

NAME:- MUKESH MEEL

CLASS:- M.Sc. (Previous)

SUBJECT:- IN-ORGANIC-CHEMISTRY

ROLL NO:- 1285828

COLLEGE:- R.K. VIGYAN (PG) COLLEGE

KALVAR, JAIPUR

(1) To prepare cis potassium  
dioxalate di aqua chromate  
 $K[Cr(C_2O_4)_2(H_2O)_2] \cdot 2H_2O$

Sturmer

(2) To prepare tetraammine cupric  
sulphate -  
 $[Cu(NH_3)_4SO_4] \cdot 2H_2O$

(3) To prepare potassium trioxalate  
ferrate  
 $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$

(4) To prepare hexa-ammine nickel  
(II) chloride  $[Ni(NH_3)_6]Cl_2$

Sturmer

(5) To prepare prussian blue  
 $Fe_4[Fe(CN)_6]$

(6) To prepare Nickel dimethyl  
glyoximate complex.  
 $[Ni(DMG)_2]$

(7) estimation of copper and Nickel  
volumetric and gravimetric  
method analysis

Sturmer

S. No.	Name of the Experiment	Page No.	Date of Experiment	Date of Submission	Remarks
(8)	Identify four acidic and basic radicals including rarer in the given inorganic mixture				
(9)	Identify four acidic and basic radical including rarer in the given in-organic mixture by dry & wet test.				
(10)	Identify four acidic and basic radical including rare in the given in-organic mixture				<u>Summed</u>
(11)	Identify four acidic & basic radical inorganic mix.				
(12)	Identify four acidic & basic radical including rarer in the given inorganic mixture.				
(13)	Identify four acidic & basic radical including rarer in the given in-organic mixture.				<u>Summed</u>



**Object:-**

To prepare cis-potassium dioxalato diqua chromate  
 $K [Cr(C_2O_4)(H_2O)_2] \cdot 2H_2O$ .

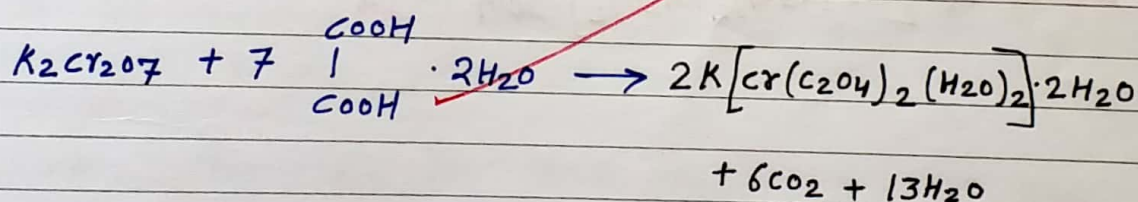
**Apparatus:-** Beaker, glass rod, funnel

**Requirements:-**

oxalic acid, crystals - 6gm  
 potassium dichromate crystals - 2gm  
 Ethanol - 40ml

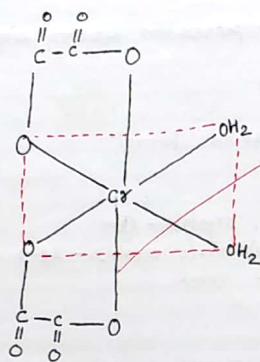
**Principle:-**

It may be prepared by the reaction of potassium dichromate with oxalic acid.

**Procedure:-**

- (1) Take a dry mortar and 6gm of oxalic acid crystals and 2gm of potassium dichromate crystals in it.
- (2) Gently grind the mixture with a pestle till the powders are quite intimately mixed.

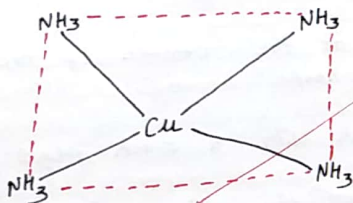




cis-isomer of  $[Cr(C_2O_4)_2(H_2O)_2]^-$  ion

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- ③ Take a china dish (10cm diameter) and moisten it with drain may the excess of water.
- ④ Introduce the mixture at the centre of the china dish in a compact heap.
- ⑤ cover the china dish with a clock glass.
- ⑥ Heat the contents in the china dish gently on a low flame.
- ⑦ After a few minutes a vigorous reactions will set in accompanied with fracturing due to evolution of carbon dioxide and water vapours.
- ⑧ soon the reactions will spread through out the mixture which will become deeply coloured liquid.
- ⑨ without waiting for the thick liquid to cool pour about 20ml some ethanol over this liquid.
- ⑩ using a metallic spatula triturate the contents until a solid is formed.
- ⑪ In case complete solidification is not achieved decant the liquid and add to it another lot of 20ml of ethanol.



12. Warm and resume titration until the product is in the form of granular crystals.
13. The crystals will take place black in diffused day light and deep purple in artificial light.

Expected yield:-

The yield of cis-potassium diqua dioxalato chromate (III) 4.1503 gm.

**Precaution:-**

- (i) pestle and mortar must be dry otherwise a reaction may set in.
- (ii) Heating should be very gentle otherwise the reaction may go out of control and cause explosion.
- (iii) since ethanol has to be added to the contents marked flame should be kept a way as far as possible.

**Result:-** Appearance Rose colour crystal  
yield - 3.19 gm

Suena  
23/4/18



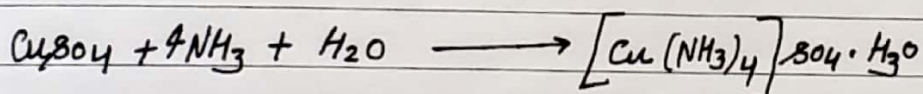
**Object:-**

To prepare tetrammonia cupric sulphate  
 $[\text{Cu}(\text{NH}_3)_4\text{SO}_4] \cdot \text{H}_2\text{O}$

Operators :- Beaker, Glass rod, funnel

**Principle:-**

It is prepared by adding ammonia to a solution of copper sulphate when cupric hydroxide is precipitated which dissolves in excess of ammonia to give a deep blue solution. The hydrated tetrammine cupric sulphate is then precipitated from the solution by adding ethyl alcohol since it is insoluble in alcohol.



tetrammine cupric sulphate (blue)

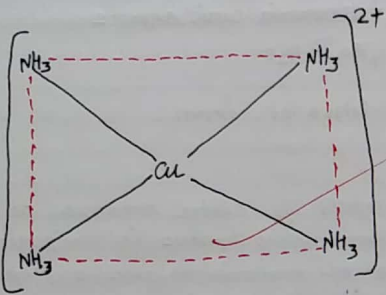
**Reagents:-**

Copper sulphate = 5 gm  
liquor ammonia = 10 ml  
ethyl alcohol = 15 ml  
dilute  $\text{H}_2\text{SO}_4$  = 2.3 ml

**Procedure:-**

The various steps for the preparation of tetrammine cupric sulphate are given below.

Teacher's Signature : \_\_\_\_\_



- Take 5gm of crystalline copper sulphate  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in a 250ml beaker dissolve it in minimum quantity of water adding a few drops of sulphuric acid if necessary to clear up the solution.
- Now add liquor ammonia solution to the beaker with constant stirring from a dropping funnel until the blue ppt of cupric hydroxide first formed completely dissolve to yield a clear deep blue solution and there should be a smell of ammonia in the beaker.
- Now add alcohol (30ml) dropwise from the dropping funnel to the beaker with constant stirring until the blue colour is nearly discharged. Heat the beaker to  $60^\circ\text{C}$  with a clock glass over the water bath for about 15-16 minutes. After this stop heating and remove the beaker from the water bath and allow it to stand long middle shaped blue crystals of tetraammine cupric sulphate separate out filter and wash crystals with a few drops of alcohol.
- Dry the crystals on a porous plate or in a desiccator weight the dry crystals.

**Result:-**

The yield of 2.9164 gm which is of blue colour.

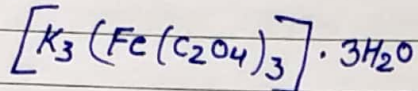
**Precaution:-**

The crystal being extremely photosensitive be preserved in dark.



**Object :-**

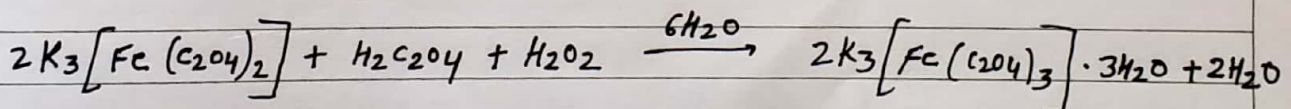
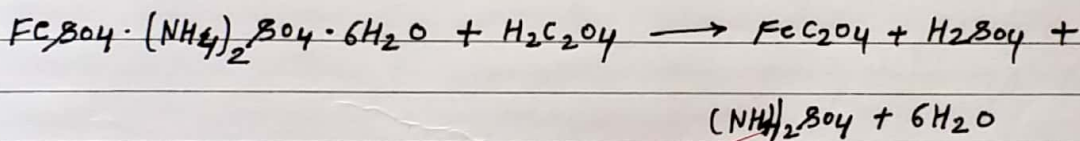
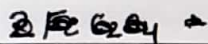
To prepare potassium trioxalato ferrate



Operators :- Beaker, glass rod, funnel

**Principle :-**

Ferrous oxalate is obtained by mixing ferrous ammonia sulphate and oxalic acid to which potassium oxalate and hydrogen peroxide are added to get the complete potassium trioxalato ferrate (III).

**Reagents :-**

Ferrous ammonium sulphate = 5.5 gm

oxalic acid = 10 gm

potassium oxalate = 10 gm

Hydrogen peroxide (20 volume) = 25 ml

H<sub>2</sub>SO<sub>4</sub> (4N)

ethyl alcohol = 50%

Acetone

Teacher's Signature : \_\_\_\_\_

**procedure :-**

- (1) dissolve 5.5 gm ferrous ammonium sulphate in 20ml of hot distilled water which is already having 4ml of  $4\text{NH}_2\text{SO}_4$  in a 250ml beaker.
- (2) stir the solution using a mechanical or a magnetic stirrer and add very slowly until of 10% oxalic acid solution to it.
- (3) Remove the beaker from the stirrer and heat it to boiling stirring the solution all the time, with a glass rod.
- (4) A precipitate of ferrous oxalate is formed which is allowed to settle down decant the supernatant solution completely. without disturbing the precipitate as far as possible.
- (5) Now add 80ml of hot distilled water to the precipitate and stir it well allow the precipitate to settle allow once again and decant the supernatant solution very carefully.
- (6) Dissolve 10gm of potassium oxalate in 30ml of hot distilled water and slowly to ferrous oxalate precipitate in the beaker.
- (7) Keep the beaker in a steam bath and add slowly and continuously 25ml of 20 volume  $\text{H}_2\text{O}_2$  stirring the solution all the time maintaining Temp. of the solution to above  $40^\circ\text{C}$  by keeping the beaker in the water bath.

Teacher's Signature : \_\_\_\_\_



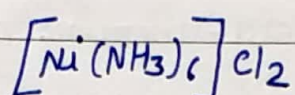
- (8.) The solution may have traces of ferric hydroxide precipitated at this stage therefore keep the beaker on wire gauze and heat the solution to boiling.
- (9.) Store it constantly and to the continuously stirred and boiling solution add very rapidly 10ml of 10% oxalic acid solution.
- (10.) Then add drop by drop 3ml more of 10% oxalic acid solution to it. Filter the hot solution through a previously weighed sintered bed ~~from funnel or sintered~~  $\text{NO}_2$ . At this stage if any crystals are formed they are dissolved in 30ml of warm ethyl alcohol.
- (11.) Collect the filtrate and keep it aside for crystallization in dark.
- (12.) Bright green coloured crystals are obtained then filter through the weighed sintered bed crucible wash them with 50% ethyl alcohol and then with a small quantity of acetone.
- (13.) Continue the solution until the crystals become dried dry further and then weigh the crystals is potassium trioxalato ferrate
- $$\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$$

**Result:-** yield of potassium trioxalato ferrate 6.2934 gm obtained with colour is green.

**Precaution:-** crystals should be preserved in dark.

**Object :-**

To prepare Hexa-ammine nickel (II) chloride.

**Glass ware requirement :-**

Beaker, Glass rod, Funnel

**Reagents :-**

Nickel chloride = 12gm

Ammonia = 24gm

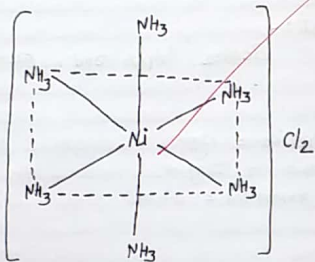
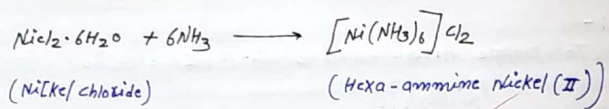
Liquor ammonia = 50ml

**Procedure :-**

- (1.) Dissolve 12gm of Nickel chloride in 20ml of warm water. Filter the solution to remove any insoluble impurities.
- (2.) Add about 24ml of concentrated aqueous ammonia slowly to the rapidly stirred solution until the green ppt of nickel hydroxide has dissolved.
- (3.) Allow the mixture to stand at room temp. for 30 minutes and then remove the crystals of hexa-ammine nickel (II) chloride by filtration on a Buchner funnel.



Chemical reactions:-



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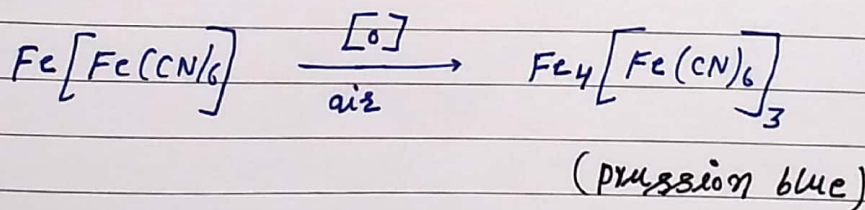
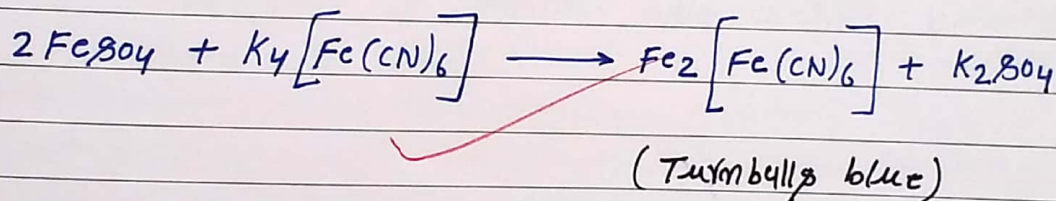
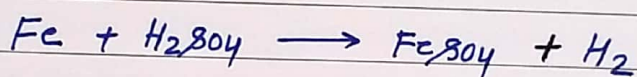
(4) Wash the ppt with ammonia (density - 0.88) about 50ml then wash it with acetone and allow the product to dry at room temp. in air.

Result:-

Yield of Hexa-ammine nickel (II) chloride  
 $[\text{Ni}(\text{NH}_3)_6]\text{Cl}_2$  4.6132 gm obtained.

**Object:-**To prepare prussian blue  $Fe_4[Fe(CN)_6]_3$ **Glass Ware required:-**Beaker, glass rod funnel  
wire gauze.**Reagents:-**

Iron filings - 0.5 gm  
 potassium ferrocyanide - 45 gm  
 Ethyl alcohol - 15 gm  
 $H_2SO_4$  (dil) - 50 ml

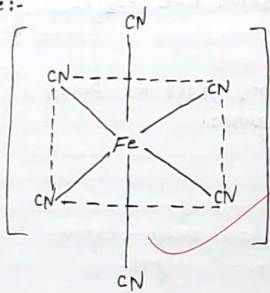
**Reactions:-****Procedure:-**

(1) Take 0.5 gm of iron filings in water to this add 10 ml of dil  $H_2SO_4$  heat in on a wire gauze.

Teacher's Signature .



Structure:-



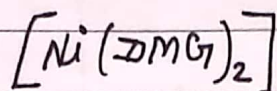
- (2.) As soon as the reaction becomes vigorous remove the burner and cool the burner and cool the solution filter it.
- (3.) To the residue add 10ml of add  $H_2SO_4$  and repeat to procedure collect the filtrate in a 250ml beaker.
- (4.) To it add a saturated solution of 4.5gm of potassium ferrocyanide heat the solution until it acquires green colour.
- (5.) Keep the green coloured product in air for nearly two hours when it change to blue or you pass the air through the green coloured product.

**Result:-**

The yield of prussian blue 5.9142 obtained which coloured is blue.

**object:-**

To prepare Nickel dimethyl glyoximate complex.

**principal:-**

Nickel dimethyl glyoximate is precipitate by treating salt solution of Nickel sulphate with alcoholic solution of DMG in presence of ammonia.

**Chemical Requirement:-**

Nickel sulphate = 5gm

DMG = 2% solution

Ammonia = 1% "

Methyl red Indicator

**Glass ware Requirement:-**

Beaker, Burner Funnel

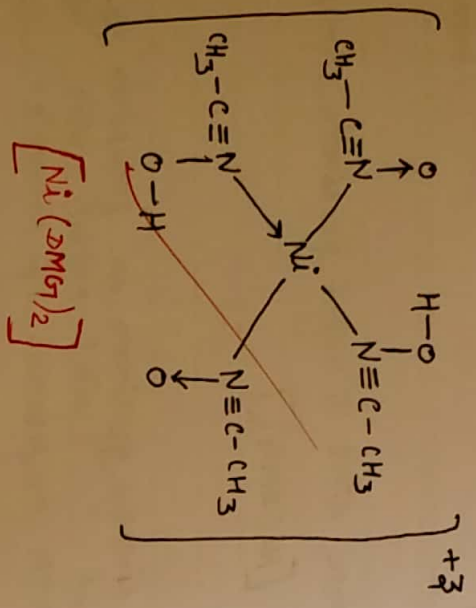
Glass rod.

**procedure:-**

- (1) We will dissolve 5gm of Nickel sulphate in minimum concentrate of water.
- (2) Then we add 1ml of acetic acid in it after if we add 1ml of methyl red indicator.



Structure:-



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- 3.) Now we add ammonia solution (1:1) till the colour change from red to yellow heat the solution to 70-80°C.
- 4.) We add 15ml 2Mg solution now add NH<sub>3</sub> solution till the small of ammonia persist. then we will observe scarlet of  $[Ni(DMG)_2]$ .
- 5.) Now we will heat the solution in water bath for 30 minutes after 30 mint. we will remove the beaker and keep it for an over night.
- 6.) Now we will filter the precipitate and wash it with hot water and then with 5ml of absolute alcohol. Now finally we will dry the ppt and weight it.

**Result:-** yield of nickel dimethyl glyoximate 3.4623gm which colour is pink.

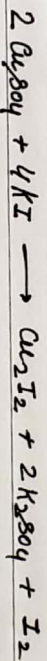
**Object:-**

Estimation of copper and nickel by volumetric and gravimetric method and analysis.

**Theory:-**

(a) Theory of estimation of copper by volumetrically-

Iodine is liberated when copper sulphate solution reacts with KI giving reddish brown colour to the solution the distorted iodine is treated against hypo solution using starch as an indicator at the end point brown colour turns of white.

**Reaction:-**

(b) Theory for estimation of copper by gravimetric method.

In a mixed solution of both copper is estimated first as cuprous thiocyanate copper is precipitated as cuprous thiocyanate from a mixed solution of copper and nickel by adding ammonia this cyanate solution is slightly acidic medium in the presence of settling agent such as  $\text{H}_2\text{SO}_4$  to reduce  $\text{Cu(III)}$  to  $\text{Cu(I)}$





Calculation for estimation of Cu by volumetrically -

1) Titration for the standard  $\text{CuSO}_4$  :-

$$N_1 = \text{Normality of standard } \text{CuSO}_4 = \frac{1}{25}$$

$$V_1 = \text{Volume of standard } \text{CuSO}_4 = 10 \text{ mL}$$

$$N_2 = \text{Normality of hypo solution} = ?$$

$$V_2 = \text{Volume of hypo used standard } \text{CuSO}_4 = 9.8$$

$$N_2 = \frac{N_1 V_1}{V_2} = \frac{1}{25} \times \frac{10}{9.8} = \frac{1}{24.5} \text{ N}$$

2) Titration for unknown solution  $\text{CuSO}_4$  :-

$$N_3 = \text{Normality of unknown } \text{CuSO}_4 = ?$$

$$V_3 = \text{Volume of unknown } \text{CuSO}_4 = 10 \text{ mL}$$

$$N_4 = \text{Normality of hypo solution} = N_2 = \frac{1}{24.5} \text{ N}$$

$$V_4 = \text{Volume of hypo solution} = 10.1$$

$$N_3 = \frac{N_4 V_4}{V_3} = \frac{1}{24.5} \times \frac{10.1}{10}$$

$$N_3 = 0.0446 \text{ N}$$

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Procedure :-

Procedure and observation for estimation of copper by volumetric method :-

Burette is washed with water and is rinsed a solution of hypo (N.25) strength it is filled with hypo solution ppt out 10ml of standard copper sulphate solution and add conical flask which is covered with a watch glass for sometime to complete the reaction. The colour of solution turn of reddish brown due to liberated iodine now - starch added new - hypo sol<sup>n</sup> is slowly added from burette to the solution in conical flask due to react.

In of Iodic hypo the solution becomes the violet. The process is repeated and concurrent volume (value) to is noted after this the ppt is washed with unknown copper sulphate solution it is then titrated against hypo solution as above method and the concurrent value of the volume is noted.

Observation - table :-

S.No.	Volume of standard $\text{CuSO}_4$	Volume of hypo (ml)			Concurrent Volume of hypo (ml)
		Initial	Final	Actual	
1.	10ml	0.0	9.8	9.8	
2.	10ml	0.0	9.8	9.8	9.8
3.	10ml	0.0	9.8	9.8	



Conc. of unknown CuSO<sub>4</sub> in gm/liter -

$$= \text{Normality} \times \text{equivalent weight}$$

$$= 0.042 \times 249.69$$

$$= 10.2872 \text{ gm/liter}$$

$$\text{Amount of copper in CuSO}_4 = \frac{63.546}{249.69} \times 10.2872$$

$$= 2.618 \text{ gm/liter}$$

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S.No.	Volume of unknown CuSO <sub>4</sub>	Volume of hypo solution			Concurrent volume at hypo (ml)
		Initial	Final	Actual	
1.	10ml	0.0	10.2	10.2	
2.	10ml	0.0	10.1	10.1	10.1
3.	10ml	0.0	10.1	10.1	

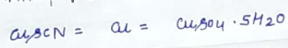
Procedure and observation for estimation of copper by gravimetric method :-

50ml of mix. of Cu-Ni is taken in a beaker now 2.5ml HCl is added to this solution 30ml Fresh H<sub>2</sub>SO<sub>4</sub> is added to this now 100ml water is added now 50ml of 10% ammonium thiocyanate is added ppt is digested on water bath till 100ml now is filtered by crucible and dried and weighted filtrate of this used estimation of nickel.

Procedure and Observation for estimation of nickel by gravimetric method :-

Filtrate of process (b) and heat it in china till solution is reached 70-80ml 35ml conc. HNO<sub>3</sub> is added 15ml conc. HCl is added to above solution is heated near dryness on stand both completely dried on water bath residue is dissolved in water and 1-2ml HCl is added solution is transferred in beaker now 150ml water is added 2-drop of methyl red is added now 30ml DMG (1:1) is added Now ammonia solution is added.

calculation for Cu estimation by gravimetric:-



$$121.54 = 63.56 = 249.69$$

$$\text{weight of copper} = \frac{63.546}{121.54} \times 0.2429$$
$$= 0.1268 \text{ gm/lit.}$$

$$\text{weight of copper sulphate} = \frac{249.69}{121.64} \times 0.2429$$
$$= 0.498 \text{ gm/lit.}$$

calculation for estimation of nickel by gravimetric:-



$$288.32 = 58.69 = \text{NiSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$$

$$\text{weight of nickel} = \frac{58.69}{288.9} \times 0.2461$$
$$= 0.04999 \text{ gm/lit.}$$

$$\text{weight of nickel ammonia sulphate} = \frac{394.98 \times 0.24}{288.9}$$
$$= 0.3364 \text{ gm/lit.}$$

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observation:-

observation of Cu:- weight of empty crucible = 29.32  
weight of crucible with ppt of  $\text{CuSCN}$  = 29.5643 gm  
weight of ppt = 0.2429 gm.

observation of Ni:- weight of empty crucible = 29.4129 gm  
weight of crucible ppt =  $[\text{Ni}(\text{HDMG})_2]$  = 23.8

$$\text{weight of ppt } [\text{Ni}(\text{HDMG})_2] = 0.2461 \text{ gm}$$

Result:-

The strength of copper & nickel in given solution is

(1) ~~Copper in copper sulphate/volumetrically~~ = 2.6180 gm/lit.

(2) ~~Copper in copper sulphate gravimetrically~~  
= 0.1268 gm/lit.

~~Nickel in nickel ammonium sulphate gravimetrically.~~

$$= 0.0499 \text{ gm/lit.}$$

Teacher's Signature : .....



**object:-** Identify for acidic (anions) and basic (cations) radicals including series in the given inorganic mixture by dry and wet test.

Glass Ware Required :-

Test tube, holder, burner, filter paper

**Test for acidic Radicals :-**

Experiment table :-

S.No.	Experiment	observation	Inference
1.)	Action of dil $H_2SO_4$ mix. + dil $H_2SO_4 + \Delta$	light brown gas involved	$NO_2$ - may be
	$Na_2CO_3 + HCl +$ fresh $FeSO_4$	black colour apper.	$NO_2$ - Confirm
2.)	Action of conc. $H_2SO_4$ mix. + conc. $H_2SO_4$	colourless gas involved with pungent odor	$Cl^-$ - may be
	mixture + conc. $H_2SO_4 +$ Heat	light brown fumes involved	$NO_3^-$ may be
	Removal of nitrite - Sodium carbonate extract + dil $H_2SO_4 + NH_4Cl +$ heat	$N_2$ is involved completely	nitrite is removed
	<b>Ring test:-</b> above solution + fresh $FeSO_4 + H_2SO_4$	brown ring is formed at two liquids	$NO_3^-$ - confirm.

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3.) other anions at converted at dil $H_2SO_4$ mix. + 2ml conc. $HNO_3$ + $(NH_4)_2SO_4$ + $\Delta$	yellow ppt obtained	$PO_4^{3-}$ confirm
4.) Test for basic Radicals (cations) :-		
- A- Flame test on burning the paste of mix. in $HCl$ with the help of $Pt$ wire	brick red coloured flame	$Ca^{+2}$
- Mixture + $NaOH$ + heat	ammonia gas not evolved	zero gas absent
- primary solutions of mix. dil $HCl$ + $\Delta$	ppt is not obtained	first group obtained (absent)
- above filtrate solution $H_2S$ gas	No. ppt is formed	second group absent
- Removal of $PO_4^{3-}$ filtrate of II <sup>nd</sup> group is bailed add conc. $H_2SO_4$ + bail + $NH_4Cl$ sol <sup>n</sup> + 0.5 $Zn(NO_3)_2$ + heat + filtrate + add $(NO_3)_2$ to the above solution	No ppt obtained	$PO_4^{3-}$ is removed
- To that above solution add solid $NH_4Cl$ + bail + $NH_4OH$ + dil $HNO_3$ + heat	White gelatinous ppt is obtained	$Al^{3+}$ may be



- ~~Residue~~ Residue + dil HCl + NaOH  
then add excess of NaOH

White ppt obtained  
ppt is disappear

$Al^{3+}$  confirm

- Filtrate of III<sup>rd</sup> group +  $NH_4Cl$  +  
 $NH_4OH$  +  $H_2S$  gas + Residue + dil.  
HCl + NaOH +  $CH_3COOH$  +  $K_4[Fe(CN)_6]$

White ppt obtained

$Zn^{2+}$  may be

White ppt obtained

$Zn^{2+}$  confirms

- filtrate of IV group + boil + cool  
+  $NH_4OH$  +  $(NH_4)_2CO_3$

$H_2S$  is removed  
White ppt is  
obtained

$Ba^{2+}$ ,  $Sr^{2+}$ ,  $Ca^{2+}$   
may be

White ppt +  $CH_3COOH$  -

i) above solution +  $K_2CrO_4$

No ppt obtained

$Ba^{2+}$  absent

ii) above sol<sup>n</sup> +  $(NH_4)_2SO_4$

No ppt "

$Sr^{2+}$  absent

iii) above solution  $(NH_4)_2C_2O_4$

White ppt

$Ca^{2+}$  confirms

Filtrate of V<sup>th</sup> group  
+  $NH_4OH$  +  $Na_2HPO_4$

No. ppt is  
obtained

V<sup>th</sup> is absent

### Test for Rarer Radicals :-

mixture + dil HCl wash ppt  
+ with hot  $H_2O$  + add  
 $NH_4OH$  to residue and  
A filter - ~~what~~ + dil HCl

White ppt obtained

$Ag^+$  / or may be

No. ppt obtained

or may be

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$\text{NH}_4\text{OH} + \text{KI} + \text{Heat}$   
3ml  $\text{SnCl}_2 + 4\text{ml}$   
conc.  $\text{HCl}$

blue ppt is  
obtained

cu is present

**Result :-**

The four acidic and basic radicals.

Acidic radicals :-  $\text{NO}_2^-$ ,  $\text{NO}_3^-$ ,  $\text{Cl}^-$ ,  $\text{PO}_4^{3-}$

basic radicals -  $\text{Al}^{3+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Cu}$

are present in the given inorganic mixture.

**Object:-** Identify for acidic (anions) and basic (cations) radicals including rarer in the given inorganic mixture by dry and wet test.

Glass ware required :-

Test tube, holder, burner, filter paper.

**Test for acid radicals:-**

Experiment	Observation	Inference
1.) Action of dil $H_2SO_4$ mixture + dil $H_2SO_4$ + heat	$CO_2$ gas evolved colourless gas evolved	$CO_3^{2+}$ may be $SO_3^{2-}$ may be
<b>Combination test:-</b> for $CO_3^{2-}$ , $SO_3^{2-}$ mixture + $K_2Cr_2O_7$ + dil $H_2SO_4$ then gas pass in water	orange sol <sup>n</sup> $CO_2$ trans it in milky in-dicator	$SO_3^{2-}$ is confirm $CO_3^{2-}$ is confirm
2.) Action of conc. $H_2SO_4$ mixture + conc. $H_2SO_4$ + heat	light brown gas evolved	$NO_3^-$ may be
Aqueous sol <sup>n</sup> of mix. + fresh $FeSO_4$ + conc. $H_2SO_4$	brown ring is formed at the junction of two liquids	$NO_3^-$ - Confirm
- mix. + conc $H_2SO_4$ + $\Delta$ + sodium carbonate extra + $CH_3COOH$ + $CaCl_2$ - solution	colourless gas I <sup>+</sup> freshly drops in the test tube obtained white ppt obtained	$F^-$ - may be $F^-$ - confirming

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### Test for basic Radicals :-

1.) Flame test :- on burning the paste of mix. in HCl through pt wire	ordinary flame appear	Interence may be
2.) Wet test :- - mixture + NaOH + Heat	ammonia gas	$\text{NH}_4^+$ may be
on taking a glass rod wetted with conc. HCl to the mouth of test tube	White fumes are formed	$\text{NH}_4^+$ confirm
- primary solution + dil HCl + $\Delta$	No-ppt is obtained	first group absent
above solution + pass $\text{H}_2\text{S}$ gas	No-ppt	second group absent
- Removal $\text{F}^-$ filtrate of 2 <sup>nd</sup> group take in porcelain dish solid + 2ml HCl and dried it 2-3 times residuc + dil HCl and make solution.	HF vapour come out side	F is Removed
- above solution + dil $\text{HNO}_3$ + $\Delta$ + solid $\text{NH}_4\text{Cl}$ + $\Delta$ + $\text{NH}_4\text{OH}$	Green ppt is obtained	$\text{Cr}^{3+}$ - may be

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Residue + Br <sub>2</sub> water + NaOH + $\Delta$ + CH <sub>3</sub> COOH + red Acetate + NaOH	yellow ppt which dissolved ppt	Cr <sup>+3</sup> confirm
- Filtrate of third group + NH <sub>4</sub> Cl + $\Delta$ + NH <sub>4</sub> OH + H <sub>2</sub> S	No ppt obtained	fourth group absent
- Filtrate of 3 <sup>rd</sup> group + boil then cool + NH <sub>4</sub> OH + (NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	H <sub>2</sub> S gas is removed ppt is not obtained	V group absent
- Filtrate of third group + NH <sub>4</sub> OH + NaNO <sub>2</sub>	white ppt is obtained	Mg <sup>+2</sup> may be
- Residue + HCl + NaOH + lith yellow reagent + $\Delta$	pink ppt is obtained	Mg <sup>+2</sup> confirm
<b>Test for Royer Radicals:-</b>		
Residue + conc. HCl + water + pass H <sub>2</sub> S gas	ppt is obtained	As may be
- Wash residue with dil HCl + ammonium solution + $\Delta$ filter it.	No - ppt is obtained	
- Filtrate + dil HCl + sodium potassium Xanthate	Red colour soli solution	Mo - confirm
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Result :-

The four acidic and basic radicals-

Acidic :-  $\text{SO}_3^{2-}$ ,  $\text{CO}_3^{2-}$ ,  $\text{NO}_3^-$ ,  $\text{F}^-$

basic :-  $\text{NH}_4^+$ ,  $\text{Cr}^{+3}$ ,  $\text{Mg}^{+2}$ ,  $\text{MnO}$

are present in the given in-organic mixture.

*Suman*  
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**object:-**

Identify four acidic (anions) and four basic (cations) radicals including verer in the given in-organic mixture by dry and wetted test.

Glass ware required:- Test tube, holder burner, filter paper.

**Test for acidic radicals:-**

S.No.	Experiment	observation	Inference
1.)	Action of dil $H_2SO_4$ mixture + dil $H_2SO_4 + \Delta$	colourless gas with smell of burning sulphur	$SO_3^{2-}$ - may be
	on keeping wetted filter paper with dil $H_2SO_4$ and $K_2Cr_2O_7$	turns filter paper in green	$SO_3^{2-}$ confirm
2.)	Action of conc. $H_2SO_4$ mixture + conc. $H_2SO_4 +$ sodium extract.	violet gas evolved	$I^-$ - may be
	dil $HNO_3 +$ acidic + $AgNO_3 + NH_4OH$	yellow ppt obtained	$I^-$ confirm
	mixture + conc. $H_2SO_4$	Insoluble pingate gas	$Cl^-$ - may be
	- Removal of $I^-$ sodium carbonate extract + dil $H_2SO_4 + NaNO_2 +$ heat	violet fumes are complete evolved	$I^-$ is removed

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**combination test :-**

mixture +  $K_2Cr_2O_7$  + conc.  $H_2SO_4$   
 $\Delta \rightarrow$   
 gas potassium  $NO_3$  sol<sup>n</sup> +  
 $CH_3COOH + (CH_3COO)_2Pb$

(3) Action of other anions which  
 not convert dil  $H_2SO_4$

- conc.  $H_2SO_4$  + take mix. +  
 conc.  $H_2SO_4 + CH_3COOH + \Delta$   
 + ignite the vapours

**Test for basic radicals :-**

1. Flame test :-  $\rightarrow$  on lowering  
 the part of wire  
 through pt wire.

2) Wet test :-

mixture +  $NaOH + \Delta$

- primary solution + dil  $HCl + heat$

- above solution dil  $HCl + pass H_2S$   
 gas residue + yellow ammonium  
 sulphate +  $NaOH$

+ add dil  $HCl + filter + add conc.$   
 $HCl + NH_4OH + oxalic acid +$   
 $pass H_2S gas$

yellow coloured

ppt is obtained

a green edged flame  
 is appeared

casassy green  
 flame appear

ammonia gas is not  
 evolved

No ppt is obtained

ppt is obtained  
 ppt is dissolved in  
 yellow ammonium sol<sup>n</sup>

ppt is obtained  
 soluble in dil  $HCl$

orange ppt obtained

$Cl^-$  confirm

$Ba^{2+}$  confirm

$Ba^{2+}$  may be

zero group absent

first group absent

II<sup>nd</sup> group may be

$Sb^{+3}, Sn^{+2}$   
 may be

$Sb^{+3}$  confirm

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Removal of  $Bo_3^{-3}$  take  
 china dish filtrate of II<sup>nd</sup>  
 group +  $\Delta + conc. H_2SO_4$   
 Repeat it 2-3 times  
 residue + dil  $HCl$

- above solution + boil + dil  
 $HNO_3 + NH_4Cl + heat + NH_4OH$

- above solution +  $NH_4Cl +$   
 $NH_4OH + pass H_2S gas$

- residue + dil  $HCl + NaOH$   
 $+ CH_3COOH + K_4[Fe(CN)_6]$

- filtrate of 4<sup>th</sup> group +  
 boil + cool +  $NH_4OH + (NH_4)_2CO_3$

- white ppt is +  $CH_3COOH$   
 + divided in 3-part

- above sol<sup>n</sup> +  $K_2CrO_4$  filtrate  
 of ~~II~~ VI group +  $NH_4OH$   
 $+ Na_2HPO_4$

**Test for basic radicals :-**

Filtrate of II<sup>nd</sup> group  
 $+ \Delta + NH_4OH$

completely dried again  
 dry it

$H_2S$  gas is removed  
 No ppt is obtained

white ppt is  
 obtained

white ppt obtained

$H_2S$  gas is removed

yellow ppt obtained

obtained No ppt is  
 obtained

$H_2S$  gas removed  
 ppt is obtained  
 brown colour

$Bo_3^{-3}$  is removed

III group absent

$Zn^{+2}$  may be

$Zn^{+2}$  confirm

$Ba^{2+}, Sr^{+2}, Ca^{+2}$   
 may be

$Ba^{2+}$  confirm

confirm VI group  
 absent

V may be

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Residue + dil HCl + 10%  $H_2O_2$   
 +  $\Delta$  + dil  $HNO_3$  +  $Pb(NO_3)_2$   
 +  $CH_3COONH_4$  + Residue +  
 dil  $HNO_3$  + alcohol +  $H_2O_2$

red colour in  
 lower layer

V - confirm

**Result :-**

The fewer acidic radicals  $SO_3^{2-}$ ,  $I^-$ ,  $Cl^-$ ,  $BO_3^{-3}$

and basic radicals  $Sb^{+3}$ ,  $Zn^{+2}$ ,  $Ba^{+2}$ ,  $V^{+2}$  are present in the given in-organic mixture.

**object:-**

Identify acidic (anions) and basic (cations) radicals including rarer in the given in-organic mixture.

**Glass ware required:-**

Test tube holder, burner filter paper.

**Test for acidic radicals:-**

Experiment	observation	Inference
1) Action of dil $H_2SO_4$ mixture + dil $H_2SO_4$	vinegar like odour	$CH_3COO^-$ may be
sodium carbonate extract + $H_2SO_4$ + diphenyl ammonie solution	deep blue colour appear	$CH_3COO^-$ confirm
2) Action of conc. $H_2SO_4$ mix. conc. $H_2SO_4$ sodium carbonate extract + dil $CH_3COOH$	colourless gas white ppt obtained	$CO_3^{2-}$ may be $CO_3^{2-}$ confirms
mixture + conc. $H_2SO_4$ + $\Delta$	pungent brown gas evolved	$Br^-$ may be
primary solution + dil $HCl$ + heat above $80^\circ C$ + dil $HCl$ + $H_2S$ gas pass	No- ppt is obtained ppt is obtained	I <sup>st</sup> group absent II <sup>nd</sup> group may be



Residue + yellow ammonium sulphate + NaOH + $\Delta$	ppt is insoluble	$\text{II}^{\text{nd}}$ group may be
+ add $\text{HNO}_3$ + heat + dil $\text{H}_2\text{SO}_4$ + $\text{C}_2\text{H}_5\text{OH}$ + $\text{NH}_4\text{OH}$	No-ppt is obtained	$\text{Ca}^{2+}$ may be
+ $\text{CH}_3\text{COOH}$ + $\text{K}_4[\text{Fe}(\text{CN})_6]$	chocolatey coloured ppt	$\text{Ca}^{2+}$ confirm.
- Removal of $\text{CO}_3^{2-}$ :- filtrate of $\text{II}$ group in china dish heat till	dried	
then cauldred + conc. $\text{HNO}_3$ + heat repeat it 2-3 times then residue + dil $\text{HCl}$	Residue and $\text{H}_2\text{O}$ formed	$\text{CO}_3^{2-}$ is removed
+ $\text{NH}_4\text{Cl}$ + $\Delta$ + $\text{NH}_4\text{OH}$	No-ppt	absent
- filtrate of $\text{II}^{\text{nd}}$ group + $\text{NH}_4\text{OH}$ + pass $\text{H}_2\text{S}$ gas	brick red coloured ppt is obtained	$\text{V}$ group may be
+ add dil $\text{HCl}$	soluble	$\text{Mn}^{2+}$ , $\text{Zn}^{2+}$ may be
- Residue + dil $\text{HNO}_3$ + $\text{PbO}_2$ + cool	Violet colour sol <sup>n</sup> appear	$\text{Mn}^{2+}$ confirm
- filtrate of $\text{IV}^{\text{th}}$ group + $\Delta$ + cool + $\text{NH}_4\text{OH}$ + $(\text{NH}_4)_2\text{CO}_3$	$\text{H}_2\text{S}$ gas is removed white ppt is obtained	$\text{Ba}^{2+}$ , $\text{Sr}^{2+}$ , $\text{Ca}^{2+}$ may be
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Residue + $\text{CH}_3\text{COOH}$ :-		
- above solution + $\text{K}_2\text{CrO}_4$	No - ppt obtain	$\text{Ba}^{2+}$ - absent
- above solution $(\text{NH}_4)_2\text{SO}_4$	No - ppt obtain	$\text{Ba}^{2+}$ - absent $\text{Sr}^{2+}$ - confirm
- filtrate of $\text{V}^{\text{th}}$ group + $\text{NH}_4\text{OH}$ + $\text{Na}_2\text{HPO}_4$	No - ppt is obtained	$\text{V}^{\text{th}}$ group is absent
<b>Test for rarer radicals :-</b>		
mixture of sol <sup>n</sup> + $\text{NaCl}$ + ferric periodate + water filtrate tube only one drop flame test	White ppt is obtained	$\text{Li}^+$ confirm

**Result :-**

Four acidic radicals -  $\text{CH}_3\text{COO}^-$ ,  $\text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{C}_2\text{O}_4^{2-}$   
and basic radicals -  $\text{Ca}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Sr}^{2+}$  and  $\text{Li}^+$  are  
present in the given inorganic mixture.



**object :-**

Identify acidic (anions) and basic (cation) in the given in-organic mixture including rarer by dry and wetted test.

**Glass ware required :-**

Test tube, holder, burner, filter paper

Experiment	Observation	Inference
1) Action of dil $H_2SO_4$ mixture + dil $H_2SO_4$	colourless $CO_2$ gas evolved	$CO_3^{2-}$ may be
- mixture + dil $H_2SO_4$ + heat	colourless gas with smell of burning sulphur	$SO_3^{2-}$ may be
<b>Combination test :-</b> for $CO_3^{2-}$ , $SO_3^{2-}$ mixture + $K_2Cr_2O_7$ + dil $H_2SO_4$	orange solution obtained	$SO_3^{2-}$ confirm
- evolved gas pass in time water Action of conc. $H_2SO_4$	$CO_2$ gas turns it in milky indicate	$CO_3^{2-}$ confirm
mix. + conc. $H_2SO_4$ + $\Delta$ + mix. conc. $H_2SO_4$ + Copper chips + $\Delta$	light-brown vapours evolution gas increase	$NO_3^-$ may be $NO_3^-$ - confirm
- mix + conc. $H_2SO_4$ + $\Delta$	colourless gas evolved	$CO_3^{2-}$ may be

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sodium carbonate -

extract + dil  $\text{CH}_3\text{COOH}$  +  $\text{CaCl}_2$ 

white ppt obtained

 $\text{C}_2\text{O}_4^{2-}$  confirm**Test for basic radicals :-**flame test :- on burning the  
paste of mix. HCl  
through pt. wirebrick red flame  
appears $\text{Ca}^{+2}$  may be

Wet test :-

mixture + NaOH + heat

ammonia gas is  
not evolved $\text{NH}_4^+$  absentprimary solution + pass dil HCl +  
 $\Delta$  +  $\text{H}_2\text{S}$  gas + above  
solution

No - ppt is obtained

first group absent

ppt is obtained

II<sup>nd</sup> group may beFilter the ppt + yellow ammonium  
sulphide + NaOH + heatppt dissolved in  
yellow amm. sul.

II B group may be

+ add. dil HCl

coloured ppt obtained

 $\text{As}^{+3}$ ,  $\text{Sb}^{+3}$ ,  $\text{Sn}^{+2}$   
may be

add conc. HCl

ppt is obtained

 $\text{As}^{+3}$  may beReduce +  $\text{HNO}_3$  +  $(\text{NH}_4)_2\text{MoO}_4$ yellow ppt is  
obtained $\text{As}^{+3}$  confirmRemoval of  $\text{C}_2\text{O}_4^{2-}$  -filtrate of II<sup>nd</sup> group take in  
china dish heat till dried + cool  
+ conc.  $\text{HNO}_3$  +  $\Delta$  + till dry it 2-3  
times + add dil HClRed residue  $\text{CO}_2$   
and  $\text{H}_2\text{O}$  formed $\text{C}_2\text{O}_4^{2-}$  is removed

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above solution + heat + conc. $\text{HNO}_3$ + $\Delta$ + $\text{NH}_4\text{Cl}$ + $\Delta$ + $\text{NH}_4\text{OH}$	$\text{H}_2\text{S}$ gas is removed ppt is obtained of brown colour	$\text{Fe}^{+3}$ may be
Residue + dil $\text{HCl}$ + $\text{KCN}$ s	Red colour sol <sup>n</sup> obtained	$\text{Fe}^{+3}$ confirm
- Filtrate of III <sup>rd</sup> group + $\text{NH}_4\text{Cl}$ + $\text{NH}_4\text{OH}$ + $\text{H}_2\text{S}$ gas	No-ppt is obtained	IV group is absent
- Filtrate of III <sup>rd</sup> group + $\Delta$ + $\text{H}_2\text{SO}_4$ gas is removed + boil + cooled	$\text{H}_2\text{S}$ gas removed	
+ $\text{NH}_4\text{OH}$ + $(\text{NH}_4)_2\text{CO}_3$	white ppt	$\text{Ba}^{+2}$ , $\text{Sr}^{+2}$ , $\text{Ca}^{+2}$ may be
Residue + $\text{CH}_3\text{COOH}$ acid - above + $\text{K}_2\text{Cr}_2\text{O}_7$ above sol <sup>n</sup> + $(\text{NH}_4)_2\text{SO}_4$	No-ppt obtained No-ppt	$\text{Ba}^{+2}$ - absent $\text{Sr}^{+2}$ - absent
above sol <sup>n</sup> + $(\text{NH}_4)_2\text{C}_2\text{O}_4$	white ppt obtained	$\text{Ca}^{+2}$ - confirm
Filtrate of I <sup>th</sup> group + $\text{NH}_4\text{OH}$ + $\text{Na}_2\text{HPO}_4$	No-ppt is obtained	II <sup>th</sup> group absent
Test for rare radicals :-		
Filtrate of II <sup>nd</sup> group + $\text{NH}_4\text{OH}$	white ppt is obtained	Ti, Zr, Be may be

Residue + dil HCl + 10%  
H<sub>2</sub>O<sub>3</sub> + BaCl

ppt is obtained

wash ppt with NH<sub>4</sub>NO<sub>3</sub>  
+ H<sub>2</sub>O<sub>2</sub> + sodium  
sulphate solution.

white ppt obtained

Zr - confirm

Result :-

The four acidic and basic radicals -

acidic radicals :- CO<sub>3</sub><sup>2-</sup>, SO<sub>3</sub><sup>2-</sup>, NO<sub>3</sub><sup>-</sup>, C<sub>2</sub>O<sub>4</sub><sup>2-</sup>

basic radicals :- As<sup>+3</sup>, Fe<sup>+3</sup>, Zr<sup>+4</sup>, Co<sup>+2</sup>

present in given  
In-organic mixture.



**object:-**

Identify acidic (anions) and basic (cations) radicals including rarer in the inorganic mixture.

**Test for acidic radicals:-**

Experiment	observation	Inference.
1. Action of dil $H_2SO_4$ mixture + dil $H_2SO_4$ + $\Delta$	light brown gas evolved	$NO_2^-$ may be
- sodium carbonate extract + HCl + fresh $FeSO_4$	Black colour solution appeared	$NO_2^-$ confirm
2. Action of conc. $H_2SO_4$ mixture + conc. $H_2SO_4$ + $\Delta$	light brown vapour formed	$NO_2^-$ may be
Removal of Nitrite - sodium carbonate extracts + dil $H_2SO_4$ + $NH_4Cl$ + $\Delta$	$N_2$ is evolved completely	Nitrite is remove
Ring test:- above solution + fresh $FeSO_4$ + conc. $H_2SO_4$	brown ring is formed of the function of two liquid	$NO_3^-$ confirm
other anions which not covered conc. $H_2SO_4$ + dil $H_2SO_4$		

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mixture + 2ml conc.  $\text{HNO}_3$   
+ 12  $(\text{NH}_4)_2\text{MnO}_4 + \Delta$

yellow ppt is  
obtained

$\text{PO}_4^{3-}$  confirm

- Sodium carbonate extract  
+ dil  $\text{HNO}_3 + \text{BaCl}_2$

white ppt is obtained

$\text{SO}_4^{2-}$  may be

- filtrate + conc.  $\text{HNO}_3 + \text{BaCl}_2$

residuals remaining  
insoluble formed

$\text{SO}_4^{2-}$  confirm

### Test for basic radicals :-

#### 1) Flame test :-

on a burning the paste of  
mixture in  $\text{HCl}$  with the  
help of pt wire

blue coloured  
flame

$\text{Cu}^{+2}$  may be

mixture +  $\text{NaOH} + \Delta$

ammonia gas evolved

$\text{NH}_4^+$  may be

on taking a glass rod fitted  
with conc.  $\text{HCl}$  to the  
mouth of test tube

white flame are  
formed

$\text{NH}_4^+$  confirm

primary solution + dil  $\text{HCl}$   
+  $\Delta$

No-ppt is obtained

I<sup>st</sup> group absent

above sol<sup>n</sup> + dil  $\text{HCl} + \text{H}_2\text{S}$  gas  
is passed

ppt is obtained

II<sup>nd</sup> group may be

+ add  $\text{HNO}_3 + \Delta + \text{dil H}_2\text{SO}_4 +$   
 $\text{C}_2\text{H}_5\text{OH} + \text{NH}_4\text{OH}$

No-ppt obtained

$\text{Cu}^{+2}$  may be

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+ $\text{CH}_3\text{COOH}$ + $\text{K}_4[\text{Fe}(\text{CN})_6]$	chocolatey coloured ppt	$\text{Cu}^{2+}$ confirm.
Removal of $\text{PO}_4^{3-}$ :-		
- Filtrate of II <sup>nd</sup> group bail + conc. $\text{HNO}_3$ + $\text{NH}_4\text{Cl}$ + bail + $\text{NH}_4\text{OH}$	$\text{H}_2\text{S}$ gas is removed No - ppt is obtained	III <sup>rd</sup> group absent
- above sol <sup>n</sup> + $\text{NH}_4\text{OH}$ + pass $\text{H}_2\text{S}$ gas	brick red coloured ppt	
+ add dil $\text{HCl}$ + bail + $\text{NaOH}$	soluble $\text{H}_2\text{S}$ is removed ppt is obtained	$\text{Mn}^{2+}$ , $\text{Zn}^{2+}$ may be
- above residue + dil $\text{HNO}_3$ + conc. $\text{HNO}_3$ + $\Delta$ + $\text{PbO}_3$ + cool	Violet colour sol <sup>n</sup> appears	$\text{Mn}^{2+}$ confirm
- Filtrate of IV <sup>th</sup> group + bail + cool + $\text{NH}_4\text{OH}$ + $(\text{NH}_4)_2\text{CO}_3$	$\text{H}_2\text{S}$ gas is removed No - ppt obtained	V <sup>th</sup> group absent
- above solution + $\text{NH}_4\text{OH}$ + $\text{Na}_2\text{HPO}_4$	No - ppt is obtained	VI group absent
Test for rare radicals :-		

Reduce + conc. HCl +  
H<sub>2</sub>O + pass H<sub>2</sub>S gas  
wash reduce with dil.  
HCl + ammonia + Δ +  
filter -

ppt is obtained

Ar or mo may be

Wash reduce with dil.  
HCl + ammonia + Δ +  
filter -

No - ppt is obtained

mo - may be

- Filtrate + dil HCl +  
sodium potassium  
dantate

red coloured  
solution

Mo - confirm

**Result :-**

The four acidic radicals are  $\text{NO}_2^-$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$ ,  
 $\text{PO}_4^{3-}$  and basic radicals are  $\text{NH}_4^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$   
and mo present in the given mixture.

*Suwendy*  
23/4/18

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