Vishwendu Vidya Prasarak Mandal's
(Regd. No: MAH 1906 / F-1614; Dt.5/3/1987)
Abhinav Vidyalay & Junior College
(Govt. Regd. No. Prim.Edu. 6-970,90-91 Dtd:16-8-90; Index No: Sec:16.17.019 / 020; H.Sec: J16.17.24)
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C11V1_Volumetric Analysis

Strong Acid Vs Strong Base

Aim: Given 0.1 N sodium hydroxide solution. Determine normality & g/liter of HCl.

Given : Flask A – HCl
Flask B – NaOH (0.1 N)

Requirements:

(l) Apparatus: Burette, pipette, conical flask, beaker, funnel, stand.
(ll) Chemicals: NaOH (0.1 N), HCl & Phenolphthalein.

Procedure:

1) Wash all the apparatus with water.
2) Rinse the burette with NaOH solution and fill it with NaOH using funnel.
3) Remove the air bubble if any. Adjust zero mark with lower meniscus.
4) Rinse the apparatus with HCl solution. Pipette out 10ml of HCl solution in conical flask. Add a drop of phenolphthalein indicator.
5) Titrate HCl solution against NaOH by adding NaOH from burette till the solution becomes light pink by constant stirring. Note this reading as a pilot reading.
6) Repeat the titration to get constant reading.

Calculations:

(NaOH) = (HCl)
N1V1 = N2V2

0.1 X C.B.R. = N2 X 10

$$N2 \text{ (N of HCl)} = \frac{0.1 \times C.B.R.}{10}$$

= 'x' N

Normality of HCl = ----------

g/lit of HCl = Normality X Eq. Wt. Of HCl
x X 36.5

Result:

Solution	Normality	Eq. Wt.	g/Lit
HCl		**36.5**	

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C11O1Crystallization_Of_Impure_Compounds

Aim: To Purify impure sample of copper sulphate by the process of crystallization

Requirement:
Evaporating dish, glass rod, tripod stand wire gauze,beaker,funnel,filter paper.etc.
Impure copper sulphate,distilled water

Therory: Crystallization may be defined as the process of obtaining a solid in the crystalline form its solution. Solubility of the given solvent is always high at higher temperature which decreases with lowering of temperature.

Procedure -
1) Heat 100 ml of distilled water in a beaker.
2) Add powdered impure copper sulphate to the hot distilled water, with constant stirring till saturated solution is obtained
3) Fitter the hot solution to remove the insoluble impurities and collect the clear filtrate in an evaporating dish.
4) Heat the evaporating dish slowly using a wire gauze and a glass rod till the point of crystallization is reached.(Take a drop of hot solution on a glass rod and cool it by blowing. if it immediately solidifies, it indicates the point of crystallization.)
5) Place the evaporating dish on an asbestos sheet and allow it to cool slowly.
6) Blue coloured prismatic crystals of pure copper sulphate separate out.
7) The crystals thus obtained.are filtered and washed with water which are then dried either using dessicator or between filter papers
8)The crystals may be further purified by recrysatallization.
Result-Blue prismatic crystals of pure copper sulphate are obtained.

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C11O2_Melting_and_Boiling_Point

Melting point

Aim: Determination of melting of an organic compound.

Apparatus : Thieles tube , stand, thermometer, capillary tube, bunsen burner,, thread.

Chemicals: Liquid paraffin

Procedure:
Take a capillary tube , seal it at one end by rotating it into the extreme edge of bunsen flame . Fill the finely powdered dry organic compoud in the capillary tube by pressing its open end in the powder push the compound down by tapping the capillary tube. Repeat the process until the height of compound in the capillary tube isabout 1 cm. Tie the capillary tube to the termometer by using a thread in such a way that the end of the capillary tube containing the powder is in contact with the mercury bulb.Suspend the thermometer in the thieles tube containing paraffin liquid in such away that mercury bulb is at the mouth of
The side arm and open end of the capillary tube is above the level of paraffin oil.Heat the side arm with a burner using a low oxidising flame. Note down the temperature at which the whole solid melts and turns into liquid.This temperature is the melting point of the organic substance.

RES ULT:
The melting point of the given organic compoud = t+273 K

Boiling point

Aim: Determination of boiling of an organic compound.
Apparatus : Thieles tube , stand, thermometer, capillary tube , fusion tube, bunsen burner,, thread.

Chemicals: Liquid paraffin
Procedure: Take small quantity of the given organic sample in a dry fusion tube . Insert capillary tube sealed at one end in the fusion tube with the open end inside the liquid. Tie the fusion tube to a thermometer by using a thread in such a way that the liquid touches the bulb. Suspend the thermometer in the thieles tube containing paraffin oil in such away that the bulb is at the mouth of the side arm and the open end of the fusion tube is above the level of the paraffin oil .The side arm is then heated with an oxidising flame .At first a few bubbles escape from the bottom of the capillary tube from time to time. Note down the temperature at which a continuous fast stream of bubbles come out of the lower end of the capillary tube.This temperature is the boiling point of the substance.

RESULT: The boiling point of the given liquid =----------K

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C11P1_Preparation_of_Lyophilic_and_Lyophobic_Solutions

Lyophilic solution

Aim: To prepare a lyophilic sol of starch

Requirements: 1) Apparatus: 250 mL beaker, glass rod.
2) Chemicals: Starch Powder, distilled water.

Procedure:
1) Take 100 mL distilled water in a 250 mL beaker and boil the contents of the beaker using wire gauze
2) Take 1 gm of starch powder in another beaker and add some water to make a thin paste
3) Now, add this starch paste into the boiling water with constant stirring using a glass rod.4) After cooling a turbid solution of starch sol is obtained.
Note: To avoid coagulation of sol use
A) Clean Apparatus
B) Only distilled water

Lyophobi c solution

Aim To prepare lyophobic sol of ferric hydroxide.

Requirement: Apparatus: Two 250 ml beaker, glass rod, dropper.
Chemicals: Ferric Chloride solid distilled water.

Theory: When ferric chloride solid is boiled with distilled water, due to hydrolysis, reddish brown sol of ferric hydroxide is obtained.

$$FeCl_3 + 3H_2O \xrightarrow{\quad\quad} Fe(OH)_3 + 3HCl$$
(solid) (sol)

Procedure:
1) Dissolve 2 gm of solid ferric chloride in 100 ml of distilled water. Taken in a 250 ml beaker, to make 2 % solution of ferric chloride.
2) Boil 100 ml of distilled water in another 250 ml beaker.
3) Now add 2% ferric chloride solution, dropwise to the boiling distilled water, till a white gelatinous ferric hydroxide sol is obtained
4) Allow the sol to cool at room temperature without stirring or shaking

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C11 I1_Qualitative -Analysis_Tests

Aim: To detect anion (acidic radical) and cation (basic radical) in given inorganic sample.

Requirements: Test tubes, Test tube stand, Test tube holder, Watch glass, Platinum wire, Glass rod, Plastic beaker (100 ml), Spirit lamp, funnel, conical flask, filter paper

Qualitative analysis of salt involves the following steps:

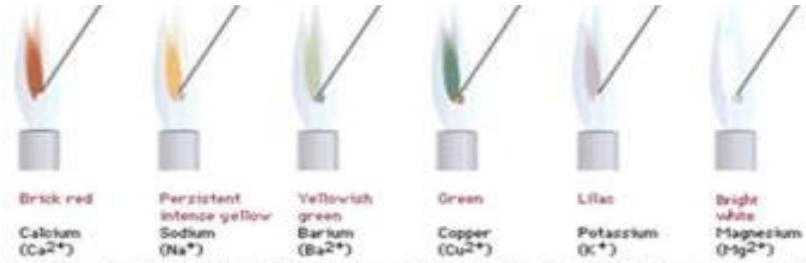
1. **Preliminary tests.**
2. **Dry test for cations.**
3. **Dry test for anions .**
4. **Preparation of original solution (O.S).**
5. **Wet test for cations.**
6. **Wet test for anions.**
7. **Molecular formula of the compound.**
8. **Reactions.**

1. Preliminary Test:

	Tests	**Observations**	**Inference**
1	***Color***	I) Blue II) Green III) Pale yellow IV) White or Colourless v) Red or violet	I) Cu^{2+} may be present. II) Cu^{2+}, Cr^{3+}, Ni^{2+} may be present. III) Cd^{2+} may present. IV) Zn, Ca, Ba, Mg, K salts may be present. v) Co salts may be present
2(a)	***Nature: pinch of salt*** ***+1/2 T.T.water*** ***(shake well)***	I) Crystalline (Water soluble) II) Amorphous (Insoluble in water)	I) Cl^-, Br^-, I^-, NO_3^-, SO_4^{2-}, or compounds of K^+, NH_4^+,may be present II) Insoluble salts of CO_3^{2-}, S^{2-}, SO_3^{2-}, may be present
2(b)	***Nature***	II) Hygroscopic (Absorbs moisture from air and becomes wet.)	II) Chlorides or nitrates of Cu^{2+}, Ni^{2+}, Ca^{2+} may be present

2. Dry test for Cations:

	Tests	**Observations**	**Inference**
1	**Effect of heat:** Take a small quantity of Salt in a dry test tube and heat it strongly in oxidizing flame (blue flame) and observe the changes. (if residue is obtained in the above test, let it cool and observe the colour of residue again)	I) Salt fuses. (melts) II) Decrepitation (Crackling noise) III) Water vapour condenses on the cooler parts of the test tube IV) White sublimation V)Gas evolved : a) Colourless gas with smell of NH_3 b) Colourless gas with no smell of NH_3	I) Salts of K^+, $NH4^+$,Ca^{++} and hydrated salts present. II) Chlorides and nitrates of K^+, Pb^{++}, Ba^{+2}may be present. III) Salts containing water of crystallization may be present. IV) NH_4+ may be present a) NH_{4+} present b) Co_3^{2-}, Cl^- may be present

		c) Brown gas d) Violet gas VI) Change of colour of residue : Hot / Cold a) Yellow / White b) Reddish Brown / Yellow c) Brown / Yellow VII) Blue salt turning white VIII) Residue left in the test tube a) Coloured residue b) White residue	c) NO_3^-, Br^- may be present d) Ipresent a) Zn^{++} may be present b) Pb^{++} may be present c) Cd^{++}may be present VII) $CuSO_4 5H_2O$ present VIII) a) Cu^{++}, Cd^{++}, Cr^{+}, Ni^{++}salts present. b) Al^{3+}, Zn^{++},Ba^{++}, Ca^{++},K^+ NH_4^+ salts present.
2	Charcoal cavity test Take salt and solid Na_2CO_3 in the ratio 1:2 on a watch glass. Add few drops of water and prepare a paste. Place this in a charcoal cavity and heat it with a blow pipe in a reducing flame (red flame)	I) Redscales II) Metallic bead with yellow incrustation III) Decripitation IV) White residue and fumes with smell of NH_3 V)White residue VI) Coloured or black residue	I) Cu^+ may be present II) Pb^{++} may be present III) Pb^{++} may be present IV) NH_4^+ may be present V) Mn^{2+}, Zn^{2+}, Al^{3+}, Mg^{2+}, Ba^{2+}, Sr^{2+}, Ca^{2+} may be present VI) Cu^{2+}, Ni^{2+}, Fe^{3+}, Co^{2+} may be present
3	***Flame test :*** Clean a platinum wire with conc. HCl. Moisten a small quantity of the substance with conc. HCl and dip the wire in it and heat it in oxidising flame. Observe the color of the flame.	I) Pinkish blue or violet II) Bluish green III) Apple green IV) Brick red V) Bluish white VI) Colour not distinct	I) K^+ may be present II) Cu^{++} may be present III) Ba^{++}may be present IV) Ca^{++} may be present V) Pb^{++} may be present VI) K^+ ,Cu^{++},Ba^{++},Ca^{++} may be absent.
	 The flame test is a well established laboratory procedure to identify the presence of a particular element in a chemical sample. The elements shown give their characteristic colours to the flame.		
4	***Borax-Bead test :*** (For colored salts only) Make a small loop at the end of the clean platinum wire. Heat it strongly and deep it in borax powder .Heat it again till a glassy bead is obtained. Now touch the hot bead with a small crystal of salt and heat it again in oxidizing flame. Observe the colour of the bead.	I) Greenish blue II) Dark green III) Reddish brown	I) Cu^{++} may be present II) Cr^{+3} may be present III) Ni^{++}may be present
5	***Test for NH_4^+:*** Salt + NaOH heat to boiling	Smell of NH_3 gas	NH_4^+ may be present

3. Dry test for Anions:

	Tests	Observations	Inference
1	***Action of dil. HCl*** (Salt + dilute HCl). If effervescence obtained, then pass it through lime water (use downward delivery tube)	Colourless gas with brisk effervescence in cold turning lime water milky	Co_3^{-2} may be present
2	***Action of Conc.H_2SO_4*** *(Salt + conc. H_2SO_4 + heat)*	I) Colourless gas giving dense white fumes with NH_4OH held on a glass rod. II) Brown gas III) Violet gas	I) Cl^- may be present II) NO_3^- , Br^- may be Present III) I^- may be present
3	***Action of MnO_2 and Conc. H_2SO_4*** ($Salt + MnO_2 + H_2SO_4$)	I)Colorless or Greenish yellow gas that bleaches moist litmus paper II)Reddish brown gas turning starch paper yellowish brown III) Light Brown fumes, no action on starch paper. IV)Violet gas turning starch paper bluish black	I) Cl^- may be present II) Br^- may be present III) NO_3^- may be present IV) I^- may be present

Conclusion: The probable radicals are

1. Cation _____

2. Anion _____

4. Preparation of Original Solution / Water Extract (O.S. / W.E.)

For only water soluble compounds: 2g of salt + 20ml of water

5. Wet test for Cations:

A) Analysis of group VI (NH_4^+, K^+)

	Tests	Observations	Inference
1	W.E + NaOH, Warm	Smell of NH_3, vapours turning moist litmus paper blue or moist turmeric paper reddish brown.	NH_4^+ present
	C.T. For NH_4^+ W.E. + Nessler's reagent	Brown precipitate	NH_4^+ confirmed
2	(If NH_4^+ is absent, perform this test) W.E + CH_3COOH+ Sodium cobaltinitrate.	Yellow ppt.	K^+ present
	C.T. For K^+ W.E. + picric acid, shake vigorously	Yellow crystalline ppt	K^+ confirmed

B)Group Detection

<table>
<tr><td colspan="5">Step (1) : O.S. + dil. HCl</td></tr>
<tr><td rowspan="4">ppt Gr-I present And proceed for the analysis of Gr-I.
If no ppt. follow step (2)</td><td colspan="4">Step (2) : O.S. + dil. HCl + H_2S water</td></tr>
<tr><td rowspan="3">Black or yellow ppt.Gr-II present And proceed for the analysis of Gr-II.
If no ppt. follow step (3)</td><td colspan="3">Step (3) : O.S. + NH_4Cl + NH_4OH till alkaline</td></tr>
<tr><td rowspan="2">White gelatinous ppt. Gr-IIIA present And proceed for the analysis of Gr-IIIA.
If no ppt. follow step (4)</td><td colspan="2">Step (4) : O.S. + NH_4Cl + NH_4OH till alkaline+ H_2S water</td></tr>
<tr><td>Black or white ppt. Gr-IIIB present And proceed for the analysis of Gr-IIIB.
If no ppt. follow step (5)</td><td>Step (5) : O.S. + NH_4OH + $(NH_4)_2CO_3$

White ppt. Gr-IV present And proceed for the analysis of Gr-IV.
If no ppt. reject the solution</td></tr>
</table>

C)Analysis of the particular group.

1.Analysis of Gr - I (Pb^{++}) :

O.S. + dil. HCl	White ppt	Pb^{++} present
C.T. for Pb^{++}		
i) O.S. + KI solution	i) Yellow ppt	i) Pb^{++} confirmed
ii) OS + K_2CrO_4	ii) Yellow ppt	ii) Pb^{++} confirmed

2. Analysis of Gr - II (Cu^{++} , Cd^{++}) :

O.S. + NaOH in excess	**a)** Blue ppt.	a) Cu^{++} present
C.T. for Cu^{++}	b) White ppt.	b) Cd^{++} present
OS + CH_3COOH+ $K_4Fe(CN)_6$	Chocolate brown ppt	Cu^{++} confirmed
C.T. for Cd^{++}		
i) OS + $K_4Fe(CN)_6$	i) White ppt.	i) Cd^{++} confirmed
ii) O.S. + dil. HCl + H_2S water	ii) Yellow ppt.	ii) Cd^{++} confirmed

3. Analysis of Gr - IIIA (Al^{+3} , Cr^{+++}) :

O.S. + NH_4Cl + NH_4OH till alkaline	a) White gelatinous ppt.	a) Al^{+3} present
C.T. for Al^{+3}	b) Green ppt.	b) Cr^{+++} present
OS + Na_2HPO_4 in excess	White ppt.	Al^{+3} confirmed.
C.T. for Cr^{+++}	i) Green ppt soluble in excess of NaOH	i) Cr^{+++} confirmed.
i) OS + NaOH		
ii)OS +PbO_2 + NaOH boil it and allow it to stand, a bright yellow colouration, acidify it with acetic acid.	ii) Yellow ppt.	ii) Cr^{+++} confirmed

4.Analysis of Gr - IIIB (Ni^{++},Zn^{++}) :

O.S. + NH_4OH till alkaline + H_2S water	**a)** Black ppt.	a) Ni^{++} present
C.T. for Ni^{++}	b) White ppt.	b) Zn^{++} present
O.S. + NH_4OH till alkaline + dimethyl glyoxime	Scarlet red ppt.	Ni^{++} confirmed.
C.T. for Zn^{++}		
OS+ dil.HCl + $K_4Fe(CN)_6$	Yellow ppt.	Zn^{++} confirmed.

5.Analysis of Gr - IV (Ba^{++} , Ca^{++}) :

O.S. + K_2CrO_4	Yellow ppt.	Ba^{++} present
C.T. for Ba^{++}		
OS + dil. H_2SO_4	White ppt.	Ba^{++} confirmed
OS + NH_4OH till alkaline + ammonium oxalate $(NH_4)_2C_2O_3$	White ppt. of CaC_2O_4	Ca^{++} present
C.T. for Ca^{++}		
Flame test	Brick red flame	Ca^{++} confirmed

5. Wet test for Anions:

	Test	**Observation**	**Inference**
1	**Test for Halides** W.E.+ 3-4 ml of dil. HNO_3 till no effervescence, boil, cool and add $AgNO_3$	a)Curdy white ppt. completely soluble in NH_4OH. b)Pale yellow ppt. partly soluble or insoluble in NH_4OH	a) Cl^- present. b) Br^-, I^- present.
	Distinction between Cl^-, Br^-, I^- W.E. + Dil. H_2SO_4, till acidic+ Chloroform+ Cl_2 water in excess, shake vigorously and observe.	a)Chloroform layer colourless. b)Chloroform layer yellowish brown c)Chloroform layer violet.	a) Cl^- present. b) Br^- present. c) I^- present.
	C.T. For Cl^- (Chromyl Chloride test) Salt+solid $K_2Cr_2O_7$(1:3)+Conc.H_2SO_4, heat, collect the reddish brown vapours of chromyl chloride in another test tube, add NaOH, Shake add CH_3COOH+ $(CH_3COOH)_2Pb$	Yellow precipitate	Cl^- confirmed
	C.T. for Br^-, I^- W.E. + MnO_2 + Conc. H_2SO_4 heat	a) Reddish brown gas b) Violet gas	a) Br^- confirmed b) I^- confirmed
2	***Test for SO_4^{-2}*** W.E + Dil.HNO_3 till acidic, add $Ba(NO_3)_2$ ***C.T. for $SO4^{-2}$*** W.E. +CH_3COOH+ $(CH_3COOH)_2Pb$	White Ppt. insoluble in Dil.HNO_3 White Ppt. soluble in Ammonium acetate	SO_4^{-2} present SO_4^{-2} is Confirmed

3	***Test for CO_3^{-2}*** W.E. +Dil. HCl ***C.T. for CO_3^{-2}*** W.E. + Phenolphthalein	Colorless gas with brisk effervescence turning lime water milky Pink color decolorizes slowly.	CO_3^{-2} present CO_3^{-2} confirmed
4	***C.T. for NO_3^{-2}:*** W.E. + Conc. H_2SO_4(add carefully), cool well under tap water, add freshly prepared $FeSO_4$ solution by the side of the test tube slowly (do not shake)	Brown ring is formed at the junction of two solutions	NO_3^{-2} present and confirmed

Inference : The given radicals are: **1. Cation _____**
2. Anion _____
The given salt is _____

Reactions:

1) Action of conc. H_2SO_4

$2KCl + H2SO4 \rightarrow K2SO4 + 2HCl$

$NH_3 + HCl \rightarrow NH_4Cl$

2) Action of conc. H2SO4 and MnO2

2KCl + MnO2 + 3H2SO4 ? 2KHSO4 + MnSO4 + H2O + Cl2?

3) Test for group VI radical i.e. potassium (K+) [C.T.]

(i) *O.S.+ Sodiumcobaltinitrate → Yellow ppt.*

$3KCl + Na_3[Co(NO_2)_6] \rightarrow K_3[Co(NO_2)_6] + 3NaCl$

(ii) *O.S.+ Picric Acid → Yellow ppt.*

$KCl + C_6H_2(NO_2)_3OH \rightarrow C_6H_2(NO_2)_3OK + HCl$

(iii) *O.S.+ Perchloric acid →Yellow ppt.*

$KCl + HClO_4 \rightarrow KClO\downarrow + HCl$

(iv) *O.S + SodiumHydrogentartarate → White ppt. of potassium hydrogen tartarate.*

$KCl + NaHC_4H_4O_6 \rightarrow NaCl + KHC_4H_4O_6$

4) Test for halides :

$KCl + AgNO_3 \rightarrow AgCl\downarrow + KNO_3$

5) C.T. for Cl- :

O.S.+ lead acetate→ white ppt. of $PbCl_2$

$2KCl + Pb(CH_3COO)_2 \rightarrow 2CH_3COOK + PbCl_2$

Now as an example, let us consider the reactions for positive tests for various groups of basic radicals(B.R).

1) B.R. of Group I :

$Pb(NO_3)_2 + 2HCl \rightarrow PbCl_2\downarrow + 2HNO_3$

2) B.R. of Group II :

(i) $Pb(NO_3)_2 + H_2S \rightarrow PbS\downarrow + 2HNO_3$

(ii) $CuSO_4 + H_2S \rightarrow CuS\downarrow + H_2SO_4$

(iii) $CdCl_2 + H_2S \rightarrow CdS\downarrow + 2HCl$

3) B.R. of Group IIIA :

(i) $AlCl_3 + 3NH_4OH \rightarrow Al(OH)_3\downarrow + 3NH_4Cl$

(ii) $CrCl_3 + 3NH_4OH \rightarrow Cr(OH)_3\downarrow + 3NH_4Cl$

4) B.R. of Group IIIB :

(i) $ZnCl_2 + H_2S \rightarrow ZnS\downarrow + 2HCl$

(ii) $NiCl_2 + H_2S \rightarrow NiS\downarrow + 2HCl$

(iii) $CoCl_2 + H_2S \rightarrow CoS\downarrow + 2HCl$

5) B.R. of Group IV :

(i) $BaCl_2 + (NH_4)_2CO_3 \rightarrow BaCO_3\downarrow + 2NH_4Cl$

(ii) $CaCl_2 + (NH_4)_2CO_3 \rightarrow CaCO_3\downarrow + 2NH_4Cl$

6) B.R. of Group V :

$MgCl_2 + Na_2HPO_4$ (*in presence of* NH_4Cl & NH_4OH) $\rightarrow MgNH_4PO_4\downarrow + 2NaCl$

7) Nesssler's reagent test for NH_4^+ :

(i) $HgCl_2 + 2KI \rightarrow HgI_2 + 2HCl$ & $HgI_2 + 2KI \rightarrow K_2HgI_4$

(K_2HgI_4+NaOH is Nessler's reagent.)

(ii) $2K_2HgI_4 + 3NaOH + NH_4OH \rightarrow HgNH_2OHgI\downarrow + 4KI + 3NaI + 3H_2O$

(Ammonium salt is changed to NH_4OH by NaOH, then the above reaction takes place)