PREPARATION AND STANDARDIZATION OF SOLUTIONS

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INTRODUCTION: Standard solutions

- The solutions with known strength.
- Calibration of other solutions and reagents depends upon the accurate strength of these solutions.
- Prepared by using certain substances (known as standard substances) having typical characteristics.

- Two Types of substances which are generally employed for preparing standard solutions:
  - Primary standard substances
  - Secondary standard substances

Primary standard substances

- Substances which can easily be obtained in pure and crystalline form e.g. Oxalic acid, sodium carbonate etc

Characteristics of primary standard substances

- It should be easy to obtain, to purify and to preserve.
- It must not be hygroscopic.
- It should not decompose at ordinary temperature.
- It should be readily soluble under the conditions in which it is used.
- Its reaction with other reagents should be quantitative and practically quick.
- It should have high eq. wt. so that the error due to weighing is minimized.
- It should be fairly cheap.

Sodium Carbonate

Primary standard substances used for Acid- Alkali titration

- *Sodium carbonate (eq. wt. 53 for anhydrous)*
  - Can be easily obtained in pure state
  - Its standard solution is prepared by directly dissolving a known weight of it in water and making the solution to a known volume.

Normality of solution	N/10	N/20
Sodium carbonate (anhydrous)	5.3g/l of solution	2.65g/l of solution

  - Used for finding strength of HCl, H_2SO_4 etc whose standard solutions cannot be prepared directly

- *Oxalic acid $(COOH)_2$ (eq. wt. 63 as it has two molecules of water of hydration)*
  - available in pure state

Normality of solution	N/10	N/20
Oxalic acid (hydrated)	6.3g/l of solution	3.15g/l of solution

  - Employed to find the strength of solutions of alkalies (NaOH and KOH) whose standard solutions cannot be prepared by the direct method

Preparation of some primary standard solutions

- Prepared by using standard substances.
- Known quantity of standard substance is dissolved in distilled water and desired volume is made.
- These substances have

Constant weight | High purity | Non hygroscopic

- Therefore, solution obtained is of

Known and definite concentration

The examples of such solutions:

Standard N/10 oxalic acid solution (Primary standard)

- Weigh 6.3g Oxalic acid (Hydrated salt)
- Dissolve in distilled water
- Make up the volume to one liter in volumetric flask
- used to find the strength of solutions of alkalies like NaOH, KOH (Secondary standards) whose standard solutions can not be made by direct weighing.

Standard N/10 sodium carbonate solution (Primary standard)

- Weigh 5.3g pure anhydrous salt
- Dissolve in distilled water
- Make up the volume to one liter in volumetric flask
- is used to find out the strength of solutions of acids like HCl, H_2SO_4, HNO_3 etc whose standard solutions cannot be prepared directly.

Secondary standard substances

- Those substances or reagents which cannot be obtained in a sufficient pure state
- e.g. NaOH, KOH, HCl, H_2SO_4, HNO_3

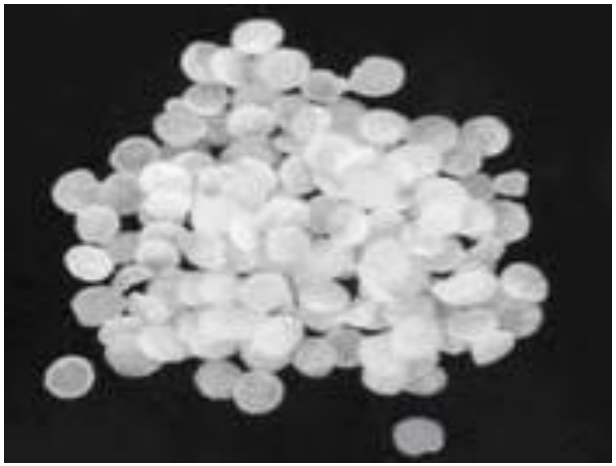


Fig. Sodium Hydroxide pellets

Preparation of some secondary standard solutions

For example: 1. Standard alkalies
2. Standard acids

Preparation of standard alkalies
N/10 NaOH

- Prepare concentrated stock solution
  - Say, 50% of NaOH by dissolving equal parts of NaOH pellets (50 gm) & water (50 gm) in a flask
- Keep it tightly stoppered for 3-4 days
- Use the clean, supernatant liquid for preparing N/10 solution
- Approximately 8 ml of this stock solution (50%) is required per litre of distilled water.

- This will give ***approximate solution***
- For standardization, take this solution i.e. approximate N/10 NaOH solution in burette

- Take 10 ml of standard N/10 oxalic acid (primary standard) solution in conical flask
- Add 2- 3 drops of phenolphthalein indicator to it.
- Add approximate N/10 NaOH solution from burette to the conical flask containing standard oxalic acid solution by continuous mixing by swirling the flask

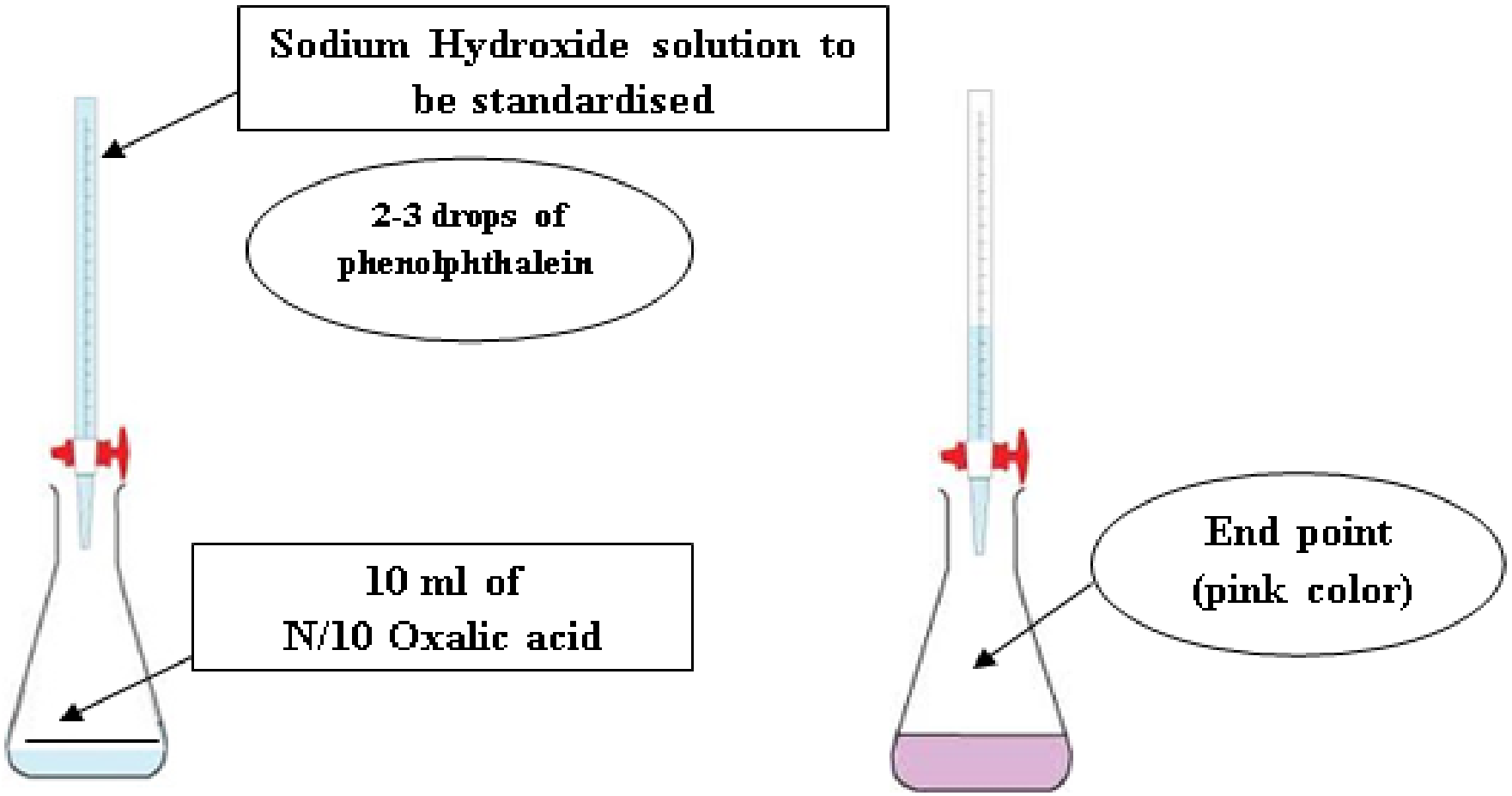
Till apearance of pink colour

- Note down the volume of approximate N/10 NaOH solution used in the titration of 10 ml of standard oxalic acid.
- Calculate the normality of the unknown sodium hydroxide solution by using Normality equation

$$N_1V_1 = N_2V_2$$

(Base) (Acid)

Note: Ideally for 10 ml of N/10 oxalic acid solution, exact 10 ml of N/10 NaOH solution should be consumed.



Standard Solutions

If volume of approx. NaOH used in the titration is less than 10 ml, means solution is strong and its normality is not N/10, so dilute the basic solution and again standardize with standard oxalic acid solution till normality of approximate solution is same as that of standard solution.

$$N1 = \frac{N2V2}{V1}$$

N_1 = Normality of NaOH solution. (?)

V_1 = Volume of NaOH solution used. (ml)

N_2 = Normality of standard oxalic acid solution. (0.1 N)

V_2 = Volume of standard oxalic acid solution. (10 ml)

If volume of approx. NaOH used in the titration < 10 ml

Solution is strong and its normality is not N/10

dilute the basic solution

and

again standardize with standard oxalic acid solution

Till

Normality of approximate solution is same as that of standard solution

Preparation of standard acids: before going for the preparation of standard acids, first let us see the strength of concentrated acids generally available

Approximate Strength of Concentrated Acids Generally Available

Sr. No.	Acids	Sp. Gr.	Approx. Strength
1	Acetic acid glacial CH_3COOH	1.05	16N
2	Hydrochloric acid HCl	1.16	12N
3	Nitric acid HNO_3	1.42	16N
4	**Sulphuric acid H_2SO_4**	**1.84**	**36N**

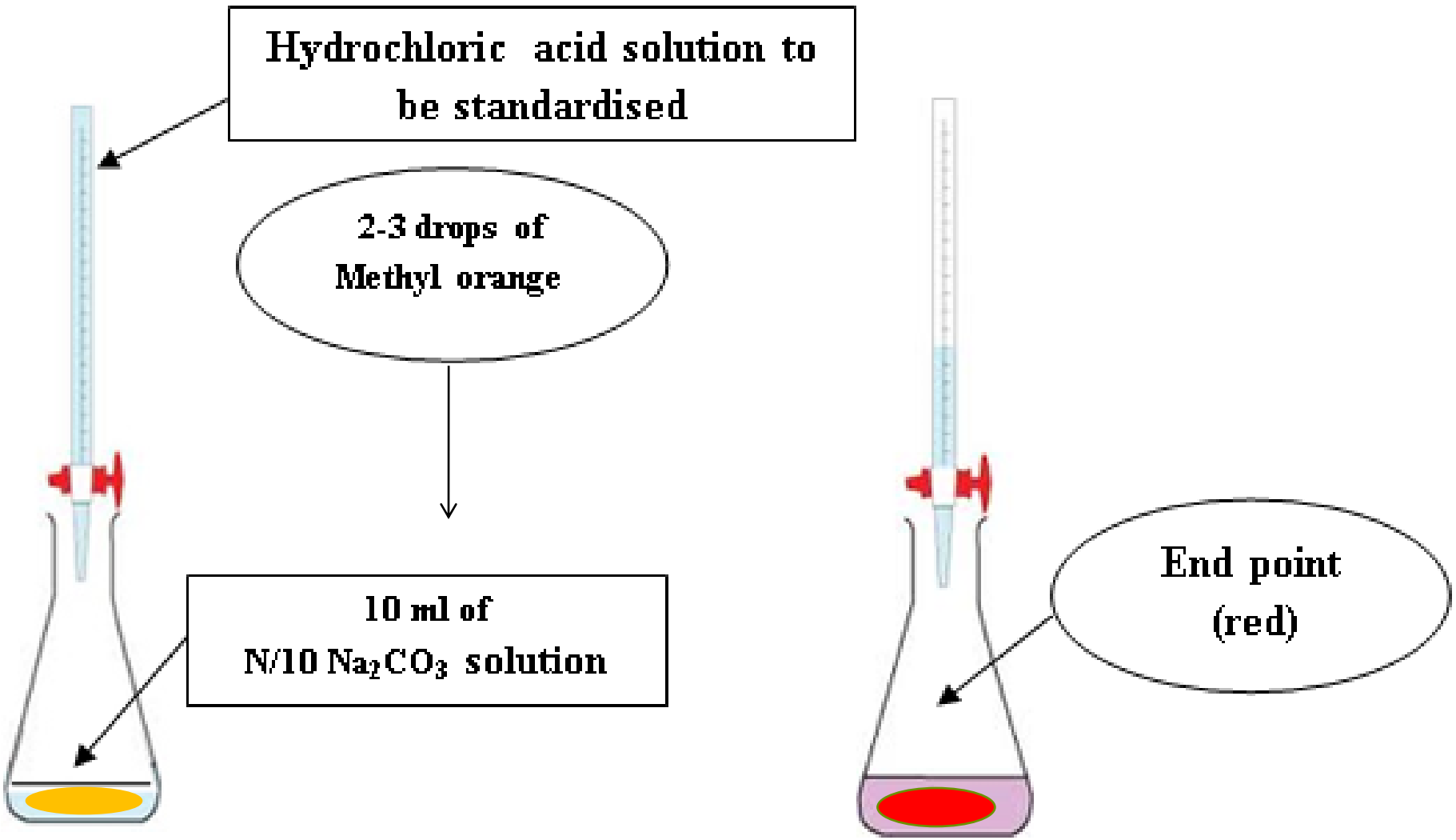
Preparation of N/10 HCl: following two ways can be used:

- First, prepare approximately 0.1 N solution on the basis of the strength given on the label (usually 12 N) by diluting it 120 times with distilled water

- Standardize it against standard N/10 Na_2CO_3 using methyl orange as an indicator.

- Firstly, prepare approximately 0.1 N solution on the basis of the strength given on the label (usually 12 N) by diluting it 120 times with distilled water

- Standardize it against standard N/10 NaOH which is already standardized against N/10 oxalic acid, using Phenolphthalein indicator



Standard Solutions

Preparation of N/10 H_2SO_4 : before its preparation, one must observe the cautions for handling H_2SO_4

- **Precautions :**
  - **Concentrated H_2SO_4 is very corrosive in nature, therefore, it should be handled carefully.**
  - **<u>Always remember</u>: "ADD ACID TO WATER" under cold conditions. This is done to avoid bumping due to the heat generated.**

- For preparation of N/10 H_2SO_4, take 10 ml of concentrated H_2SO_4 (usually about 36 N)
- Dilute 36 times by adding acid in small quantity to distilled water in a cold water bath to make it 1N
- Dilute this 1N solution further 10 times to make it N/10
- Standardize against standard N/10 NaOH or N/10 KOH using phenolphthalein indicator

Preparation of N/10 HNO_3

- Take 10 ml of concentrated HNO_3 (usually about 16 N)
- Dilute 16 times by adding acid to distilled water to make it 1N
- Dilute this 1N solution further 10 times to make it N/10.
- Standardize against standard N/10 NaOH or N/10 KOH using phenolphthalein indicator.

Preparation of Some Other Reagents

1 Preparation of chromic acid (Cleaning solution) H_2CrO_4

- Dissolve 50 g of $K_2Cr_2O_7$ in 50 ml of water in a beaker kept in cold water.
- Add slowly 500 ml of concentration H_2SO_4 and cool.

- It is a very corrosive solution and care should be taken to avoid its coming in contact with the skin.
- It is almost a saturated solution of $K_2Cr_2O_7$ in concentrated H_2SO_4.
- Here also, during preparation of chromic acid, observe the precaution for handling of conc. H_2SO_4 i.e. 'ADD ACID TO WATER', under cold conditions (in a cold water bath)

4. Preparation of 0.1 N sodium thiosulphate solution ($Na_2S_2O_3.5H_2O$)

1. Dissolve approximately 24.8 g of sodium thiosulphate crystals in previously boiled and cooled distilled water.
2. Make the volume to 1000 ml.
3. Store the solution in a cool place in a dark colored bottle.
4. After storing the solution for about two weeks, filter if necessary and standardize.

Standardization of sodium thiosulphate

- Weigh accurately about 5.0 g of finely ground potassium dichromate which has been previously dried to a constant weight at 105 ± 2° in to a clean 1.0 litre volumetric flask.
- Dissolve in water make up to the mark; shake thoroughly and keep the solution in dark place.
- Pipette 25.0 ml of this solution into a clean glass stoppered 250 ml conical flask.
- Add 5.0 ml of concentrated hydrochloric acid and 15.0 ml of 10% potassium iodide solution.
- Allow to stand in dark for 5 minutes and titrate the mixture with the solution of sodium thiosulphate using starch solution as an indicator towards the end.

End point: Blue color to Green color

- The normality (N) of the sodium thiosulphate can be calculated as:

- $N = 25W \div 49.03V$

Where,

- W is weight in g of the potassium dichromate
- V is volume in ml of sodium thiosulphate solution required for the titration.

5. Preparation of 0.1 N standard solution of silver nitrate

1. Dissolve slightly more than the required quantity (17.2 g instead of 16.989 g) of reagent grade silver nitrate in distilled water.
2. Dilute to one liter in a volumetric flask.
3. Weigh accurately 0.5844 g of NaCl (dried at 110°C before weighing) and transfer to a 100 ml volumetric flask and add 50 ml of halogen free water to dissolve the material. Make up the volume with distilled water to the mark and mix the contents.
4. Pipette out 10 ml of the prepared standard sodium chloride solution in 100 ml conical flask and add 2-3 drops of potassium chromate indicator (5% solution in water).
5. Titrate with silver nitrate solution until a perceptible reddish brown color appears.

- Carry out a blank titration using 10 ml of distilled water instead of sodium chloride solution.
- Deduct the blank reading from the reading for the standard sodium chloride solution.

Normality of the silver nitrate solution is calculated using normality equation:

- $N_1V_1 = N_2V_2$

Where,

N_1 = Normality of standard sodium chloride solution (0.1N)

V_1 = Volume in ml of sodium chloride used for titration (10 ml).

N_2 = Normality of prepared silver nitrate solution.

V_2 = Volume in ml of prepared silver nitrate solution used for titration.

6. Preparation of EDTA solutions

1. **Preparation of 0.01 M EDTA solution**:

   Dissolve 3.8 g of disodium ethylene diamine dihydrogen tetra acetate (EDTA, M.Wt. 372.25) in distilled water

   Volume made to 1 liter with distilled water

   Mix well and store in polyethylene reagent bottle

   It is standardized against 0.01 M $CaCO_3$ or $CaCl_2$

2. **Preparation of 0.01 M $CaCl_2$ solution:**

Prepare standard Ca solution i.e. 1ml = 1mg $CaCO_3$, M.wt. 100

Weigh 1g $CaCO_3$ into 500ml conical flask or beaker

Add dilute HCl through funnel until $CaCO_3$ is dissolved

Add 20 ml water, boil to expel CO_2 and cool.

Add few drops of methyl red indicator

[adjust colour intermediate orange (brownish red) with dilute NH_4OH or HCl as required].

Transfer quantitatively to 1 L volumetric flask and make up volume to the mark.

Shake it well and store it well and store in air-tight reagent bottle.

3. **Erichrome Black T indicator**:

Dissolve 0.5 g of Erichrome black T in 100 ml of triethanolamine

OR

0.4 g in 100 ml methanol

4. **Buffer solution**:

Dissolve 16.9 g NH_4Cl in 143 ml NH_4OH

Dilute to 250 ml with water.

Store in tightly stoppered Pyrex of plastic bottle

Dispense from bulb-operated pipette

Discard after 1 month or when 1-2 ml added to sample fails to produce pH 10.0±0.1 at end point titration.

7. Standardization of EDTA solution:

- Rinse and then fill burette with prepared EDTA solution.
- Pipette 25 ml of standard $CaCO_3$ solution into 250 ml Erlenmeyer flask
- **Add** 1 ml ammonia buffer

  (to raise the pH as reaction takes place at high pH)
- **Add** 3-4 drops of Erichrome black T indicator
- Titrate the EDTA solution until colour changes from wine red to dark blue with no reddish tinge remaining.
- Calculate the molarity of EDTA ($M_1V_1 = M_2V_2$),

[if excess, follow the procedure for the standardization, recheck the molarity and it should be 0.01 M]

8. Preparation of Fehling solution

- Used for the estimation of reducing sugars
- Generally prepared fresh by mixing equal quantities of Fehling's A and Fehling's B which are prepared separately as follows:

a) Fehling's A

- Dissolve 34.639 g $Cu(SO)_4.5H_2O$ in distilled water
  - Add 0.5 ml concentrated H_2SO_4, mix and
  - Make the volume to 500 ml. Filter if necessary.

b) Fehling's B

- Dissolve 173 g of Rochelle salt (Na K tartarate) and 50 g of NaOH in distilled water.
- Allow to stand for two days.
- Filter if necessary.

Standardisation of Fehling's solution:

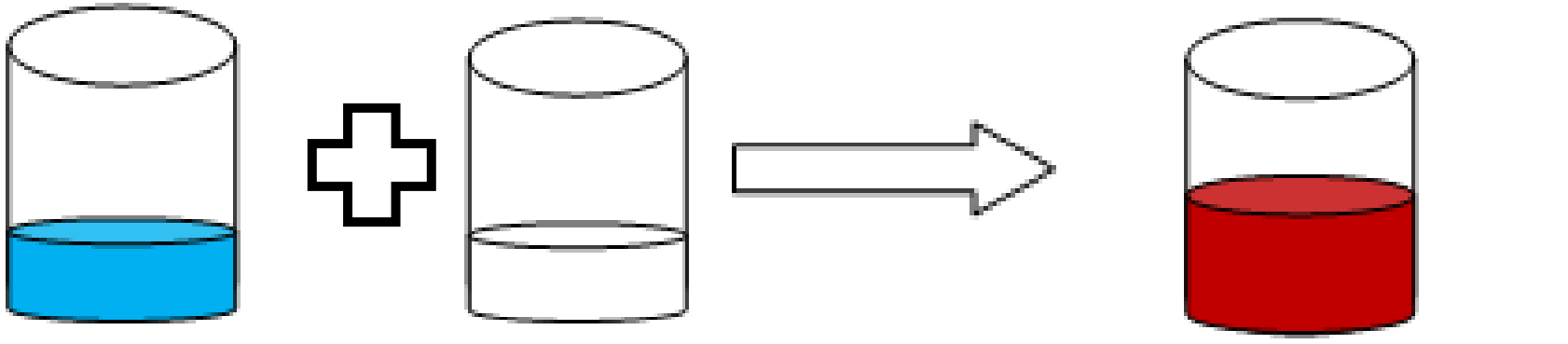
1. 5 ml of Fehling's solution A + 5 ml of Fehling's solution B

using two separate pipettes in a 250 ml Erlenmeyer flask

2. Fill up a burette with the standard lactose solution
3. Heat the content of the flask to boiling over burner or heater and maintain moderate boiling for 2 min (add some inert boiling chips to prevent bumping)
4. Add 3 to 4 drops of methylene blue indicator (0.2% in water) without removing from the flame.
5. Titrate the content of the flask against standard lactose solution (0.5%) from the burette until the blue colour disappears and the bright brick-red colour of precipitated Cu_2O appears.

- 6. At the end point the Cu_2O suddenly settles down giving a clear supernatant.
- 7. Note the volume of lactose solution required for the standardization of Fehling's solution.
- 8. After this preliminary titration, further titration or titrations should be carried out, adding practically the whole of the standard lactose solution volume (one ml less than required as observed in first titration) required for the titration before commencing the heating.
- 9. Let the contents boil for 2 minutes. Now, add 3-4 drops of methylene blue indicator, continue heating and complete the titration within 3 min from the commencement of boiling.
- 10. Let V_1 ml be the titre for this experiment.

- 11. Multiply the titre value by mg/ml lactose of the standard solution to obtain total lactose required to reduce the copper and term the value a "Factor F".



Fehling A solution Fehling B solution brick-red Precipitates of Cu_2O

Note: Carefully note the first disappearance of blue colour. Once missed, it is difficult to ascertain the end point. Maintain the boiling at a uniform rate during the titration.

9.Preparation of pH indicator solutions

- **Phenolphthalein indicator solution:**

Weigh 1.0 g phenolphthalein

place the powder in a 100 ml volumetric flask containing about 50ml of 95% ethanol

Stopper and shake vigorously for a few minutes

add 20ml more ethanol and shake until a clear solution is formed

make the volume to 100 ml

- **Methyl orange indicator solution**

1. Dissolve 1.0 g of methyl orange powder in distilled water
2. dilute to one litre
3. Filter, if necessary

Thank You

2. Preparation of Gerber sulphuric acid

- In Gerber fat test, Gerber Sulphuric acid is used to dissolve casein in milk.
- **concentrated H_2SO_4** → Causes → Charring of organic matter

  Dilute H_2SO_4 → Causes → Casein is precipitated & not dissolved

- Therefore, the concentration of sulphuric acid → So adjusted to just strong enough to dissolve the casein without charring the fat.
- Also, acid produces necessary heat to keep the fat in the liquid state

Gerber H_2SO_4

- Strength → 90-91%
- Sp. Gr. → 1.807 to 1.812

Concentrated H_2SO_4

- Strength → 97-99%
- Sp. Gr. → 1.835

For practical purposes:

900ml conc. H_2SO_4 + 100ml Distilled water

1000ml Gerber acid

Precaution for handling of concentrated H_2SO_4 and other acids

ADD ACID TO WATER under cold conditions

Preparation

1. Take required vol. of water (say 100 ml) in a flask
2. Keep the flask in a basin of ice-cold water
3. Carefully, add the required quantity of concentrated H_2SO_4 (say 900 ml) in small quantities at a time keeping the container sufficiently cold.
4. Mix gently.

3. Testing the amyl alcohol (a by product of fuel oil refinery) used for fat determination

- Iso-amyl alcohol (also called Iso-butyl carbinol) used in Gerber fat test

Should be clear, colorless

and

Free from impurities particularly fatty mater.

Perform various tests to know purity of iso-amyl alcohol:

- **Density**: At 27°C, Density shall be between 0.803 to 0.805 g/ml (using specific gravity bottle).
- **Boiling point**: Boiling point shall be 128-129°C (can be checked using boiling point apparatus).
- 95% of the liquid shall get distilled between 130-132°C.
- **Test for absence of furfural and other impurities**: 5 ml iso-amyl alcohol + 5 ml H_2SO_4 (97%) → Observe the color → shall not show more than a yellow or light brown color.
- **Test for absence of fatty matter**: Carry out a blank Gerber fat test using distilled water in place of milk. If any fat separation is observed → indicates impurities of some fatty matter.