Inorganic salts

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Introduction

The aim of Exercise 9 is to analyze three inorganic salts (one of them poorly soluble) - and detect both cation and anion, which are two components of each salt, belonging to the groups mentioned below:

- Group I cations: Ag^{+}, Hg_2^{2+}, Pb^{2+}
- Group IIA cations: Pb^{2+}, Hg^{2+}, Cu^{2+}, Cd^{2+}, Bi^{3+}
- Group III cations: Fe^{3+}, Mn^{2+}, Cr^{3+}, Co^{2+}, Ni^{2+}, Zn^{2+}, Al^{3+}
- Groups IV and V cations: Ca^{2+}, Ba^{2+},Sr^{2+}, Mg^{2+}, Na^{+}, K^{+}, NH_4^{+}
- Group I anions: Cl^{-}, Br^{-}, I^{-}, $[Fe(CN)_6]^{4-}$, $[Fe(CN)_6]^{3-}$
- Groups II and V anions: NO_2^{-}, CH_3COO^{-}, NO_3^{-}, MnO_4^{-}
- Group III anions: SO_3^{2-}, CO_3^{2-}, $C_2O_4^{2-}$, BO_3^{3-}, $C_4H_4O_6^{2-}$
- Group IV and VI anions: PO_4^{3-}, $S_2O_3^{2-}$, CrO_4^{2-}, SO_4^{2-}

The qualitative analysis of single salt consists of preliminary research and analysis of cation and anion separately. For this purpose, the salt must be dissolved in some suitable solvent to prepare a clear and transparent solution.

All inorganic salts can be divided into soluble in water and insoluble in water and the solubility depends strongly on the composition of the salt (cation and anion). Of course, there are some rules which are helpful in prediction of whether the salt will dissolve in water and they are presented in the table below:

SOLUBLE SALTS	Na^{+}, K^{+}, and NH_4^{+} ions form *soluble salts* for instance NaCl, KNO_3, $(NH_4)_2SO_4$, Na_2S, K_2CO_3
	nitrates NO_3^{-} form *soluble salts*, for instance $Cu(NO_3)_2$, $Fe(NO_3)_3$, $Ca(NO_3)_2$
	acetates CH_3COO^{-} form *soluble salts*, for instance $Co(CH_3COO)_2$, $Ba(CH_3COO)_2$, CH_3COOK
	chlorides Cl^{-}, bromides Br^{-}, and iodides I^{-} generally form *soluble salts*, except the salts of the Pb^{2+}, Hg_2^{2+}, Ag^{+}, and Cu^{+} ions
	sulfates SO_4^{2-} generally form *soluble salts*, except $BaSO_4$, $SrSO_4$, and $PbSO_4$, which are insoluble, and Ag_2SO_4, $CaSO_4$, and Hg_2SO_4, which are slightly soluble
INSOLUBLE SALTS	sulfides S^{2-} are usually *insoluble*, except Na_2S, K_2S, $(NH_4)_2S$, MgS, CaS, SrS, and BaS
	chromates CrO_4^{2-} are usually *insoluble*, except Na_2CrO_4, K_2CrO_4, $(NH_4)_2CrO_4$, $MgCrO_4$
	phosphates PO_4^{3-} and carbonates CO_3^{2-} are usually *insoluble*, except salts of the Na^{+}, K^{+}, and NH_4^{+} ions
	sulfates SO_4^{2-} of the salts $BaSO_4$, $SrSO_4$, and $PbSO_4$ are insoluble, and Ag_2SO_4, $CaSO_4$, and Hg_2SO_4, which are slightly soluble
	chlorides Cl^{-}, bromides Br^{-}, and iodides I^{-} of the salts of the Pb^{2+}, Hg_2^{2+}, Ag^{+}, and Cu^{+} ions
	all carbonates are insoluble except those of sodium, potassium, and ammonium

There are two main steps in preliminary research:

1. **Solubility test -** which always begins with dissolving the sample in distilled water. In the first, attempts are made at room temperature and then solutions are warmed up until the boiling point of solvent, due to the lack of solubility. If the salt is not soluble in water, the following solvents are used one after another in a systematic order:
   - diluted CH_3COOH
   - diluted HCl
   - concentrated HCl
   - diluted HNO_3
   - concentrated HNO_3
   - *aqua regia* - mixture of concentrated HCl and HNO_3 acids in ratio 3:1
   - strong bases

The form and color of analyzed salt may suggest its solubility and the presence of particular ions like Cu^{2+} (blue), Ni^{2+} (green), Mn^{2+} (light pink), Co^{2+} (rose), Cr^{3+} (green or purple), CrO_4^{2-} and Fe^{3+} (yellow), MnO_4^- (purple) and then the solution of dissolved salt should also be colored. The crystalline form of the salt suggests its good solubility in water, whereas the powder form of a salt indicates its poor solubility in water. Also, if dissolving any salt in water is followed by appearance of gelatinous residue, different from starting one, it may imply salt hydrolysis. For instance, this is observed with Bismuth salts.

2. **Checking pH of the solution -** to specify the acidity or basicity of an aqueous solution, which gives an initial orientation about the type of the salt:
   - ✓ neutral pH is observed for salts of strong acids and strong bases or salts of weak acids and weak bases (NaCl, NH_4NO_2);
   - ✓ basic pH is observed for salts of strong bases and weak acids (CH_3COOK, Na_2CO_3);
   - ✓ acidic pH is observed for salts of weak bases and strong acids (NH_4NO_3, $MgSO_4$), as well as acid salts ($KHSO_4$).

Identification of salt samples

Student must identify three unknown solid salts. Two of them are soluble in water, the third is insoluble in water. Nevertheless, each simple salt is composed of one cation and one anion and they have to be identified. Therefore, some helpful procedure is given below to follow:

1. Take a look at the sample - specify its form (crystal or powder) and color (colorless, white or other). Remember that hydrated copper(II) salts and anhydrous cobalt salts are blue, hydrated iron(II), chromium(III), nickel(II) salts are green, hydrated cobalt salts are red, calcium(II), zinc(II) or cadmium(II) nitrates or sulfates are white.
2. Take a small amount of salt to a clean test tube and dissolve it in a small amount (5-10 mL) of distilled water.

If the sample dissolves in water, the obtained solution can be diluted with additional amount of distilled H_2O and it is ready for cation and anion identification. However, it is still worth checking the pH of the solution (important information given in Introduction part). Then, you can start with cation identification. For this purpose:

3. Take small amount of obtained solution and start the analysis with the use of the group reagent. As a first, use HCl as the 1st group reagent. The occurrence of the precipitate points at the presence of the cation from the 1st group cations. The lack of the precipitate means, that cation belongs to another group (II, III, IV or V). Therefore, the reactions with other group reagents (procedures described in book entitled *"Selected Topics in General and Inorganic Chemistry" part C*) should be conducted.
4. Confirmation of the group to which cation belongs, should be followed by characteristic reactions to identify specific cation (exercises 1-4).

After identification of the cation you can start the determination of the anion (exercises 5-8).

Example of the analysis

Sample 1

1. Visual test – white crystalline powder
2. Solubility in water – soluble
3. Cation identification:
   a) number of the group:
      1st group reagent (HCl solution) – no precipitate
      2nd group reagent (HCl / H_2S aq) – black precipitate – means, that cations belongs to the 2nd group

Conclusion *– the salt is white, so Pb(II) and Hg(II) ions are under consideration*

   b) characteristic reactions:
      for Pb(II): reaction with KI, K_2CrO_4 or diluted H_2SO_4 – yellow precipitate obtained in reaction with KI and K_2CrO_4 and white precipitate with H_2SO_4
      for Hg(II): drop of the analyzed solution placed on the copper plate – no reaction

Conclusion: Sample contains Pb^{2+} ions

4. Anion identification
   a) number of the group: reactions with $AgNO_3$aq and $BaCl_2$aq

Observation: no precipitate with $AgNO_3$ and $BaCl_2$

Conclusion *– the salt is white, so CH_3COO^-, NO_2^- and NO_3^- are the only possible anions*

   b) characteristic reactions:
      for CH_3COO^-: reaction with solid $KHSO_4$ – detection of acetic acid odor (vinegar odor)
      for NO_2^- and NO_3^-: the reaction with Mohr's salt in the presence of diluted and concentrated H_2SO_4, respectively – no reaction

Conclusion *– CH_3COO^- is present in the analyzed salt*

Final conclusions: Sample 1 is $Pb(CH_3COO)_2$

Sample 2

1. Visual test – green powder
2. Solubility in water – insoluble
3. Solubility in CH_3COOHaq – soluble
4. Solubility in diluted HCl - soluble
3. Cation identification:
    a) number of the group
        1st group reagent (HCl solution) – no precipitate
        2nd group reagent (HCl / H_2S aq) – no precipitate
        3rd group reagent (solutions of NH_3, NH_4Cl and H_2Saq) – **REMEMBER that the salt was dissolved in the acid, so the obtained solution has acidic pH. Therefore, the reaction with the 3rd group reagent must be followed in excess of NH_3 and NH_4Cl to obtain alkaline solution (check the pH with indicator paper)**

    Observation – the appearance of black precipitate

Conclusion *– the salt is green, so Ni(II) ion is the most probable one*

    b) characteristic reactions for Ni(II): the reaction with dimethylglyoxime (HDMG) in the presence of NH_3 solution

    Observation – the appearance of characteristic rose-red precipitate

Conclusion: Sample contains Ni^{2+} ions

4. Anion identification
    a) Dissolution of salt sample is accompanied by the release of colorless gas

Conclusion *– the salt contains CO_3^{2-} anions which are decomposed and carbon dioxide gas is released, visible as gas bubbles*

Final conclusions: Sample 2 is $NiCO_3$