

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

These simple solids can be:

metal (Cu), oxides (ZnO , Al_2O_3), hydroxides ($\text{Al}(\text{OH})_3$), acids ($\text{H}_2\text{C}_2\text{O}_4$) 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) $\text{CH}_3\text{COOH}_{\text{diluted}}$ - 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 ($\text{HCl} + \text{HNO}_3$)
 - g) diluted weak bases ($\text{NH}_3 \cdot \text{H}_2\text{O}$) - AgCl
 - h) diluted strong bases (NaOH) - $\text{Al}(\text{OH})_3$
 - i) concentrated bases: $\text{NH}_3 \cdot \text{H}_2\text{O}$, NaOH
 - j) melting with sodium carbonate (Na_2CO_3) - BaSO_4 , Al_2O_3

Analysis of the simple solid sample

Preliminary studies.

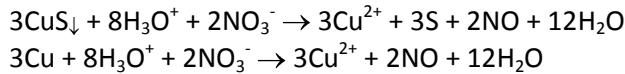
1. Transforming the solid sample into solution in **the simplest way**

- a) dissolving in H_2O , np. $\text{NaCl} \rightarrow \text{Na}^+ + \text{Cl}^-$
- b) dissolving in CH_3COOH , e.g.. $\text{CaCO}_3 \downarrow + 2\text{CH}_3\text{COOH} \rightarrow 2\text{CH}_3\text{COO}^- + \text{Ca}^{2+} + \text{CO}_2 \uparrow + \text{H}_2\text{O}$

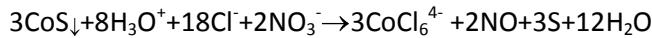
- c) dissolving in non-oxidizing acid
$$\text{ZnS} + 2\text{H}_3\text{O}^+ \rightarrow \text{Zn}^{2+} + \text{H}_2\text{S} \uparrow + 2\text{H}_2\text{O}$$
$$\text{Al}(\text{OH})_3 + 3\text{H}_3\text{O}^+ \rightarrow \text{Al}^{3+} + 6\text{H}_2\text{O}$$
$$\text{Zn} + 2\text{H}_3\text{O}^+ \rightarrow \text{Zn}^{2+} + \text{H}_2 \uparrow + 2\text{H}_2\text{O}$$

(check the electromotive series- hydrogen displacement)

- d) dissolving in oxidizing acid:



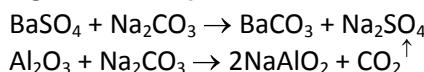
- e) dissolving in aqua regia



- f) dissolving in bases

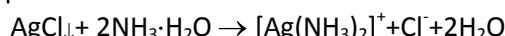


- g) melting with Na_2CO_3



- h) other ways of dissolving:

- i. complexation reactions



- ii. cation reduction

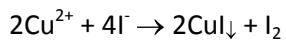


After dissolving of the solid sample identify the cation and (possible) anion. Sometimes the heavy metal ions disturb in the analysis of anions and have to be separated using the so-called **sodium extract**.

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:



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

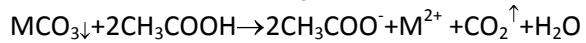
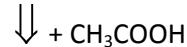


solution: Na^+ , CO_3^{2-} , anions, K^+ , NH_4^+ , amphoteric:
 AlO_2^- , ZnO_2^{2-} , $[\text{Fe}(\text{C}_2\text{O}_4)_3]^{3-}$, $[\text{Cr}(\text{C}_2\text{O}_4)_3]^{3-}$,
 $[\text{Sn}(\text{C}_2\text{O}_4)_4(\text{H}_2\text{O})_2]^{4-}$



Preliminary tests on the anions

precipitate: alkaline earth carbonates, heavy metals, hydrogencarbonates, hydrooxides, AgI (silver halides), some phosphates



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.