## Classical qualitative analysis. 6. Analysis of the simple solid sample

These simple solids can be:
metal (Cu), oxides (ZnO, $Al_2O_3$), hydroxides ($Al(OH)_3$), acids ($H_2C_2O_4$) or salts ($NaNO_3$)

**Preliminary tests on the solid sample:**

1. color
2. behavior during heating – evolution of gases
3. attempt to stain flame on a platinum wire
4. behavior during the melting with phosphate or borax bead (phosphate or borax beads tests)- colors
5. solubility test at room temperature and after heating - order of examination of the simplest dissolution:
   a) $H_2O$ (determination of pH)
   b) $CH_3COOH_{diluted}$ - carbonates
   c) diluted non-oxidizing acids (HCl, $H_2SO_4$) - Zn
   d) diluted oxidizing acid ($HNO_3$) - Cu
   e) concentrated acids: $CH_3COOH$, HCl, $H_2SO_4$, $HNO_3$
   f) aqua regia (HCl + $HNO_3$)
   g) diluted weak bases ($NH_3 \cdot H_2O$) - AgCl
   h) diluted strong bases (NaOH) - $Al(OH)_3$
   i) concentrated bases: $NH_3 \cdot H_2O$, NaOH
   j) melting with sodium carbonate ($Na_2CO_3$) - $BaSO_4$, $Al_2O_3$

**Analysis of the simple solid sample**
Preliminary studies.

1. Transforming the solid sample into solution in **the simplest** way
   a) dissolving in $H_2O$, np. $NaCl \rightarrow Na^+ + Cl^-$
   b) dissolving in $CH_3COOH$, e.g.. $CaCO_3\downarrow + 2CH_3COOH \rightarrow 2CH_3COO^- + Ca^{2+} + CO_2\uparrow + H_2O$
   c) dissolving in non-oxidizing acid
      $ZnS + 2H_3O^+ \rightarrow Zn^{2+} + H_2S\uparrow + 2\,H_2O$
      $Al(OH)_3 + 3H_3O^+ \rightarrow Al^{3+} + 6H_2O$
      $Zn + 2H_3O^+ \rightarrow Zn^{2+} + H_2\uparrow + 2H_2O$
      (check the electromotive series- hydrogen displacement)
   d) dissolving in oxidizing acid:
      $3CuS\downarrow + 8H_3O^+ + 2NO_3^- \rightarrow 3Cu^{2+} + 3S + 2NO + 12H_2O$
      $3Cu + 8H_3O^+ + 2NO_3^- \rightarrow 3Cu^{2+} + 2NO + 12H_2O$
   e) dissolving in aqua regia
      $3CoS\downarrow + 8H_3O^+ + 18Cl^- + 2NO_3^- \rightarrow 3CoCl_6^{4-} + 2NO + 3S + 12H_2O$
   f) dissolving in bases
      $Al(OH)_3\downarrow + OH^- \rightarrow Al(OH)_4^-$; $Al_2O_3\downarrow + 2OH^- \rightarrow 2AlO_2^- + H_2O$
   g) melting with $Na_2CO_3$
      $BaSO_4 + Na_2CO_3 \rightarrow BaCO_3 + Na_2SO_4$
      $Al_2O_3 + Na_2CO_3 \rightarrow 2NaAlO_2 + CO_2\uparrow$
   h) other ways of dissolving:
      i. complexation raeactions
         $AgCl\downarrow + 2NH_3 \cdot H_2O \rightarrow [Ag(NH_3)_2]^+ + Cl^- + 2H_2O$
      ii. cation reduction
         $2AgI\downarrow + Zn \rightarrow 2Ag^0\downarrow + 2I^- + Zn^{2+}$

After dissolving of the solid sample identify the cation and (possible) anion. Sometimes the heavy metal ions disturb in the analysis of anions and have to be separated using the so-called **sodium extract**.

**Sodium extract** – boiling with a saturated solution of $Na_2CO_3$

In order to separate the heavy metal ions that interfere with the analysis of anions,

e.g, $Cu^{2+}$ ions with the detection of oxidizing anions:

$$2Cu^{2+} + 4I^- \rightarrow 2CuI\downarrow + I_2$$

Before preparation of sodium extract check the presence of $Na^+$ and $CO_3^{2-}$

**Preparation of sodium extract solution**:

Boil a sample with saturated solution of $Na_2CO_3$

⇓

| solution: $Na^+$, $CO_3^{2-}$, anions, $K^+$, $NH_4^+$, amphoteric: $AlO_2^-$, $ZnO_2^{2-}$, $[Fe(C_2O_4)_3]^{3-}$, $[Cr(C_2O_4)_3]^{3-}$, $[Sn(C_2O_4)_4(H_2O)_2]^{4-}$ | precipitate: alkaline earth carbonates, heavy metals, hydrogencarbonates, hydrooxides, AgI (silver halides), some phosphates |
|---|---|
| ⇓ | ⇓ + $CH_3COOH$ |
| Preliminary tests on the anions | $MCO_3\downarrow + 2CH_3COOH \rightarrow 2CH_3COO^- + M^{2+} + CO_2\uparrow + H_2O$ |
| | cation analysis by identification reactions |

**Experimental procedure (analysis of unknown sample)**

Each student receives a simple solid sample in a dry, glass tube. It must be transformed into solution and perform the qualitative analysis. Describe the analysis of the simple solid performed with appropriate reaction equations.