



General Chemistry Laboratory

Determining the Chemical Formula of a Compound



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 lab bench (hold it down with something heavy) for ATA to sign

Collect the following items

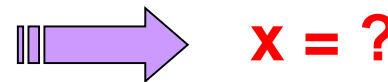
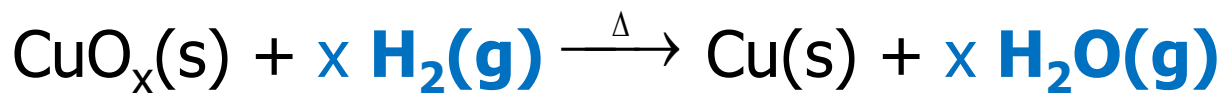
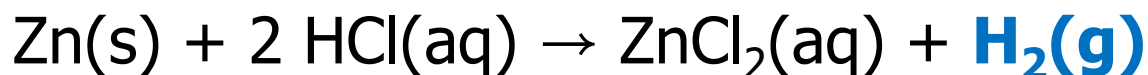
- One large test tube + one drying tube (oven)
- 250 mL Erlenmeyer flask, thistle tube, rubber tube, and alcohol burner (boxes on the central islands)
- Matches, windshield (wooden cabinet)



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

■ Chemical reactions

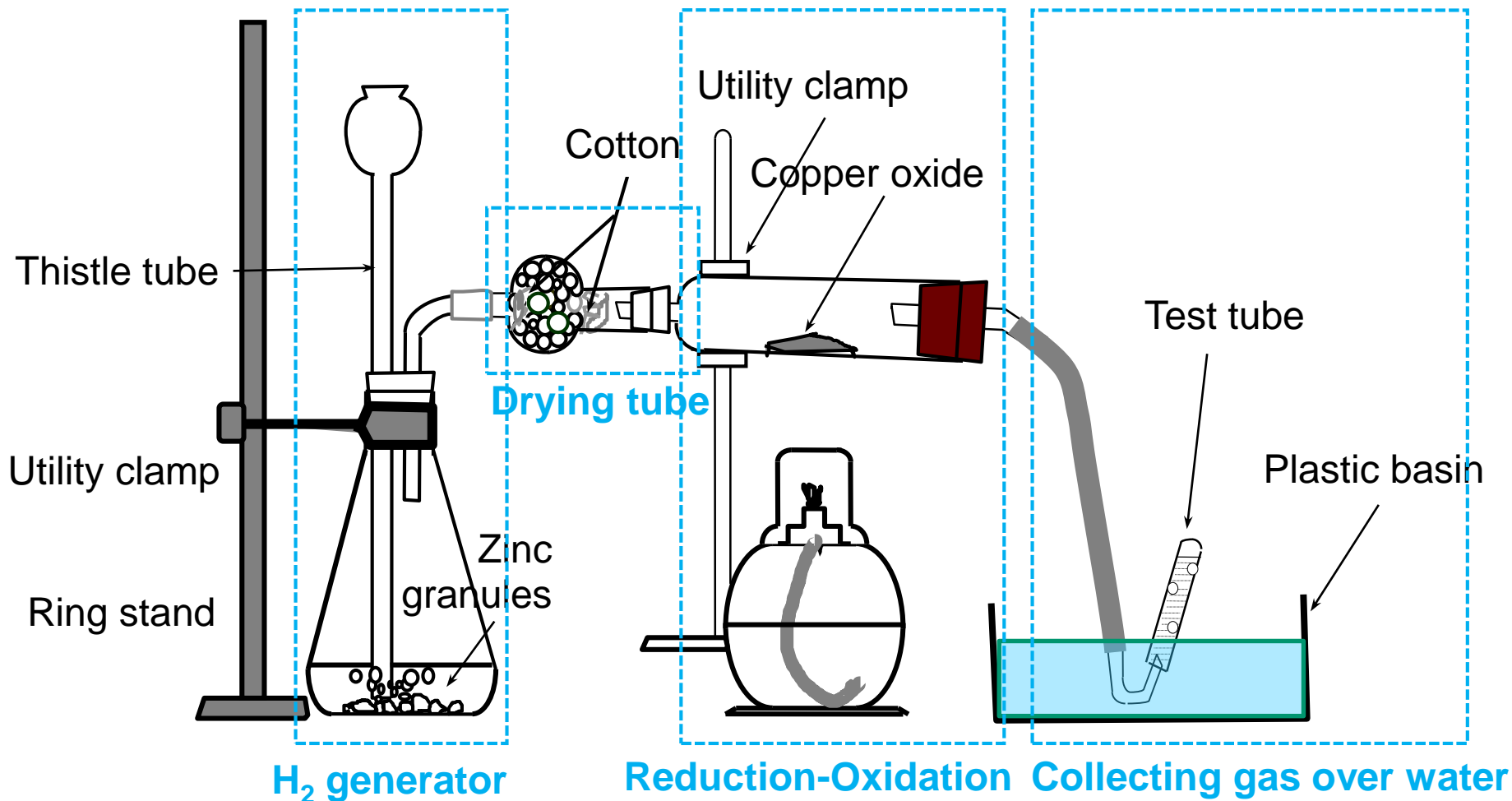


Reactant
(black powder)

Product
(red powder)



Experimental Setup



Do not remove the utility 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)

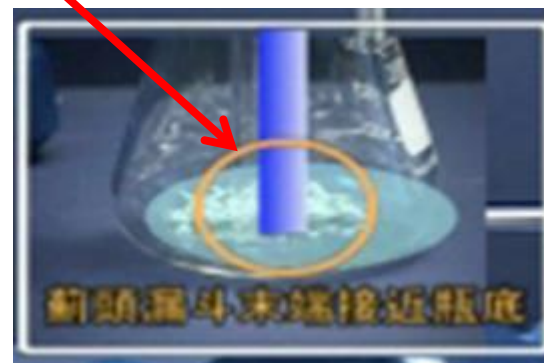
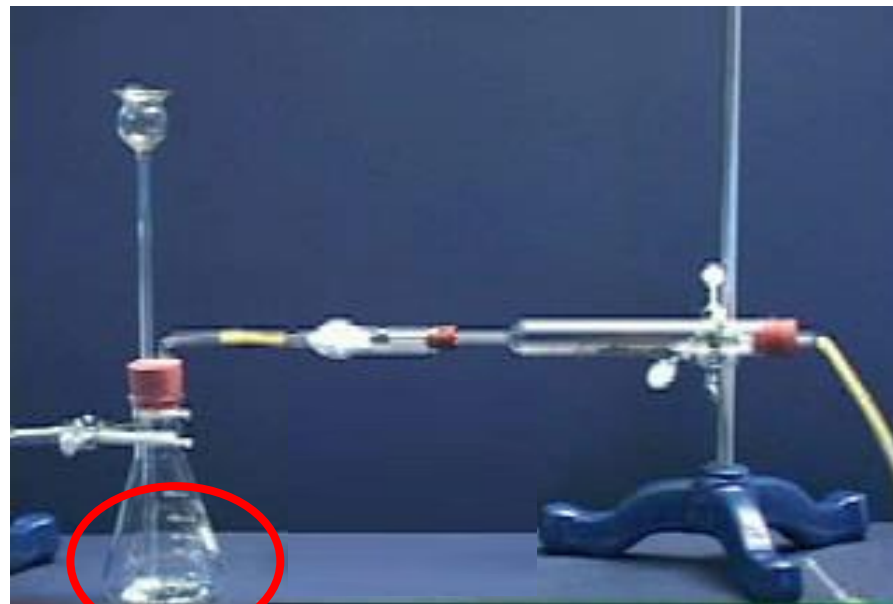
- ✓ Use the same analytical balance throughout the experiment
- ✓ Don't let the test tube touch the wind shield of the balance





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

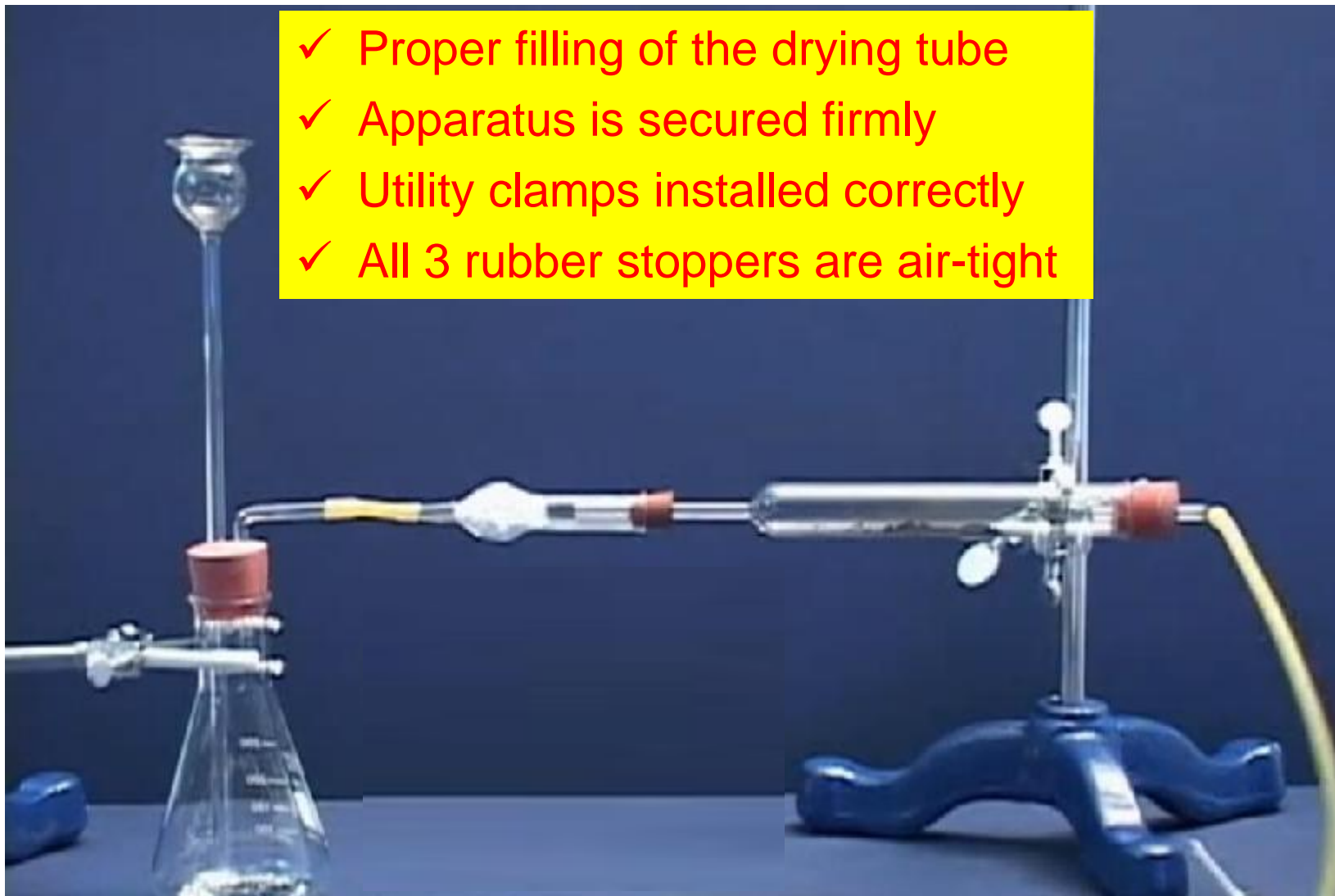
- Fill the plastic basin with water to 2/3 full
- Place 10 test tubes into water and fill them with water
- Hold the opening end of the test tube, keep it under water to avoid air from getting into the test tube





Ask a TA/ATA to Check Your Setup

- ✓ Proper filling of the drying tube
- ✓ Apparatus is secured firmly
- ✓ Utility clamps installed correctly
- ✓ All 3 rubber stoppers are air-tight

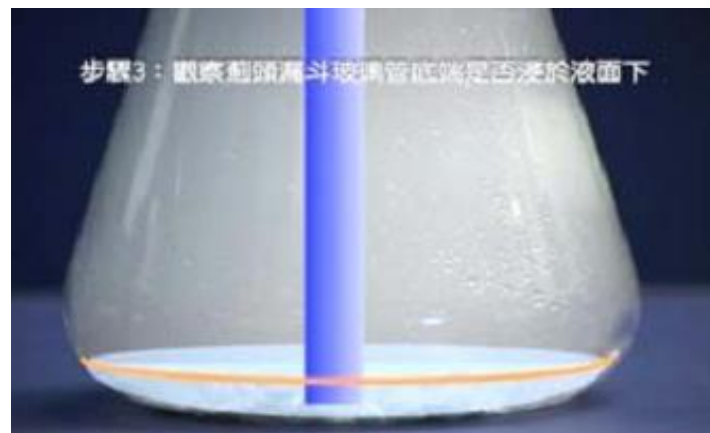




Step 5/9: Generate Hydrogen Gas

- Use 100 mL beaker to take 20 mL of 6 M HCl (wear protective gloves)
- Pour HCl through the thistle tube all at once
- 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
- ✓ H₂ 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

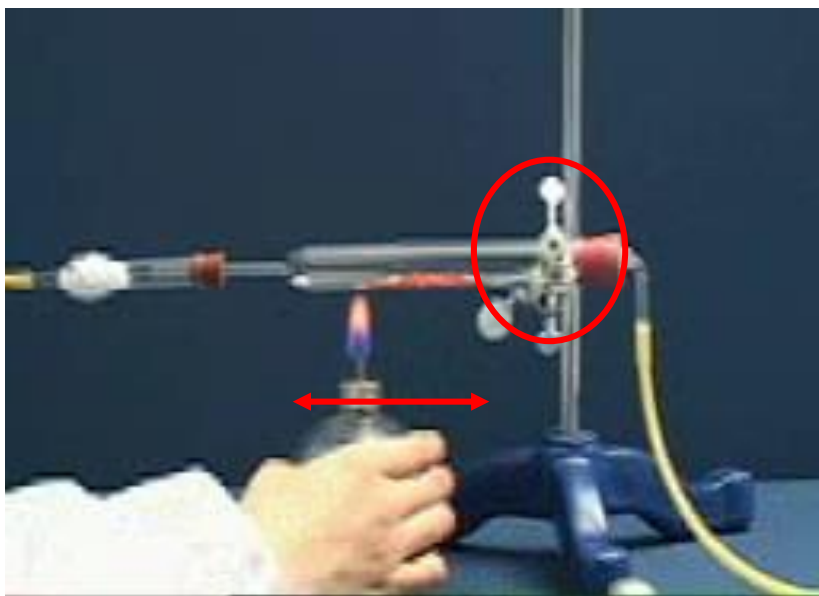


- ✓ Check the wick length of alcohol burner
- ✓ Put out the fire before refilling alcohol (should be about half filled)
- ✓ DO NOT leave the alcohol burner unattended
- ✓ Use windshield if necessary



Step 8/9: Continue Heating Evenly

- 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



- 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

✓ Do not touch the hot test tube with bare hands



Step 9/9: Weigh Cu Product

- Maintain hydrogen gas flow during the cooling process
- 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 balance (W_3)
- Calculate the mass of Cu and O

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

Molar Ratio





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 'heavy metal' recycling bin
- Brush-clean the large test tube and the drying tube
- Clean up the lab bench and check personal equipment inventory (have an associate TA signed the check list)
- This is a **Brief Report** experiment:
 - Member A: Complete calculation using correct significant figures.
Hand in prelab/lab note/report together to the TA
 - Member B: **Hand in prelab to the TA**
- Groups on duty shall stay and help clean up the lab



Lab Report Grading Rubrics

Category	Guidelines	Pts
I. Prelab exercise	1. Briefly summarize main principles and relevant equations	5
	2. List the chemicals' toxicity and physical and chemical properties	5
	3. Use flow chart to explain the experimental procedures	5
II. Lab note	4. Record data with correct significant figures and units	5
	5. Record observations, operations, and reaction conditions in details	5
III. Final report	6. Process data correctly (calculation included)	5
	7. Present final results with correct significant figures and units	5
	<i>8. Analyze the results with appropriate error discussions*</i>	<i>5</i>
	<i>9. Plot the results with correct XY axes and labeling*</i>	<i>5</i>
	<i>10. Elaborate findings and provide constructive suggestions*</i>	<i>5</i>

**Only for full reports*



Notes and Reminders

- Wear PPE (lab coat, safety goggles, closed-toe shoes, long pants) at all times in the laboratory
- Bring a scientific calculator (smartphone use is not allowed)
- Communicate with your lab buddy
- Communicate with TA/ATA should you have any question