

Ex 2

## SALT ANALYSIS

H<sub>2</sub>SO<sub>4</sub>/HNO<sub>3</sub>

CLASSMATE

Date \_\_\_\_\_

Page \_\_\_\_\_

- o) Identification of cation and anions in given salt is called salt analysis.
- o) Salt analysis is test of cation and anions.
- o) Diff test of cation and anions gives some specific observable changes which gives information about the cation and anions.

(Mainly salts here)

Test of Ionic radical

Test of cation

(basic & radical)

→ Dry test

→ Flame test

→ wet test

Test of anion

(acidic radical)

→ Wet test

Interfering radicals some of anions are act as interfering radical during the test of cations. They are called interfering radicals. systematic  
eg PO<sub>4</sub><sup>3-</sup>, C<sub>2</sub>O<sub>4</sub><sup>2-</sup>, BO<sub>3</sub><sup>3-</sup>, F<sup>-</sup>, SiO<sub>4</sub><sup>4-</sup>/SiO<sub>3</sub><sup>2-</sup>

During salt analysis anions are identify before test of cations because some of anions are interfering and they can interfere during systematic procedure to identify cations

If any such radical is present it must be removed by suitable method before test of cations.

## Test of Anions

### Classification

#### Class A

ion which produce volatile product with  $H_2SO_4$

#### Class B

anions which don't produce volatile product with  $H_2SO_4$

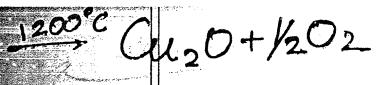
group I  
if  $H_2SO_4$ )  
similarly

subgroup II  
(conc  $H_2SO_4$ )

subgroup I  
(By ppt for)

subgroup II  
(which identify by  
redox)

$O_3^{2-}$	$F^-$	$SO_4^{2-}$	(orange) $Cr_2O_7^{2-}$ chromate
$CO_3^{2-}$	$Cl^-$	$PO_4^{3-}$	
$SO_3^{2-}$	$Br^-$	greenate $AsO_4^{3-}$	(yellow) $Cr_2O_7^{2-}$ chromate
$SO_3^{2-}$	$I^-$	$CrO_4^{2-}$	
$S^{2-}$	$NO_3^-$	$MnO_4^-$	(purple) $MnO_4^-$ manganate (green) $MnO_4^-$ manganate
$S_2O_3^{2-}$	$C_2O_4^{2-}$	$Po_4^{3-}$	
$NO_2^-$	$BO_3^{3-}$		
$H_3COO^-$			



classmate

Date \_\_\_\_\_

Page \_\_\_\_\_

# CLASS A

Subgroup (I) (dil H<sub>2</sub>SO<sub>4</sub>/dil HCl)

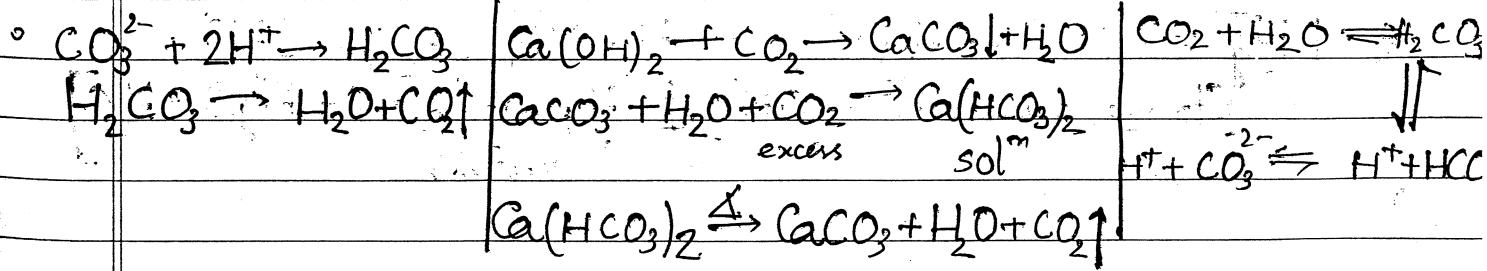
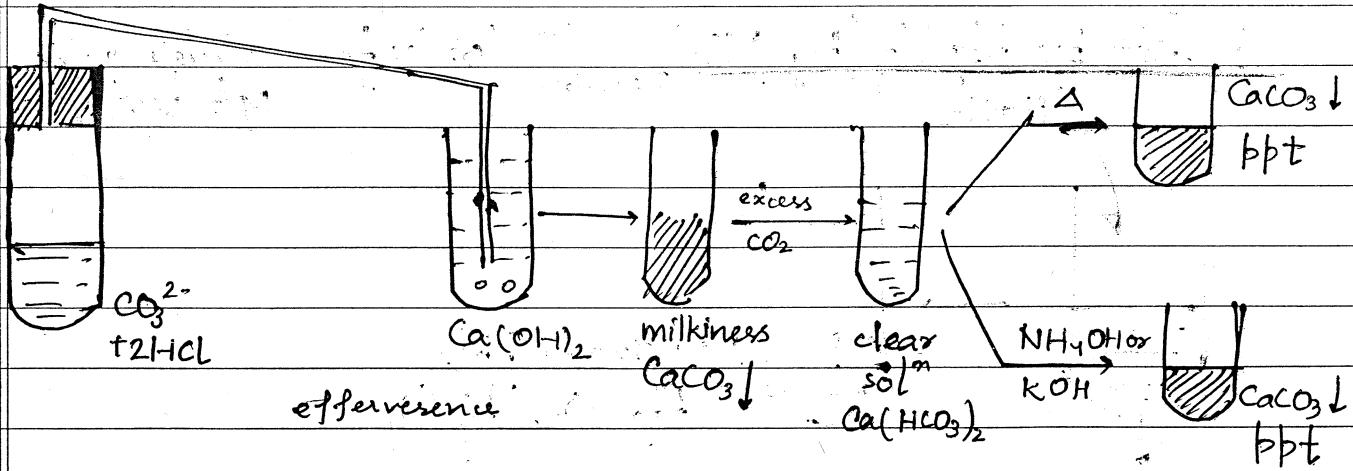
(Acids: non redox, non dehydrating)

## TEST OF CO<sub>3</sub>

- (1) All carbonates are water insoluble except IA carbonate (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>. [Li<sub>2</sub>CO<sub>3</sub>: insoluble]  
sparingly soluble

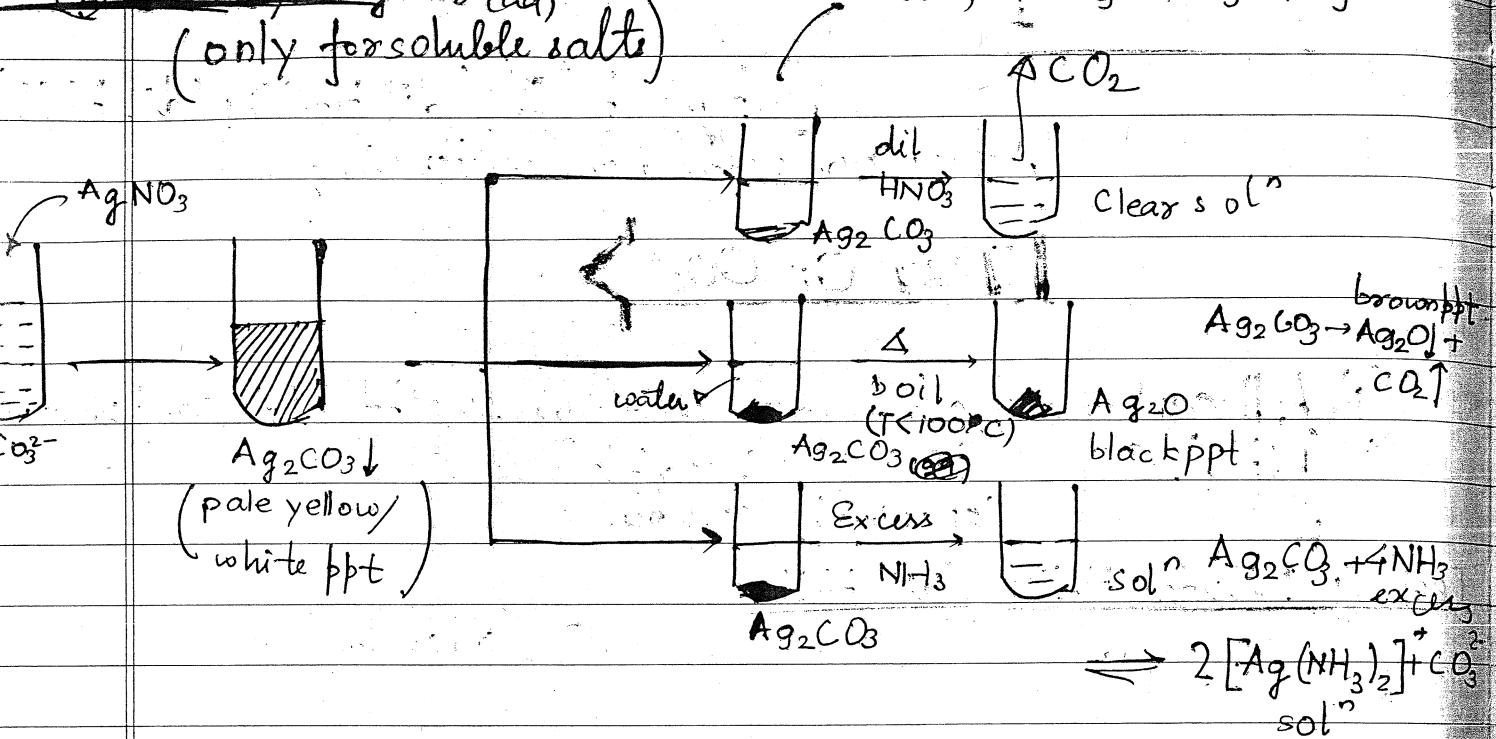
Test (1) Dilute H<sub>2</sub>SO<sub>4</sub>/dil HCl

CO<sub>2</sub> (odourless)



② Test by  $\text{AgNO}_3$  (aa)  
(only for soluble salts)

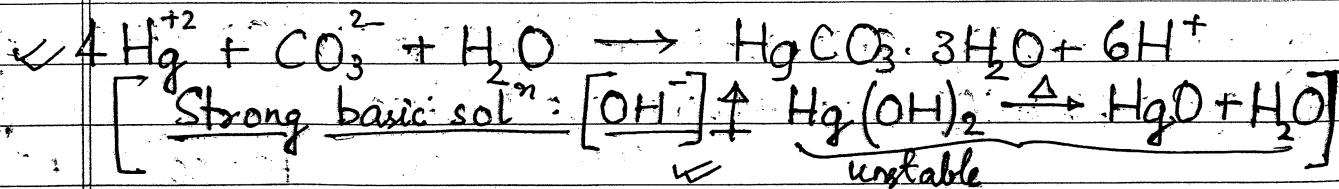
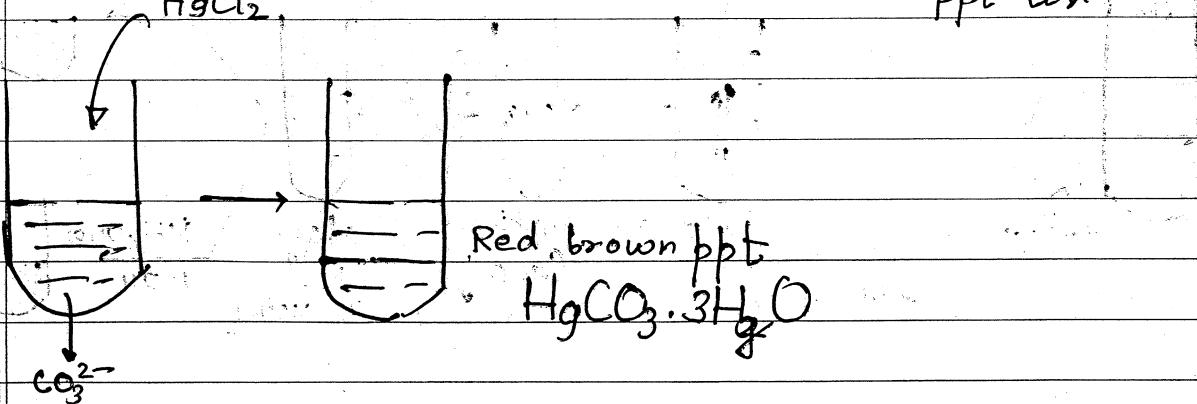
diff. from  $\text{AgCl}$ ;  $\text{AgBr}$ ;  $\text{AgI}$



③ Test by  $\text{HgCl}_2$ /  $\text{Hg}(\text{NO}_3)_2$  soluble  
(only for soluble salts)

$\text{HgCl}_2$

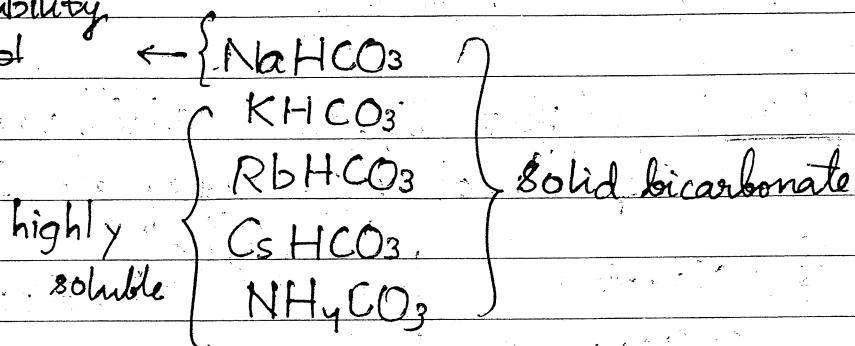
ppt test



## TEST OF BICARBONATE

Solubility

sparingly sol  
soluble

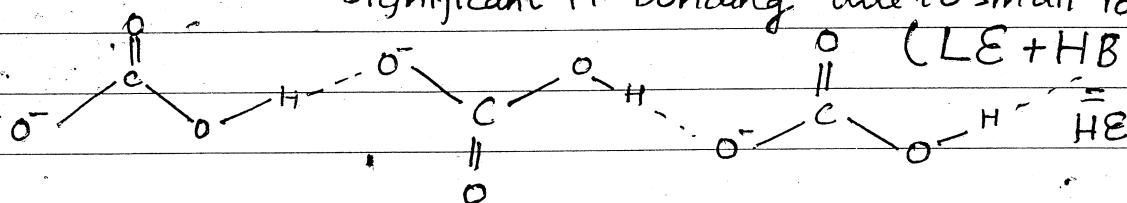


$\text{NaHCO}_3$

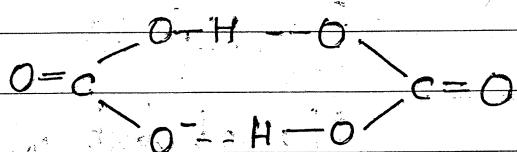
(s)

significant H-bonding due to small ion

(LE + HBE)



$\text{KHCO}_3$  (s)

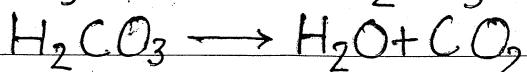
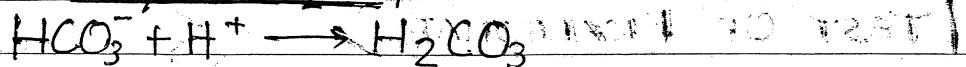


However due to large size of  $\text{K}^+$ , extended H-bonding not possible.

- II<sup>b</sup> carbonates do not exist in solid form due to high solubility polarisability of cation (Thermal stability ↓)

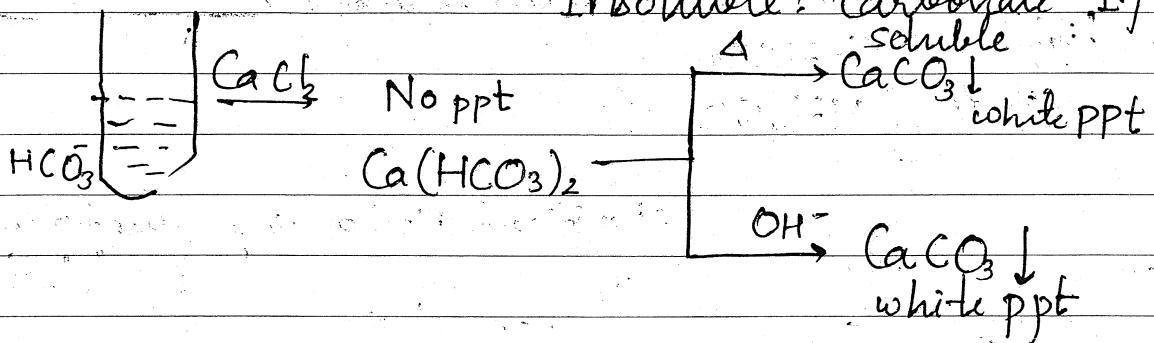
- After potassium bicarbonate the separation becomes significant to prevent ft bonding

t ① Test by dil. HCl/dil H<sub>2</sub>SO<sub>4</sub>

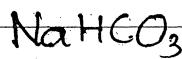
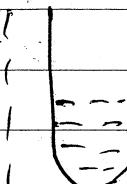


t ② Test by BaCl<sub>2</sub>/CaCl<sub>2</sub> (first dissolve in distill water)

Insoluble: carbonate If



Q



$\text{CaCl}_2$ : white ppt  
 $\text{CaCO}_3$

No ppt but white ppt on  $\Delta$

$\text{FeCl}_3$ : Red brown ppt  
 $\text{Fe(OH)}_3$   
 pink  
 (in oilphalim)

No ppt

colourless

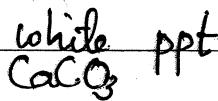
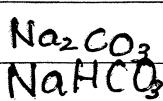
$(\text{NO}_3)_2$ : white ppt

No ppt

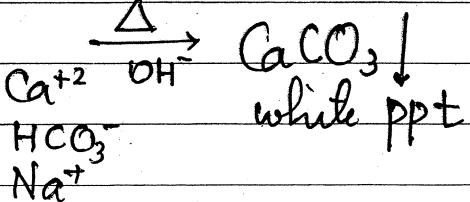
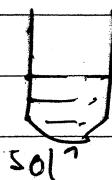
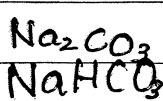
To prove simultaneous presence

prove carbonate

Q



$\xrightarrow{\Delta}$



proves  
bicarbonate

• also Hph pink

( $\Delta \text{CO}_2$   
of solid crystals)

aqua regia

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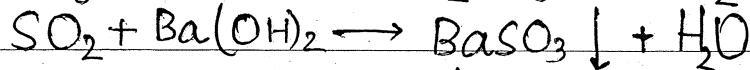
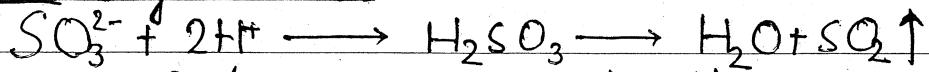
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Page \_\_\_\_\_

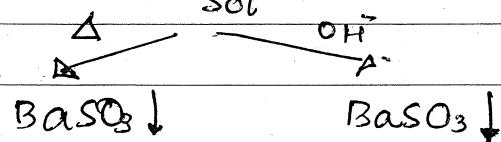
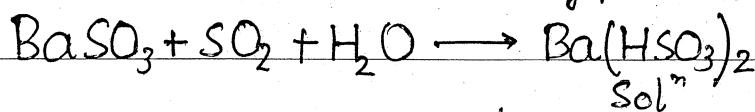
## TEST OF $\text{SO}_3^{2-}$

solubility of sulphite salt same as carbonate salt  
 $(\text{Li}_2\text{SO}_3)$  soluble

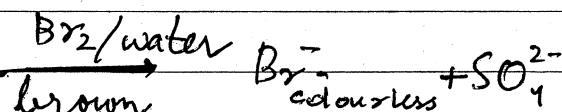
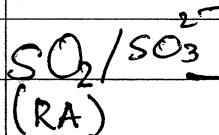
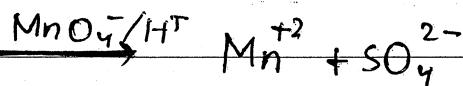
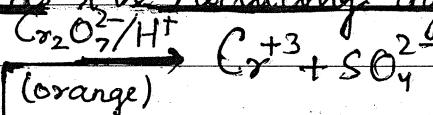
### ① Test by dil HCl



white ppt  
(milky)



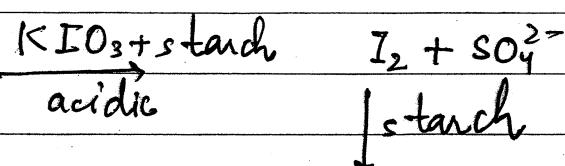
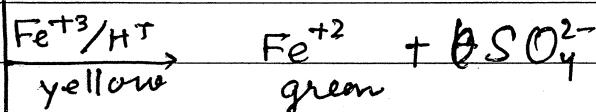
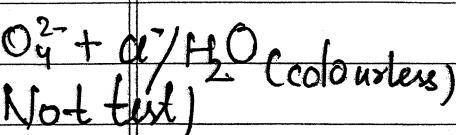
### ② Test of $\text{SO}_2$ based on its reducing agent



$\text{Cl}_2$

or (colourless)

$\frac{1}{2}\text{O}_2 \downarrow$



blue black

(starch iodate test : )  
all RA give test

$\text{SO}_2$  solubility >  $\text{SO}_3$   
(fairly)

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Date \_\_\_\_\_

Page \_\_\_\_\_

$\text{SO}_2$  better RA

Q  $\text{H}_2\text{S}$  is only reducing agent but  $\text{SO}_2$  is OA as well as RA. Explain?

A  $\text{H}_2\text{S} \quad \text{S}^{2-}$  (-II to VI)

$\text{SO}_2 \quad \text{S}^{4+}$  (-II to VI) both possible

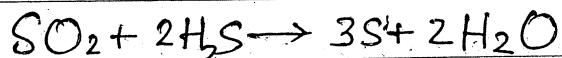
Q What happens when  $\text{H}_2\text{S}$  is passed through aq. soln of  $\text{SO}_2$ ?

A  $2\text{H}_2\text{S} + \text{SO}_2 \rightarrow 3\text{S} + 2\text{H}_2\text{O}$  (Comproportionation rxn)  
(yellow ppt turbidity)

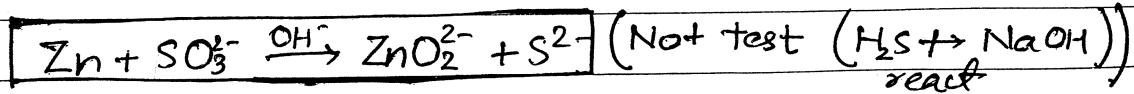
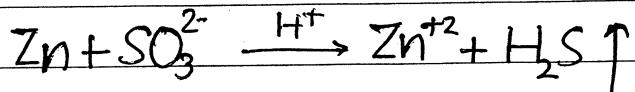
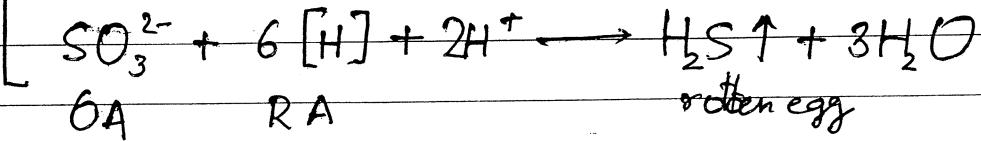
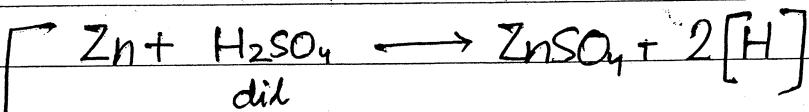
- Colloidal sulphur obtained in this rxn (fine particles)
- pure sulphur obtained by above rxn on heating

③ Test of  $\text{SO}_2$  based on its oxidising nature

(i) Test by  $\text{H}_2\text{S}$



(ii) Test by ( $\text{Zn} + \text{dil. H}_2\text{SO}_4$ ) (iron)



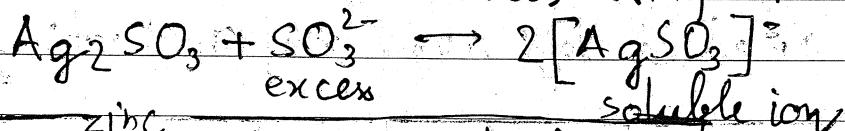
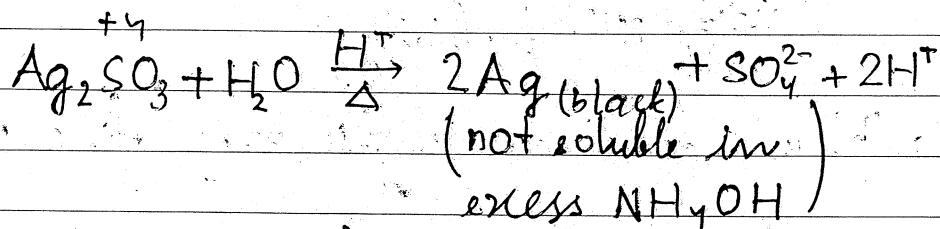
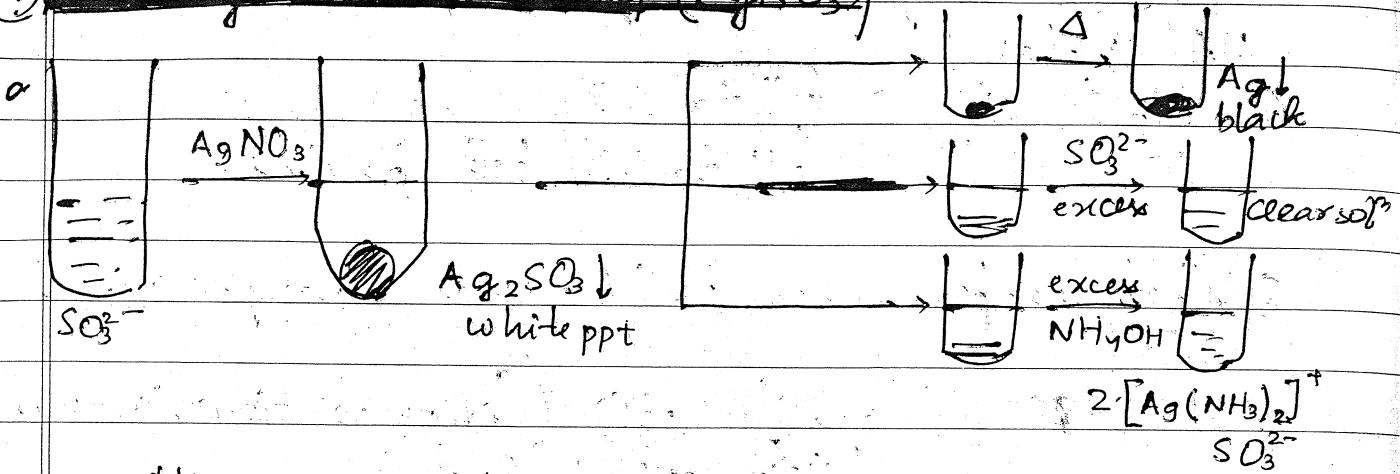
In nitric acid, ppt may dissolve to some extent ( $\text{Ag}_2\text{SO}_4$  ppt])

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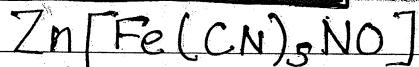
Date \_\_\_\_\_

Page \_\_\_\_\_

iv) Test by silver nitrate ag ( $\text{AgNO}_3$ )



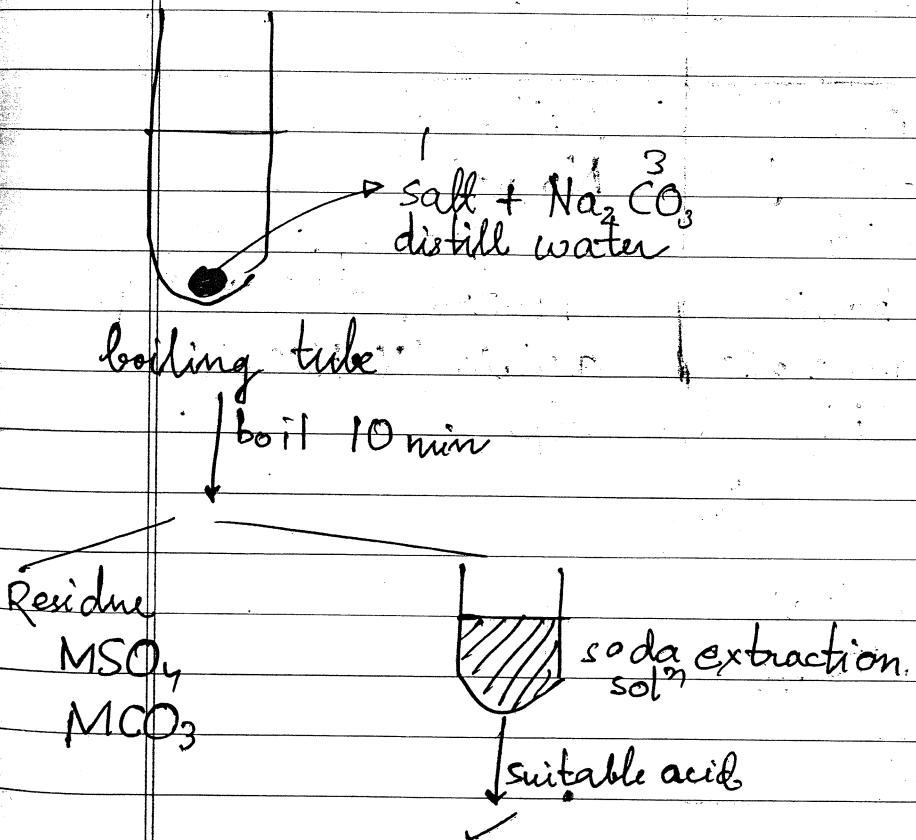
v) Sodium nitroprusside test

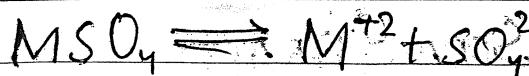


Salmon red colour of zinc nitroprusside is converted to red colour when it is placed with  $\text{SO}_2$  or  $\text{SO}_3^{2-}$  due to formation of unknown composition

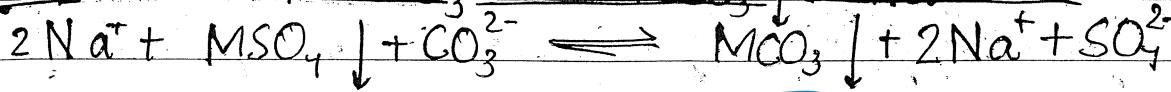
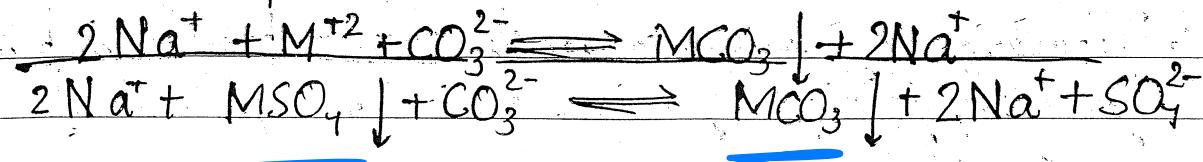
## PREPARATION OF SODA EXTRACT

1. Preparation of sodium carbonate extract provide imp method for identification of anions which are given in form of insoluble salt
2. This method is based on ppt exchange rxn or double displacement method (very slow)
3. Sodium carbonate is preferred to prefer extract because of sodium salt are water soluble
4. Excess of sodium carbonate present in extract is neutralised by suitable acid
5. Soda extract is used for test of anions (except carbonates and bicarbonate)





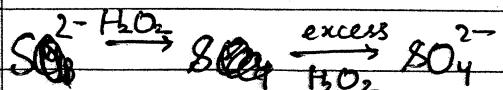
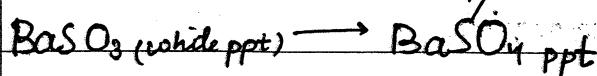
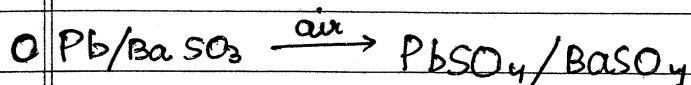
$$K_{sp} = 10^{-5}$$



- Suitable acid so that anion of acid does not interfere during test of unknown anion
- Cation test not done because present in residue
- Reagent attack on solid slow (10min) ✓

Test of  $HSO_4^-$  → (Solubility same as bicarbonate)

	$Na_2SO_3$	$NaHSO_3$
1. dil HCl	$SO_2 \uparrow$	$SO_2 \uparrow$
2. $MnO_4^- / H^+$	$Mn^{+2} + SO_4^{2-}$	$Mn^{+2} + SO_4^{2-}$
3. $BaCl_2$	$BaSO_3 \downarrow$ white	No ppt
4. litmus	basic	neutral ( $HSO_4^- K_a$ )
5. $H_2O_2$ / litmus	neutral	acidic ( $NaHSO_3$ )
6. Zn / dil $H_2SO_4$	$H_2S$	$H_2S$
		$Na_2SO_4$



lime water :  $\text{Ca(OH)}_2$

slaked lime : ~~o.ppt for conc. solution~~  
 $\text{Ca(OH)}_2$  of  $\text{Ca(OH)}_2$  (Moss salt)

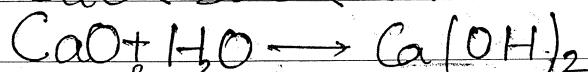
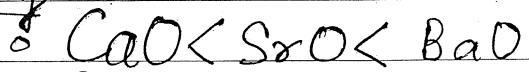
### Test of $S^{2-}$

### SOLUBILITY

All sulphide are water insoluble but sulphides of first A and II A are sparingly soluble and are fairly soluble

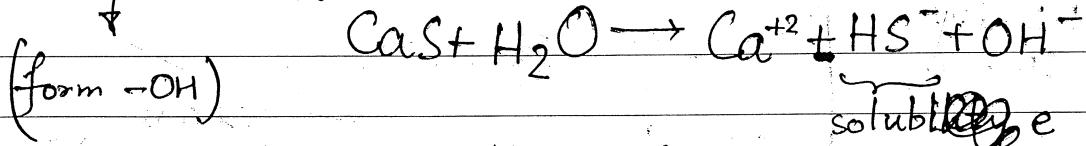
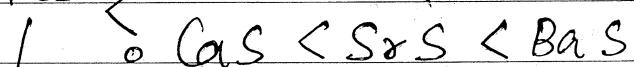
solubility of II A sulphides gradually change on contact with water and  $(\text{NH}_4)_2\text{S}$  water soluble

### Solubility

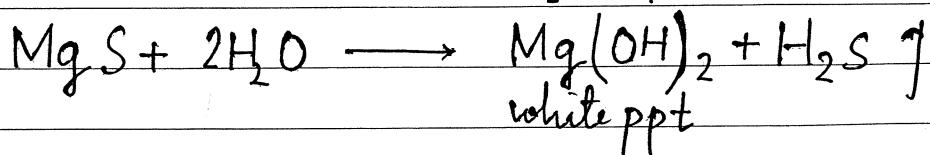
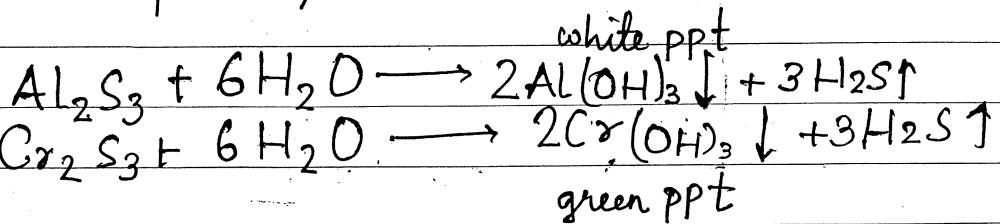


of  $\text{O}^{2-}$ ,  $\text{S}^{2-}$  same as  $\text{OH}^-$

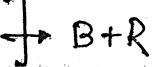
BES



- $\text{Cr}_2\text{S}_3$ ,  $\text{Al}_2\text{S}_3$ ,  $\text{MgS}$  does not exist in water (in solid state present)



solet blue 1  
purple red 1



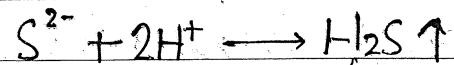
o Reagent not in excess classmate  
when not given  
No further

Date \_\_\_\_\_

Page \_\_\_\_\_

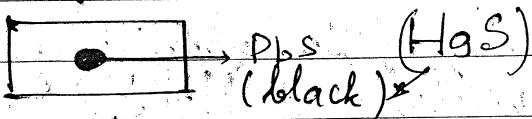
## Tests

### ① dil HCl



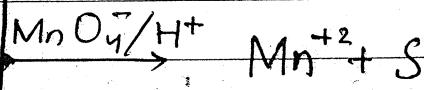
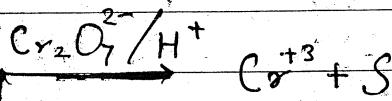
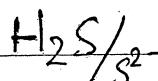
(rotten egg g)

$\left. \begin{array}{l} \text{• PbS ppt} \\ \rightarrow HNO_3 \times \\ \text{MnS not ppt} \\ \rightarrow HNO_3 \checkmark \end{array} \right\}$



### ② Test based on its reducing nature of $H_2S$

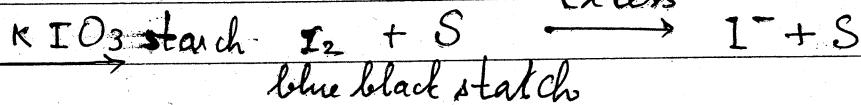
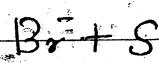
(RA)



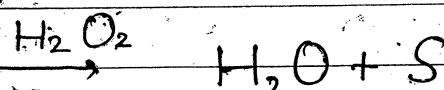
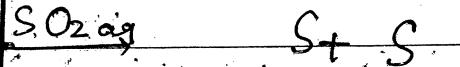
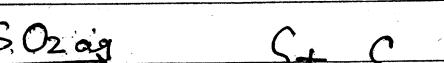
1, 2 dry  $S^2$

$Cl_2$  water

$Br_2$  water

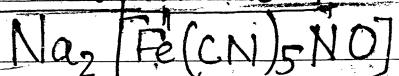
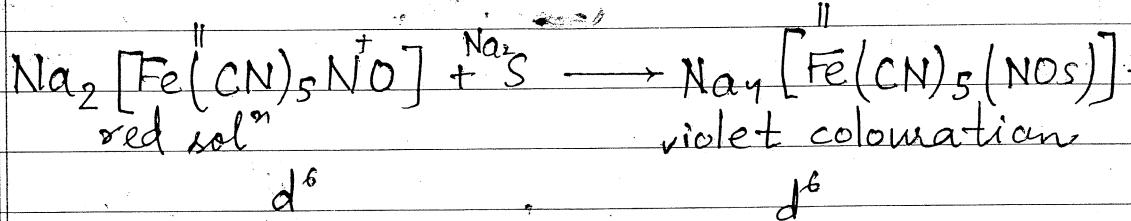
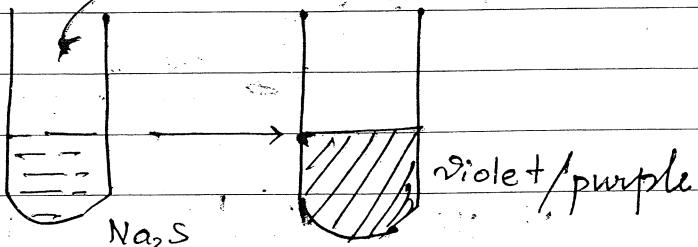


↓  
fuzziness



3

Test by sodium nitroprusside

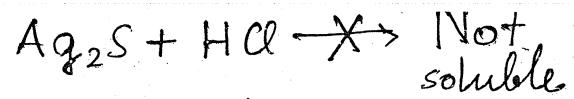
CaS  
responds

- Sod. nitroprusside does not produce violet colouration with  $\text{H}_2\text{S}$  but in alkaline medium sodium nitroprusside can produce violet colouration with  $\text{H}_2\text{S}$   
[Alkaline medium  $\text{H}_2\text{S} + 2\text{OH}^- \rightarrow \text{S}^{2-} + 2\text{H}_2\text{O}$ ]

- It is not much sensitive and requires suff.  $\text{S}^{2-}$  ion

- filter paper  $\text{H}_2\text{S}$  dissolve sufficiently but diss. less

$\rightarrow$  filter paper soaked with  $\text{H}_2\text{S}$   $\rightarrow$  No test  
Medium basic necessary



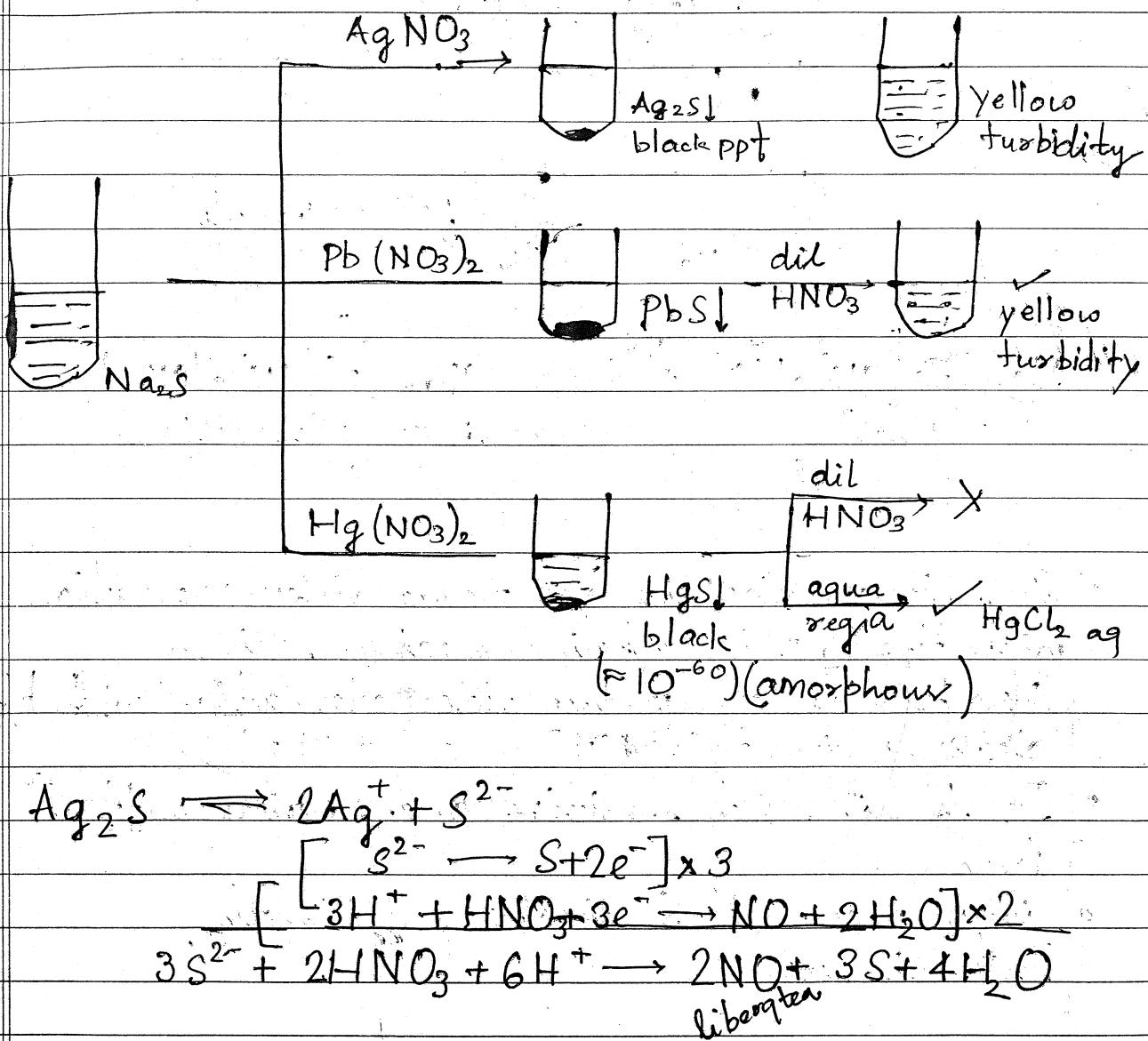
classmate

Date \_\_\_\_\_

Page \_\_\_\_\_

- To relate solubility see cation

(4) Test by  $AgNO_3$  /  $Pb(NO_3)_2$  /  $Hg(NO_3)_2$  >

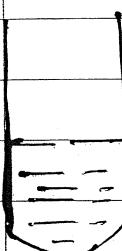


No response to  $S^{2-}$ 

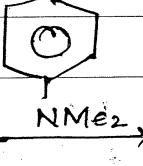
(5) Taty



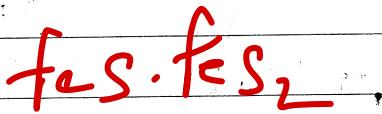
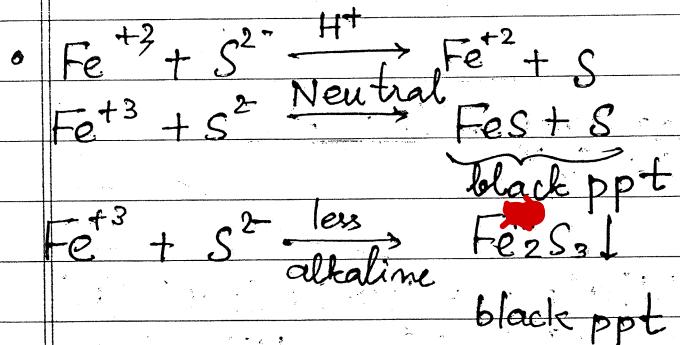
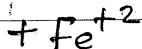
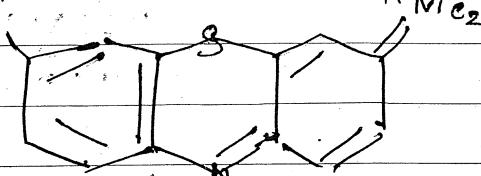
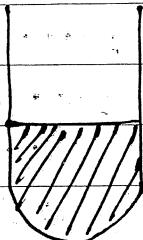
para am

 $N, N\text{-dimethyl para amine anilime}$   
 $\text{NMe}_2 + \text{Fe}_2(\text{SO}_4)_3^-$ 


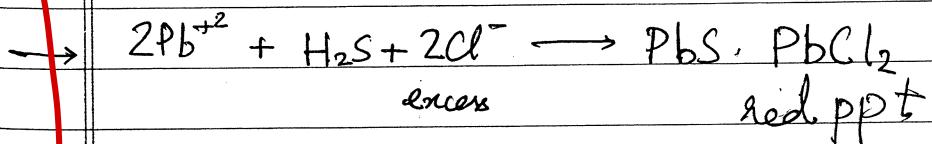
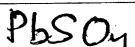
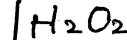
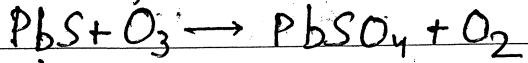
responds  
to  
[S] produced

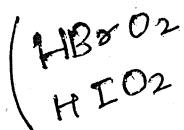


Methylene blue  
(indicator dye  
stuff)



Black ppt of  $\text{PbS}$  convert to white  $\text{PbSO}_4$ .





salt  
soluble  
acid not

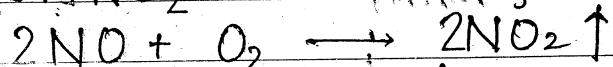
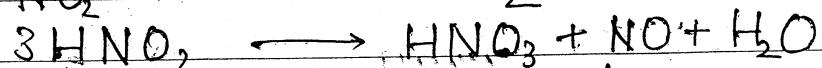
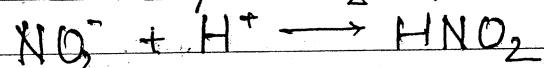
chlorite,  
hyposulphurous  
 $\text{HNO}_2$   
nitroso acid

classmate  
Date \_\_\_\_\_  
Page \_\_\_\_\_

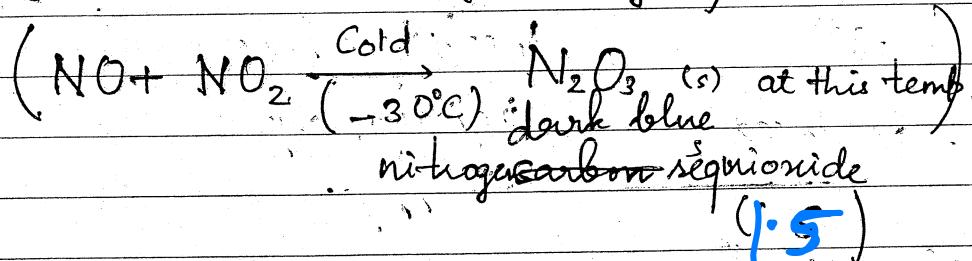
## NITRITE ION

All nitrites are water soluble except  $\text{AgNO}_2$   
(~~is~~ which is sparingly soluble.)

### ① Test by dil $\text{HCl}$ / dil $\text{H}_2\text{SO}_4$

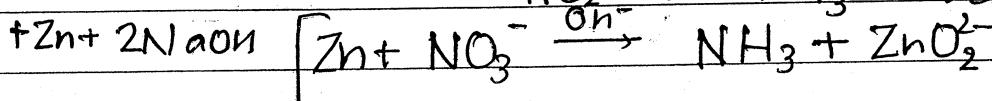
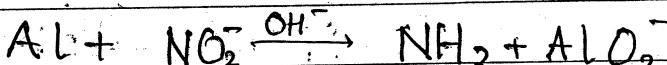
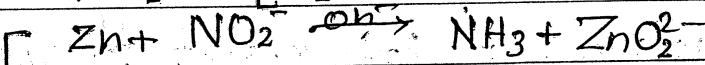
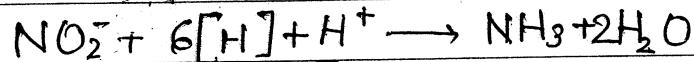
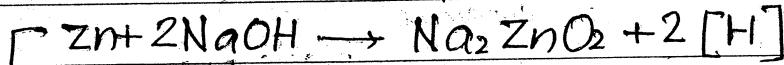
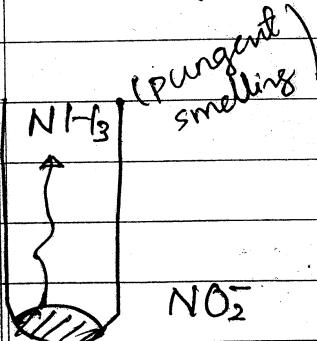


(brown gas)



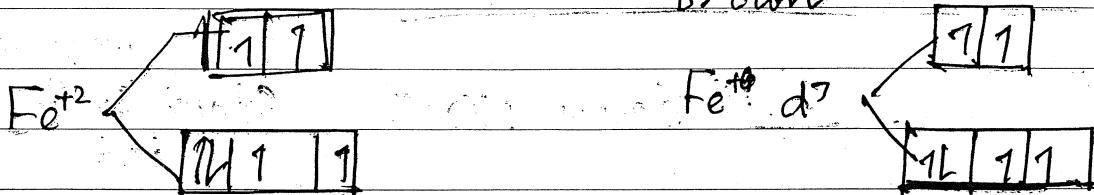
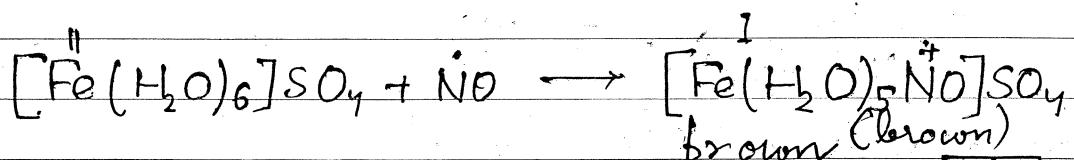
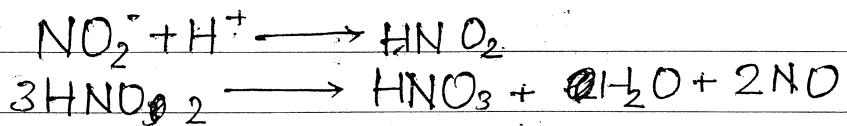
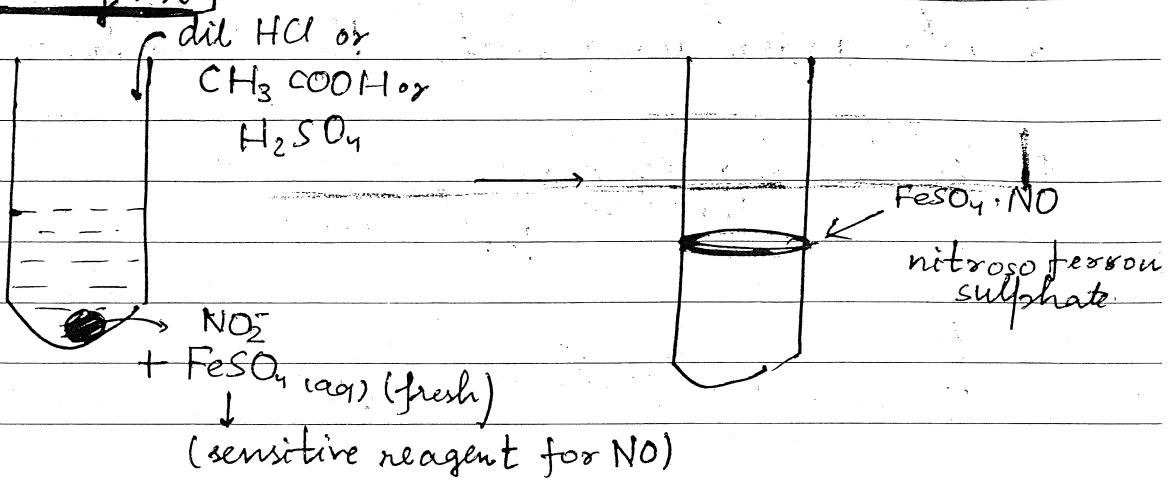
### ② Test by $\text{Zn}/\text{Al}/\text{Devarda alloy} + \text{NaOH}$

$(\text{Zn} + \text{Al} + \text{Cu})$

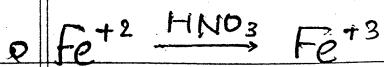


• Test also given by Nitrate in some ~~a~~ ~~1~~ conditions

### 8 Brown ring test



- brown ring test of nitrite is done by freshly prepared  $\text{FeSO}_4$  ( $\text{Fe}^{+2} \xrightarrow{\text{air}} \text{Fe}^{+3}$ ) and dil  $\text{H}_2\text{SO}_4$
- brown ring test is actually due to absorption of NO by  $\text{FeSO}_4$
- brown ring of nitrite is preferred to perform in soda extract because  $\text{M}^{n+}$  which produce insoluble sulphate create turbulence during test
- brown ring complex is unstable due to +1 oxidation state of iron
- on rapid mixing, standing or heating brown colour disappear and yellow colour of  $\text{Fe}^{+3}$  leaving behind in test tube.

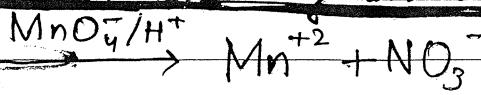


to

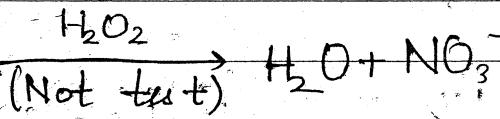
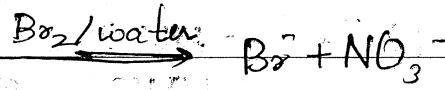
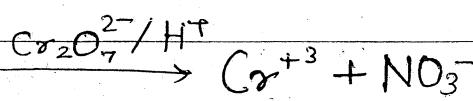
o long standing NO may be oxidised by NO<sub>3</sub><sup>-</sup>

o acid added along sides No disturbance in medium

(4) Test based on its reducing nature

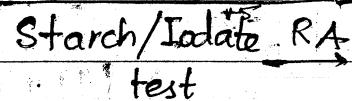


RA

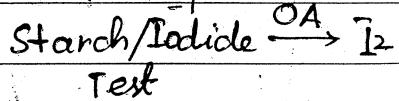
II  
NO<sub>2</sub><sup>-</sup>

(5) Test based on its oxidising nature

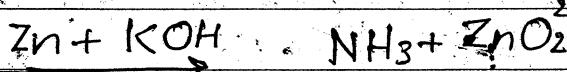
\* always remembers individual



RA

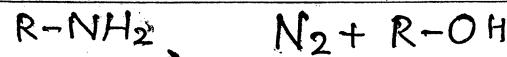
I<sub>2</sub>

Test

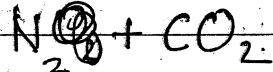
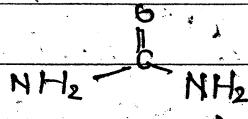
II  
NO<sub>2</sub><sup>-</sup>

Test by organic

reagent

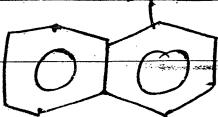


(in acid).



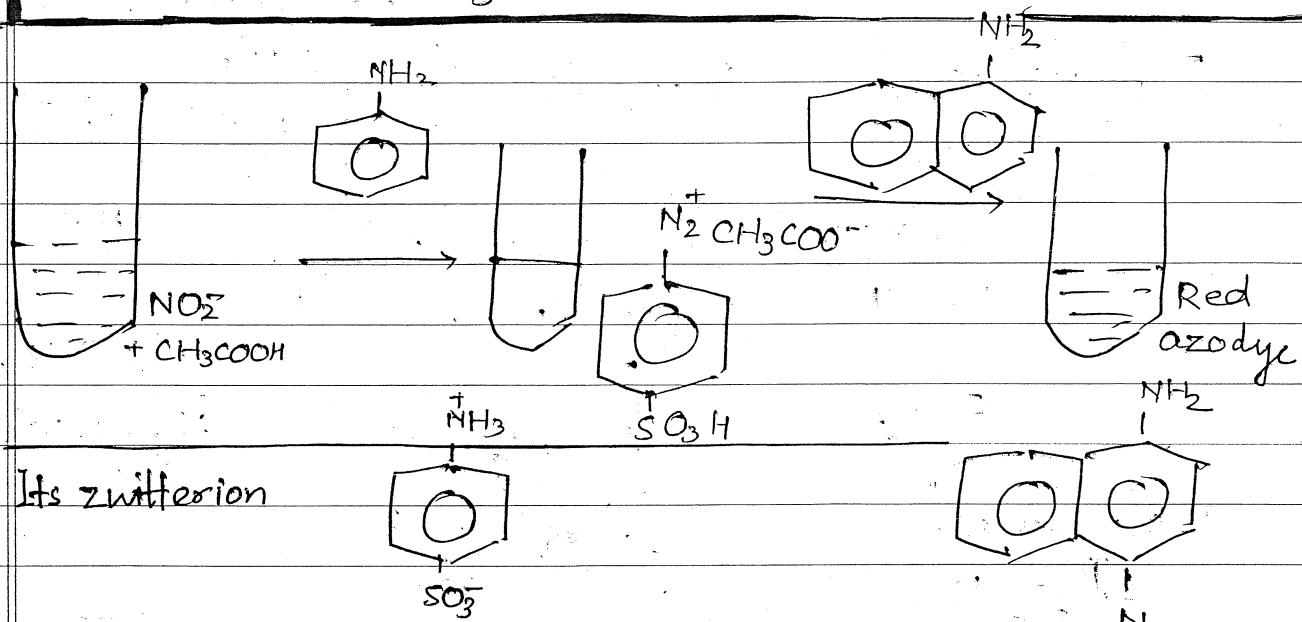
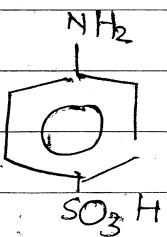
NH<sub>2</sub> | naphtylamine test

⑥ Test by sulphanilic acid +

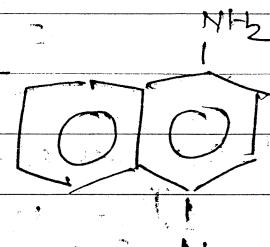
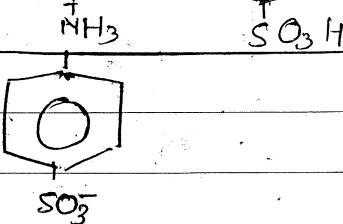


Griess

Ilosavay  
test



o Its zwitterion

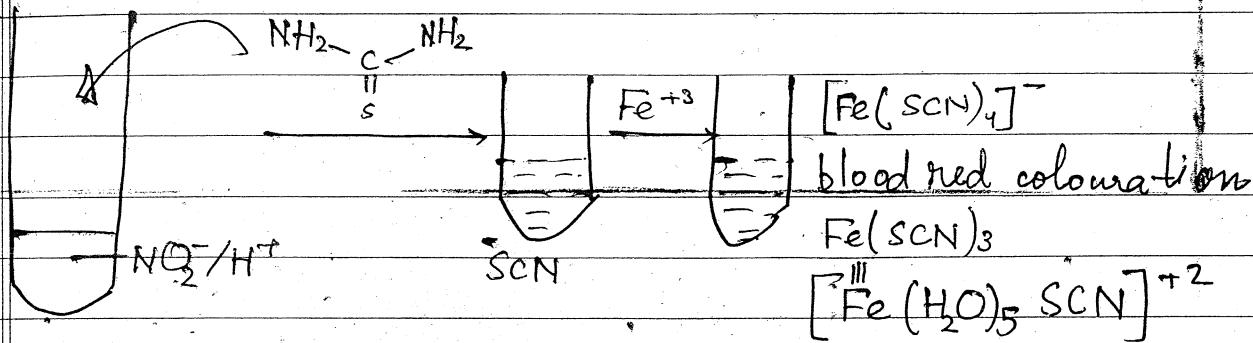


To keep it in deprotonated form (for rxn)  
 $\text{CH}_3\text{COOH}$  added

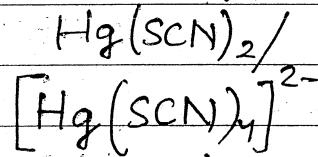
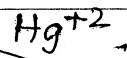
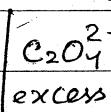


$\text{Fe}^{+3}$  configuration nearly colourless  
 [Laporte forbidden transition]  
 spin

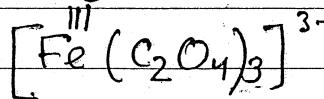
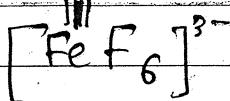
(7) Test by thiourea



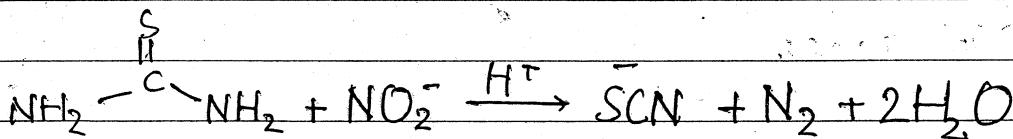
Excess  $\text{F}^-$



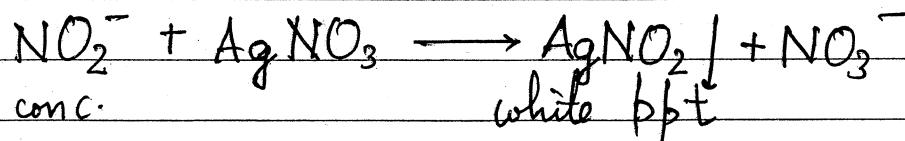
Colourless sol



green

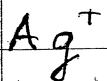
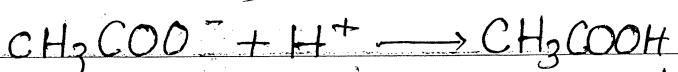


(8) Test by  $\text{AgNO}_3$

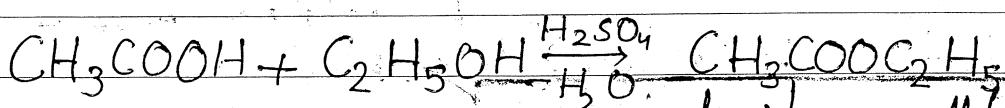


TEST OF  $\text{CH}_3\text{COO}^- \rightarrow$ 

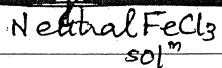
All acetates are water soluble except  $\text{Hg}^{2+}$ ,  $\text{Cu}^{2+}$ ,

(1) Test by dil  $\text{H}_2\text{SO}_4 \rightarrow$ 

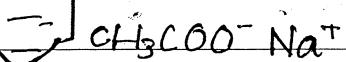
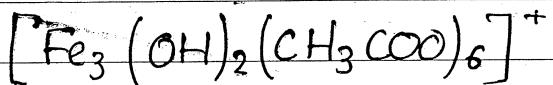
vinegar like odour

(2) Test by  $\text{C}_2\text{H}_5\text{OH} + \text{Conc. H}_2\text{SO}_4 \rightarrow$ 

fruity smell (ester)

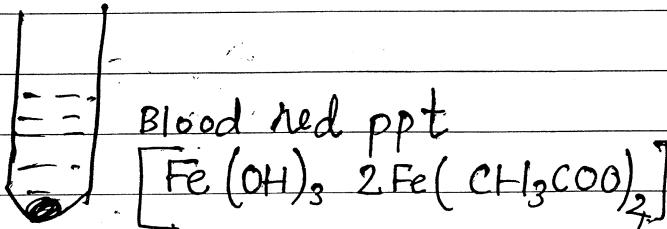
(3) Test by neutral  $\text{FeCl}_3 \text{ sol}^m$ 

Blood red colouration



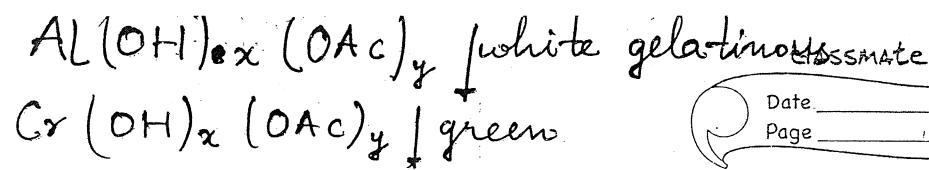
Standing

→ Neutral sol<sup>m</sup> added  
since initially basic  
 $\text{FeCl}_3$  acidic



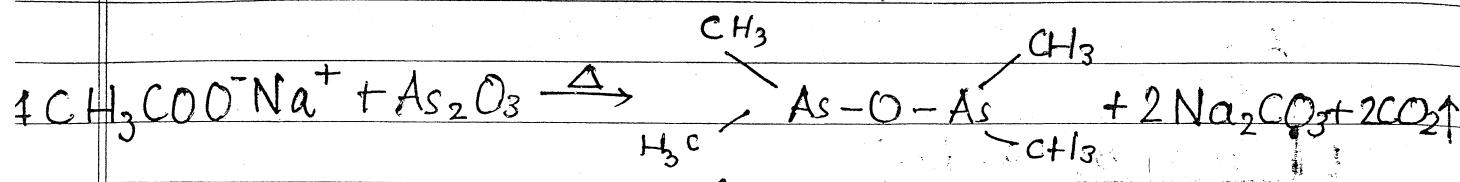
→ When acetate ions are identified by  $\text{FeCl}_3$ ,  $\text{FeCl}_3$  sol<sup>m</sup> must be neutralised by suitable base (1 to 2 drop  $\text{NH}_4\text{OH}$ ) because  $\text{FeCl}_3$  sol<sup>m</sup> is acidic which can protonate acetate ion and decrease concentration of  $\text{OH}^-$  ion

$\text{Li}(\text{em})_3$



o let

## (4) Test by $\text{As}_2\text{O}_3$ (Cacodyl test)



(Cacodyl oxide)

(organometallic)

(poisonous irritating gas)

Test of thiosulphate ( $\text{S}_2\text{O}_3^{2-}$ )  $\checkmark$  acid not present isolated

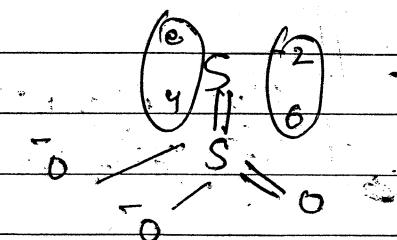
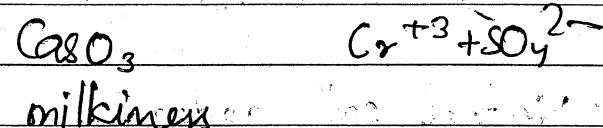
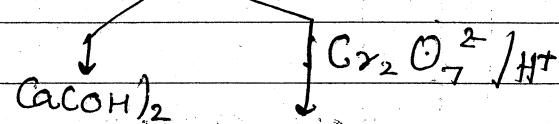
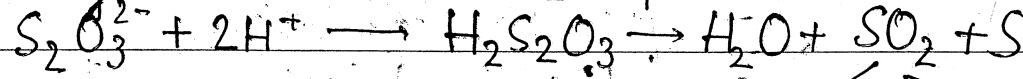
IA : Soluble

IIA : Soluble (except:  $\text{Ba}_2\text{S}_2\text{O}_3$ )

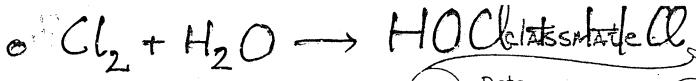
(Some other  $\text{Pb}^{+2}$ ,  $\text{Bi}^{+3}$ ,  $\text{Ag}^+$ ,  $\text{Hg}^{+2}$ ,  $\text{Cu}^+$  all are insoluble)

$\text{Fe}^{+2}$ : soluble There is only one  $\text{S}_2\text{O}_3^{2-}$  which exists in hydrate form  
( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ )

### (1) Test by dil $\text{HCl}$ or dil $\text{H}_2\text{SO}_4$



(S)



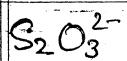
$\bullet \text{O}X^-$  stable

Date \_\_\_\_\_  
Page \_\_\_\_\_

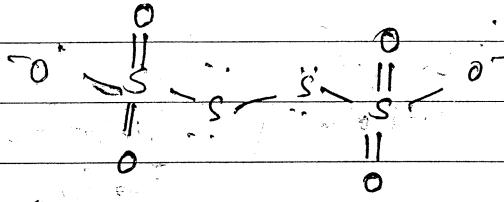
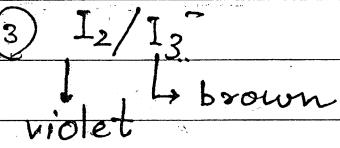
2 Test of  $\text{S}_2\text{O}_3^{2-}$  based on its reducing nature

Strong OA

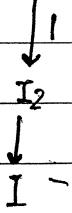
- ①  $\text{MnO}_4^- / \text{H}^+$
- ②  $\text{Cr}_2\text{O}_7^{2-} / \text{H}^+$
- ③  $\text{MnO}_4^- / \text{OH}^-$  (No S)
- ④  $\text{Cl}_2 / \text{water}$
- ⑤  $\text{Br}_2 / \text{water}$
- ⑥  $\text{H}_2\text{O}_2$

Mild oxidising agent

- ①  $\text{Fe}^{+3} / \text{H}^+$
- ②  $\text{Cu}^{+2}$



- $\text{KIO}_3$  stands no significant test



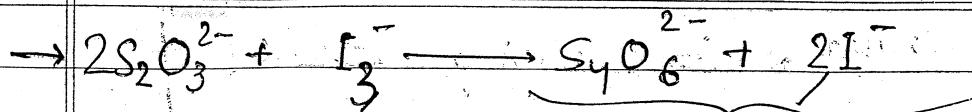
# Tincture iodine $KI_3$

$(KI + I_2)$

classmate

Date \_\_\_\_\_

Page \_\_\_\_\_



brown

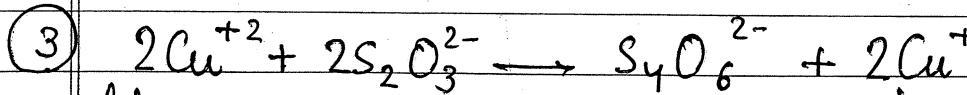
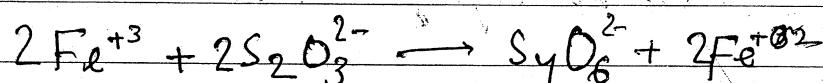
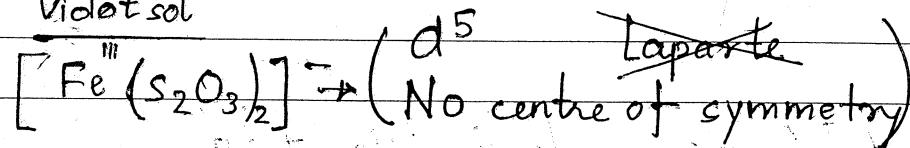
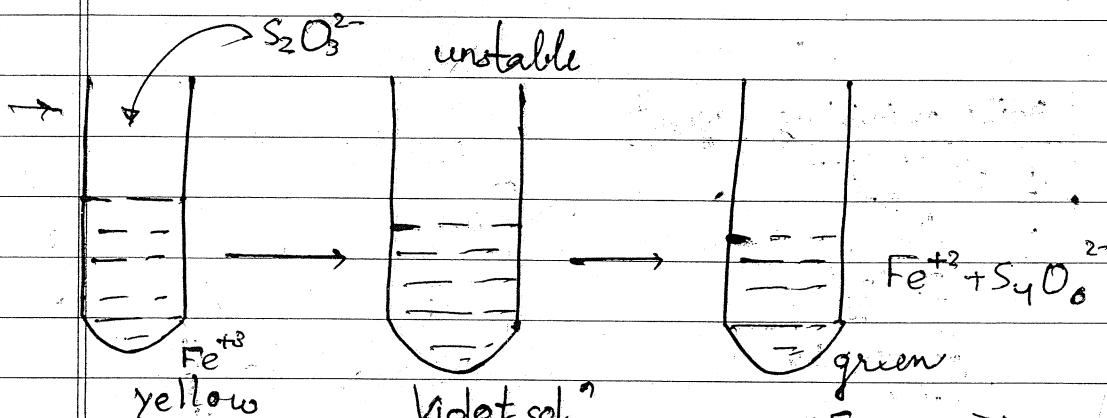
colourless

→ Used as antichlor in bleaching industry

• removes excess chlorine

→  $Na_2S_2O_3$  (hypo)

→ Removes excess  $I_2$



blue

excess

$Cu_2S_2O_3 \downarrow$

white ppt ( $Cu^+$ )

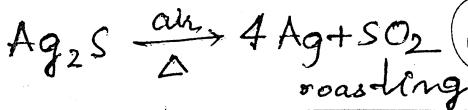
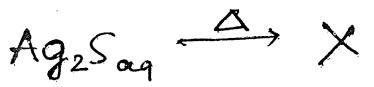
excess  $S_2O_3^{2-}$

$[Cu_6(S_2O_3)_5]^{4-}$

(bridging ligand)

complex

& structure



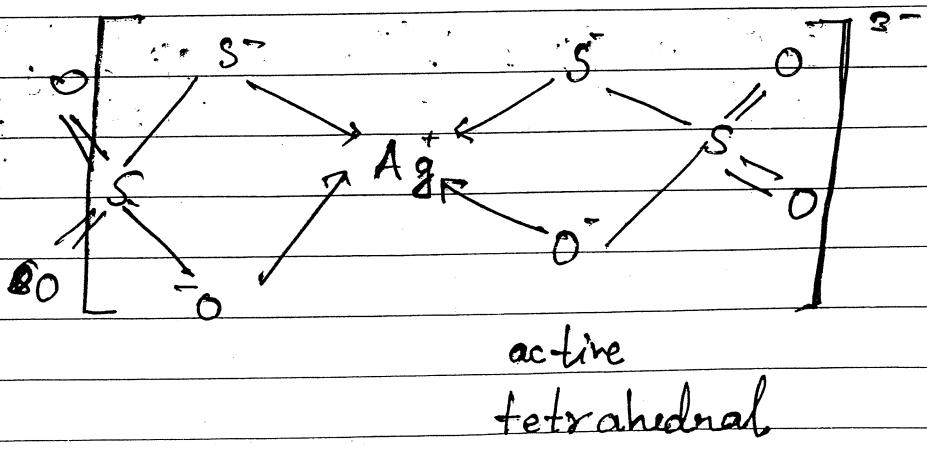
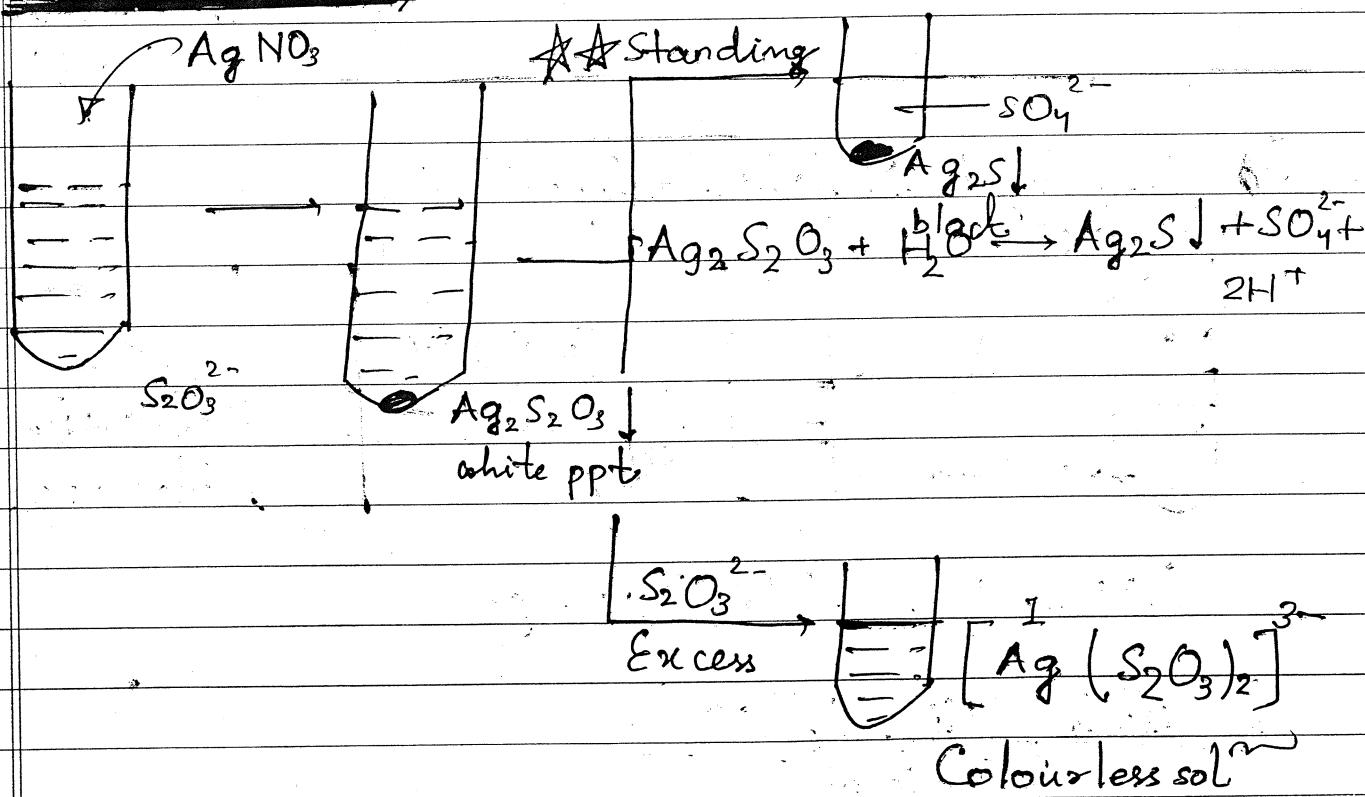
classmate

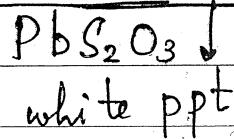
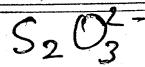
Date \_\_\_\_\_  
Page \_\_\_\_\_

### ③ Test by $CaCl_2 / BaCl_2$

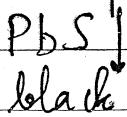
	$CaCl_2$	$BaCl_2$
$CO_3^{2-}$	$CaCO_3 \downarrow$	$BaCO_3 \downarrow$
$SO_3^{2-}$	$CaSO_3 \downarrow$	$BaSO_3 \downarrow$
$S_2O_3^{2-}$	No ppt	$BaS_2O_3 \downarrow$ white

### ④ Test by $AgNO_3$

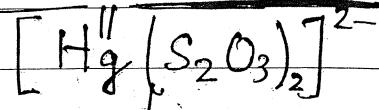
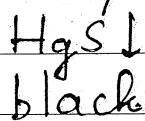
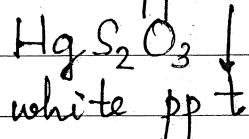
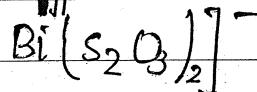
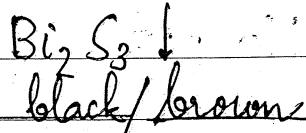
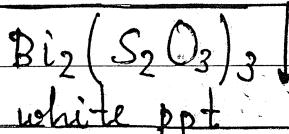
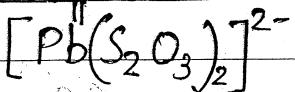




Standing of  
white ppt

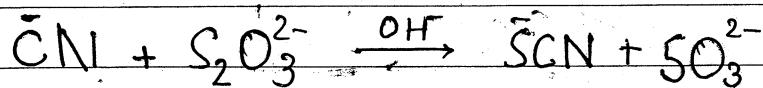
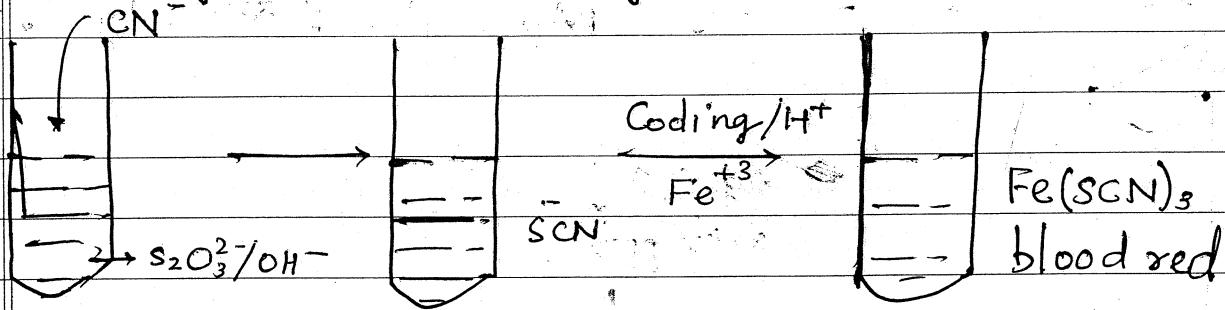


White ppt react  
with excess  $S_2O_3^{2-}$



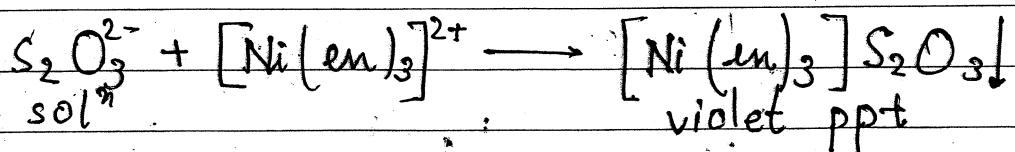
all colourless  
sol<sup>n</sup>

### 5 Test by $KCN$ followed by $Fe^{+3}$

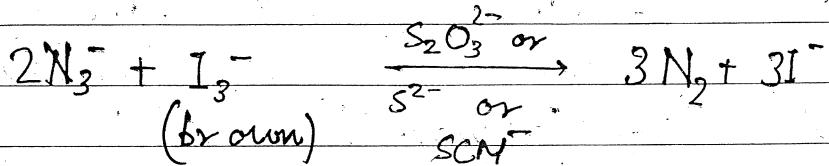
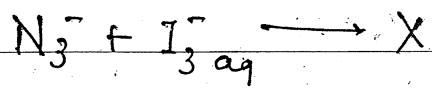


### 6 Test by $[Ni(en)_3]SO_4$

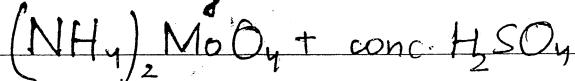
violet



7) Tetrahydrotetraiodide test



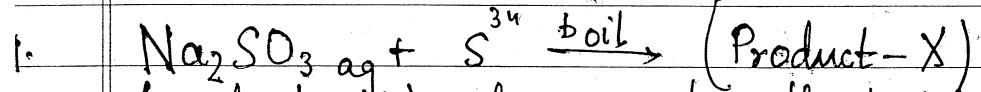
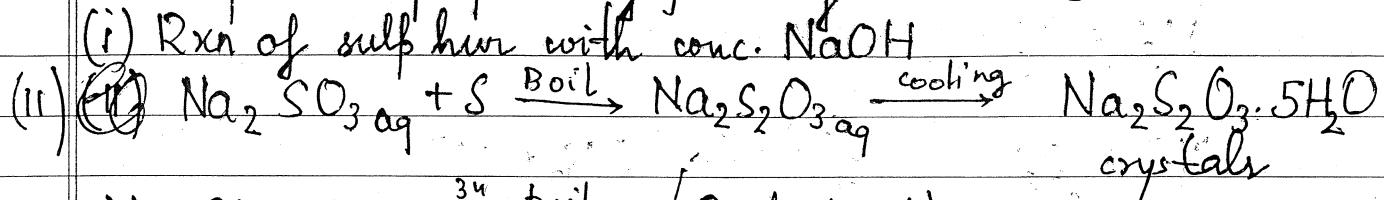
8) Blue ring test



Thiosulphate soln when treated it produce a temporary blue colour at jn pt and is called blue ring test

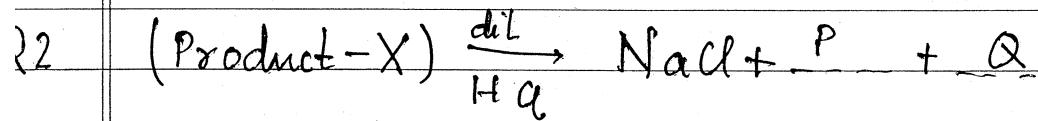
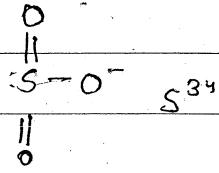
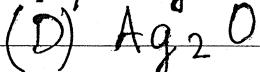
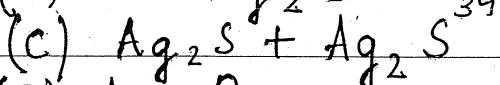
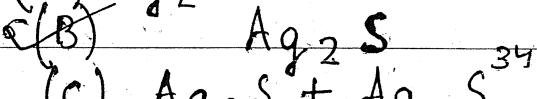
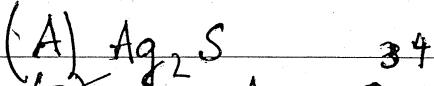
~~graph~~

dium thiosulphate is prepared by following two methods

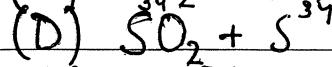
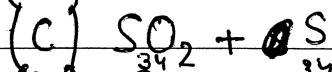
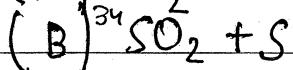
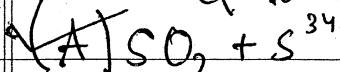


(Product-X) when react with  $\text{AgNO}_3$  gives white ppt(Y) which becomes black on standing

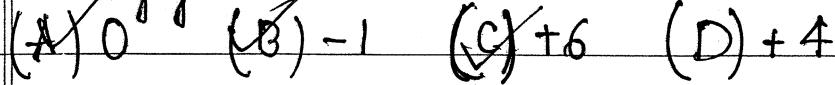
Z is

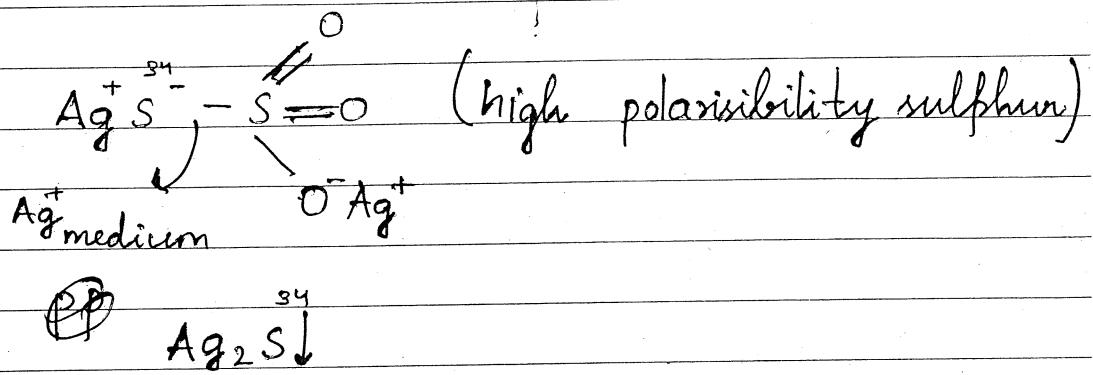
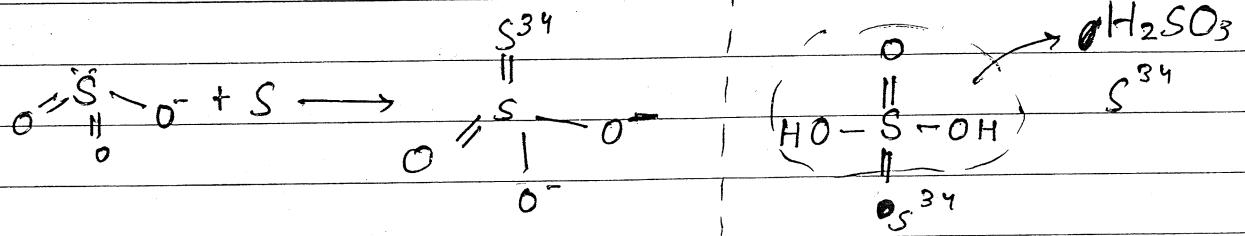


P and Q are



3. What is ON of sulphur in product  $\text{Na}_2\text{S}_2\text{O}_3$  is strongly heated





Q3

