પુ•ના International School

Shree Swaminarayan Gurukul, Zundal

Class – XI | Subject: Chemistry(Practical)(Term-1&2) | Experiment (2021_22)

Exp. No	Aim
	QUANTITATIVE ANALYSIS(Term-1)
1	Prepare 250 ml of 0.1MSolution of Oxalic Acid From Crystalline Oxalic Acid
2	Determination of Concentration/Molarity of Sodium hydroxide Solution by Titrating it against a 0.1M Standard Solution of Oxalic acid
3	Determination of Concentration/Molarity of dilute hydrochloric acid Solution by Titrating it against a Standard Solution of Sodium carbonate
	QUALITATIVE ANALYSIS(Term-2)
4	To Identify the given inorganic salt [Ba(NO3)2]
5	To Identify the given inorganic salt [Pb(CH3COO)2]
6	To Identify the given inorganic salt Pb(NO3)2
7	To Identify the given inorganic salt PbCl2
8	To Identify the given inorganic salt MgSO4
9	To Identify the given inorganic salt [(NH4)3PO4]
10	To Identify the given inorganic salt [Sr(NO3)2]
	CONTAIN BASED EXPERIMENT(Term-1&2)
11	Purification of sample of Copper Sulphate by Crystallisation
12	Determination of melting point of a solid organic compound.

EXPERIMENT 2.1

Oxalic acid 

Aim

Preparation of 250 mL of 0.1M standard solution* of oxalic acid.

Theory

A solution of exactly known concentration is considered to be a standard solution. There are various ways of expressing the concentration of a standard solution. Standard solution of an acid/base is used to determine the unknown concentration of a solution of bases / acids by volumetric analysis. For example, a standard solution of oxalic acid can be used to determine the unknown concentration of an alkali solution. The strength of a standard solution is usually expressed in moles per litre. The formula of hydrated crystalline oxalic acid is

$$\begin{matrix}COOH\\|\\COOH\end{matrix} \cdot 2H_2O$$

*Learn more about standard solution in Unit-6.

and its molar mass is 126 g. If 126 g of oxalic acid is present in one litre of the solution, it is known as one molar (1.0 M) solution.

For the preparation of one litre of 0.1 M oxalic acid solution, we require $\frac{126}{10} = 12.6\,g$ of hydrated oxalic acid. Therefore, for preparing 250 mL of 0.1 M oxalic acid solution, we require:

$\frac{12.6\,g \times 250\ mL}{1000\ mL} = 3.1500\,g$ of hydrated oxalic acid.

In general for preparing a solution of required molarity, the amount of substance to be weighed can be calculated by using the formula given below :

$$\text{Molarity (M)} = \frac{\text{Mass of solute is grams} \times 1000}{\text{Molar mass of solute} \quad \text{(volume of solution to be prepared in mL)}}$$

Material Required

- Measuring flask (250 mL) : One
- Funnel : One
- Weighing tube/Watch glass : One
- Wash bottle : One
- Iron stand with ring clamp : One

- Oxalic acid : As per need

Procedure

Oxalic acid 

(i) Weigh an empty, clean and dry watch glass/weighing tube accurately (Weight 1).

(ii) Weigh 3.1500 g oxalic acid by placing it on the above watch glass/in a weighing tube (Weight 2). Always note weight up to the fourth decimal place and clean the balance before and after weighing the chemical.

(iii) Transfer oxalic acid carefully from the watch glass/weighing tube into a clean and dry measuring flask using a funnel. Weigh the empty watch glass again (Weight 3) and find out the mass of oxalic acid transferred to the measuring flask by substracting this mass (Weight 3) from the combined mass of watch glass and oxalic acid (Weight 2). Calculate the exact molarity of solution from this mass. Wash funnel several times with distilled water by using a wash bottle to transfer the sticking particles if any into the measuring flask. While washing the funnel, add water in small amounts so that its volume in the flask does not exceed th of the volume of the measuring flask as shown in Fig. 2.27 a, b.

(iv) Swirl the measuring flask till solid oxalic acid is completely dissolved. Add more distilled water with shaking. Make up the volume with distilled water to the etched mark by adding last few mL dropwise. Stopper the flask and shake it thoroughly to make the solution uniform throughout (Fig. 2.27 c, d). Label it as 0.1 M oxalic acid solution.

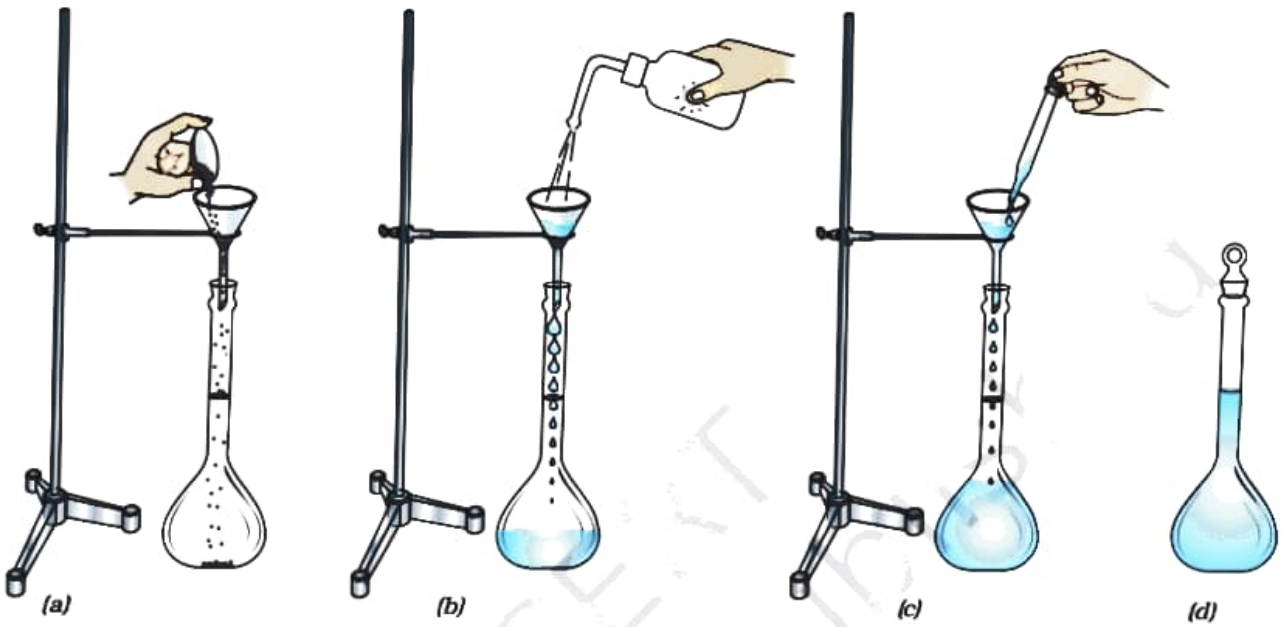


Fig. 2.27 : Making standard a solution
(a) Transfering oxalic acid *(b) Diluting the solution*
(c) Adding last few mL dropwise *(d) Standard solution*

Precautions

(a) The pan of the balance should be cleaned before and after weighing.
(b) Never touch the weights with hand. Use forceps to transfer weights from the weight-box to the pan of the balance.
(c) Always use spatula to transfer the reagent from the bottle on to the watch glass.
(d) Stopper the reagent bottle immediately after withdrawing the substance.
(e) Always use distilled water to prepare the standard solution.
(f) Always check the adjustment of the balance before weighing the substance.
(g) Care should be taken while weighing the chemicals. These should not be spilled on the pan of the balance.
(h) Watch glass/weighing bottle and funnel should be washed several times by using small amounts of distilled water each time.
(i) While making the solution, water should be added carefully so that the lower part of the meniscus just touches the etched mark of the measuring flask.
(j) To ensure uniform composition of the solution, stopper the flask and shake it carefully and thoroughly.

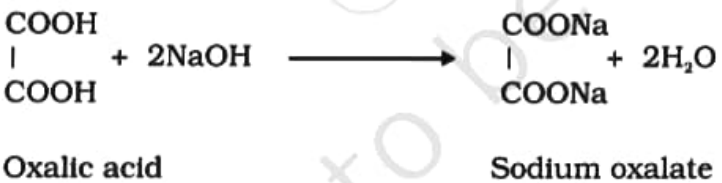
EXPERIMENT 6.1

Aim

Determination of the concentration (strength) of a given sodium hydroxide solution by titrating it against a standard solution of oxalic acid.

Theory

In the titration of a strong acid with a strong base, the amount of acid and base becomes chemically equivalent at the end point and the chemical reaction is called neutralization reaction. Near the end point there is a sudden change in the pH of the solution. If after end point even a small amount of base/acid is added the solution would become slightly alkaline or acidic respectively. In the titration between oxalic acid (weak acid) and sodium hydroxide (strong base), following reaction takes place:



In this titration phenolphthalein (HPh) is used as an indicator. The concentration of unknown solution is calculated in g/L. Molarity of the solution can be calculated by using the formula

$$a_1 M_1 V_1 = a_2 M_2 V_2 \qquad ...(4)$$

where a_1, M_1, V_1 are respectively basicity, molarity and volume of acid used and a_2, M_2 and V_2 are acidity, molarity and volume respectively of base used in the titration.

Material Required

- Burette (50 mL) : One
- Pipette (10 mL) : One
- Conical flask (100 mL) : One
- Burette stand : One
- Funnel : One
- White glazed tile : One
- Measuring flask (100 mL) : One

- Oxalic acid : As per need
- Sodium hydroxide solution : As per need
- Phenolphthalein indicator : As per need

Procedure

Oxalic acid 

Sodium Hydroxide 

(A) Preparation of 0.1M Standard Solution of Oxalic Acid

Follow the procedure as described in Experiment No. 2.1.

(B) Titration of Sodium Hydroxide and Oxalic Acid Solution

(i) Clean the burette thoroughly, wash it with distilled water and finally rinse it with sodium hydroxide solution. (Always rinse the burette (Fig. 2.17) with the solution, which is to be taken in it). Clamp the burette vertically in a burette stand.

(ii) Fill sodium hydroxide solution into the burette through a funnel above the zero mark.

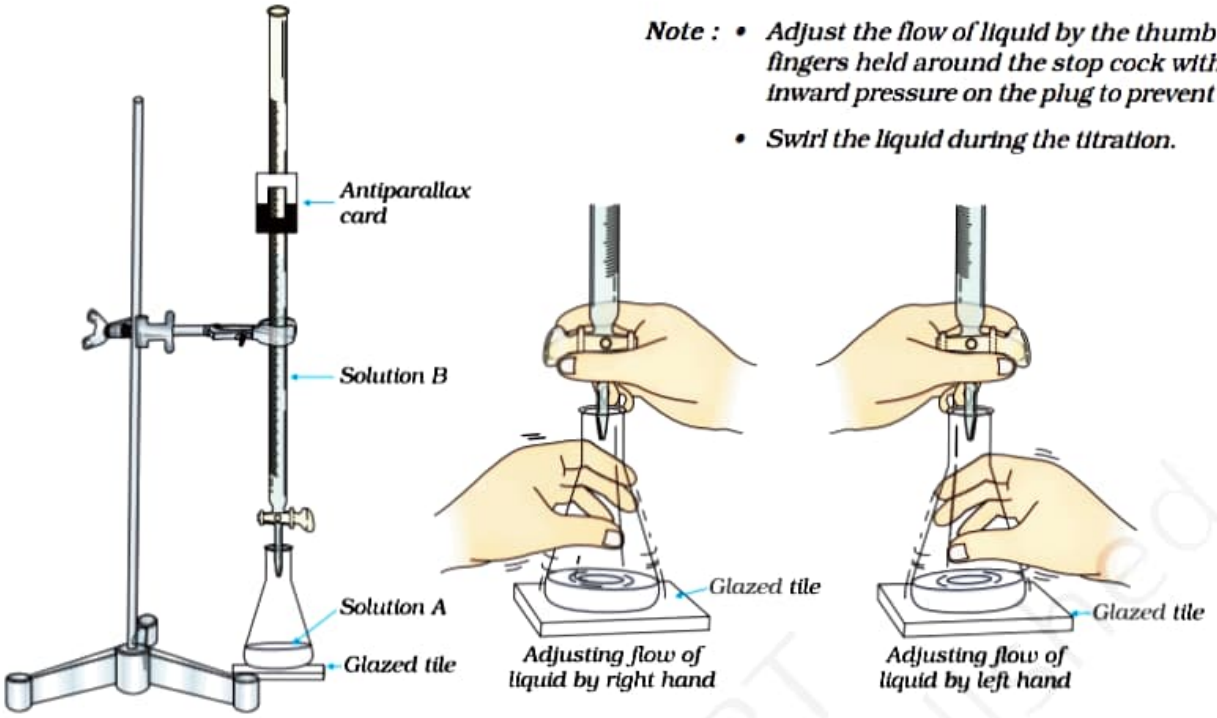
(iii) **Remove the air gap, if any, from the nozzle** of the burette by running the solution forcefully from the burette nozzle.

(iv) **Remove the funnel before noting initial reading of the burette.** Also while noting the reading, see that no drop of the liquid is hanging at the nozzle of the burette.

(v) Note the initial reading by keeping the eye exactly at the same level as the meniscus of the solution.

(vi) Pipette out 10 mL of oxalic acid solution in a washed and dried conical flask. Always wash the pipette with water and rinse (Fig. 2.21) with the liquid to be measured before pipetting out the liquid.

(vii) Add 1-2 drops of phenolphthalein indicator to the conical flask. Place the flask over the glazed tile as shown in Fig. 6.3 Titrate the acid with sodium hydroxide solution till a very faint permanent pink colour is obtained. Add sodium hydroxide solution in small amounts initially and then dropwise.



Note : • *Adjust the flow of liquid by the thumb and two fingers held around the stop cock with a slight inward pressure on the plug to prevent leakage.*

• *Swirl the liquid during the titration.*

Fig. 6.3 : Titrating the solution

(viii) Read the lower meniscus of the solution in the burette again and record it as final reading.

(ix) Repeat the procedure until three concordant readings are obtained. Record your readings as in Table 6.1.

Table 6.1 : Titration of sodium hydroxide vs oxalic acid solution

Sl. No.	Volume of oxalic acid solution taken in conical flask each time V_1 mL	Burette readings		Volume of sodium hydroxide solution used V_2 mL = (y–x) mL	Concordant reading in mL
		Initial reading (x)	Final reading (y)		

Calculations

Molarity of NaOH solution can be calculated by using the equation:

Oxalic acid Sodium hydroxide

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

where, M_1 and V_1 are the molarity and volume of the oxalic acid solution.

M_2 and V_2 are the molarity and volume of the sodium hydroxide solution.

a_1 and a_2 are respectively the basicity of oxalic acid and acidity of sodium hydroxide. In this case $a_1 = 2$ and $a_2 = 1$.

Also, Molar mass of oxalic acid, $(COOH)_2.2H_2O = 126\ g\ mol^{-1}$ and Molar mass of sodium hydroxide (NaOH) = $40\ g\ mol^{-1}$

Calculate the concentration of sodium hydroxide solution in g/L by using the equation given below.

Concentration (strength) in g/L = Molarity Molar mass

Result

Concentration of NaOH solution is ———————— g/L.

Precautions

(a) Always rinse the burette with the solution, which is to be taken in it.
(b) Remove the air gap if any, from the burette before titrating the solution. Make sure that the nozzle of burette is also filled.
(c) Never forget to remove the funnel from the burette before noting the readings of the burette and ensure that no drop is hanging from the nozzle of the burette.
(d) Always read the lower meniscus for all transparent solutions and upper meniscus for coloured solutions.
(e) To note the burette readings place the eye exactly at the level of the meniscus.
(f) Never hold the pipette at the bulb.
(g) Never use the pipette and burette with a broken nozzle.
(h) Never suck a strong acid or an alkali with the pipette.
(i) Always keep the lower end of the pipette dipped in the liquid while sucking the liquid.

Experiment 6.3

Aim

Determination of the strength of a given solution of dilute hydrochloric acid by titrating it against a standard solution of sodium carbonate.

Theory

The strength of hydrochloric acid is determined by titrating it against a standard solution of sodium carbonate. The following reaction takes place:

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

In this titration, methyl orange, a weak base (yellow in the unionised form) is used as an indicator.

In this experiment also, the titration follows the usual course, i.e., the proton furnished by the addition of the acid first neutralises sodium carbonate solution. When the entire sodium carbonate solution is neutralised, the last drop of the acid added from the burette produces the pinkish red colour change, which is the end point.

The concentration (strength) of the unknown solution is calculated in g/L. It is calculated from the molarity of the solution.

Here, the molarity equation is written as

$$\underset{a_1 M_1 V_1}{\text{Base}} = \underset{a_2 M_2 V_2}{\text{Acid}}$$

where, a_1 and a_2 are the acidity and basicity of the alkali and the acid respectively. M_1 and M_2 are the molarities, V_1 and V_2 are the volumes of the base and acid respectively used to neutralise each other.

Material Required

- Burette (50 mL) : One
- Pipette (10 mL) : One
- Conical flask (100 mL) : One
- Burette stand : One
- Funnel : One
- Glazed tile (white) : One
- Measuring flask (100 mL) : One

- Hydrochloric acid : As per need
- Sodium carbonate : As per need
- Methyl orange solution : As per need

Procedure

Hydrochloric acid 

(A) Preparation of 0.1 M standard solution of sodium carbonate

Follow the procedure as described in Experiment 2.1.

(B) Titration of hydrochloric acid and standard sodium carbonate solution.

Follow the procedure as given in the Experiment 6.1.

In this case, hydrochloric acid is taken in the burette and sodium carbonate solution in the conical flask. Methyl orange is used as an indicator. The colour change at the end point will be from yellow to pinkish-red. Record your observations in Table 6.2.

Table 6.2 : Titration of Hydrochloric acid with standard sodium carbonate solution

Sl. No.	Volume V_1 of Na_2CO_3 solution taken in the conical flask each time in mL	Burette readings		Volume of HCl solution used V_2 mL = (y–x) mL	Concordant reading in mL
		Initial reading (x)	Final reading (y)		

Calculations

Calculate the strength of HCl solution by using the equation

Na_2CO_3 solution		HCl solution
$a_1M_1V_1$	=	$a_2M_2V_2$

where M_1 and V_1 are the molarity and volume of sodium carbonate solution respectively and a_1 is the number of moles of OH^- (aq) ions supplied by one mole of the base (i.e. the acidity of the Na_2CO_3 solution).

$\therefore \quad a_1 = 2$

M_2 and V_2 are the molarity and volume respectively of hydrochloric acid solution.

a_2 is the number of moles of H^+ (aq) ions supplied by one mole of the acid (i.e. the basicity of HCl).

$\therefore \quad a_2 = 1$

Molar mass of Na_2CO_3 = 106 g mol^{-1}, Molar mass of HCl = 36.5 g mol^{-1},

$\therefore$ Concentration (Strength) of HCl solution in g/L = Molarity Molar mass

Result

The concentration (strength) of the given HCl solution is _____ g/L.

Precautions

(a) Care should be taken while handling the acid and base.
(b) Always rinse the burette and the pipette with the solution which is to be taken in them.
(c) Remove the air gap if any, from the burette before titration.
(d) Never forget to remove the funnel from the burette before noting the initial reading of the burette and ensure that no drop is hanging from the nozzle.
(e) Always read the lower meniscus for all transparent solutions and upper meniscus for the coloured solutions.
(f) Never use burette and pipette with a broken nozzle.
(g) Never suck a strong acid or an alkali with the pipette, use pipette bulb.
(h) Always keep the lower end of the pipette dipped in the liquid while sucking the liquid.
(i) While transferring the solution to the flask, do not blow out the last drop of the solution from the jet of the pipette.
(j) The strength of the solution must be calculated up to the fourth decimal place.

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→ Aim:- To identify the given inorganic salt [$Ba(NO_3)_2$].

	Experiment	Observation	Inference.
*	Primary test.		
1.	Colour	White	Absence of Cu^{2+}, Fe^{3+}, Co^{+2}, Mn^{+2}
2.	Smell	No specific	NH_4^+, S^{2-}, CH_3COO^- absent.
3.	Gas evolved	A reddish brown gas evolved, which turned $FeSO_4$ sol^n black.	NO_3^- may be Present.
4.	Sublimation	No Sublimation	NH_4^+, I^- may be absent
5.	Description	No description	$[Pb(NO_3)_2]$, NaCl, KBr absente.
6.	Residue	White	Zn^{2+}, Pb^{2+} may be absent
7.	Flame Test. Prepare a paste Salt + conc. HCl	Persistent grassy green flame on prolonged heating	Ba^{2+} may be present
8.	Salt + dil H_2SO_4 (warm)	No gas evolves	CO_3^{2-}, S^{2-}, NO_2 may be absent.
9.	Salt + dil H_2SO_4 adding drops of $KMnO_4$	Pink colour of $KMnO_4$ is lost.	Cl^-, Br^-, I^-, $C_2O_4^{2-}$, Fe^{2+} may be absent.
10.	Heat a pinch of salt and conc. NaOH	No ammonia gas evolved	NH_4^+ absent.
11.	Preparation of (0.5) Shake a Salt + water mix.	Sol^n obtained.	label it as the original Solution.

Teacher's Signature _____

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12.	To a part of (CO.5) add 1.2ml of dil. HCl	NO PPt formed.	Gr.I. Absent. (Pb^{2+} absent).
13.	To a part of Soln pass H_2S gas.	NO PPt formed.	Gr.II Absent. (Pb^{2+}, Cu^{2+}, Ag^{3+} absent)
14.	To remaining soln add solid NH_4Cl Boil, cool down, add a few drops NH_4OH.	NO PPt formed	Gr.III absent. (Fe^{2+}, Al^{3+}, absent)
15.	Through a part of this soln, pass H_2S gas.	NO PPt formed.	Gr.IV absent. (Zn^{2+}, Mn^{2+}, Ni^{2+}, Co^{2+}, absent).
16.	To the remaining ammonical solution add ammonium carbonate.	white ppt formed.	Gr.V present. (Ca^{2+}, Ba^{2+}, Sn^{2+} may be present).
*	CONFIRMATORY TEST		
17.	For Nitrate.		
a)	copper chips test. heated a pinch of the salt with conc. H_2SO_4.	Reddish Brown Gas	NO_3^- Confirmed.
b)	Ring Test.	Dark Brown ring is observed.	NO_3^- Confirmed.
18.	For Ba^{2+}		
a)	Potassium Chromate test.	Yellow PPt.	Ba^{2+} confirmed.
b)	Perform flame test with salt.	Grassy green flame	Ba^{2+} confirmed.

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→ Result.

i) Acid Radical NO_3^-

ii) Basic Radical. Ba^{2+}

→ Precaution

i) Handle reagents properly

ii) Never heat a wet test tube

iii) Keep processing test-tube away from body

iv) Don't inhale unknown/poisonous gases.

Teacher's Signature

Expt. No./Name: Experiment - 5. Date / / Page No. 13

→ Aim:- To identify the given inorganic salt [$Pb(CH_3COO)_2$]

	Experiment.	Observation	Inference.
★	PRIMARY TEST		
1.	Colour	white	Shows absence of Cu^{2+}, Ni^{2+}, Fe^{3+}, Mn^{2+}, Co^{2+}.
2.	Smell	Vinegar like smell	Shows presence of CH_3COO^-
3.	Density	heavy	Salt of Pb^{2+} or Ba^{2+} Carbonate may be present.
4.	Diliquescene	No deliquescence	Shows absence of $Zn(NO_3)_2$ & Cl of Zn^{+2}, Mg^{+2} etc.
★	DRY HEATING TEST.		
5.	Gas evolved	Colourless gas with characteristics vinegar like smell.	CH_3COO^- may be present
6.	Sublimate formed	No Sublimation	NH_4^+ & I^- are absent.
7.	Description	No decrepitation	Salts like $Pb(NO_3)_2$, NaCl, KI are absent.
8.	Residue	white salt become black on heating	CH_3COO^- may be present.
★	FLAME TEST.		
9.	Make a paste of salt and conc. HCl & Perform the test.	Dull bluish white flame.	Pb^{2+} may be present.
10.	Dil. H_2SO_4 test. treat a pinch of salt with dil. H_2SO_4 (Heat)	No gas evolved.	CO_3^{2-}, S^{2-}, NO_2^-, SO_3^{2-} may be absent.

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11.	$KMnO_4$ Test: To a pinch of salt add dil H_2SO_4 & heat then add $KMnO_4$.	Pink color of $KMnO_4$ wasn't discharged.	Cl^-, Br^-, I^-, $C_2O_4^{2-}$, Fe^{2+} may be absent.
12.	Heat a pinch of salt with conc. NaOH	No ammonia gas is evolved.	NH_4^+ absent.
13.	Preparation of CO.S) Shake mix. of salt & water.	Solution is obtained	label it as the original solution.
14.	To a part of 0.5 add 2ml of dil. HCl.	white ppt is obtained.	• Group I is present. • Pb^{2+} might be present.

→ Result: i) Acid Radical: CH_3COO^-
ii) Basic Radical: Pb^{2+}

→ Precautions: i) Don't heat wet test tube
ii) Don't inhale gases, they might be poisonous
(iii) Keep test-tube far from face, while dry Heating
(iv) Handle reagents carefully

Teacher's Signature _____

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→ Aim: To identify the given inorganic salt $Pb(NO_3)_2$.

	Experiment	Observation	Inference
★	PRIMARY TEST		
1.	Colour	White	Shows absence of NH_4^+, CH_3COO^-, S^{2-}
2.	Smell	NO specific odour	Shows absence of Cu^{2+}, Ni^{+}, Fe^{2+}, Ca^{2+}
3.	Density	Heavy / Thick	Salt of Pb^{2+} or Ba^{2+} carbonate
4.	Deliquescence	NO deliquescene	Shows absence of $Zn(NO_3)_2$, chlorides of Zn^{2+}, Mg^{2+} etc.
★	DRY HEATING TEST:		
5.	Gas evolved	A reddish brown gas evolved which turned $FeSO_4$ solution, black	NO_3^- may be present.
6.	Sublinate formed	NO sublimation	Shows absence of NH_4^+ & I^-
7.	Descrepitation	The salt descripitates	$Pb(NO_3)_2$, NaCl, KBr may be present.
8.	Swelling	NO swelling	Shows absence of indicated PO_4^{3-}.
9.	Residue	Heat → Brown cold → Yellow	Pb^{2+} might be present.
★	FLAME TEST.		
10.	Prepare a paste of salt with concentrated HCl and perform flame test.	Dull bluish-white flame	Pb^{2+} may be present.

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11.	Dil H_2SO_4 test: Treat a pinch of salt with dil H_2SO_4 & Heat.	NO gas evolved.	Pb^{2+} may be present.
12.	$KMnO_4$ Test: To a pinch of salt add dil H_2SO_4 (hot) and then add a drop of $KMnO_4$.	Pink colour of $KMnO_4$ was not discharged	Cl^-, Br^-, I^-, $C_2O_4^{2-}$, Fe^{2+} may be absent.
13.	conc. H_2SO_4 Test. Salt + conc. H_2SO_4 + (Heat if required)	A reddish brown gas evolved, which turned $FeSO_4$ soln into black.	NO_3^- may be present.
14.	Confirmatory tests for Nitrate.		
a)	'Cu' chip test Heat a small quantity of salt with conc. H_2SO_4 and a few 'Cu' chips.	Reddish brown gas evolved.	NO_3^- is confirmed $2KNO_3 + H_2SO_4 \rightarrow K_2SO_4 + 2HNO_3$
b)	Ring test - 2-3 ml of salt soln + $FeSO_4$ soln Add conc. H_2SO_4 along sides of test tube.	Solution obtained of dark brown colour, 2 liquids at the function.	label it as original soln.
15.	Preparation of (0.5) Shake a pinch of salt with water.	Solution obtained.	label it as original soln.
16.	To a pinch of 0.5, add 1-2 ml of dil HCl.	white ppt is formed.	Group I, Pb^{2+} may be present.

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11. confirmatory test for Pb^{2+} dissolve white ppt with distilled water & divide it in two parts:		
a) KI Test: To one Part add KI solution	Yellow ppt obtained.	Pb^{2+} is confirmed. $PbCl_2 + 2KI \rightarrow PbI_2 + 2KCl$
b) K_2CrO_4 Test: To one part add K_2CrO_4 soln.		Pb^{2+} is confirmed. $PbCl_2 + K_2CrO_4 \rightarrow PbCrO_4 + 2KCl$.

→ RESULT:

- Acid Radical : NO_3^-
- Basic Radical : Pb^{2+}

→ Precaution:-

1) Don't heat wet test tube
2) Don't inhale gases directly, they might be harmful.
3) Keep test tube away from face while dry heating
4) Handle reagents carefully.

Teacher's Signature

Expt. No./Name: Experiment 1 Date / / Page No. 18

Aim:- To identify the given inorganic salt $PbCCl_2$.

Experiment.	Observation.	Inference.
1. Colour [primary test]	white.	Shows absence of Cu^{2+}, Ni^{2+}, Fe^{2+}, Fe^{3+}, Mn^{2+}, Ca^{2+}
2. Smell	No specific smell	Shows absence of NH_4^+; CH_3COO^-
3. Density	Heavy	Salt of Pb^{2+} or Ba^{2+} carbonate may be present.
4. Deliquescene	no deliquescene	Shows absence of $Zn(NO_3)_2$ & chlorides of Zn^{2+}.
* Dry HEATING TEST		
5. Gas evolved.	Colourless gas with pungent smell; white fumes with ammonia → white PPt with $AgNO_3$.	Cl^- may be present.
6. Descreption	No descreption.	$PbCNO_3)_2$, Nacl are absent.
7. Residue	Hot → Brown cold → Yellow	Pb^{2+} might be present.
* FLAME TEST.		
8. Make a paste of salt + conc. HCl	Dull bluish white flame	Pb^{2+} may be present.
9. Dil. H_2SO_4 Test. Treat a pinch of salt with dil H_2SO_4 and heat.	No gas evolved.	CO_3^{2-}, S^{2-}, NO_2^-, SO_3^{2-} may be present.

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10.	Conc. H_2SO_4 + Salt + Heat (if required)	colourless gas, pungent smell white fumes with ammonia and white $AgNO_3$ ppt.	Cl^- may be present.
11.	Heat a pinch of salt with conc. NaOH.	No ammonia gas evolved.	NH_4^+ absent.
12.	Preparation (0.5) of shaking a mix of salt and water.	Solution obtained.	label it as the original sol^n.
13.	To a part of 0.5 add 1.2 ml of dil HCl.	white coloured ppt is obtained.	Group I is present Pb^{2+} may be present.
14.	Heat a pinch of salt with conc. NaOH.	No NH_3 gas is released	Absence of NH_4^+ ions.

→ Result:

- Acid Radical - Cl^-
- Basic Radical - Pb^{2+}.

→ Precautions:-

i) Never heat wet test-tube
ii) No direct inhalation of gases
iii) Keep tube away from face while dry heating
iv) Handle reagents carefully.

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Expt. No./Name: Experiment-8 Date / / Page No. 20

→ Aim:- To identify the given inorganic salt $MgSO_4$.

→ Experiment.	Observation	Inference.
✱ Primary Test:-		
1. colour.	white.	shows absence of Cu^{2+}, Ni^{2+}, Mn^{2+}, CO^{2+}, NH_4^+, CH_3COO^-
2. Smell	odourless.	S^{2-}, SO_3^{2-}. NH_4^+, CH_3COO^- are absent.
3. Gas evolved.	No gas evolved.	S^{2-}, SO_3^{2-}, Cl^-, CH_3COO^-, NH_4^+, NO_3^- are absent.
4. Sublimate formed.	No sublimation	NH_4^+, I^- are absent.
5. Descripitation.	No descripitation	$Pb(NO_3)_2$; NaCl, KBr, are absent.
6. Residue	white residue that glows on heating	Ba^{2+}, Sr^{2+}, Ca^{2+}, Mg^{2+}, Al^{3+} maybe present.
✱ Flame test:-		
7. Make a paste of salt + conc. HCl.	No specific flame colour.	Ca^{2+}, Sr^{2+}, Ba^{2+}, Cu^{2+}, Zn^{2+}, Pb^{2+} may be present.
8. Dil H_2SO_4 Test. Treat a pinch of salt + dil H_2SO_4 and heat.	No gas evolved.	CO_3^{2-}, S^-, NO_2^-, SO^{2-} might be absent.
9. $KMnO_4$ Test. A Pinch of salt added to dil. H_2SO_4 + heat. then add $KMnO_4$.	Pink colour of $KMnO_4$ wasn't discharged.	Cl^-, Br^-, I^-, $C_2O_4^{2-}$, Fe^{2+}, maybe Absent.
10. conc. H_2SO_4 + Salt + Heat (If required)	No gas evolved.	Cl^-, Br^-, I^-, NO_3^-, CH_3COO are absent.

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11.	Heat a pinch of Salt with conc. NaOH.	No ammonia gas evolved.	Cl^-, Br^-, I^-, NO_3^-, CH_3COO^- are absent. & NH_4^+ absent.
12.	Shake a mix. of Salt with water.	Solution obtained.	Label as original solution.
13.	To a part of 0.5 add 1-2 mL of dil HCl.	No ppt formed.	Group I absent (Pb^{2+}) ~~Ag^+, Hg_2^{2+} etc~~).
14.	Through the above formed solution Pass H_2S gas.	No ppt formed.	Group II absent (Pb^{2+}, Cu^{2+}, ~~Ag^+~~ etc).
15.	To remaining soln add a pinch of solid NH_4Cl, Boil the soln and add excess NH_4OH.	No ppt formed.	Group III absent (Fe^{2+}, ~~Al^{3+}~~ absent).
16.	To remaining soln add ammonium carbonate.	No ppt formed.	Group IV absent (Ca^{2+}, Ba^{2+} absent).
17.	Through a part of the above soln, Pass H_2S gas.	No ppt formed.	Group IV absent. (Zn^{2+}, Mn^{2+}, Ni^{2+}, Ca^{2+} absent).

→ Result:-

- Acid Radical - SO_4^{2-}
- Basic Radical - Mg^{2+}

→ Precaution:-

i) Don't heat wet test tube
ii) Don't inhale any gases
(iii) Keep tube away from face while heating dry.
(iv) Handle reagents carefully

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Expt. No./Name: **Experiment: 11** Date / / Page No. 26

★ **Aim:** To identify the given inorganic salt $(NH_4)_3PO_4$.

★ Experiment	Observation	Inference.
→ Primary test.		
1. Colour	White.	Shows absence of Ni^{2+}, Fe^{3+}, Co^{2+}, Mn^{2+}, ~~NH_4^+ is present.~~
2. Smell	Ammonical smell	NH_4^+ is present.
→ Dry Heating test.		
3. Gas evolved.	colourless gas, pungent and sweet smell, white fumes.	NH_4^+ may be Present.
4. Sublimation	White Sublimate.	NH_4^+ may be present.
5. Swelling	Salt swells	PO_4^{3-} may be present
6. Flame test. Make a paste of Salt + conc. HCl & Perform flame test.	NO specific smell.	Ca^{+2}, Sr^+, Ba^+, Cu^{2+}, Zn^{2+}, Pb^{2+} are absent.
7. Dilute H_2SO_4 treated with a pinch of salt and heat.	NO gas is evolved	CO_3^-, SO_3^-, S^{-2}, NO_2^- are absent.
8. To a pinch of Salt add dil. H_2SO_4 (warm) & then add $KMnO_4$	Decolourise $KMnO_4$ from Pink.	Cl^-, Br^-, I^-, $C_2O_4^{2-}$ and Fe^{2+} are absent.

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9.	Conc. H_2SO_4 + Salt + Heat (If required)	NO gas evolved.	Cl^-, Br^-, I^-, NO_3^-, CH_3COO^- absent.
10.	Heat a pinch of Salt with conc. NaOH.	colourless gas with Ammonical smell evolved.	Group 3 is present (NH_4^+ Present)

→ Result.

Acid Radical - PO_4^{3-}

Basic Radical - NH_4^+

→ Precaution

1) Never heat a wet test tube

2) Don't inhale gases which are unknown/poisonous

3) Handle reagent very carefully.

Teacher's Signature

Expt. No./Name: Experiment: 15

Date 1/1

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→ Aim: To identify the given inorganic salt $Sr(NO_3)_2$.

	Experiment	Observation	Inference
1.	colour	white.	shows absence of Ni^{2+}, Fe^{2+}, Fe^{3+}, Co^{2+}.
2.	Smell	No specific smell	NH_4^+, CH_3COO^-, S^{2-} are absent.
3.	Gas evolved.	NO_2 gas. Red brown gas turns $FeSO_4$ into black.	NO_3^- may be present.
4.	Sublimate formed.	No sublimation	$Pb(NO_3)_2$, NaCl, HBr is absent.
5.	Decrepitation.	No decrepitation	NH_4^+, I^- are absent.
6.	Residue	white residue which glows on heating.	Ba^{2+}, Sn^{2+}, Co^{2+}, Mg^{2+}, may be present.
7.	Flame Test: Salt + conc. HCl & perform test.	Crimson Red flame	Sr^{2+} may be present.
8.	Salt + dil. H_2SO_4 and heat sol^n	No gas evolved.	CO_3^{2-}, S^{2-}, NO_3^-, SO_3^{2-} is absent.
9.	Salt + dil. H_2SO_4 & Heat + few drops of $KMnO_4$	Pink colour of $KMnO_4$ was ~~dissolved~~ discharged.	Cl^-, Br^-, I^-, $C_2O_4^{2-}$, Fe^{2+} may be absent.
10.	Heat a pinch of salt with conc. NaOH.	No ammonia gas evolved.	NH_4^+ absent
11.	Shake mix. of salt + water.	sol^n is obtained.	label as original sol^n.
12.	To a part of 0.5 add 1-2ml of dil. HCl.	No PPt obtained.	Group I absent, Pb^{++} absent
13.	Through apart.	No PPt formed.	Group II absent.

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	of this soln pass H_2S gas.		
14.	Through a part of this soln pass H_2S gas.	No ppt formed.	Group V present. (Ca^{2+}, Ba^{2+}, Sr^{2+} may be present).

Result:

- Acidic Radical : NO_3^-
- Basic Radical : Sr^{2+}.

Precaution:

- Don't heat wet test tube
- Handle reagents carefully
- Don't inhale unknown gas.

Teacher's Signature

EXPERIMENT 3.1

Aim

Purification of sample of any one of the following Potash alum, Copper sulphate or Benzoic acid by crystallisation.

Theory

Crystallisation is one of the techniques for the purification of an impure compound particularly when the original crude material obtained after a reaction is in a very impure condition. First step of the process involves choosing a single solvent or a mixture of solvents, which dissolves the crude material readily when hot, but only to a small extent when cold. The crude substance is then dissolved in the minimum amount of boiling solvent to obtain a saturated solution. Insoluble impurities are removed by filtering the hot solution. It is then checked for crystallisation point and then cooled slowly when the solute crystallises out leaving the greater part of impurities in the solution. The crop of crystals is collected by filtration and the process is repeated until the crystals of pure substance are obtained. Sometimes during cooling minute quantity of the substance (solid which is being purified) is added to the solution to facilitate the initial crystallisation. This is called **seeding**. The added tiny crystal acts as a 'nucleus' for the growth of new crystals. Growth of crystals depends upon the conditions in which crystallisation is carried out. For obtaining good crystals, rapid cooling should be avoided because it results into small or disfigured crystals.

Purity of crystals is often judged from the colour of the crystals. For example, pure crystals of alum, copper sulphate and benzoic acid are white, blue and

greenish white respectively. Impurities impart colour to the crystals; therefore, impure crystals have a colour different from pure crystals.

Material Required

- Beaker (250 mL) : One
- Glass funnel : One
- Tripod stand : One
- Porcelain dish : One
- Glass rod : One
- Sand bath : One

- Potash alum, Copper sulphate and Benzoic acid : As per need

Procedure

Copper sulphate 

(i) Take 30-50 mL distilled water in a beaker and prepare a saturated solution of potash alum/copper sulphate in it at room temperature by adding the impure solid sample in small amounts with stirring. Stop adding the solid when it does not dissolve further. To prepare saturated solution of benzoic acid use hot water.

(ii) Filter the saturated solution so prepared and transfer the filtrate into a porcelain dish. Heat it on a sand bath till nearly ¾ of the solvent is evaporated. Dip a glass rod into the solution, take it out and dry it by blowing air from the mouth. If a solid film deposits on the rod, stop heating.

(iii) Cover the porcelain dish with a watch glass and keep the content of the dish undisturbed for cooling.

(iv) When crystals are formed, remove the mother liquor (liquid left after crystallisation) by decantation.

(v) Wash the crystals of potash alum and copper sulphate, thus obtained first with very small quantity of alcohol containing small amount of cold water to remove the adhering mother liquor and then with alcohol to remove moisture. Wash the crystals of benzoic acid with cold water. Benzoic acid is soluble in alcohol. Do not use alcohol to wash its crystals.

(vi) Dry the crystals between the folds of a filter paper.

(vii) Store the dry crystals thus obtained at a safe and dry place.

(viii) Repeat steps (ii-vii) for obtaining maximum amount of pure substance.

Precautions

(a) Do not evaporate the entire solvent while concentrating the solution.

(b) Do not disturb the solution while it is being cooled.

(c) Use the washing liquid in 3-4 very small installments rather than in one installment.

EXPERIMENT 3.2

Aim

Determination of melting point of a solid organic compound.

Theory

The kinetic energy of molecules of a substance increases on heating. When it becomes high enough to overcome the attractive forces operating between the molecules, the lattice structure of the solid breaks, the solid melts and comes into the liquid state. Melting point of a substance is the temperature at which solid state of a substance begins to change into the liquid state, when the pressure is one atmosphere.

Material Required

- Thiele's tube /Kjeldhal's flask/beaker : One
- Thermometer : One
- Capillary tubes : As per need
- Iron stand with clamps : One

- Liquid paraffin /Conc. H_2SO_4 : As per need
- Organic Compound (Naphthalene/ *p*-Dichlorobenzene/ *p*-Toluidine) : As per need

Procedure

(i) Take a capillary tube of approximately 8 cm in length. Seal its one open end by heating it in a Bunsen flame. Rotate the capillary while sealing to ensure complete closure of the opening.

(ii) Crush the desired substance (about 100 mg) into fine particles and fill the substance in the capillary tube up to nearly 1cm length. For filling the capillary, dip its open end in to the powder. Hold the sealed end between the index finger and the thumb and tap the upper end gently with the other hand so that solid particles are tightly packed and capillary is prevented from breaking.

(iii) Moisten the capillary tube with liquid paraffin and stick it to the thermometer. It will stick to the thermometer by cohesive forces. See that the lower ends of the capillary tube and the thermometer bulb are at the same level. The thermometer is fitted into a rubber cork, which has a groove on its side for the escape of air and vapours.

(iv) Take a Thiele's tube (Fig. 3.1 a) and fill it with 50 to 60 mL liquid paraffin so that it crosses the bent portion of the Thiele's tube. Alternatively, Kjeldahl flask's may be used in place of Thiele's tube.

(v) Dip the thermometer along with the capillary tube in liquid paraffin and adjust the rubber cork in such a way that the thermometer bulb and the filled portion of the capillary is completely dipped in the liquid paraffin and the open end of the capillary remains in the air as shown in Fig. 3.1 a. The thermometer and the capillary tube should not touch the sides of the Thiele's tube.

(vi) Now start heating the side arm of the Thiele's tube with a low flame from the side opposite to that of the capillary tube and note the temperature when the solid starts melting.

p-Dichlorobenzene

p-Toluidine

Naphthalene

Hazard Warning

- *Avoid contact with skin and eyes and don't inhale vapours of these chemicals.*

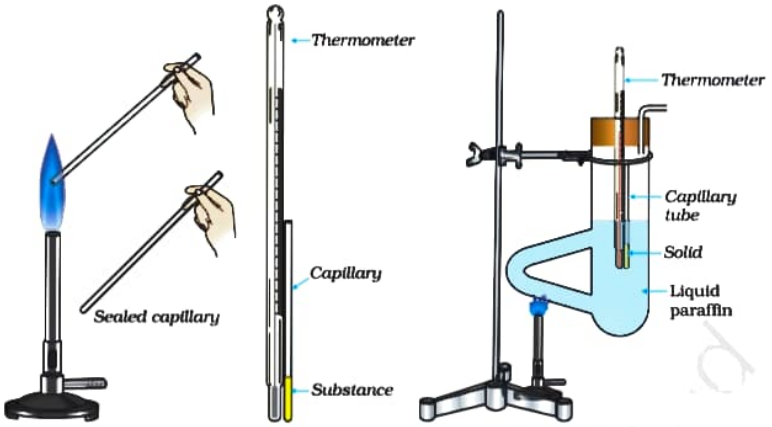

***Fig. 3.1** : (a) Determination of melting point using Thiele's tube*

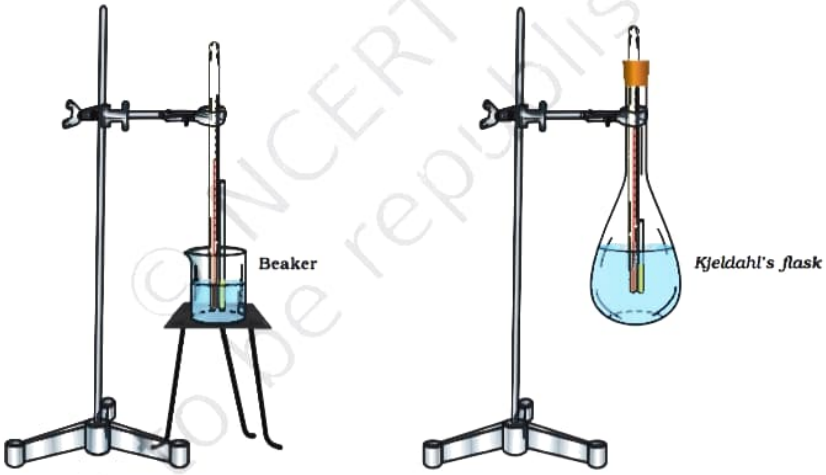

***Fig. 3.1** : (b) Different apparatus used for determining melting point*

This temperature is the melting point of the solid. If you have taken Kjeldahl flask, heat it by revolving the flame around the bottom of the flask to ensure uniform heating. For this, hold the burner in your hand and also keep a sand bath below the flask while heating. It will prevent spilling of acid in case of accident. Repeat the experiment with other solids.

Precautions

(a) Keep the lower end of the capillary tube and the thermometer at the same level.
(b) Capillary tube should not be very thick.
(c) Packing of the powder should be uniform without any big air gaps in between the solid particles.
(d) Thiele's tube should be heated at the side arm by using a low flame.
(e) The cork of the Thiele's tube or Kjeldhal flask holding the thermometer should have a side groove so that vapours can escape through it during the process of heating to prevent bursting of the tube or flask.
(f) Never fill the bulb of Kjeldahl flask's more than half.

***Note :** Paraffin can be safely heated upto 220°C. Therefore for determination of melting point of a substance*