



Volumetric Measurement Techniques

Technique #1 – Use of a Burette

Technique #2 – Use of a Pipette

Technique #3 – Use of a Volumetric Flask

Technique #4 – Use of a Bottle-Top Dispenser

#1 - Techniques on the Use of a Burette

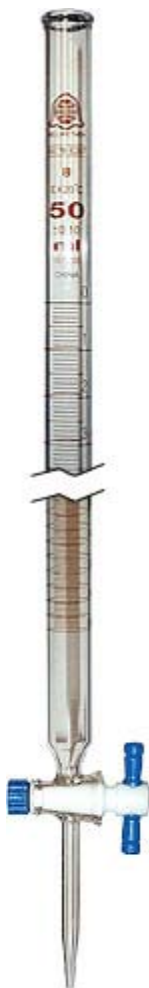


Figure 1:
A 50 mL
burette

A burette is used to deliver variable volumes of solution precisely and accurately (Figure 1). It is used for titrations, where the solution in the burette is called the *titrant*. When the stopcock valve is aligned with the burette, as shown in Figure 1, the valve is in the open position and the titrant flows from the burette into the collecting vessel. Rotate the stopcock valve 90 degrees to close the valve. A commonly used burette in our lab delivers a total volume of 50 mL and is calibrated in 0.1 mL increments. This means that the finest division on the burette is 0.1 mL (Figure 2).

When reading the burette, the last digit is the digit where you estimate to within the finest division. Therefore, a burette reading of the 50 mL burette is recorded to two decimal places.

Steps to prepare and use a burette:

1. A burette is usually stored with distilled water and capped.
2. Empty the burette of distilled water from the top and check that the stopcock is liquid-tight. If the stopcock feels loose when it is turned, hold onto the stopcock handle and rotate the screw clockwise to tighten. The stopcock should rotate smoothly. If it doesn't, you should disassemble the stopcock to clean the stopcock and where it touches the glass surface of the burette. Reassemble the stopcock with all its parts as laid out in Figure 3.

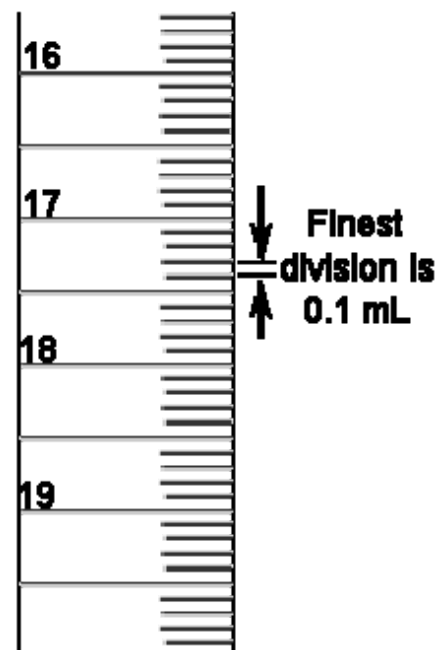


Figure 2: Division
markings on a
50 mL burette

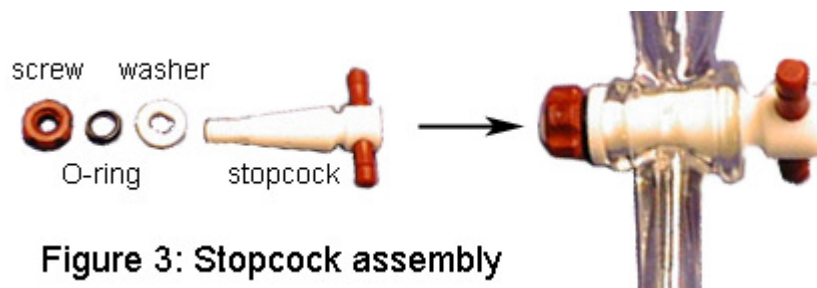


Figure 3: Stopcock assembly

3. Close the stopcock and using a small beaker, add approximate 10 mL of the titrant from the top. Hold the burette horizontally, rotate the burette to ensure the titrant touches the interior surface of the burette. Open the stopcock to allow the titrant

to drain through the tip. Drain the rest of the titrant from the top of the burette. This step known as **acclimatization** is done at least twice.

4. Fill the burette well above the zero mark. Rotate the stopcock to let some solution flow through the tip.
5. Look for any trapped air bubble below the stopcock (Figure 4). To dislodge the air bubble, take your burette over to the sink, rapidly rotate the stopcock and allow small quantities of titrant to pass. Close the stopcock and check if the bubble is gone. If the bubble remains, open the stop stopcock and tap the burette near the stopcock, or give the burette with a quick up-and-down jerk. A combination of the above procedures should dislodge the trapped bubble

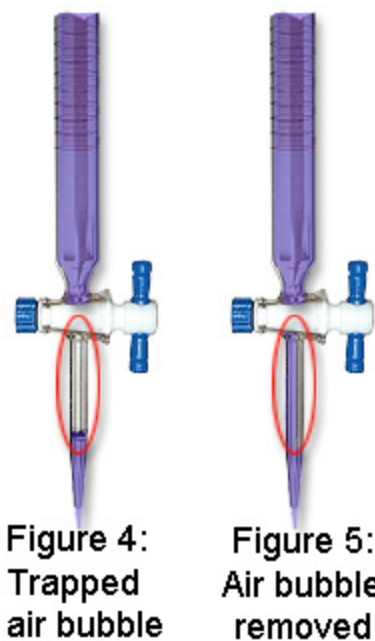
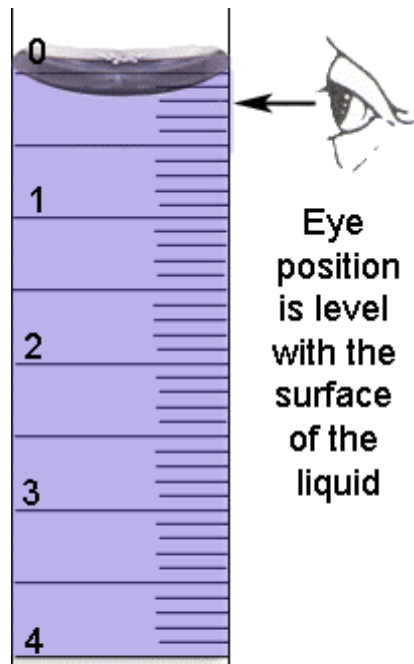


Figure 4:
Trapped
air bubble

Figure 5:
Air bubble
removed



**Figure 6: Initial reading
of the burette.**
(eg - $V_i = 0.20$ mL)

- (Figure 5).
6. Finally, refill the burette to above the zero mark. Lower the level of the titrant below the zero mark. The eye should be level with the meniscus for a proper reading. The correct reading is where the bottom of the meniscus touches the scale. Record the initial volume (V_i) to 2 decimal places (Figure 6).
 7. Place a receiving flask (usually an Erlenmeyer flask) under the tip of the burette. Adjust the burette height so that the tip of the burette is inside the neck of the Erlenmeyer flask (Figure 25).
 8. Dispense the titrant through the burette until a desired volume is reached or until a chemical analysis is complete. **Do not let the solution drain past the last mark on the burette.**

9. Record the final volume (V_f). The volume of solution dispensed is found by subtracting V_i from V_f .

Final Burette Reading, V_f (mL)	23.45
Initial Burette Reading, V_i (mL)	0.20
Volume delivered (mL)	23.25

10. To clean up, drain the solution from the top of the burette into a proper waste container. Rinse the burette 2-3 times with distilled water. Each time, open the stopcock to drain some of the rinse water through the tip and then empty the rest through the top of the burette.

11. Store the burette with distilled water.



**Figure 25 - A
titration setup**

#2 - Techniques on the Use of a Pipette

Pipettes are for the transfer of known volumes of liquid from one container to another. Pipettes that deliver a fixed volume are called volumetric or transfer pipettes (Figure 7a).

Other pipettes are known as measuring pipettes. They are calibrated with graduated markings along the side so that any volume up to the maximum capacity can be delivered (Figure 7b).

Liquids are drawn into the pipette through a slight vacuum by using a pipette bulb (Figure 8). ***Never use your mouth to pipette.***

The volume of a volumetric pipette is recorded to four significant figures (i.e. – 10.00 mL). To read the volume of a measuring pipette, estimate the last digit to within the finest division on the pipette.

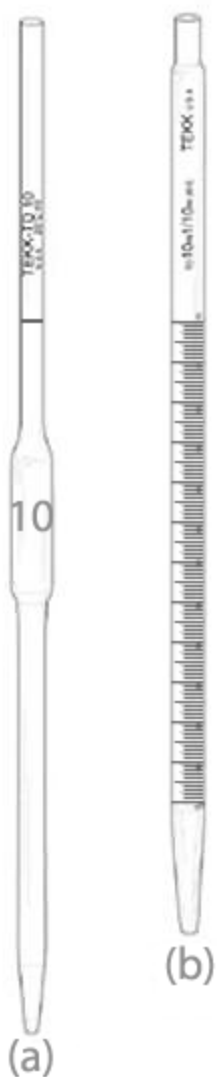


Figure 7:
Pipettes

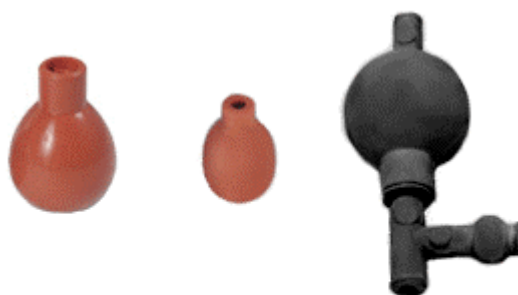


Figure 8: Different types of
pipette bulbs

Steps to prepare and use a volumetric pipette:

1. Inspect the pipette to make sure that the tip is not chipped and the top end of the pipette is smooth and flat. If the top end of the pipette is chipped, or not smooth, it is very difficult to pipette.
2. To fill a pipette, a rubber bulb (Figure 8) is used to provide suction to pull up the solution. Keep the tip of the pipette below the surface of the liquid. It is easier to draw up solution from a smaller, narrower container (i.e. 50 mL beaker) to avoid sucking up air. ***Avoid sucking liquid into the pipette bulb.***
(Note: Never dip a pipette into the reagent bottle. Obtain the solution with a small clean and dry beaker. Pipette the solution out of the beaker.)

3. Acclimatize the pipette with the solution to be used in the analysis by drawing up a small amount of the solution. Remove the bulb and tilt the pipette horizontally to rinse the inner walls of the pipette. Drain completely. Repeat this step three times.
4. Fill the pipette with solution past the calibration mark. Remove the bulb and quickly place the index finger of the hand holding the pipette over the exposed end of the pipette.

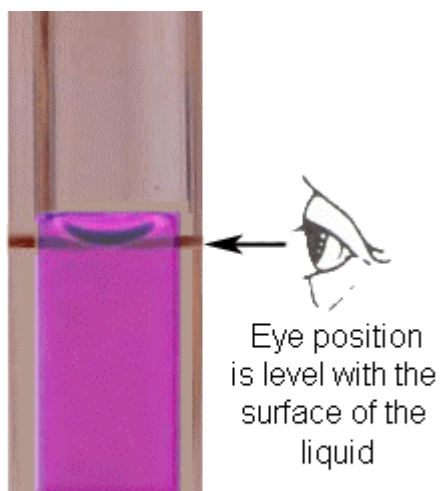
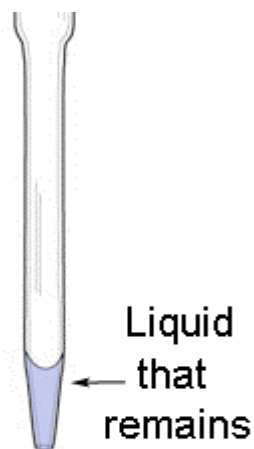


Figure 9: The bottom of the meniscus sits at the calibration mark

5. Tilt the pipette slightly and wipe away any liquid on the outside surface. Slowly release pressure on the index finger so that the bottom of meniscus approaches the calibration mark at eye level. At the mark, apply pressure on the index finger to stop the level of the liquid.
6. Touch the tip of the pipette on the wall of the container. This will drain any drop that remains on the pipette tip.

7. Transfer the pipette to the receiving container and release the pressure on the index finger. Drain the solution into the receiving container with the pipette tip touching the wall of the container. After draining, wait 10 seconds before removing the pipette. At this point, the calibrated amount of liquid has been transferred. Look closely at the tip of the pipette. A small portion of solution remains in the tip (Figure 10). ***Do not blow out the pipette.***
8. After use, clean the pipette by rinsing with distilled water.



**Figure 10:
A small portion
of the liquid
remains.**

#3 - Techniques on the Use of a Volumetric Flask



Figure 11: A 100.0 mL Volumetric flask

A volumetric flask is calibrated to contain one specified volume. It is used to prepare standard solutions and for the dilution of solutions. Volumetric flasks are cleaned and thoroughly rinsed but rarely do they need to be dried. When the meniscus touches the mark that is etched on the neck of the flask, the calibrated volume is contained in the volumetric flask.

When preparing a solution with a solid solute, the solid should not be emptied directly into the volumetric flask. First, dissolve the solid in a beaker using about half to two-thirds of the volume of the final solution. Second, quantitatively transfer the solution to the volumetric flask. Details of preparing a standard solution and quantitative transfer are covered in *Technique #7*.

The volume measured in a volumetric flask is recorded to four significant figures (i.e. – 100.0 mL).

Steps to make a Dilution using a Volumetric Flask

1. Pipet the solution to be diluted into the volumetric flask directly.
2. Add distilled water to fill the flask about two-thirds full.
3. Stopper the flask and invert to mix the solution.
4. Add more distilled water and invert to mix the solution again.
5. Bring the level close to the mark and allow time for drainage. Then use a Pasteur pipette to make the final addition (Figure 12). The eye should be level with the meniscus and the mark to make a correct reading for the volume (Figure 13).



Figure 12: Pasteur pipette

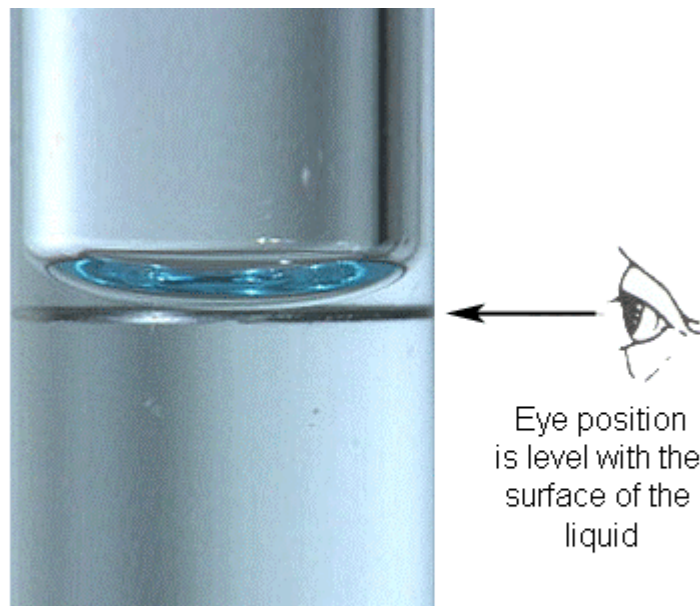


Figure 13: Fill the volumetric flask such that the bottom of the meniscus touches the mark on the neck of the flask

6. Firmly stopper the flask and invert repeatedly (at least 15 times) to assure uniform mixing.

#4 – Techniques on the Use of a Bottle-Top Dispenser



**Figure 19:
A Bottle-Top Dispenser**

A bottle-top dispenser is a hand operated pump that screw on to a reagent bottle. These dispensers are surrounded by a clear plastic sleeve, which protects the glass cylinder from breaking, and eliminates the risk of hazardous spills. The volume is accurate to about 1-2%. The volume to be dispensed is set by adjusting the volume knob on the plunger.

Steps to Use a Bottle-Top Dispenser

1. Prior to use, check the dispenser to see whether the dispenser volume is set correctly.
 2. Place a graduated cylinder at the outlet.
 3. Pull the top of the dispenser up to as far as it will go.
 4. Push down gently and completely so that the liquid is collected in the graduated cylinder. While the liquid is dispensing into the graduated cylinder, observe that there is no air bubble in the liquid. If air bubbles are present, it could be that the container is near empty or the dispenser is malfunctioning. Consult your instructor to refill the solution and check the mounting of the dispenser.
 5. Read the graduated cylinder and make sure that the volume collected is within 2% of the preset volume.
 6. Place a clean, empty container at the outlet.
- Repeat steps 3 and 4 to dispense the solution into the container.