

## Introduction

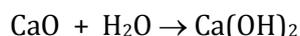
The aim of Exercise 10 is to analyze three inorganic substances in liquid or solid state. These group of compounds contains metals, nonmetals, metal and nonmetal oxides, hydroxides, inorganic acids and inorganic salts (described in the instruction for Exercise 9).

Metals are elements with low electronegativity and they form positive ions in compounds. They are solid under normal conditions except for Hg, which is liquid at room temperature. The color of most of metals is silvery or grey except for Cu (reddish-orange) or Au (yellow). Metals are characterized by bright luster (more reactive metals form oxide layer on the surface), hardness, ability to resonate sound, they are also excellent conductors of heat and electricity. Due to their ductility and malleability, they can be drawn into wire and can be beaten into very thin sheets.

Nonmetals are elements electronegative in character. Most of the nonmetals exist in two out of three states of matter at room temperature like gases (O<sub>2</sub>) and solids (S), and only Br<sub>2</sub> is liquid. Nonmetals are very brittle and cannot be rolled into wires or pounded into sheets. They have no metallic luster and do not reflect light. They are also poor conductors of heat and electricity. The colors of nonmetals vary, for instance sulfur is yellow and Br<sub>2</sub> brownish red.

Metal oxides are solid under normal conditions. They have different colors (ZnO is white, PbO yellow, CuO black) and have no luster.

Metal oxides exhibit basic properties:



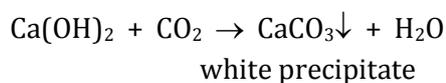
and the more electropositive the central atom the more basic the oxide.

Nonmetal oxides are gaseous in general (SO<sub>2</sub>, CO<sub>2</sub>) and they dissolve in water to give the oxo anion of particular acid:



therefore we can say that they show acidic properties and the more electronegative the central atom, the more acidic the oxide.

Hydroxides are compound which contain hydroxyl groups OH<sup>-</sup>. In most cases, they are white solid powder, granules or pills. Hydroxides of alkali metals (NaOH, KOH) are strong bases and show good solubility in water. Alkaline earth metals hydroxides (Mg(OH)<sub>2</sub>, Ca(OH)<sub>2</sub>, Sr(OH)<sub>2</sub>, Ba(OH)<sub>2</sub>, are moderately strong bases, and poorly soluble in water, however their solubility increases within the group together with increasing atomic number. Amphoteric hydroxides Be(OH)<sub>2</sub>, Zn(OH)<sub>2</sub>, Pb(OH)<sub>2</sub>, Sn(OH)<sub>2</sub>, Al(OH)<sub>3</sub>, Sb(OH)<sub>3</sub>, Cu(OH)<sub>2</sub> are also poorly soluble in water, but they dissolve in acids and bases. Some of the hydroxides is used for the identification of other compounds. For instance, a solution or suspension of Ca(OH)<sub>2</sub>, known as limewater, can be used for detection of CO<sub>2</sub> gas:



Inorganic acids are also called as mineral acids and the most popular are sulfuric acid  $\text{H}_2\text{SO}_4$ , hydrochloric acid  $\text{HCl}$  and nitric acid  $\text{HNO}_3$ . They are liquid except for boric acid  $\text{H}_3\text{BO}_3$ , which is white crystalline solid. Mineral acids are soluble in water and insoluble in organic solvents. Inorganic acids range from acids of great strength (e.g.: sulfuric acid) to very weak (e.g.: boric acid).

Inorganic salts also belong to inorganic substances, but they have already been described in the instruction for Exercise 9.

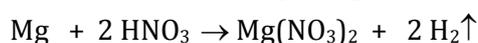
## SUPPORTIVE INFORMATIONS FOR GENERAL ANALYSIS

1. Starting the analysis of given samples it is good to describe its color (yellow, colorless), form (liquid, solid, crystalline, amorphous) and odor (odorless, characteristic odor). **REMEMBER NOT TO TASTE THEM!!!**
2. If the sample is solid powder, check its solubility in water first, then in acid or base – it allows to determine the chemical character of the sample. Observe the sample while dissolving to see potential additional “special effects” like the release of gas bubbles, the change of the color of the solution.
3. If the sample is liquid or soluble in water check pH of the solution to specify the acidity or basicity of the solution:
  - basic pH suggest the presence of basic oxides ( $\text{MgO}$ ,  $\text{CaO}$ ), basic salts ( $\text{CH}_3\text{COONa}$ ,  $(\text{NH}_4)_2\text{CO}_3$ ) or hydroxide solution ( $\text{NH}_3$  aq,  $\text{NaOH}$  aq)
  - acidic pH suggests the presence of inorganic acid ( $\text{HNO}_3$ ,  $\text{HCl}$ ) or acidic salt ( $\text{NH}_4\text{NO}_3$ ).
  - neutral pH may be observed for boric acid and solutions of neutral salts ( $\text{NaCl}$ ,  $\text{KNO}_3$ ).
4. After dissolving, cation (in case of metals, metal oxides, hydroxides) or anion (in case of acids) must be identified.
5. The final formula of the compound must be given in the report at the end of the analysis.

## SUPPORTIVE INFORMATIONS FOR THE ANALYSIS OF INDIVIDUAL GROUPS OF INORGANIC SUBSTANCES

### METALS

Metals are available in many forms like ribbon, granules, foil, wire or powder. The more the metal is crumbled the faster it dissolves. Usually metals dissolve in mineral acids (metals are oxidized). **The electrochemical series (activity series)** helps in predicting the choice of acid for dissolving analyzed sample. All metals placed above hydrogen ( $\text{Cd}$ ,  $\text{Ni}$ ,  $\text{Co}$ ,  $\text{Mn}$ ,  $\text{Pb}$ ,  $\text{Fe}$ ,  $\text{Mg}$ ) displace hydrogen from acids to form  $\text{H}_2$  while those placed below hydrogen do not displace hydrogen from acids. For example,  $\text{Co}$  reacts with  $\text{HCl}$  to form  $\text{H}_2$ , whereas  $\text{Cu}$  does not:



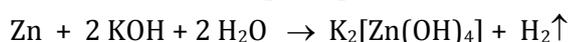
Metal dissolution should be initiated at room temperature. If the dissolving process is slow, the sample can be heated but it should be done carefully.

Metals placed below hydrogen in activity series like Bi, Cu, Hg, Ag dissolve in oxidizing acids (HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>) and NO, NO<sub>2</sub> or SO<sub>2</sub> are formed, instead of H<sub>2</sub>. For instance:



This reaction is not a simple oxidation of copper. The metal is oxidized to Cu(II) by the nitrate ions of the acid, accompanied by the formation of brown nitrogen dioxide gas NO<sub>2</sub>.

Aluminum and zinc are amphoteric metals and they dissolve both in acids and strong bases solutions with the release of H<sub>2</sub> gas:



After dissolving the sample of metal the cation must be identified (Exercises 1-4, procedures described in book entitled *"Selected Topics in General and Inorganic Chemistry" part C*).

### NONMETALS

Student do not obtain gaseous sample for identification, therefore only nonmetals like S (yellow solid), C (black), I<sub>2</sub> (dark violet crystals) can be taken into consideration as potential samples.

Sulfur and carbon do not dissolve in water, whereas I<sub>2</sub> dissolves in ethanol and aqueous solution of KI.

### METAL OXIDES

Metal oxide can be basic (MgO) or amphoteric (PbO), nevertheless they can be identified in the same manner like metal samples (cation identification - information above). However, some of metal oxides can absorb carbon dioxide (CO<sub>2</sub>) from the air to form metal carbonates (MgCO<sub>3</sub>, PbCO<sub>3</sub>). The presence of carbonates in given sample reduces its solubility in water. In that case, the sample should be dissolved in hydrochloric acid HCl and carbonates treated as contamination only.

The color of the solution after dissolving the sample can indicate the presence of certain cations. For example: Cu<sup>2+</sup> forms blue solutions, Ni<sup>2+</sup> green, Mn<sup>2+</sup> light pink, Co<sup>2+</sup> pink or rose, Cr<sup>3+</sup> greenish gray.

### HYDROXIDES

To identify any hydroxide only cation must be detected (Exercises 1-4, procedures described in book entitled *"Selected Topics in General and Inorganic Chemistry" part C*).

### INORGANIC ACIDS

If the analyzing substance is inorganic acid, an anion should be detected (instructions for Exercises 5-8).

### INORGANIC SALTS

The procedure of inorganic salt analysis is described in the instruction for Exercise 9.

## EXAMPLES OF THE ANALYSIS

### Example 1 – liquid, colorless

1. Checking pH – universal indicator paper changed color into red. *Conclusion*: this sample may be the aqueous solution of acid, therefore an anion should be identified.
2. Anion identification - reactions with  $\text{AgNO}_3\text{aq}$  and  $\text{BaCl}_2\text{aq}$  give no precipitate with both group reagents. *Conclusions*: searching anion may be  $\text{CH}_3\text{COO}^-$  or  $\text{NO}_3^-$  (the solution is colorless,  $\text{MnO}_4^-$  ions are excluded). The  $\text{CH}_3\text{COO}^-$  can be identified with solid  $\text{KHSO}_4$ . *Observation*: no characteristic odor of acetic acid. *Conclusion*:  $\text{CH}_3\text{COO}^-$  are absent.
3.  $\text{NO}_3^-$  ions can be identified with Mohr's salt (the instruction for Exercise 6). *Observation*: brown ring is formed in the reaction mixture. *Conclusion*:  $\text{NO}_3^-$  are present.

*Final conclusion*: the analyzed sample is nitric acid  $\text{HNO}_3$

### Example 2 – solid, metallic

1. Visual test – sample is solid, silvery gray, lustrous. *Conclusion*: it may be metal.
2. Test of the solubility in water – insoluble.
3. Test of the solubility in dilute  $\text{HCl}$ aq – *Observations*: it dissolves slowly and faster after small heating. Additionally, colorless gas is released during dissolving – visible bubbles.
4. Visual test of the solution after dissolution of the sample in the acid – *Observations*: solution is colorless. *Conclusions*: some metals like Co, Cr, Ni can be eliminated, because they form color solutions when dissolved. The sample dissolves in  $\text{HCl}$ , therefore metals such as Pb, Zn or Al should be considered in further analysis.
5. Cation identification:
  - a) the sample dissolves in  $\text{HCl}$ , therefore 1<sup>st</sup> group cations is not taken into consideration
  - b) reaction with  $\text{H}_2\text{S}_{\text{aq}}$  (no need to add  $\text{HCl}$ , because the sample is already dissolved in  $\text{HCl}$ ).  
*Observation*: yellow precipitate. *Conclusions*: it indicates presence of  $\text{Cd}^{2+}$ , because only  $\text{CdS}$  is yellow among other sulfides of 2<sup>nd</sup> group cations.

*Final conclusion*: Cadmium metal (Cd) is the analyzed sample

### Example 3 – solid, powder

1. Visual test – sample is solid and white powder. *Conclusion*: it may be metal oxide or salt.
2. Test of the solubility in water – insoluble
3. Test of the solubility in dilute  $\text{HCl}$  – *Observations*: good solubility, the resulting solution is colorless.
4. Cation identification - subsequent reactions with group reagents indicate the presence of the cation from the 3<sup>rd</sup> group of cations. The color of the initial sample suggest Zn or Al cations (both are amphoteric). The reaction with  $\text{NH}_3\text{aq}$  leads to white precipitate which dissolve in excess of  $\text{NH}_3\text{aq}$ . *Conclusions*: it is  $\text{Zn}^{2+}$ .
5. Anion identification - reactions with  $\text{AgNO}_3\text{aq}$  and  $\text{BaCl}_2\text{aq}$  give no precipitate with both group reagents. The characteristic reactions for residual anions ( $\text{NO}_3^-$ ,  $\text{CH}_3\text{COO}^-$ ) also give no visual effects and odor. *Conclusion*: it is oxide not salt.

*Final conclusions*: the analyzed sample is  $\text{ZnO}$  (zinc oxide)