



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

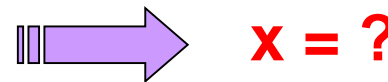
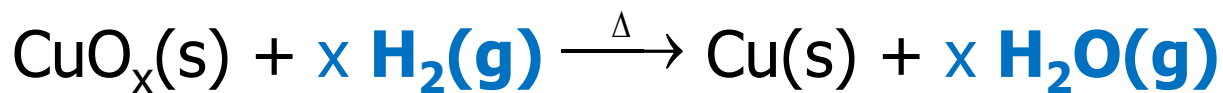
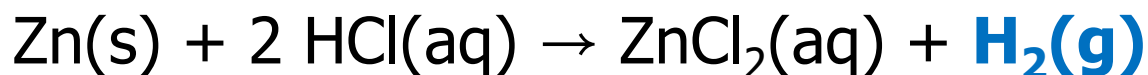
- One large test tube + one drying tube (oven)
- 250 mL Erlenmeyer flask, thistle tube, rubber tube, and alcohol burner (boxes on the central islands)
- Matches, windshield (wooden cabinet)
- An iron stand and two three prong clamps



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

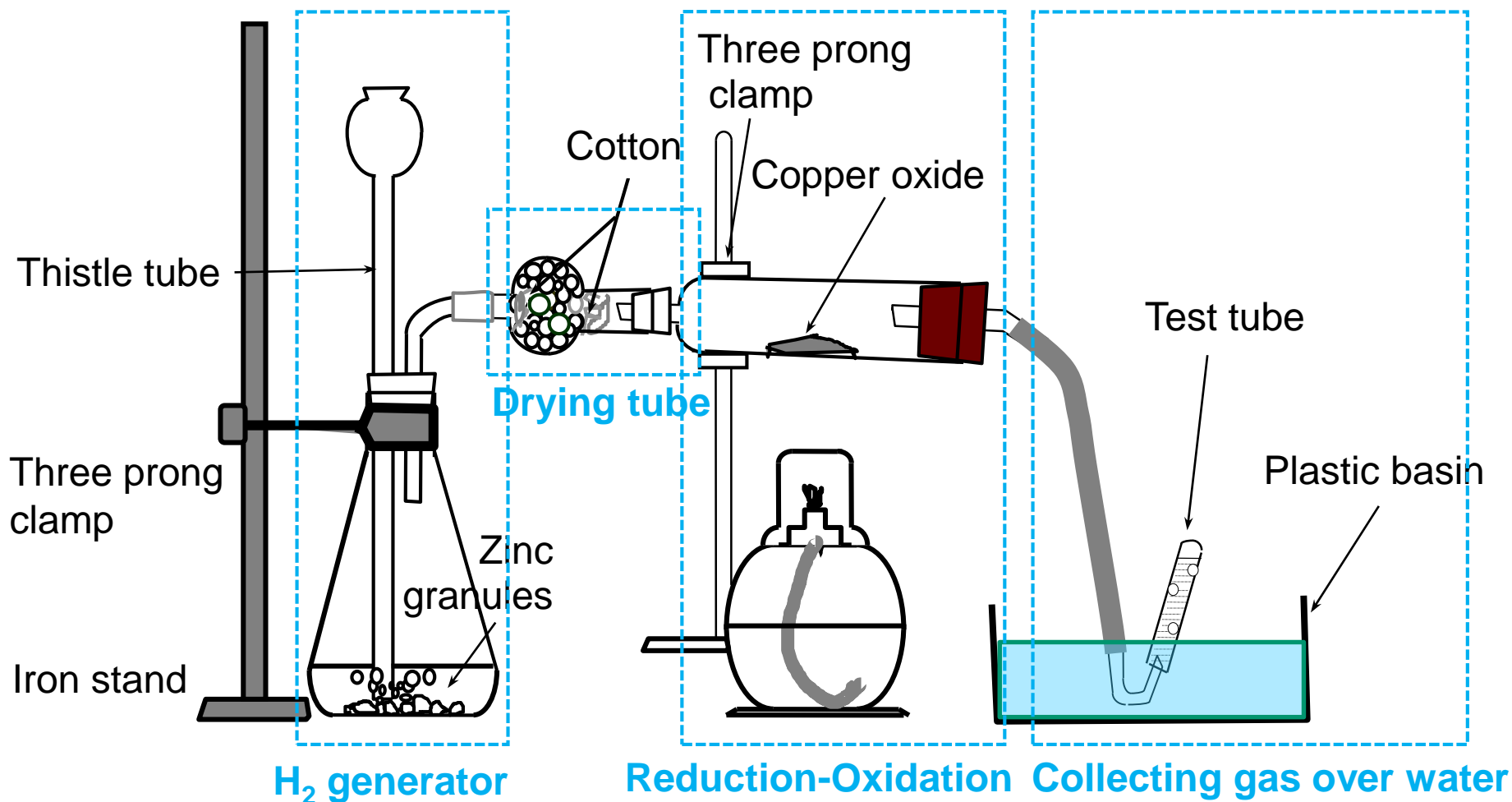


Reactant
(black powder)

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)

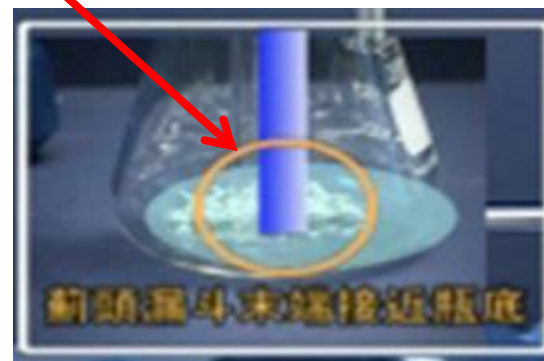
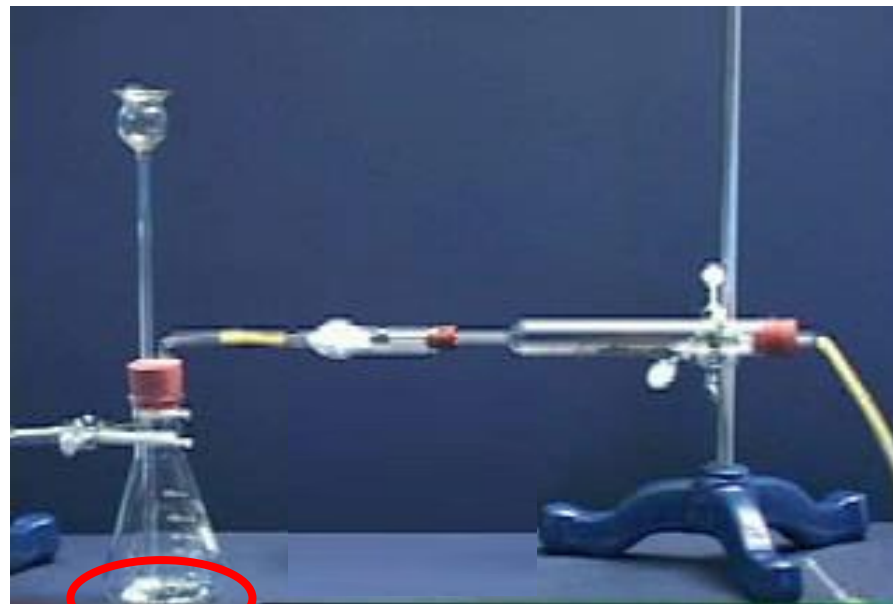
- ✓ Use the same analytical balance throughout the experiment
- ✓ Don't let the test tube touch the wind shield of the balance





Step 3/9: Set up the Apparatus

- Measure 15 g zinc granules into Erlenmeyer flask
- Use two three prong clamps to fix the Erlenmeyer flask and the test tube, separately
- 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

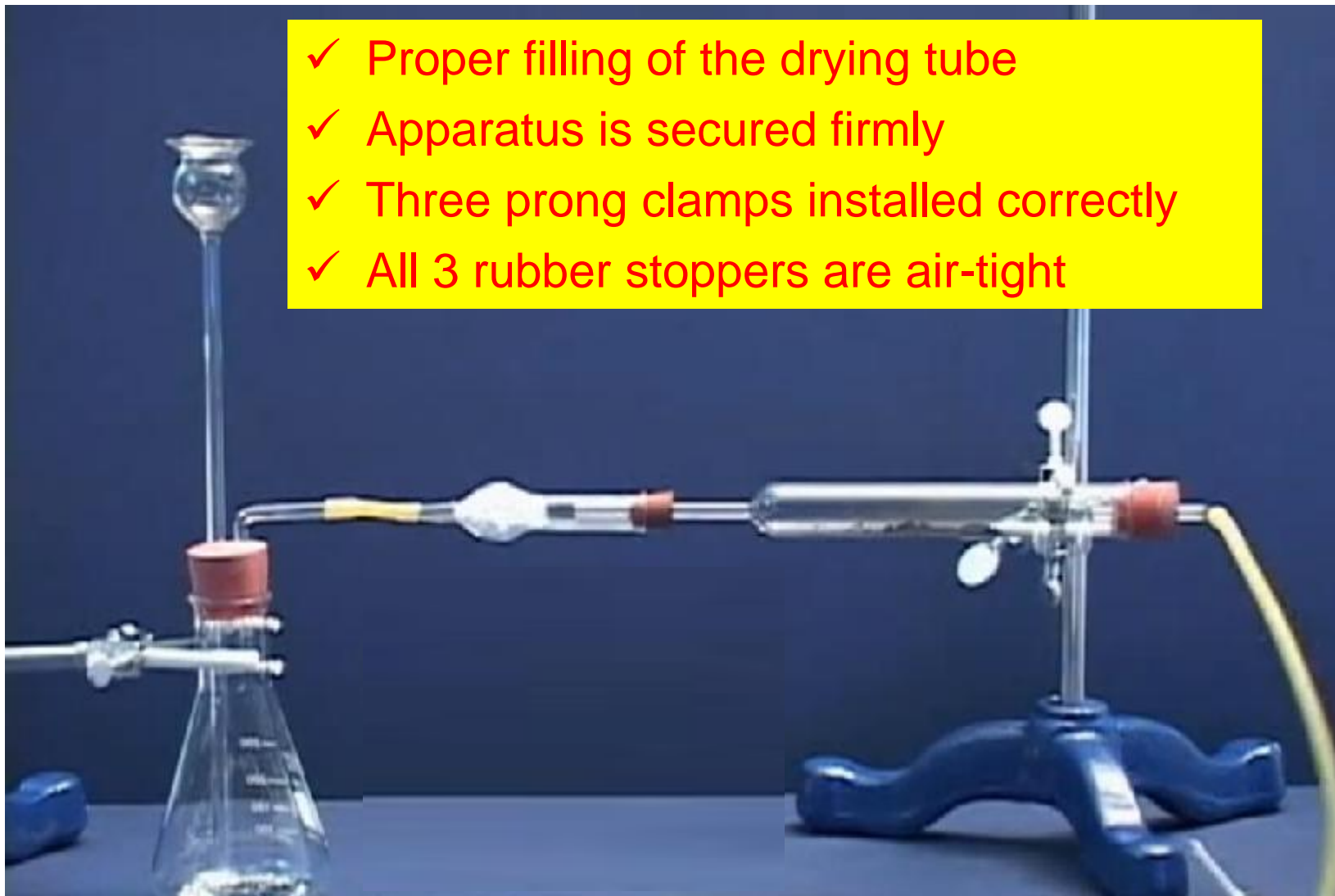
- Fill the plastic basin with water to 2/3 full
- Place 10 test tubes into water and fill them with water
- Hold the opening end of the test tube, keep it under water to avoid air from getting into the test tube





Ask a TA/ATA to Check Your Setup

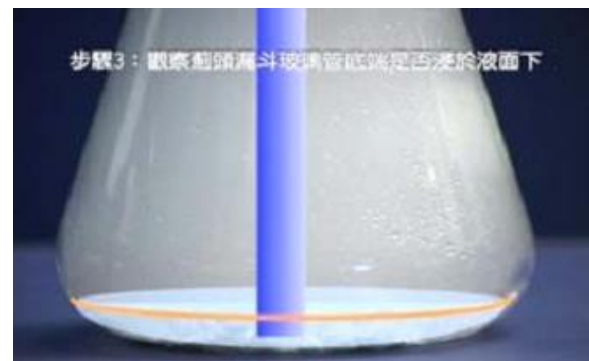
- ✓ Proper filling of the drying tube
- ✓ Apparatus is secured firmly
- ✓ Three prong clamps installed correctly
- ✓ All 3 rubber stoppers are air-tight





Step 5/9: Generate Hydrogen Gas

- Use 100 mL beaker to take 20 mL of 6 M HCl (wear 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

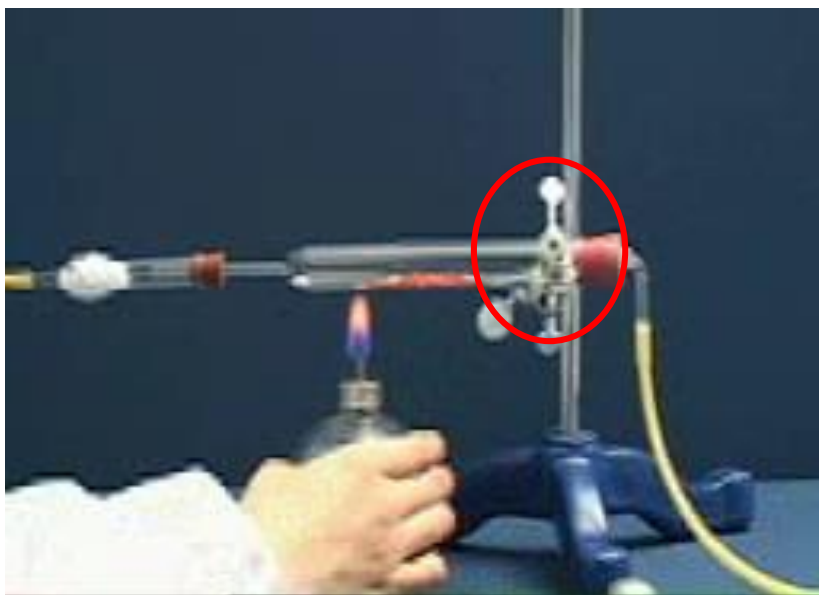


- ✓ Check the wick length of alcohol burner
- ✓ Put out the fire before refilling alcohol (should be about half filled)
- ✓ DO NOT leave the alcohol burner unattended
- ✓ Use windshield if necessary



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

$$\text{Cu} : \text{O} = \frac{\text{Mass of Cu}}{63.55} : \frac{\text{Mass of O}}{16.00}$$

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:
 - Complete calculation using correct significant figures
 - **Hand in prelab/lab note/report together to the TA**
- Groups on duty shall stay and help clean up the lab



Notes and Reminders

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



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
- 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



Electronic Balance (resolution 0.01 g)



Analytical Balance (resolution 0.0001 g)



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
- 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

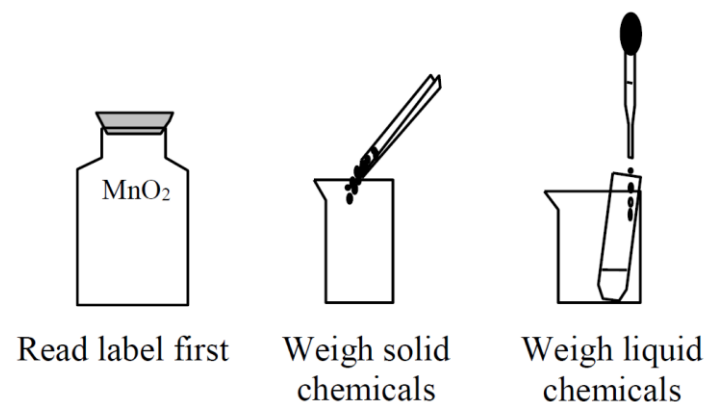
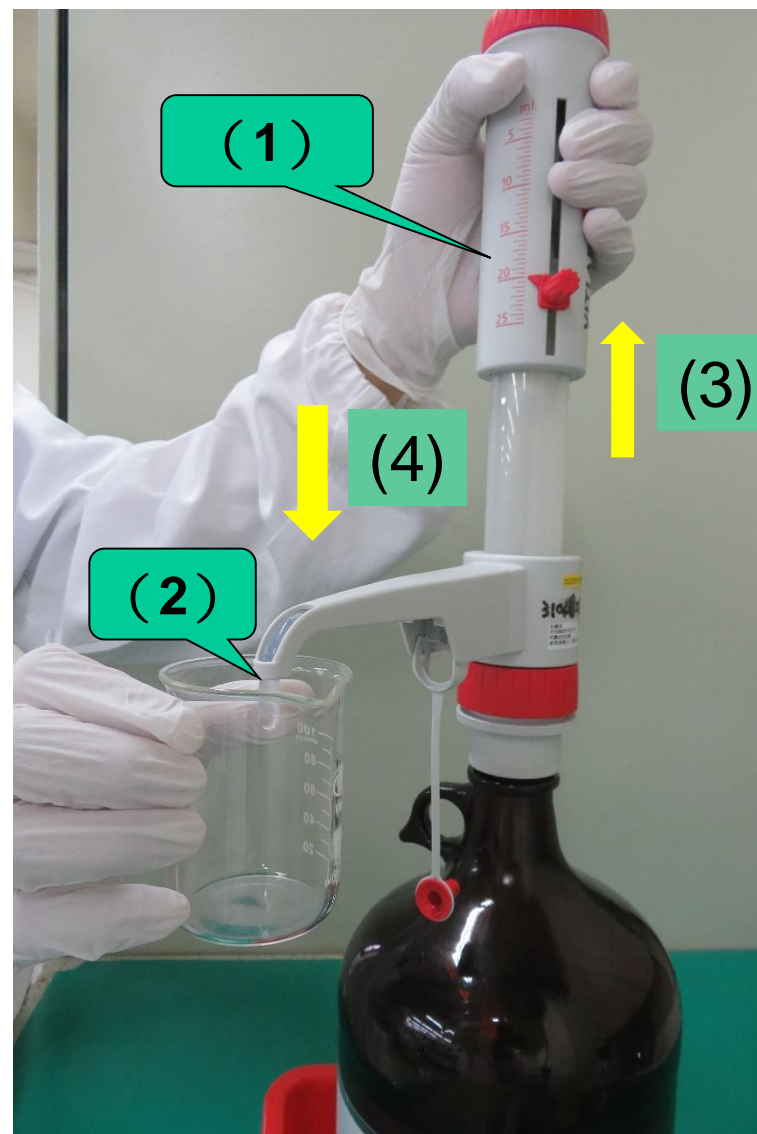


Figure T10-1 Weighing chemicals



Lab Dispenser

- (1) Check the pre-set volume on the dispenser. Do not change the setting unless instructed to do so
- (2) Place the receiving flask under the tip of dispenser
- (3) To remove the air bubbles in the dispenser, lightly pull the piston pump up and down for several times
- (4) Gently pull the piston pump up until it reaches the end of travel range, then slowly push the piston down to obtain the solution





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

