

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

These simple solids can be:

metal (Cu), oxides (ZnO, Al₂O₃), hydroxides (Al(OH)₃), acids (H₂C₂O₄) or salts (NaNO₃)

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) H₂O (determination of pH)
 - b) CH₃COOH_{diluted} - carbonates
 - c) diluted non-oxidizing acids (HCl, H₂SO₄) - Zn
 - d) diluted oxidizing acid (HNO₃) - Cu
 - e) concentrated acids: CH₃COOH, HCl, H₂SO₄, HNO₃
 - f) aqua regia (HCl + HNO₃)
 - g) diluted weak bases (NH₃·H₂O) - AgCl
 - h) diluted strong bases (NaOH) - Al(OH)₃
 - i) concentrated bases: NH₃·H₂O, NaOH
 - j) melting with sodium carbonate (Na₂CO₃) - BaSO₄, Al₂O₃

Analysis of the simple solid sample

Preliminary studies.

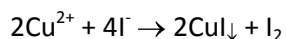
1. Transforming the solid sample into solution in **the simplest way**
 - a) dissolving in H₂O, np. NaCl → Na⁺ + Cl⁻
 - b) dissolving in CH₃COOH, e.g.. CaCO₃↓ + 2CH₃COOH → 2CH₃COO⁻ + Ca²⁺ + CO₂↑ + H₂O
 - c) dissolving in non-oxidizing acid
$$\text{ZnS} + 2\text{H}_3\text{O}^+ \rightarrow \text{Zn}^{2+} + \text{H}_2\text{S}^{\uparrow} + 2\text{H}_2\text{O}$$
$$\text{Al(OH)}_3 + 3\text{H}_3\text{O}^+ \rightarrow \text{Al}^{3+} + 6\text{H}_2\text{O}$$
$$\text{Zn} + 2\text{H}_3\text{O}^+ \rightarrow \text{Zn}^{2+} + \text{H}_2^{\uparrow} + 2\text{H}_2\text{O}$$
(check the electromotive series- hydrogen displacement)
 - d) dissolving in oxidizing acid:
$$3\text{CuS}\downarrow + 8\text{H}_3\text{O}^+ + 2\text{NO}_3^- \rightarrow 3\text{Cu}^{2+} + 3\text{S} + 2\text{NO} + 12\text{H}_2\text{O}$$
$$3\text{Cu} + 8\text{H}_3\text{O}^+ + 2\text{NO}_3^- \rightarrow 3\text{Cu}^{2+} + 2\text{NO} + 12\text{H}_2\text{O}$$
 - e) dissolving in aqua regia
$$3\text{CoS}\downarrow + 8\text{H}_3\text{O}^+ + 18\text{Cl}^- + 2\text{NO}_3^- \rightarrow 3\text{CoCl}_6^{4-} + 2\text{NO} + 3\text{S} + 12\text{H}_2\text{O}$$
 - f) dissolving in bases
$$\text{Al(OH)}_3\downarrow + \text{OH}^- \rightarrow \text{Al(OH)}_4^-; \text{Al}_2\text{O}_3\downarrow + 2\text{OH}^- \rightarrow 2\text{AlO}_2^- + \text{H}_2\text{O}$$
 - g) melting with Na₂CO₃
$$\text{BaSO}_4 + \text{Na}_2\text{CO}_3 \rightarrow \text{BaCO}_3 + \text{Na}_2\text{SO}_4$$
$$\text{Al}_2\text{O}_3 + \text{Na}_2\text{CO}_3 \rightarrow 2\text{NaAlO}_2 + \text{CO}_2^{\uparrow}$$
 - h) other ways of dissolving:
 - i. complexation reactions
$$\text{AgCl}\downarrow + 2\text{NH}_3\cdot\text{H}_2\text{O} \rightarrow [\text{Ag(NH}_3)_2]^+ + \text{Cl}^- + 2\text{H}_2\text{O}$$
 - ii. cation reduction
$$2\text{Ag}\downarrow + \text{Zn} \rightarrow 2\text{Ag}^0\downarrow + 2\text{I}^- + \text{Zn}^{2+}$$

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 Na₂CO₃

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

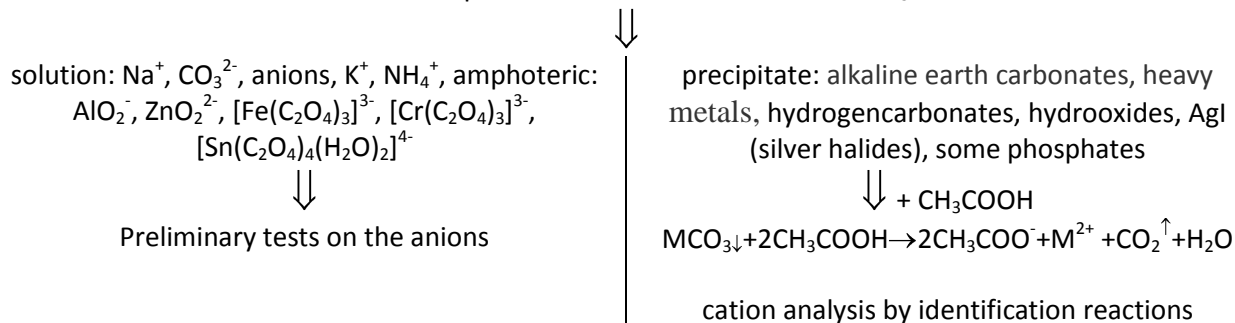
e.g, Cu^{2+} ions with the detection of oxidizing anions:



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



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.