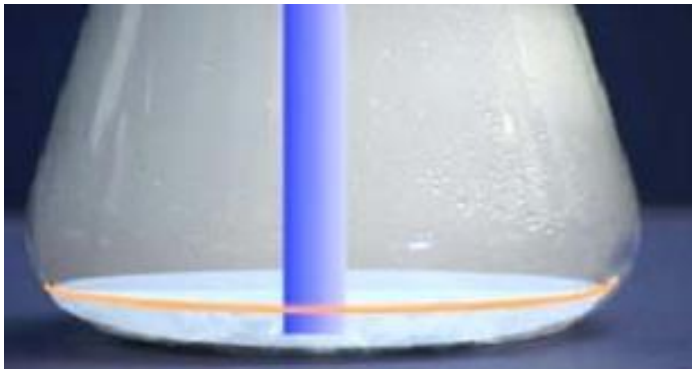
- Fill the plastic basin with water to 2/3 full
- Place 10 test tubes into water and fill them with water
- Hold the opening end of the test tube, keep it under water to avoid air from getting into the test tube



Ask a TA/ATA to Check Your Setup



Step 5/9: Generate Hydrogen Gas

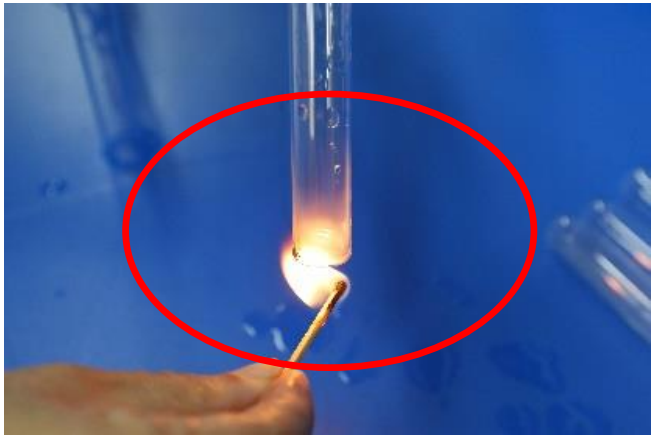


- Use 100 mL beaker to take 20 mL of 6 M HCl
- Pour HCl(aq) through the thistle tube <u>all at once</u>
- The end of the thistle tube should be immersed in the solution
- Start collecting gas with small test tubes right away

✓ HCl(aq) is a strong acid

✓ H_2 is explosive (keep lab windows and safety doors opened)

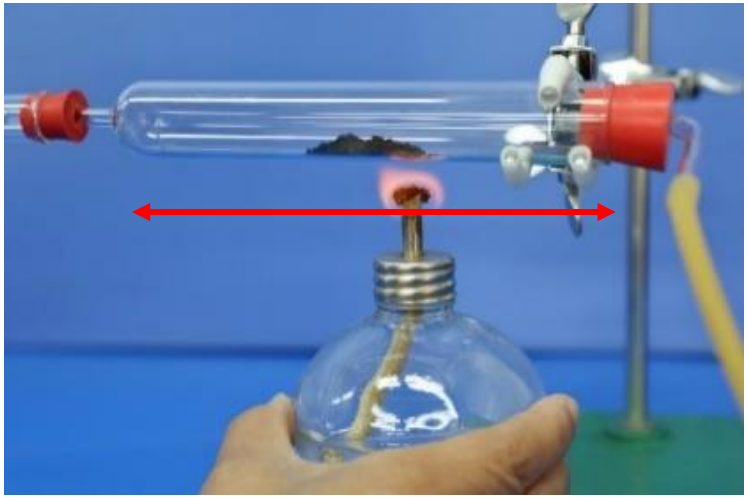
Step 6/9: Collect Gas over Water



- Hold test tubes upside-down in water, then put the rubber tube into the opening end
- Once filled (no water), keep the opening end downward and place the test tube on the table (collect 10 tubes at once)
- Light a match and bring the flame to the opening end of the test tube. Test for a loud squeaky sound (H_2/air mixture)
- The squeaky sound should reduce significantly as the system is being filled with H_2

✓ Only start heating after air has been purged out of the system

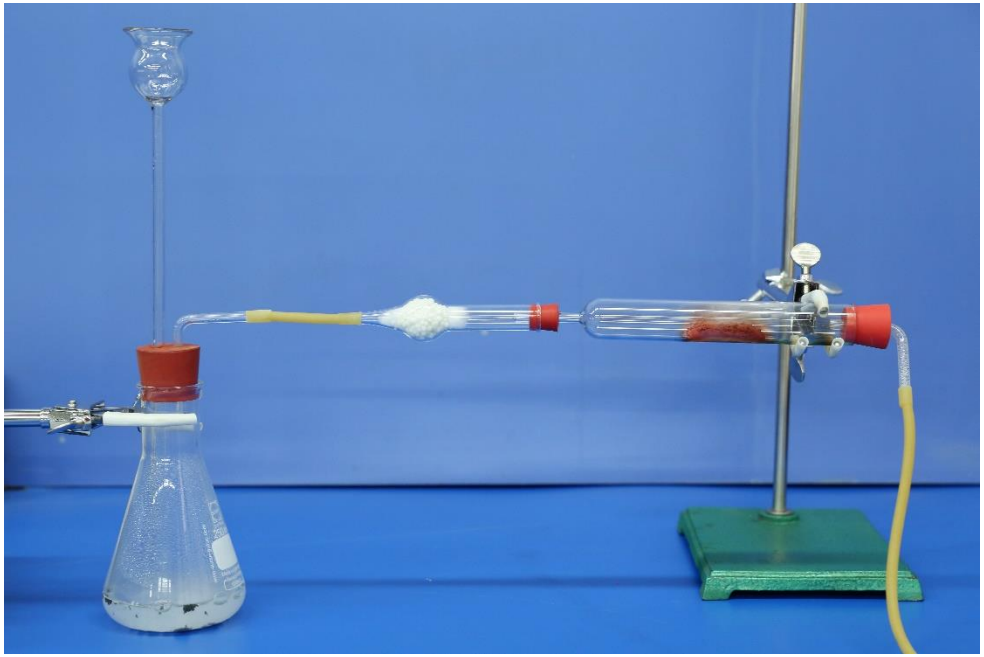
Step 7/9: Start the Reducing Reaction



- Remove the rubber tube from the water basin
- Add another 20 mL HCl to maintain the hydrogen gas supply
- Light the alcohol burner and start heating
- Heat both ends of the large test tube first (why?)
- Move alcohol burner horizontally for evenly heating
- Do not burn the rubber stoppers and the clamp

✓ Check the wick length of alcohol burner
✓ DO NOT leave the alcohol burner unattended
✓ Use windshield if necessary

Step 8/9: Cooling the System



- Observe and record any change, wait until the reaction is complete (keep hydrogen gas flowing)
- Put out the alcohol burner and let the system cool down
- Maintain hydrogen gas flow during the cooling process

✓ Do not touch the hot test tube with bare hands

Step 9/9: Weigh Cu Product

- Disconnect the test tube only after cooling to room temp (or Cu may be oxidized again)
- Accurately weigh the test tube and the metallic copper product using the same analytical balance **(W_3)**
- Calculate the **mass** and **molar ratio** of Cu and O, then give the **empirical formula** of copper oxide

$$\text{Cu : O} = \frac{Mass\ of\ Cu}{63.546} : \frac{Mass\ of\ O}{15.9994}$$

Molar Ratio

CuO_x → x = ?

Lab Note: Observations

- Use ball pens and avoid correction tapes
- Record operations and raw data (value + units)
- Changes that can be observed:
  - The appearances of reactants and final products
  - What happens as HCl(aq) is added to Zn (initial-during-after)
  - Sounds from ignition of each test tube
  - Color change on CuO and the heated region
  - ……

Test tube #	*Sound*
1-3	*Silent*
4-7	*Loud*
8-10	*Muffled*

Final Report (Brief Version)

- List the experimental data on your note book or lab manual
- List the detailed calculations in the blank space
- **Conclusion**: Use 1-2 sentences to summarize your experiment formally
- Need not answer the questions and discussion in the report sheet

I. Experimental Data and Results (show all calculations)

1. Weight of empty large test tube (W_1) **40.5110 g**
2. Weight of test tube and copper oxide (W_2) **41.7540 g**
3. Weight of copper oxide ($W_2 - W_1$)
4. Weight of test tube and copper (W_3)
5. Weight of copper ($W_3 - W_1$)
6. Weight of oxygen ($W_2 - W_3$)
7. Empirical formula of copper oxide

The simplest whole number ratio

3. $W_2 - W_1$ = 41.7540 g − 40.5110 g
= 1.2430 g

4….

II. Conclusion

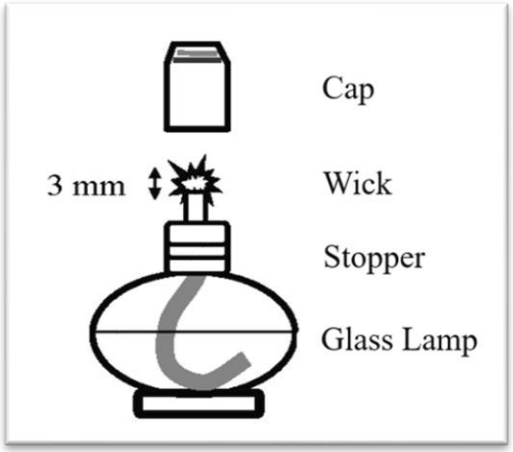
Clean-Up and Check-Out

- Recycle the cotton wool, $CaCl_2$, zinc granules (rinse with water), and the produced copper into designated containers
- Pour the waste solution into the 'heavy metal' recycling bin
- Brush-clean the test tubes, large test tube and the drying tube
- Clean up the lab bench and check personal equipment inventory (have an associate TA sign the checklist)
- This is a **Brief Report** experiment
  - Complete calculation using correct significant figures
  - Give the conclusion
  - **Hand in prelab/lab note/report together to the TA**

- Groups on duty shall stay and help clean up the lab

T1 – Alcohol Burner

- Inspect the burner before use – make sure that there are no cracks, chips or defects in the glass body
- Adjust the wick length to ~ 3 mm from the top of the stopper
- Fill with denatured (or 95%) ethanol through a funnel to 1/2 - 2/3 full
- Use a match to light the wick of the burner (do NOT use a burner to light another burner)



- When in use, keep the burner in an upright position and away from combustible materials (e.g. paper, clothing, etc.)
- If necessary, use a metallic windshield (not papers or books) to block wind
- Do not use books or other items to raise the height of alcohol burner (adjust the height of the heated object instead)
- Use the cap to put out the flame (do not blow off the flame)
- If the burner is overturned and causes a small fire, cover the fire quickly with a wet rag (do not remove the rag right away or flame may reignite). In the case of bigger fire, use a fire extinguisher instead. Inform lab instructor ASAP after the fire is put out, and open the windows to disperse the alcohol vapor

T1 Video (YouTube link)

T4 – Collecting Gas Over Water

- This technique is used to collect water-insoluble or slightly water-soluble gases (e.g. hydrogen H_2, oxygen O_2, and nitrogen N_2)
- Water-soluble gases such as NH_3, HCl are not suitable to be collected by this method
- Because the density of gas is lower than that of water, when the gas is introduced into the collection device (test tube in Figure T4-1) water will be displaced out

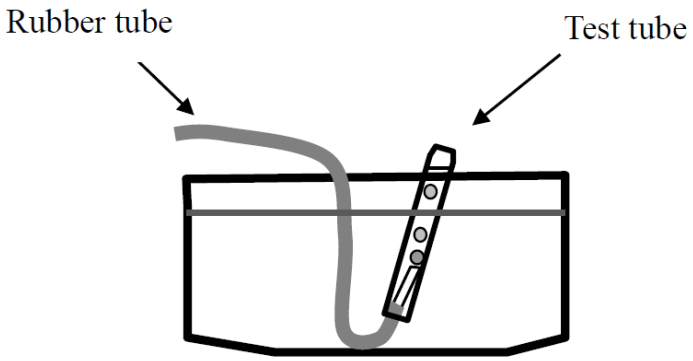


Figure T4-1 Collection gases over water

- Operation:
  1. Fill a test tube with water and immerse it in a water basin (the open end points down)
  2. Place the rubber tube into the open end of the test tube
  3. When gas bubbles start to emerge from the open end of test tube, remove the rubber tube
  4. Place the test tube upside-down on the lab bench, or seal it with a rubber stopper

T4 Video (YouTube link)

T9 – Electronic Balance

- Unless instructed, do not move the balance so that proper calibration is maintained
- Do not overload the balance (the maximum load is 610 grams for *electronic balance*, and 210 grams for *analytical balance*)
- Before use, warm up the balance for at least 30 min and ensure that it is level and clean

Electronic Balance
(resolution 0.01 g)

Analytical Balance
(resolution 0.0001 g)

- Do not put chemicals directly on the weighing pan – use a folded weighing paper, a weighing boat, or a beaker (mind the weight limit) as container
- Close all windshields on the *analytical balance* before zeroing and recording values
- Maintain the tidiness inside and outside the balance; use the provided soft brush to clean spillages
- Do not weigh hot objects as the convective airflow will affect the measured mass

T9 Video (YouTube link)

T10 – Weighing Chemicals

- Read the label on the chemical bottle carefully before proceeding to weigh
- For solid chemicals, place a folded weighing paper or a beaker on the electronic balance to hold chemicals. Use a clean and dry spatula to move chemicals
- For liquid chemicals, use a clean and rinsed dropper pipet

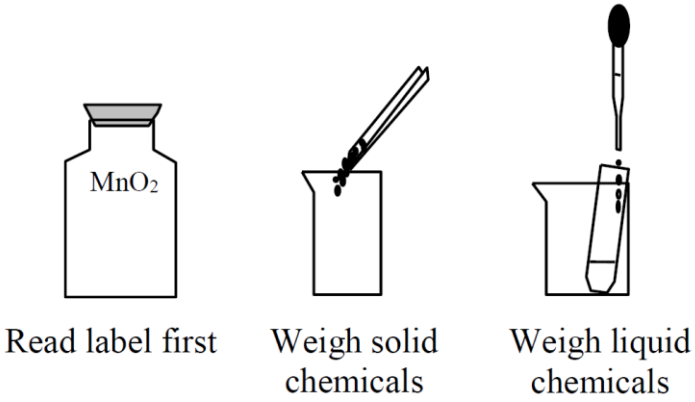


Figure T10-1 Weighing chemicals

- Unless specifically instructed, never return any excess chemical to the original bottle to avoid contamination – use the designated waste bin
- Maintain the tidiness inside and outside the balance – move appropriate amount with spatula to avoid any spillage, and use the provided soft brush to clean scattered chemicals
- Close the cap of chemical bottle immediately after use

T10 Video (YouTube link)

T16-Dispenser

1. Check the pre-set volume and do not change the volume setting afterwards.
2. Position the flask under the tip of dispenser.
3. Lightly pull the piston pump up and down several times to get rid of the bubbles.
4. Lightly pull piston pump up to the top, then slowly push down to obtain the measured solution.