

Mixture Separation

(Qualitative Analysis)

Aim

- To separate and identify some common cations present in an inorganic mixture of salts by performing various tests.

Theory

- Qualitative analysis is the systematic approach that involves precipitation reaction to remove cations sequentially from a mixture. The behavior of the cations toward a set of common test reagents differs from one cation to another and furnishes the basis for their separation.

Materials and Tools Required

1. Test tubes
2. Boiling tubes
3. Test tube holder
4. Test tube stand
5. Flame
6. Reagents
7. Centrifuge

Group I Cations

(Ag^+ , Hg_2^{2+} and Pb^{2+})

silver mercurous lead

Reagent : dil. HCl

Cations form insoluble chlorides with hydrochloric acid. When diluted HCl is added to the solution, white precipitates of AgCl, Hg_2Cl_2 and PbCl_2 are formed. Other metallic cations remain in solution.

Group II Cations

IIA: (Hg^{2+} , Cd^{2+} , Cu^{2+} , Bi^{3+})

mercuric

cadmium

cupric

bismuth

IIB: (As^{3+} , Sb^{3+} and As^{5+})

stannous

antimony

arsenic

Reagent : dil. $\text{HCl} + \text{H}_2\text{S}$

The pH of the solution is adjusted to 0.5 and then H_2S is added. Since the concentration of sulfide ion (S^{2-}) is very low at low pH, only Gr.II sulfides having very low K_{sp} values will precipitate. Cations with larger K_{sp} values for Gr.IV sulfides remain in solution.

Group III Cations

(Al^{3+} , Fe^{3+} , Fe^{2+} and Cr^{3+})

Aluminum ferric ferrous chromic

Reagent : $(\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}) = \text{Buffer solution}$

Since the solution is basic Al^{3+} , Fe^{3+} and Cr^{3+} form insoluble hydroxides and are also separated from the solution.

Group IV Cations

(Co^{2+} , Ni^{2+} , Zn^{2+} and Mn^{2+})

cobalt nickel zinc manganese

Reagent : $(\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}) + \text{H}_2\text{S}$

After isolating the insoluble sulfides in acidic medium, the solution is made basic and the metallic sulfides having larger K_{sp} values such as ZnS , NiS , CoS and MnS precipitate.

Group V Cations

(Ca^{2+} Sr^{2+} and Ba^{2+})

calcium strontium barium

Reagent : $(\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}) + (\text{NH}_4)_2\text{CO}_3$

These three metallic cations form soluble chlorides and sulfides and hence are separable from group 1, 2, 3 and 4 cations. However, their carbonates precipitate in a mixture of ammonium carbonate.

Group VI Cations

(NH_4^+ , Mg^{2+} , K^+ and Na^+)

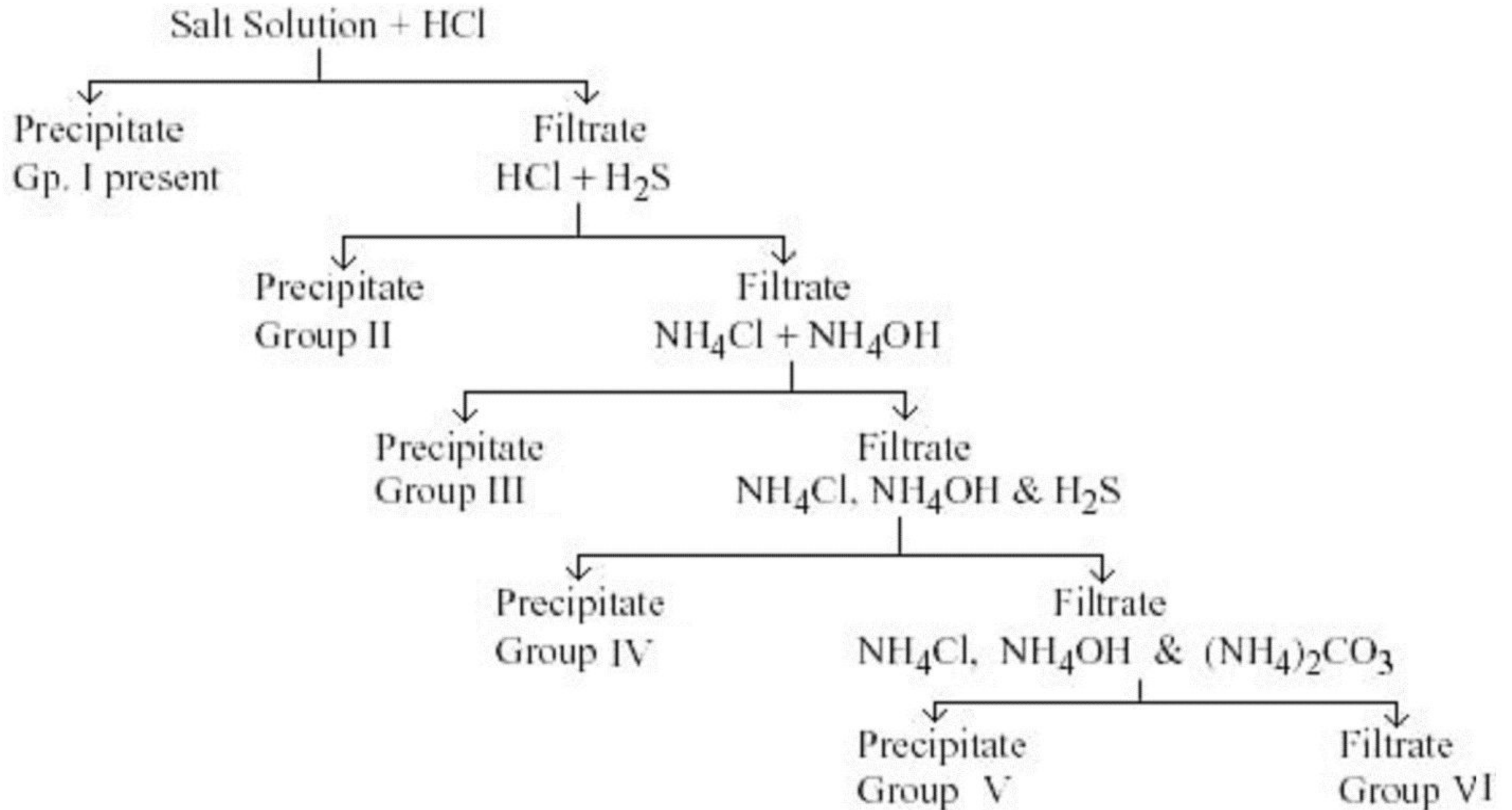
Ammonium magnesium potassium sodium

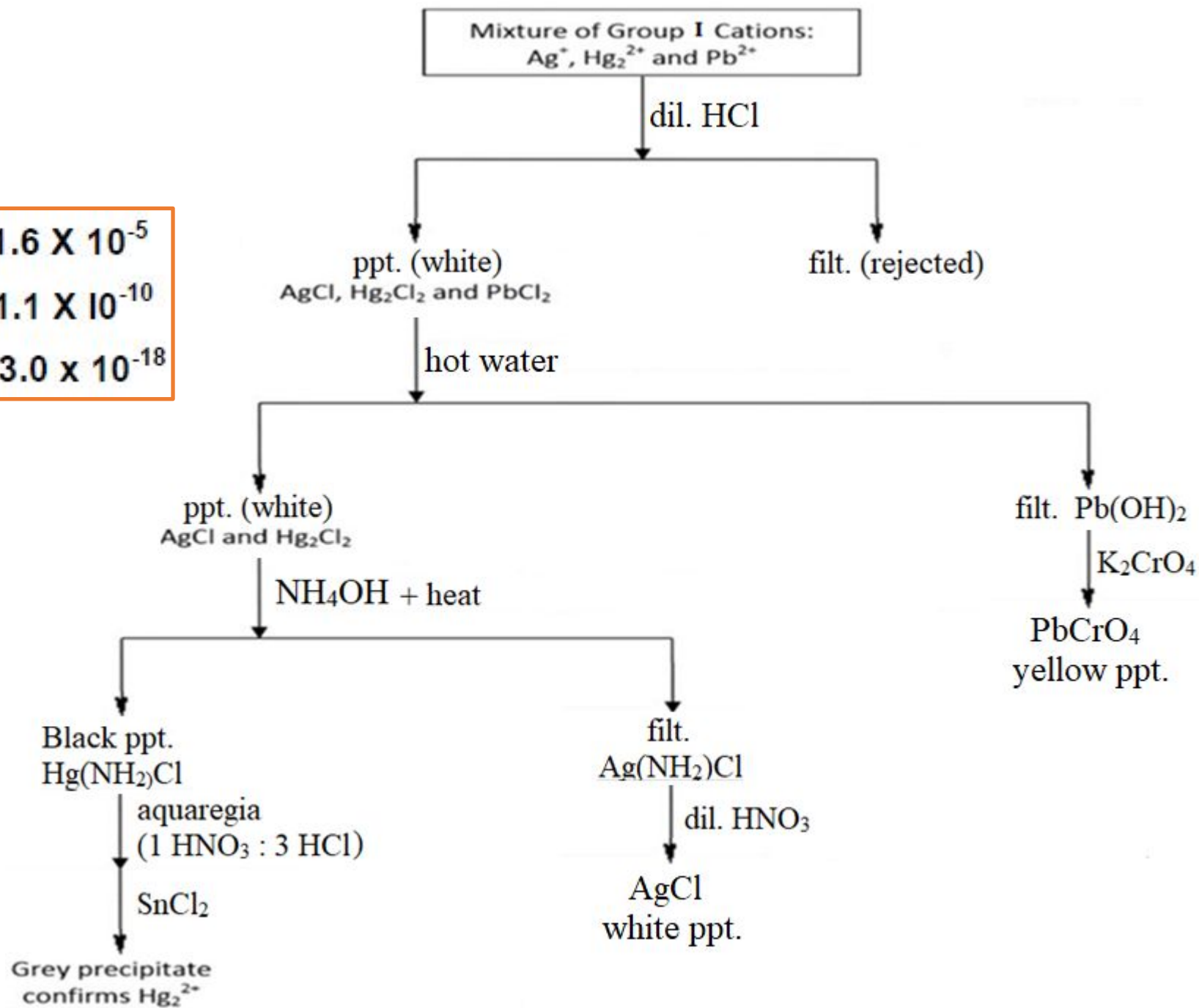
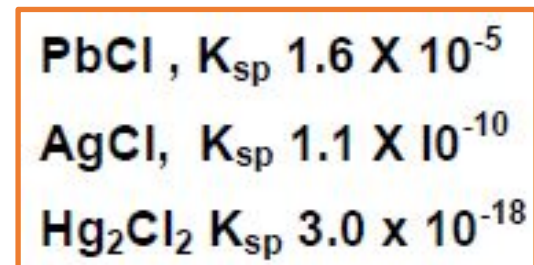
No specific reagent

None of the cations in this group form precipitates in the separation processes of group 1-5 cations and thus remain in the final solution.

Color	Inference
Blue or bluish green	Copper salts
Green	Nickel salts
Dark green	Chromium salts
Dark brown	Ferric salts
Light pink or flesh color	Manganese salts
Colorless	Absence of Cu^{2+} , Ni^{2+} , Fe^{3+} , Mn^{2+} and Co^{2+} salts

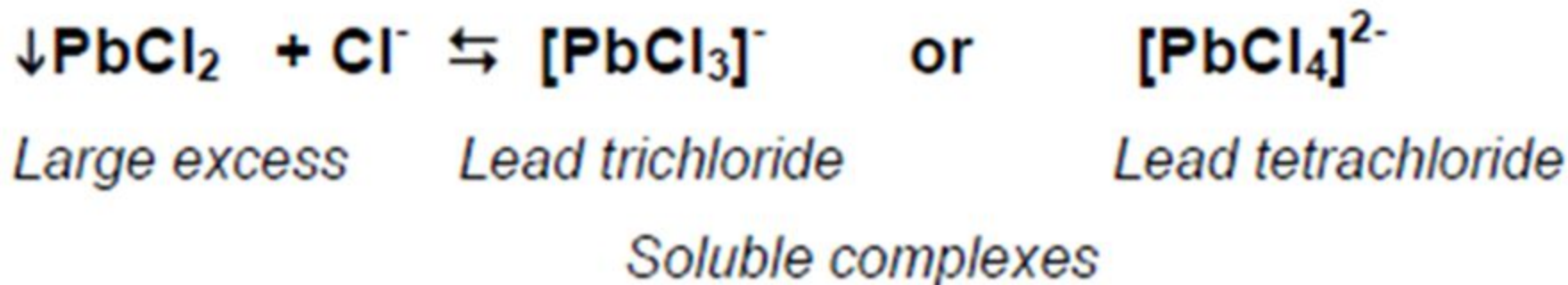
Systematic Separation of Cations in Qualitative Analysis





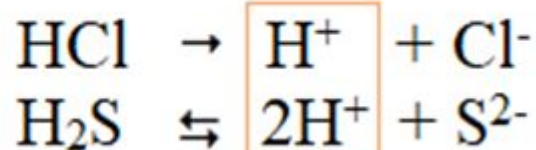
Too large excess of the acid is avoided:

Dissolves the lead chloride by complex formation.
Mercury (I) and silver chlorides do not dissolve because they are very unstable.



- Differentiate between mercuric and mercurous.?
- How can you separate and identify mixture of Hg_2^{2+} / Hg^{2+} ?

excess



Mixture of group II cations
IIA (Hg^{2+} , Cu^{2+} , Cd^{2+} , Bi^{3+})
IIB (Sn^{2+} , Sb^{3+} and As^{5+})

dil. HCl + H_2S

ppt.

Sulphides of gp. II Cations

$(\text{NH}_4)_2\text{S}$ amm. sulphide

filt. (rejected)

ppt.

Insoluble (II- A sulphides)
(HgS , Bi_2S_3 , CuS and CdS)

c. HNO_3

Black ppt.
 $\text{Hg}(\text{NH}_2)\text{Cl}$

aquaregia
(1 HNO_3 : 3 HCl)

SnCl_2

Grey precipitate
confirms Hg^{2+}

filt.

soluble nitrates of
 Bi^{3+} , Cu^{2+} , Cd^{2+}

NH_4OH (excess)

white ppt.
 $\text{Bi}(\text{OH})_3$

filt.
 $\text{Cu}(\text{NH}_3)_4$ / $\text{Cd}(\text{NH}_3)_4$
blue yellow

filt.

Soluble (II-B sulphide) complexes
 $(\text{NH}_4)_3\text{AsS}_4$, $(\text{NH}_4)_3\text{SbS}_4$, $(\text{NH}_4)_2\text{SnS}_3$ etc

c. HCl

Yellow ppt.
 (As_2S_3)

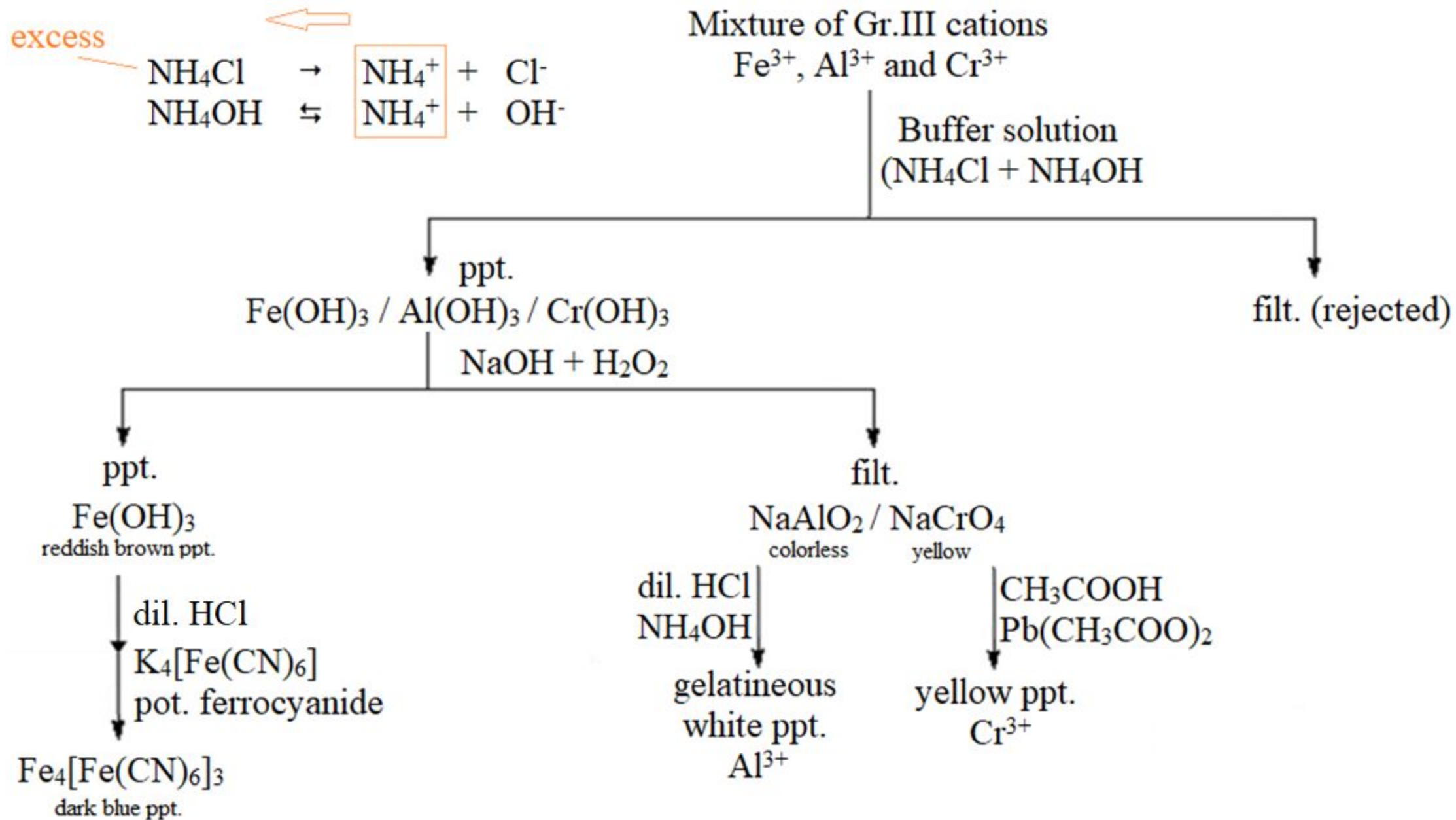
filt.

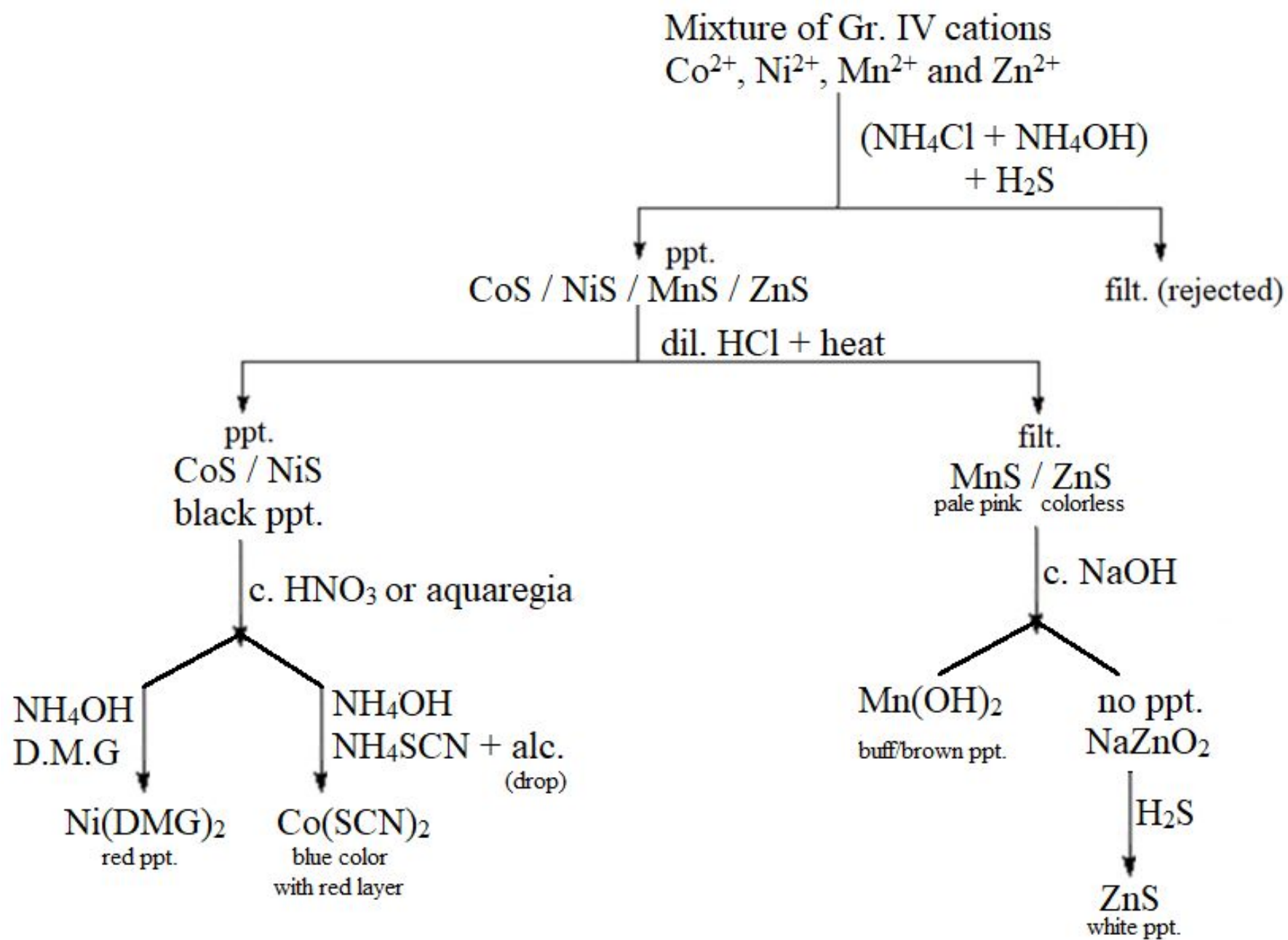
SnCl_2 / SbCl_3

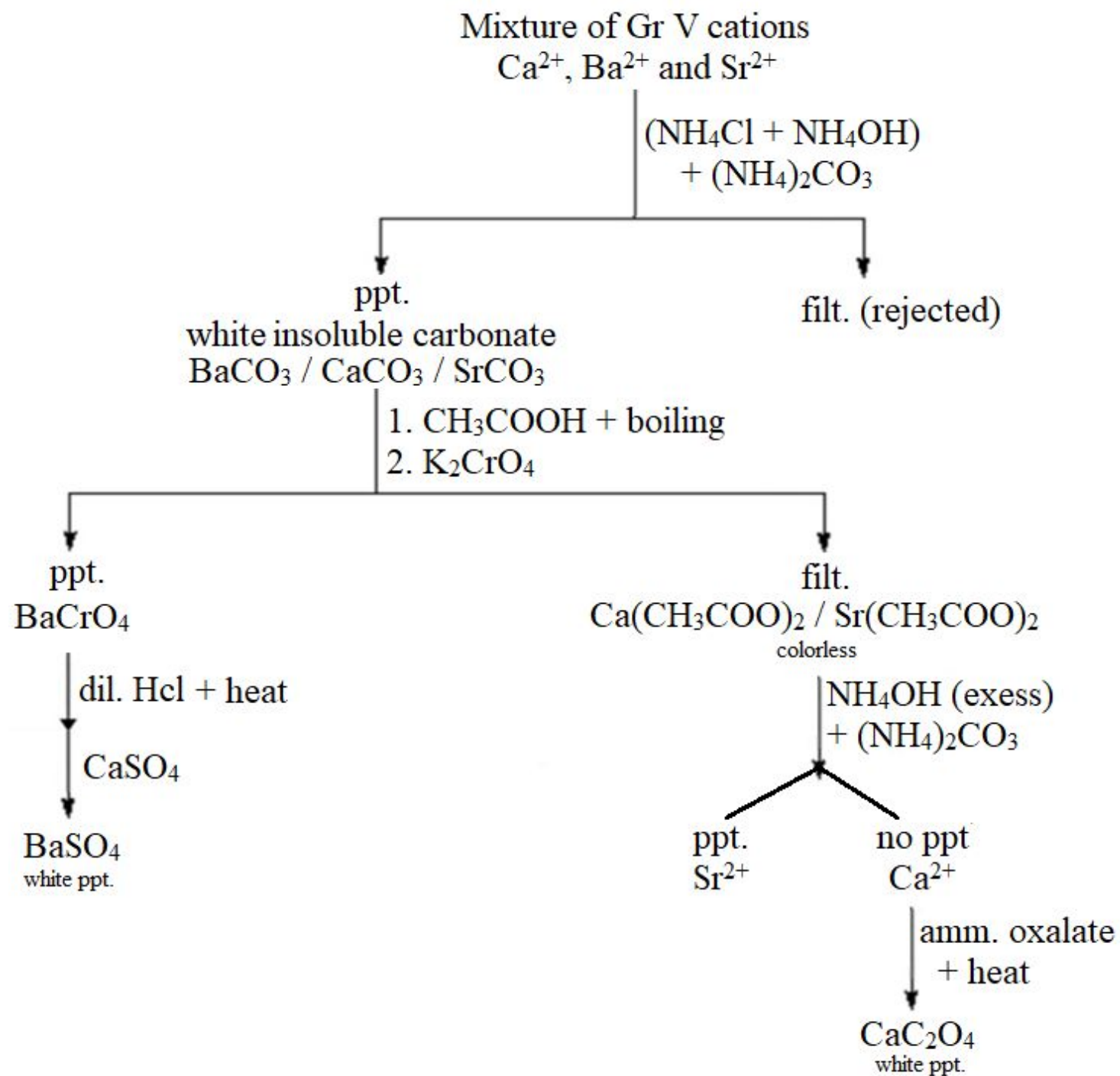
NH_4OH
oxalic acid

orange ppt.
 Sb^{3+}

no ppt.
 Sn^{2+}

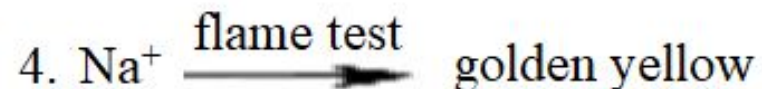
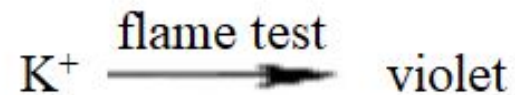
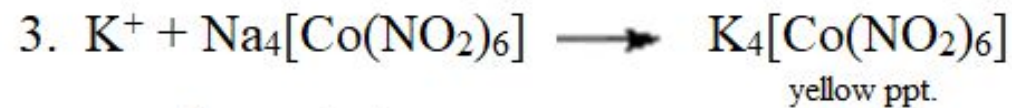
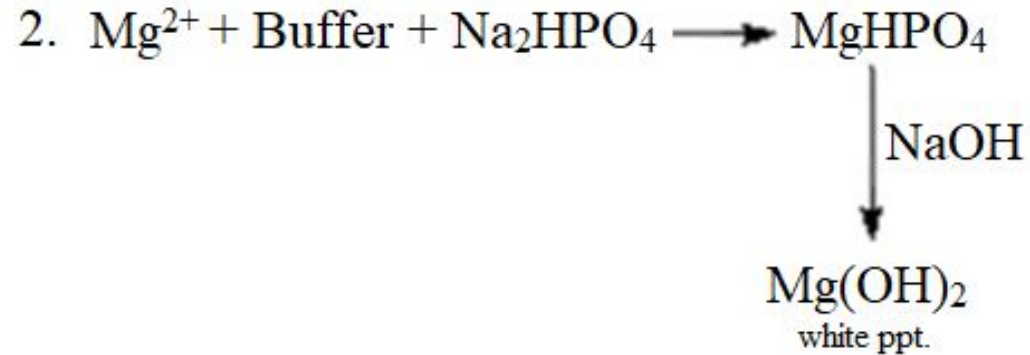
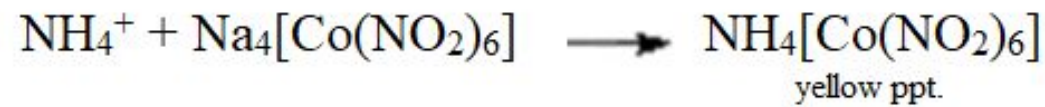






Gr. VI cations

1. $\text{NH}_4^+ + \text{c. NaOH} + \text{heat} \longrightarrow \text{NH}_3$ gas forms white clouds
when exposed to dil. HCl



Scheme for separation of cations

HCl or a soluble chloride, preferably NH_4Cl , added to unknown; filtered

precipitate:
contains
chlorides
of lead
(Pb), silver
(Ag), and
mercurous
mercury
(Hg)

PbCl_2
(white)
 AgCl
(white)
 Hg_2Cl_2
(white)

Group I

solution: H_2S passed into the acid solution; filtered

precipitate: treated with
ammonium sulfide
(NH_4)₂S; filtered

precipitate:
contains
cupric (black)
cadmium, (yellow)
bismuth (brown)
and
mercuric (black)
sulfides

Group IIa

solution:
contains
arsenic,
antimony,
and tin
cations

Group IIb

solution: neutralized with NH_4OH and NH_4Cl ; filtered

precipitate:
contains
aluminum
(Al), chro-
mium (Cr),
and
ferric (Fe)
hydroxides

$\text{Al}(\text{OH})_3$
(white)
 $\text{Cr}(\text{OH})_3$
(gray-green)
 $\text{Fe}(\text{OH})_3$
(brown)

Group III

solution: H_2S passed into alkaline solution;
filtered

precipitate:
contains
cobalt (Co),
nickel (Ni),
manganese
(Mn), and
zinc (Zn)
sulfides

CoS (black)
 NiS (black)
 MnS (buff)
 ZnS (white)

Group IV

solution: NH_4OH and NH_4Cl
and (NH_4)₂ CO_3
added; filtered

precipitate:
barium,
strontium,
and
calcium
carbonates
(all white)

Group V

solution:
contains
magnesium,
sodium,
and potas-
sium ions

Group VI

Report

- Physical Properties

Colour	
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- Chemical Properties

Experiment	Observation	Result

Experiment	Observation	Result

The cations are &