

Qualitative Analysis of Group 1 Cations

Objectives

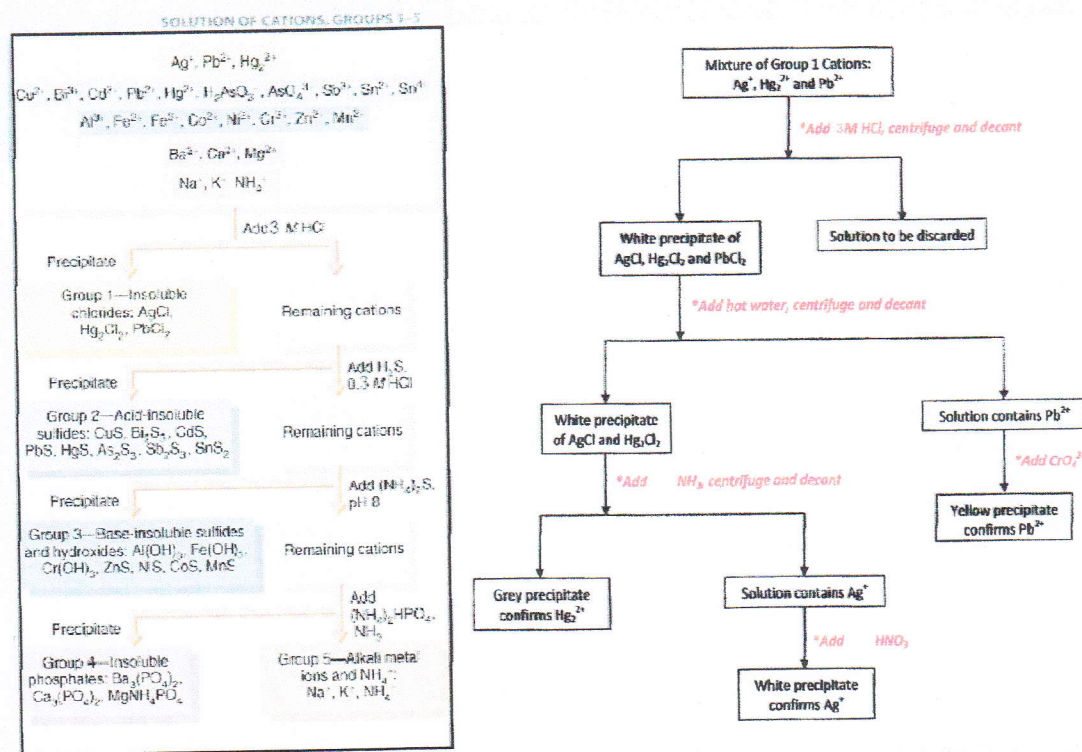
The objectives of this laboratory are to follow a classic analytical scheme to separate and identify the ions in a *known* mixture of Group 1 cations, and then to then apply this scheme to identify the ions in an *unknown* mixture of Group 1 cations.

Background

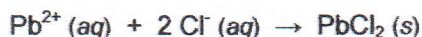
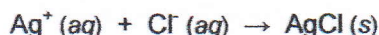
One common task in analytical chemistry is the identification of the various ions present in a particular sample. For example, if you are an environmental chemist your job may be to recover soil or water samples in order to determine the presence of toxic ions such as Pb^{2+} or Hg_2^{2+} . A common experimental method used to identify ions in a mixture is called **qualitative analysis**.

In qualitative analysis, the ions in a mixture are separated by **selective precipitation**. Selective precipitation involves the addition of a carefully selected reagent to an aqueous mixture of ions, resulting in the precipitation of one or more of the ions, while leaving the rest in solution. Once each ion is isolated, its identity can be confirmed by using a chemical reaction specific to that ion.

Cations are typically divided into **Groups**, where each group shares a common reagent that can be used for selective precipitation. The classic qualitative analysis scheme used to separate various groups of cations is shown in the flow chart below.



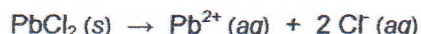
Note that Ag^+ , Pb^{2+} , and Hg_2^{2+} are called the **Group 1 cations** since they are the first group separated from the larger mixture. Since these ions all form insoluble chlorides, their separation from all other ions may be accomplished by the addition of 3M HCl (aq) resulting in the precipitation of AgCl (s), PbCl₂ (s), and Hg₂Cl₂ (s):



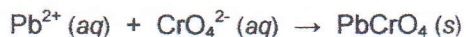
The sample must then be centrifuged (spun rapidly), which separates the solid precipitates from the ions still in solution. The solids settle to the bottom, and the solution containing the remaining ions (Groups 2 – 5) remains on top of the solid. This solution is called the **supernatant solution**, and must be carefully decanted (poured off without disturbing the solid), and saved for further study. The Group 1 cations contained within the collected precipitate must then be separated from each other in order for the presence of each ion to be confirmed.

Separation and Confirmation of Group 1 Cations

Lead(II) chloride can be separated from the other two chlorides based on its increased solubility at higher temperatures. This means that lead(II) chloride will dissolve in hot water, leaving the mercury(I) chloride and the silver chloride in solid form:



The presence of Pb^{2+} in the aqueous solution can then be confirmed by the formation of a yellow precipitate of PbCrO₄ upon the addition of aqueous K₂CrO₄:



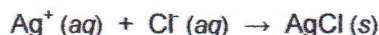
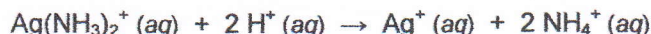
Next, Hg_2^{2+} and Ag^+ cations can be separated by adding 3 M NH₃ (aq) to the solid mixture of the two chlorides. Silver chloride will dissolve since it forms a soluble complex ion with ammonia:



However, mercury(I) chloride reacts with the ammonia yielding what appears to be a gray solid which is actually a mixture of black Hg (l) and white HgNH₂Cl (s). The presence of this gray solid is confirmation of the presence of Hg₂²⁺.



The presence of Ag^+ can be confirmed by the appearance of a white precipitate upon adding 6 M HNO₃ (aq) to the solution. The nitric acid reacts with the ammonia and thus destroys the complex ion containing the silver cation. Once in solution again the silver cation precipitates with the chloride as indicated by the following reactions:





(a) Precipitation of group 1 cations
by adding HCl(aq):
 $\text{AgCl}(s) + \text{Hg}_2\text{Cl}_2(s) + \text{PbCl}_2(s)$



(b) Confirmation test for Lead by
addition of $\text{Na}_2\text{CrO}_4(\text{aq})$: $\text{PbCrO}_4(s)$



(c) Confirmation test for mercury
by adding $\text{NH}_3(\text{aq})$:
 $\text{Hg}(l) + \text{Hg}(\text{NH}_2)\text{Cl}(s)$



(d) Confirmation test for Silver by
adding HCl(aq): $\text{AgCl}(s)$

كما وأشرنا أعلاه أنه سيتم فصل أيونات المجموعة الأولى فقط (Pb^{+2} , Hg^{+2} , Ag^{+}) من المحلول وذلك بترسيبها على هيئة كلوريدات ($AgCl$, Hg_2Cl_2 , $PbCl_2$) ويتم الكشف عن كل أيون حسب المخطط التالي :

