ANALYTICAL CHEMISTRY

Basic Skills in Chemical Laboratory

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Chapter Description

- Expected Outcomes
  - Understand the basic calculations in chemical analysis.
  - Describe the handling and applications of common laboratory apparatus and chemicals.

Contents

- Analytical Balance
- Buoyancy
- Volume Measurement
  - Burette
  - Volumetric Flasks
  - Pipette
  - Syringe
- Filtration
- Drying
- Calibration of Glassware

LABORATORY SAFETY

Your Responsibilities:

- ✓ Familiarize yourself with all the laboratory facilities available
- ✓ Wear your goggles at all times
- ✓ **Contact lenses** should not be worn in the laboratory
- ✓ Rubber gloves must be used when transferring concentrated acids
- ✓ Never eat or drink in the laboratory
- ✓ Volatile solvents and fuming concentrated acids must be placed and handled in a fume cupboard
- ✓ You must wear a gas mask when handling reagents in the form of powders
- ✓ Any chemical spills have to be cleaned immediately
- ✓ Know how to use the fire extinguisher or the emergency fire blanket
- ✓ All bottles and containers should have **complete labels**

DISPOSAL OF CHEMICAL SUBSTANCE

- Pour the waste into the sink and dilute with pipe water.
- Keep the waste for proper disposal at approved disposal site.
- Treat the waste to reduce its hazard and then pour it into the sink and dilute with water or keep it for proper disposal at approved disposal site.
- Recycle the waste.

EXAMPLE OF DISPOSAL METHOD

Chemical waste	Disposal method
Dichromate	$Cr_2O_7^{2-}$ is reduced to Cr^{3+} using $NaHSO_3$ and treated with a hydroxide to form $Cr(OH)_3$ and evaporated and the solid is disposed at a disposal site.
Acid	Mix the acid waste with a base to neutralize (check with a litmus paper) and pour the mixture into the sink.
Iodate	IO_3^- is reduced to I^- with $NaHSO_3$, neutralized with a base and pour it into the sink
Lead ion	Solution containing Pb^{2+} is treated with sodium metasilicate (Na_2SiO_3) to precipitate $PbSiO_3$ that can be disposed at the disposal site.
Silver and gold ions	Treated to retrieve the elements.
Gas	The experiment should be performed under a fume hood; the gas is passed through a chemical trap or ignites in a flame to prevent it from escaping from the fume cupboard.

LABORATORY LOG BOOK

- The notebook is used to record all **activities performed and observations** made during the experiments.
- The content of the notebook must be legible by others. You have to use full sentences to avoid ambiguity.
- Should clearly explain what has been done and observed to enable other people to repeat your experiments.
- A hard copy of your important computer-generated data such as spectra or chromatograms should be placed on the page of your notebook.

ANALYTICAL BALANCE

- Principle of using analytical balance:
  1. Place a suitable receiving container on the pan of the balance.
  2. Ensure that the container is not too heavy that will exceed the maximum capacity of the balance.
  3. Use "tare" button if necessary.
  4. Add the chemical substance into the container appropriately and read the new reading.
  5. Do not place any chemicals on the balance pan to avoid corrosions on the balance and to ensure cleanliness.

WEIGHING BY DIFFERENCE

- Convenient for hygroscopic substances that can easily absorb moisture from the air.
- First, weigh a capped weighing bottle that contains the dry reagent.
- Quickly transfer the reagent into another container and immediately recap the weighing bottle and weigh it.
- The difference in mass is the weight of the substance that has been transferred from the weighing bottle.

WEIGHING ERRORS

Tips to achieve good weighing:

- Use a dry, clean piece of paper or tissue paper to lift or hold weighing bottle or container because finger prints can change the weight registered.
- The sample has to be at ambient temperature to avoid errors caused by air convection flow.
- Samples removed from a hot oven take about 20-30 minutes to cool down to room temperature. It is advisable to place the sample in a desiccators while cooling to avoid the adsorption of moisture.
- All windows of the electronic balance must be closed while weighing to avoid the effect of air movement from the vicinity.
- Highly sensitive electronic balance should be placed on a sturdy and secure bench such as concrete table to reduce vibrations.
- A factory-calibrated electronic balance may show slightly different mass as the gravitational force at the factory may not be exactly the same as that in your laboratory.

BUOYANCY

- If an object is placed on the balance pan, it will displace a volume of air equivalent to the volume of the object.
- When this happens, the apparent weight of the object will be less than its original weight. The difference in weight is equal to the weight of the air displaced by the object.
- The force that causes the decrease in the measured weight is known as buoyancy.
- A correction has to be made if the density of the object is not the same as the density of the standard weight.
- The actual weight, m, i.e. the mass of the object weighed in vacuum can be calculated from the following equation:

$$m = m' + m' d_a \left(\frac{1}{d} - \frac{1}{d_w} \right)$$

EXAMPLE

In a weighing of benzene (density 0.878 g/mL) the measured weight is 10.000 g. Calculate the actual weight of the sample and the error (%w/w) if the measurement does not consider the buoyancy effect using the stainless steel weight (density 7.8 g/mL).

Solution

Insert the value into the buoyancy equation:

$$m = 10.000 + 10.000 \times 0.0012 \left(\frac{1}{0.878} - \frac{1}{7.76} \right) = 10.0121 \text{ g}$$

$$\text{Error} = \frac{10.012 - 10.000}{10.000} \times 100 = 0.12\%$$

VOLUME MEASUREMENT

Volumetric apparatus are labelled by the manufacturer as to the temperature and how the apparatus was calibrated.

- Apparatus marked with **"TD"** meaning "to deliver" are for delivering a volume at the indicated calibration temperature.
- Apparatus marked with **"TC"** meaning "to contain" and/or those that have frosted ring at the upper part are assigned to contain a volume at the indicated temperature.

EFFECT OF TEMPERATURE ON VOLUME MEASUREMENT

- Not only the volume of the liquid, but also the volume of the apparatus can also expand with the increase in temperature.
- At ambient room temperature, an aqueous solution expands at a rate of 0.025% per °C (A change of 1°C will change the volume by 0.025%)
- All volumetric measurements are based on a specific standard temperature and the usual temperature used is 20°C.
- The volume at 20°C can be calculated from the following equation:

$$V_{20^\circ} = V_T + 0.00025(20 - T)(V_T)$$

- where
  - V_{20° volume of liquid at 20°C
  - V_T volume of liquid at measurement temperature (T)
  - T measurement temperature, °C

EXAMPLE

A sample (40.0 mL) is taken from a refrigerator at 5°C. Calculate the volume of the sample at 20°C.

Solution

$V_{20°} = V_{5°} + 0.00025(20 - 5)(40.00)$

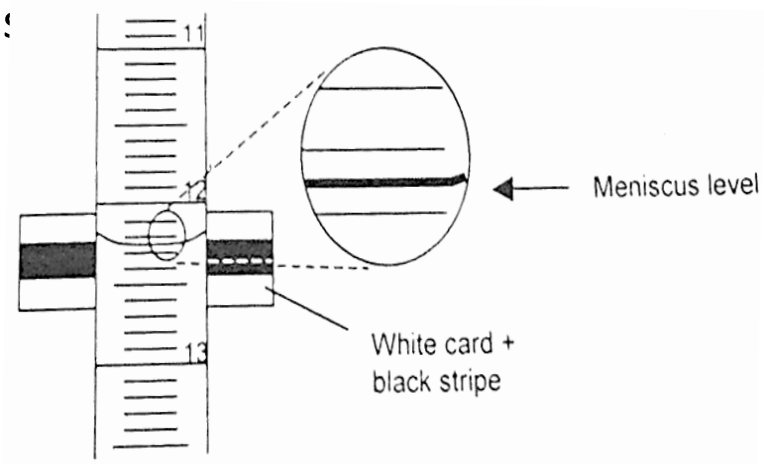
$= 40.00 + 0.15$

$= 40.15$ mL

BURETTE

Tips of how to use a burette

- Rinse the burette a few times with the titrant us
- Degas the burette before using it
- Allow slow flow
- When approaching the end point,
  let the let titrant out slowly (less than a drop)
- Avoid parallax errors
- Read the bottom of the meniscus
- Estimate reading to 1/10 of that of the smallest scale on the burette
- Consider the thickness of the scale lines when reading the burette



BURETTE

Source: http://www.chem.yorku.ca/profs/hempsted/chemed/equipment/burette.html

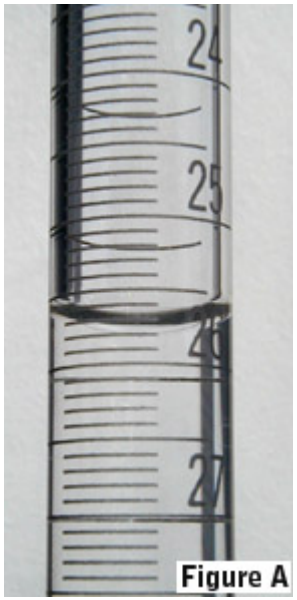


Figure A

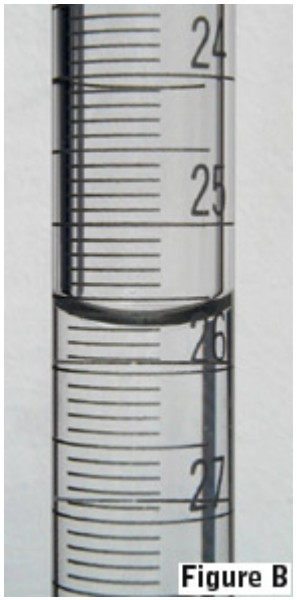


Figure B

Figure C

- Figures A, B and C below illustrate a common error that arises when reading a burette.
- This error, known as parallax, occurs if the eye is either above or below the level of the meniscus.

BURETTE

- Sometimes, air bubble can remain at the bottom of the valve.
- The bubble can give an error if it comes out during the titration.
- Get the bubble removed before the titration.
- Usually, it can easily be removed by flowing the titrant for 1-2 seconds with the valve fully open to allow the fast flow to sweep out the bubble.

Source of figures: http://www.chem.yorku.ca/profs/hempsted/chemed/equipment/burette.html

Figure 1

Figure 2

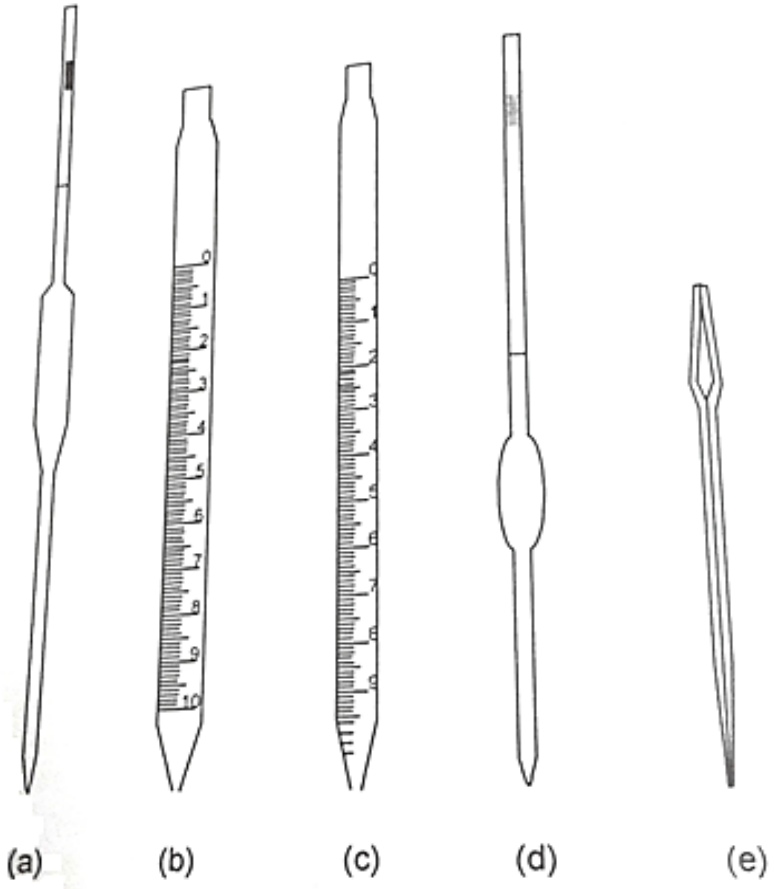
VOLUMETRIC FLASK

Principle of using volumetric flask:

1. First introduce the reagent into the volumetric flask, add enough solvent (less than the stated volume) and swirl the mixture to dissolve the reagent.
2. Add more solvent to fill almost the mark and mix the mixture.
3. Add the final volume carefully using a dropper (not a wash bottle). While adding the solvent, watch the meniscus and stop when the bottom of the meniscus is aligned with the front and the back of the mark.
4. Tighten the cap and turn the flask upside down several times to ensure complete mixing.

PIPET

There are four types of pipets, namely, **transfer** pipet, **measuring** pipet, **Ostwald-Folin** pipet and **serological** pipet:



PIPET

Technique of using pipet:

- Begin by **rinsing** your pipet with the solution that you want to use and discard the rinse.
- Use a **pipet filler** (not your mouth) to suck the liquid into the pipet. As the liquid level has risen above the graduated mark, quickly disengage the pipet filler and **replace** it with your forefinger.
- Slowly place the tip on the **bottom** of the flask; this will help to maintain a slow flow. **Wipe** any drop on the outer wall of the tip using a clean towel or tissue paper.
- **Touch** the tip to the inside wall of a beaker and let the liquid flow until the meniscus is perfectly aligned with the calibrated mark.

PIPET

…cont.:

- **Transfer** the pipet to the receiving flask and let out the liquid freely by gravity feed while **touching** the tip of the pipet to the wall of the receiving flask to avoid splashing.
- After the flow has stopped, place the tip to the inside wall of the receiving flask for a few seconds to allow **complete drain**. The pipet should be positioned almost vertical at the end of the liquid transfer.
- After using the pipet, it should be **rinsed** using distilled water or immerse it in a cleaning solution until you are ready to clean it.

MICROPIPET

Technique of using micropipet:

- Insert the tip snugly into the barrel.
- Set the volume required using the micropipet knob. Push the plunger to a stop that corresponds with the volume.
- Hold the micropipet vertically and dip the tip 3-5 mm into the reagent solution and slowly release the plunger to suck the liquid.
- While lifting the tip from the liquid container, slide the tip against the wall of the container.

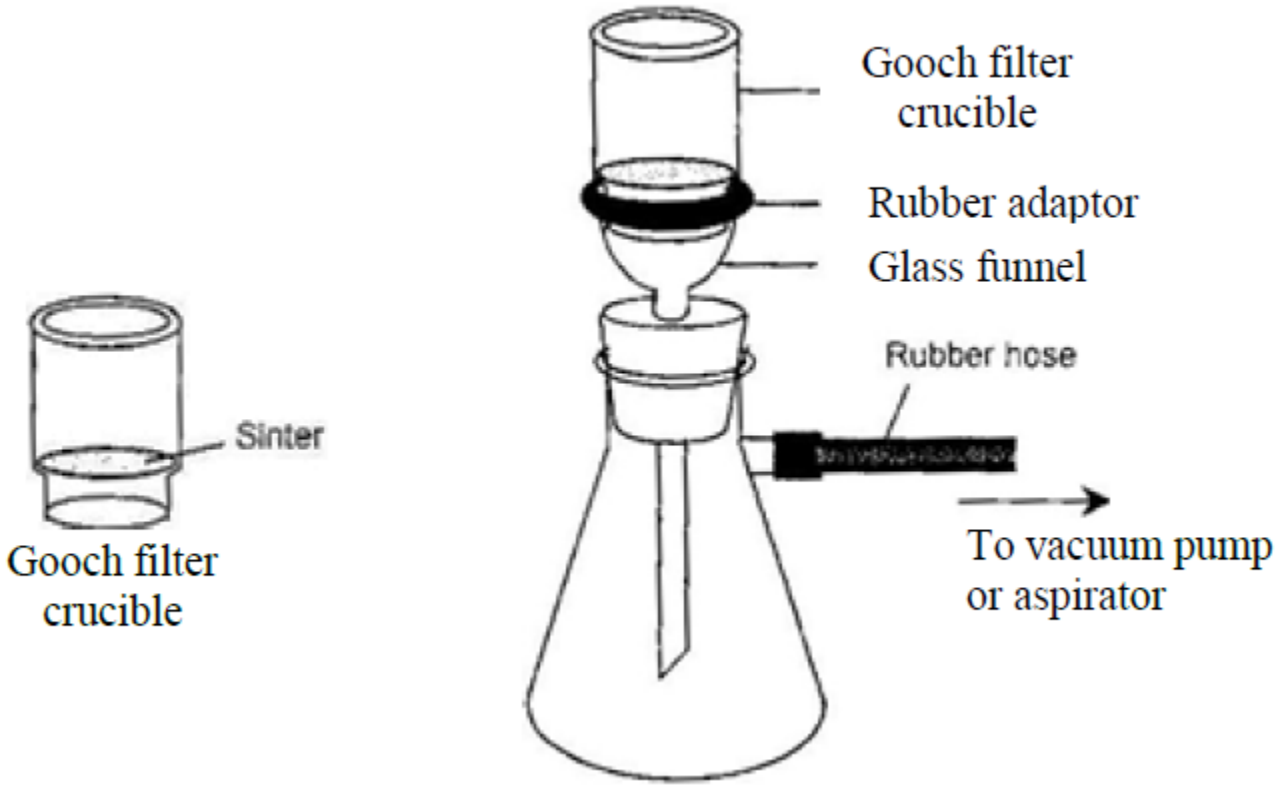
- To dispense the liquid, touch the tip end to the wall of the receiving flask and push the plunger to the set stop. Wait for a few seconds to let the liquid flow down the tip wall and push the plunger to the end to remove all liquid.
- Used tips can be discarded or washed properly for reuse.

SYRINGE

Technique of using syringe:

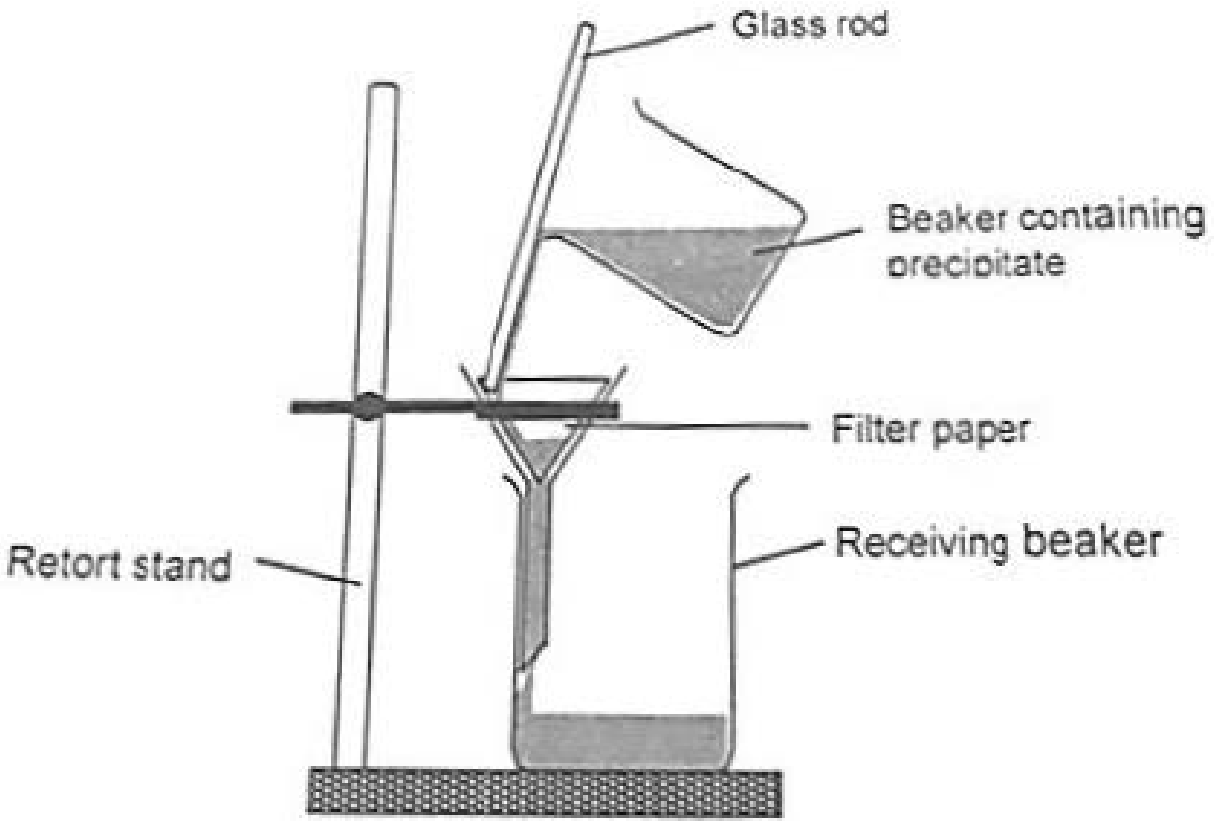
- Suck in a little liquid and discard the liquid and repeat this a few times to wash the syringe walls and to remove gas bubbles from the syringe barrel.
- The stainless steel needle can be reacted with strong acid solutions and it can contaminate the solution with ferrum ions.
- A syringe can also be used for measuring certain volume of a certain reagent.

FILTRATION



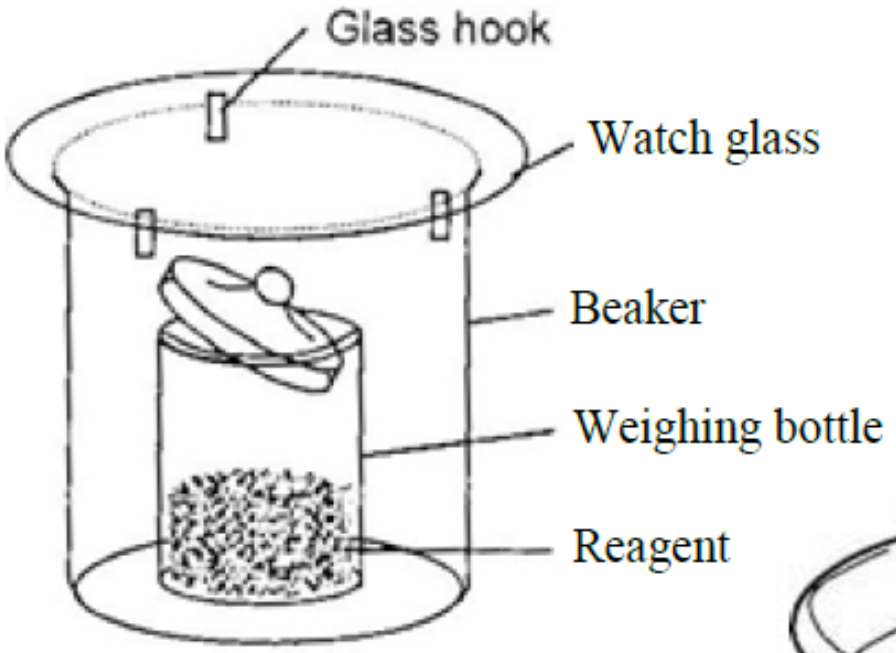
Sintered filter crucible

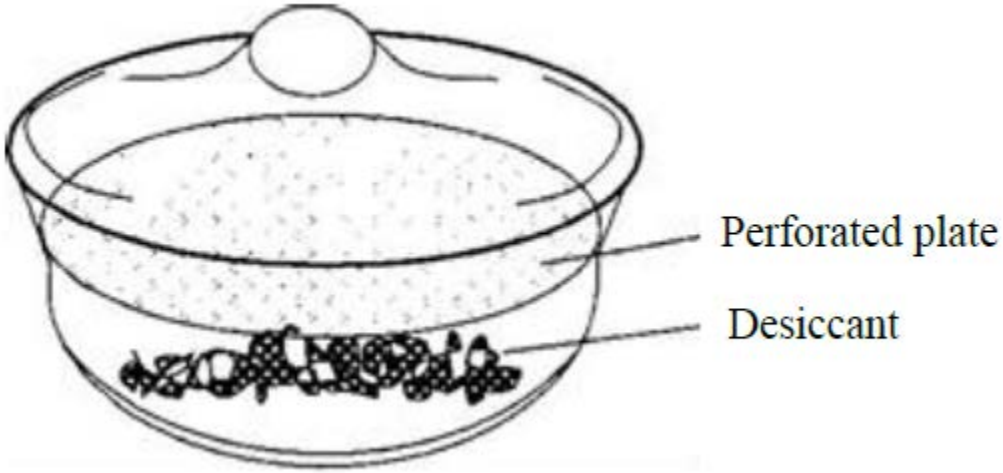
FILTRATION



Filter funnel & filter paper

DRYING





CALIBRATION OF GLASSWARE

Calculation for the Calibration of Volumetric Glassware:

- The weight of the object is corrected by taking into account the **buoyancy**.
- The volume of the glassware at calibration temperature (T) is calculated by **dividing the actual weight by the density of the liquid at the specified temperature**.
- The volume is **corrected** to the volume at the **standard temperature of 20°C**.

CALIBRATION OF GLASSWARE

i. Volumetric Pipet

ii. Burette

iii. Volumetric Flask

CALIBRATION OF GLASSWARE

Table 3.8: Volume occupied by 1.000 g of water weighed in air against a stainless steel weight.*

Temperature/°C	Volume* at temperature T	Corrected to 20°C
10	1.0013	1.0016
11	1.0014	1.0016
12	1.0015	1.0017
13	1.0016	1.0018
14	1.0018	1.0019
15	1.0019	1.0020
16	1.0021	1.0022
17	1.0022	1.0023
18	1.0024	1.0025
19	1.0026	1.0026
20	1.0028	1.0028
21	1.0030	1.0030
22	1.0033	1.0032
23	1.0035	1.0034
24	1.0037	1.0036
25	1.0040	1.0037
26	1.0043	1.0041
27	1.0045	1.0043
28	1.0048	1.0046
29	1.0051	1.0048
30	1.0054	1.0052

* Corrections for buoyancy (stainless steel weight) and changes in volume of the flask have been taken into account.

CALIBRATION OF GLASSWARE

EXAMPLE:

A 25-mL pipet delivers 24.976 g of water when calibrated against a stainless steel weight at 25°C. Use the data in Table 3.8 to calculate the volume of water delivered by the pipet at the temperature and at 20°C.

- At 25°C $V = 24.976 \times 1.0040 = 25.08$ mL
- At 20°C $V = 24.976 \times 1.0037 = 25.07$ mL

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