Classical qualitative analysis. 3. Microcrystalline analysis

Microscope – instruction of use.

1. Tilt the mirror to illuminate field of vision the best.
2. Put the specimen slide on the microscope stage.
3. Laterally looking at the microscope lower the objective lens over the specimen slide to keep distance about 0,5 cm.
4. Looking through an eyepiece with one eye(not closing the other) raise slowly stand column (rotating an adjustment knob) until the specimen comes into sharp focus. In case of failure lower the stand column again to keep distance between objective lens and specimen slide slightly smaller than 0,5 cm and again looking through the eyepiece slowly raise the column.
   DO NOT LOWER THE STAND COLUMN LOOKING THROUGH THE EYEPIECE !!!

Microcrystalline reactions.

NOTICE: If a vaporisation of a sample is necessary the specimen slide with a drop of solution should be placed in a drier, at the edge of ceramic plate or kept high above a flame of Bunsen burner. Do not add the reagent until the slide is cold.

The theoretical part

One of the most popular and common used micro method is based on identification of crystal form with the aid of microscope. Principle of operation is based on the precipitation of sparingly dissolved compounds from strongly diluted solution of appropriate salts. The precipitations are prepared directly on the speciment slides and can be examined with aid of the microscope. Recommended magnifications are 50x – 200x. Usually the method allows quickly to detect the presence of particular ions in an examined solution. One should remember that some crystals crystalize to create their specific forms only in favourable conditions. Presence of incidental substances can alter the form of creating crystals that is the main disadvantage of the microcrystalline analysis. Generally microcrystalline reactions are very sensitive: the minimum concentration 10^{-7} – 10^{-8} g (sometimes 10^{-10}g). Every microcrystalline reaction can be described with appropriate reaction equations. The description should also include colour and form of crystals (e.g. black cubes).

Experimental procedure:

Identification of K^+ ions

Put a drop of analysed solution n the specimen and vaporize it. When the slide get cold add a drop of special reagent, ~~that~~ with formula ~~is~~ $Na_2PbCu(NO_2)_6$. After 1 minute new crystals are formed and can be observed under the microscope. As a result of reaction characteristic black or dark brown cubic crystals are formed which may be written as $K_2[PbCu(NO_2)_6]$ (or $2KNO_2 \cdot Pb(NO_2)_2 \cdot Cu(NO_2)_2$ in accordance with triple salt formula. The presence of NH_4^+ ion disturbs the reaction since it creates itself similar crystals. Na^+ i Mg^{2+} ions never form crystals with the reagent mentioned above.

$2K^+ + [PbCu(NO_2)_6]^{2-} \rightarrow K_2[PbCu(NO_2)_6]$ black cubes

Identification of Na^+ ions

Evaporate a drop of solution on the speciment slide. Add a drop of uranyl acetate $UO_2(CH_3COO)_2$. After a few minutes crystals of sodium-uranyl acetete $NaCH_3COO \cdot UO_2(CH_3COO)_2$ are formed and can be observed. Crystals are regular tetra or octahedrons. If there are additionally Mg^{2+} ions in the solution then crystals of triplex salt $NaCH_3COO \cdot Mg(CH_3COO)_2 \cdot 3UO_2(CH_3COO)_2 \cdot 9H_2O$ are precipitated which shape is similar to form of $NaCH_3COO \cdot UO_2(CH_3COO)_2$.

$Na^+ + Mg^{2+} + 3UO_2^{2+} + 9CH_3COO^- + 9H_2O \rightarrow NaMg(UO_2)_3(CH_3COO)_9 \cdot 9H_2O\downarrow$ (transparent)

Identification of Ca^{2+} ions

Put a drop of investigated solution on a speciment slide, add a drop of diluted solution of H_2SO_4 next vaporize them carefully until white frame around the drop appears. Crystals of $CaSO_4 \cdot 2\ H_2O$ can be observed

$Ca^{2+} + SO_4^{2-} + 2H_2O \rightarrow CaSO_4 \cdot 2H_2O\downarrow$ (white)

Identification of Zn^{2+} ions

Put, on the specimen, a drop of examined solution that is acidulated with acetic acid, add a drop of $[Hg(CNS)_4]$ and evaporate the liquid. Observe crystals of $Zn[Hg(CNS)_4]$ and their characteristic shape of crosses and dendrites. In case of diluted solutions or solution acidified with mineral acid, created crystals are scalene triangles or wedge shaped. Such solutions should be vaporized before reagent drop is added.

$Zn^{2+} + [Hg(CNS)_4]^{2-} \rightarrow Zn[Hg(CNS)_4]\downarrow$ (white „trees")

Unknown analysis

Each student receives a test tube with 5 drops of solutions. They are obliged to identify 2 of 4 described above metallic ions. The method of analysis should be described with the appropriate reaction equations.