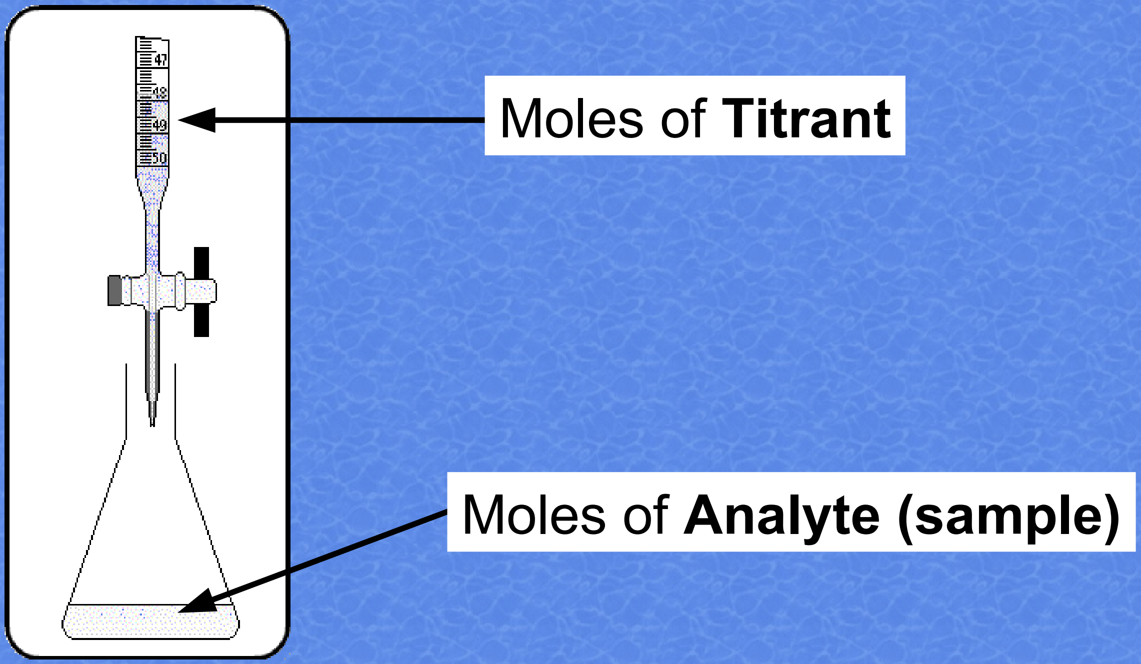
5: Titrations and Standardization

I. Titration and Equivalence
II. Primary Standards
III. Burets
IV. Today's Lab

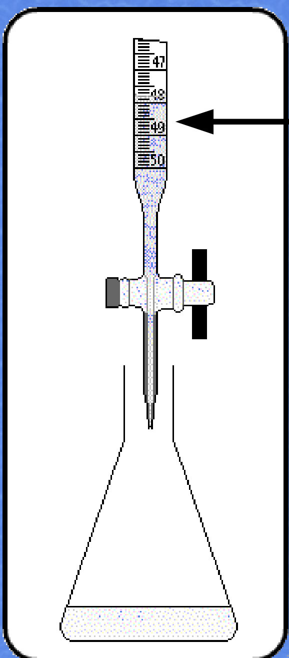
Titration

<u>Definition</u>: Determining number of **moles** of an *analyte* in *sample* by reaction with known amount of **moles** in a *titrant*.



Calculating Moles

Volume × Concentration = Moles
(L)   (mol/L)   (mol)



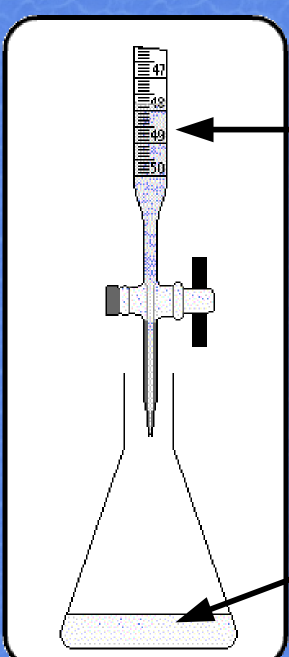
Moles of **Titrant**

ex: moles in 22.52 mL of 0.1025 M

0.02252 L × 0.1025 mol/L = 0.002308 mol
or
22.52 mL × 0.1025 mol/L = 2.308 mmol

Equivalence Point

Moles of titrant and analyte stoichiometrically equivalent



Moles of **Titrant**

Stoichiometrically equivalent

Moles of **Analyte (sample)**

Equivalence Point

$$\text{Mol titrant added} \times \frac{\text{stoic. moles analyte}}{\text{stoic. moles titrant}} = \text{mol analyte in sample}$$

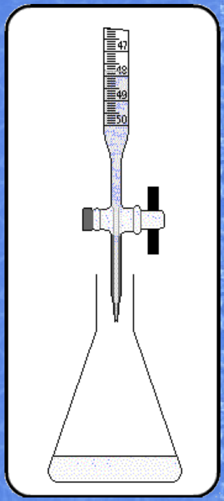
Ex: 5 t + 3 a ⟶ 2 p

Moles of **analyte** (a) in sample if 0.25 mol **titrant** (t) needed to reach the equivalence point?

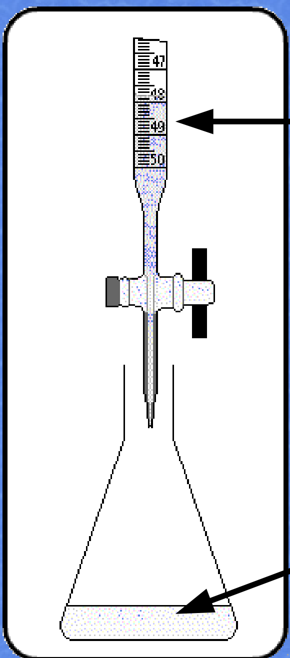
$$0.25 \text{ mol titrant} \times \frac{3 \text{ mol analyte}}{5 \text{ mol titrant}} = 0.15 \text{ mol analyte in sample}$$

Requirements for Titration

1. Known amount of sample (mass or volume)
   - Use analytical balance or volumetric pipette
2. Known concentration and volume of titrant
   - Standardize concentration
   - Use buret
3. Ability to detect equivalence point
   - Indicator, pH meter, etc.
4. Stoichiometry of reaction



Requirements for Titration



Titrant

- Known concentration
- Known volume added

Analyte (sample)

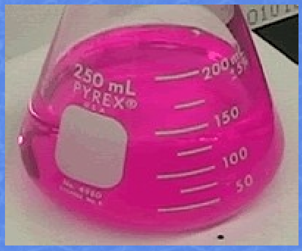
- Known volume or mass
- Calculate moles

How do we find Equivalence Point?

We cannot "see" molecules or reaction at molecular level!

<u>End point</u>: Detectable chemical or physical change in solution occurring "at" equivalence point.

- Often color change



End and Equivalence points NOT necessarily the same!!

Making Titrant

Many acids / bases purchased in "Concentrated" form

- Concentrations <u>approximate</u>

Name	% by Weight	Density (g/mL)	Molarity (mol/L)
Acetic (HCH_3CO_2)	99.8	1.05	17.4
Hydrochloric (HCl)	37.2	1.19	12.1
Nitric (HNO_3)	70.4	1.41	15.8
Phosphoric (H_3PO_4)	85.5	1.69	14.7
Sulfuric (H_2SO_4)	96	1.84	18
Ammonia (NH_3)	28	0.9	14.8

Making Titrant

$$\text{wt \%}\ \frac{(g_{solute})}{(g_{soln})} \times \text{density}\ \frac{(g_{soln})}{(mL_{soln})} \times 1/MW\ \frac{(mol_{solute})}{(g_{solute})} \times \text{Vol}\ (mL_{soln}) = \text{Moles}\ (mol_{solute})$$

$$\text{Volume (L)} \times \text{Concentration (mol/L)} = \text{Moles (mol)}$$

Primary Standards

How do you ensure the concentration of titrant is known as accurately as possible?

I. Most acids and bases used in titration (e.g., HCl) made from stocks of approximate conc.

II. Must standardize titrant against standard that can be accurately weighed (Primary Standard)

III. Primary Standards can be purchased in very pure form and heated to remove water

IV. Keep Primary Standards in desiccator

V. DO NOT CONTAMINATE Primary Standards

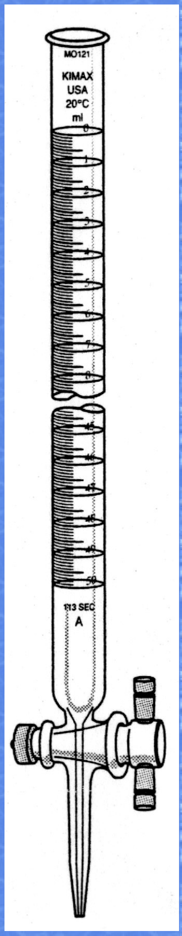
Burets

I. Must be scrupulously clean!
   A. Water sheets off clean buret
   B. Clinging drops means it's dirty!!

II. Clean with soapy water and a brush

III. Rinse several times with tap water

IV. Final rinse 3 times with distilled water



Filling Burets

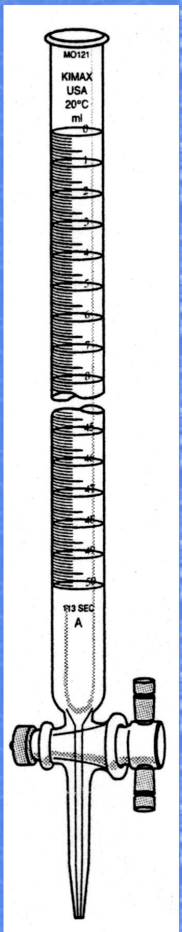
I. NEVER FILL OVER YOUR HEAD

II. Always use a funnel to fill a buret

III. Always fill buret with it in clamp

IV. Check stopcock is ***closed***

V. Place <u>waste beaker</u> under tip



Filling Buret

I. Rinse with titrant 2 times:

   A. Add small volume (<10 mL) to buret

   B. Open stopcock and drain a few mL into waste

   C. Close stopcock

   D. Turn buret on side and roll in fingers to wash walls

   E. Invert and pour contents into waste container

II. Fill buret a bit above 50 mL

   A. Drain below 50 mL to remove air in tip

   B. Do NOT waste time trying to reach 50.00 mL!

Using Buret

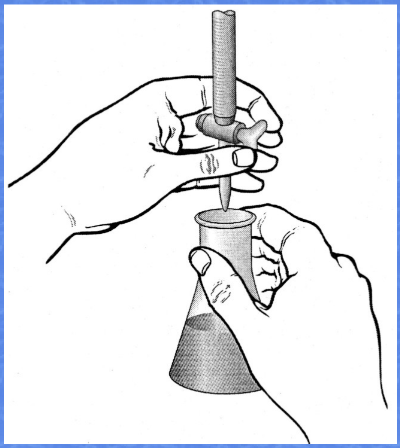
I. Buret tip BELOW top of receiving flask

II. "Opposite" grasp preferred for manipulating stopcock (as shown in the picture)

   A. Supports buret

   B. Avoids slipping and loosening of stopcock

III. Hold flask in good hand for swirling



Today's Lab

<u>Prepare and Standardize HCl</u>

- Prepare HCl from conc. HCl
- Weigh Primary Standard
- Titrate!

Today's Titrant: HCl

Needed: 1 L of 0.1M
1 L × 0.1mol/L = 0.1 mol HCl

$$\text{wt \%} \times \text{density} \times 1/\text{MW} \times \text{Vol} = \text{Moles}$$

$$0.372\ \frac{(g_{solute})}{(g_{soln})} \times 1.19\ \frac{(g_{soln})}{(mL_{soln})} \times 1/36.46\ \frac{(mol_{solute})}{(g_{solute})} \times \text{Vol}\ (mL_{soln}) = 0.1\ \text{mol}\ (mol_{solute})$$

Vol = 8.2 mL of conc HCl in 1 L soln

Today's Titrant: HCl

Needed: 1 L of 0.1M
1 L × 0.1mol/L = 0.1 mol HCl

$$\underset{(L)}{\text{Volume}} \times \underset{(mol/L)}{\text{Concentration}} = \underset{(mol)}{\text{Moles}}$$

$$\underset{(L)}{\text{Vol}} \times \underset{(mol/L)}{12.1} = \underset{(mol)}{0.1}$$

Vol = 8.2 mL of conc HCl in 1 L soln

Today's Standard

Sodium Carbonate

$$2\ HCl + Na_2CO_3 \longrightarrow CO_2 + 2NaCl + H_2O$$

Today's Standard

Sodium Carbonate

$$2\ HCl + Na_2CO_3 \longrightarrow CO_2 + 2NaCl + H_2O$$

<u>Notice</u>:

2 moles HCl : 1 mole Na_2CO_3

<u>Ratios</u>:

$$\frac{2 \text{ moles } HCl}{1 \text{ mole } Na_2CO_3} \qquad \frac{1 \text{ mole } Na_2CO_3}{2 \text{ moles } HCl}$$

Today's Standard

Sodium Carbonate

$$2\,HCl + Na_2CO_3 \longrightarrow CO_2 + 2NaCl + H_2O$$

Needed: Mass of Na_2CO_3 to react with ~25 mL of 0.1 M HCl.

25 mL × 0.1mol/L = 2.5 mmol HCl

$$2.5\text{ mmol HCl} \times \frac{1\text{ mol } Na_2CO_3}{2\text{ mol HCl}} = 1.25\text{ mmol } Na_2CO_3$$

$$1.25\text{ mmol } Na_2CO_3 \times 105.99\text{ g/mol} = 132\text{ mg} = 0.132\text{ g}$$

Today's Standard

Sodium Carbonate

$$2\,HCl + Na_2CO_3 \longrightarrow CO_2 + 2NaCl + H_2O$$

Carbon Dioxide Interference:

$$CO_2 + 2H_2O \longrightarrow H_3O^+ + HCO_3^-$$

Boil solution near end point to eliminate CO_2

Tips for Performing Titration

- DO NOT forget to add the indicator!
- Sample concentration approximately known?
  - Yes: Calculate approximate vol of titrant needed
  - No: “Waste” the first sample: titrate rapidly to find approximate end point. (Do NOT count this in final calculation)
- Perform titrations:
  - Rapidly drain titrant to within few mL of endpoint
  - Carefully titrate last few mL
  - Touch flask to buret tip to get drop
  - Rinse down flask, but not the buret tip
- SAVE YOUR HCl SOLUTION in a 1L plastic bottle for future labs.