Separation and Identification Of Group 1 Cations

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

The purpose of this experiment was to separate and identify group one cations through qualitative analysis.

Introduction

Qualitative analysis is a branch of analytical chemistry that identifies particular substances in a given sample of material. In the analysis of inorganic substances, this branch involves the analysis of both metallic constituents as cations and nonmetallic constituents as anions.

Precipitation is the formation of a solid in a solution during the chemical reaction . when the chemical reaction occurs the solid formed is called the precipitate.

Precipitate is an insoluble solid that separate from the solution .

Separations these are procedure that separate groups of ions from other groups or individual ions in a mixture of ions.

A centrifuge is an instrumental used to separate a heterogeneous mixture of solid and liquid by spinning it , after a successful centrifuge , the solid precipitate settle to the bottom of the test tube and the solution called the filter is clear .

Chemistry of the Precipitation of the Group I Cations

The precipitating reagent of Group I is a dilute solution of hydrochloric acid, and the ions precipitated are those of silver, Ag^+, mercury (I) , Hg_2^{2+}, and lead (II), Pb^{2+}. The net ionic equations are:

$Ag^+ + Cl^- \rightleftharpoons AgCl(s)$ (AgCl is white, turning purple in light)

$Hg_2^{2+} + 2Cl^- \rightleftharpoons Hg_2Cl_2(s)$ (Hg_2Cl_2 is white)

$Pb^{2+} + 2Cl^- \rightleftharpoons PbCl_2(s)$ ($PbCl_2$ is white)

A slight excess of chloride ion is used to reduce the solubility of the precipitates in accordance with the common ion effect. Even so the solubility of lead chloride is sufficiently high for an appreciable concentration of lead ion to remain in solution.

A large excess of chloride ion should be avoided in order to prevent the formation of soluble chloro complex ions which will dissolve the precipitates:

$AgCl + Cl^- \rightleftharpoons AgCl_2^-$

$PbCl_2 + Cl^- \rightleftharpoons PbCl_3^-$

$Hg_2Cl_2 + 2Cl^- \rightleftharpoons HgCl_4^{2-} + Hg$

Chemistry of the Separation and Identification of the Group I Cations

The lead chloride may be extracted from the other two chlorides which may also be in the precipitate with hot water since its solubility increases with an increase in temperature much more markedly than do the solubilities of silver chloride and mercury (I) chloride.

$$PbCl_2 + H_2O \rightleftharpoons Pb^{2+}(aq) + 2Cl^-$$

The presence of lead ion is confirmed by adding an aqueous solution of potassium chromate, K_2CrO_4, which provides chromate ion to form lead chromate, $PbCrO_4$, a birght yellow solid which is less soluble than lead chloride:

$$Pb^{2+} + CrO_4^{2-} \rightleftharpoons PbCrO_4$$

The silver chloride and mercury (I) chloride which may be in the precipitate may be separated from one another by taking advantage of the fact that only silver ion forms a soluble complex ion with ammonia. By adding ammonia water to the residue, the silver chloride is selectively dissolved due to the formation of the soluble diammine silver (I) ion.

$$\mathbf{AgCl + 2\ NH_4OH \longrightarrow [Ag(NH_4)_2]^- + Cl^- + H_2O}$$

The presence of silver ion is confirmed by acidification of the solution containing the $Ag(NH_3)_2^+$ ion with nitric acid. The nitric acid converts the ammonia of the silver complex ion equilibrium to ammonium ion:

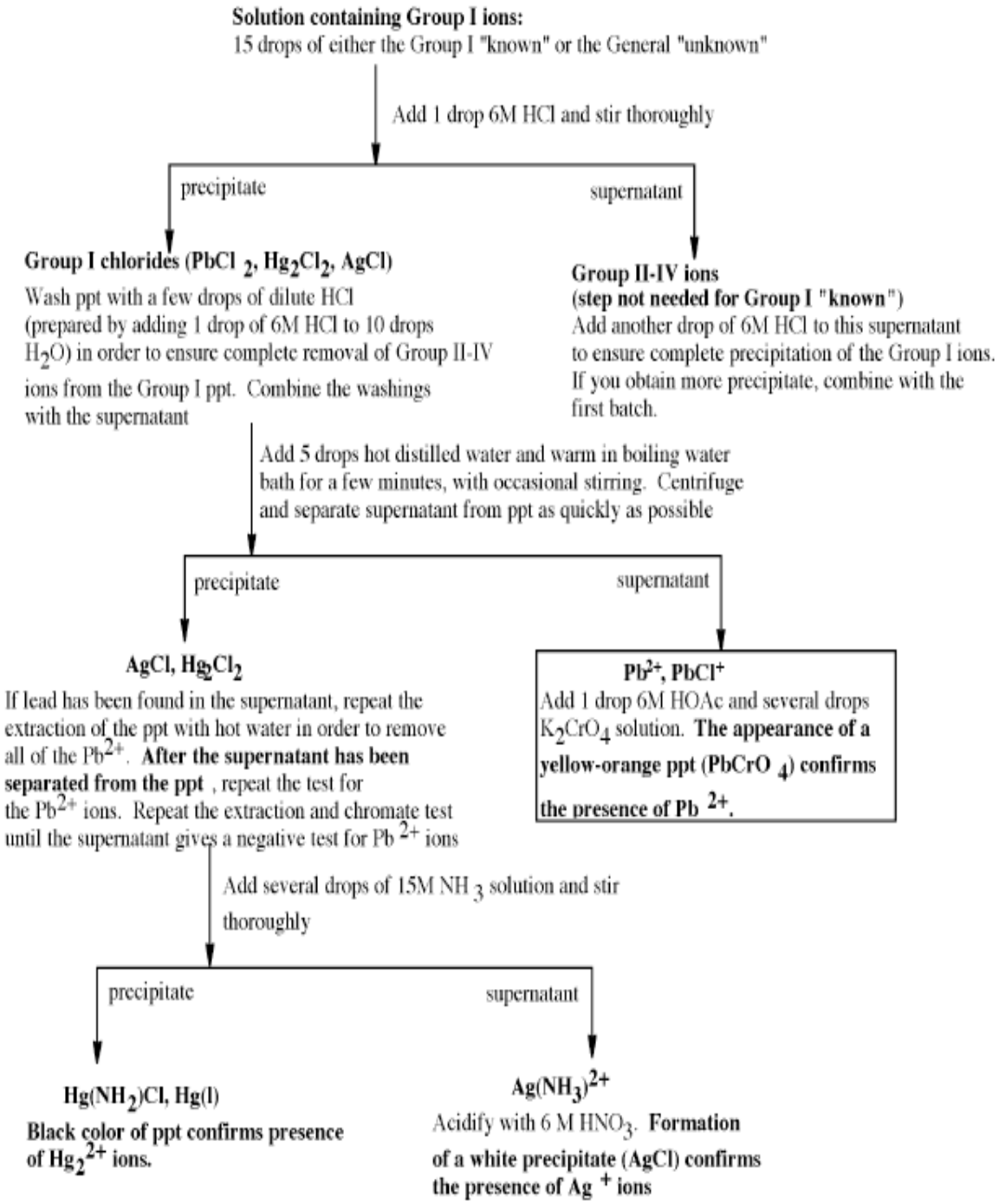
$$\mathbf{[Ag(NH_4)_2]^- + Cl^- + H_2O \xrightarrow{(2H^+)} AgCl_{(s)}(white) + 2NH_4OH}$$

The addition of ammonia converts the white mercury (I) chloride in the precipitate to a mixture of white mercury (II) amidochloride and elemental mercury, black in the finely divided condition in which it is formed:

$$Hg_2Cl_{2(s)} + 2NH_4OH \longrightarrow Hg(black) + [HgNH_4]Cl(white) + NH_4Cl + H_2O$$

This type of reaction seen with mercury is called a disproportionation reaction where the $Hg_2Cl_2(s)$ oxidizes and reduces itself to Hg(black) and $[HgNH_4]Cl$(white) ,so the mix of the two gives a gray precipitate .

The formation of the insoluble black mixture of the two principal products serves as confirming evidence for the original presence of mercury (I).


Solution containing Group I ions:
15 drops of either the Group I "known" or the General "unknown"
Add 1 drop 6M HCl and stir thoroughly
precipitate
supernatant
Group I chlorides (PbCl 2, Hg2Cl2, AgCl)
Wash ppt with a few drops of dilute HCl (prepared by adding 1 drop of 6M HCl to 10 drops H2O) in order to ensure complete removal of Group II-IV ions from the Group I ppt. Combine the washings with the supernatant
Group II-IV ions
(step not needed for Group I "known")
Add another drop of 6M HCl to this supernatant to ensure complete precipitation of the Group I ions. If you obtain more precipitate, combine with the first batch.
Add 5 drops hot distilled water and warm in boiling water bath for a few minutes, with occasional stirring. Centrifuge and separate supernatant from ppt as quickly as possible
precipitate
supernatant
AgCl, Hg2Cl2
If lead has been found in the supernatant, repeat the extraction of the ppt with hot water in order to remove all of the Pb2+. After the supernatant has been separated from the ppt , repeat the test for the Pb2+ ions. Repeat the extraction and chromate test until the supernatant gives a negative test for Pb 2+ ions
Pb2+, PbCl+
Add 1 drop 6M HOAc and several drops K2CrO4 solution. The appearance of a yellow-orange ppt (PbCrO 4) confirms the presence of Pb 2+.
Add several drops of 15M NH 3 solution and stir thoroughly
precipitate
supernatant
Hg(NH2)Cl, Hg(l)
Black color of ppt confirms presence of Hg2 2+ ions.
Ag(NH3)2+
Acidify with 6 M HNO3. Formation of a white precipitate (AgCl) confirms the presence of Ag + ions