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IDENTIFICATION OF UNKNOWN DRUG

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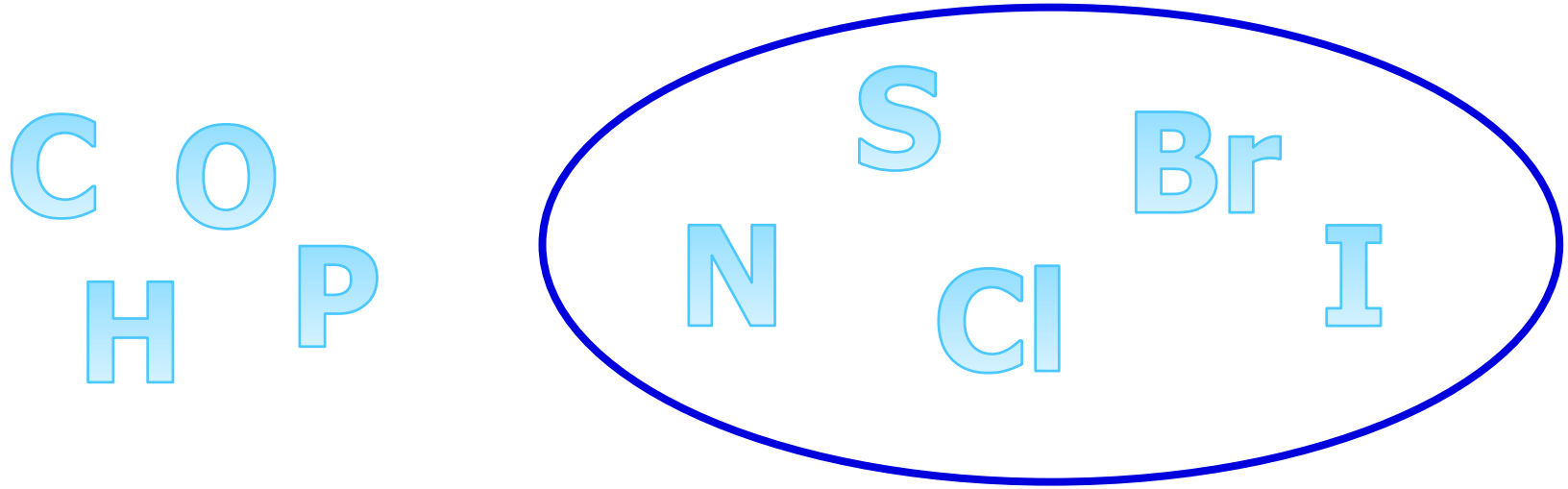
Qualitative elementary analysis

- in practical class, we evaluate the presence of nitrogen, sulfur, and halogens



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Lassaigne test

- 0.01 g of tested compound is inserted into the fusion tube and is covered with a reaction mixture (magnesium + potassium carbonate)

The space for the escape of vapors, which are created during the combustion

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Lassaigne test

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Lassaigne test

- hot fusion tube is put into the test tube containing distilled water (about 15 mL)
- the content of the broken fusion tube is released into the water

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Lassaigne test

- the content of the test tube is filtrated
- the filtrate is divided into 3 parts

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Lassaigne test

Determination of nitrogen as a cyanide

- add few drops of $FeSO_4$ and few drops of $FeCl_3$ to the 5 mL portion of the filtrate
- boil for a half minute
- acidify with dilute HCl after cooling

$$C_{org} + N_{org} \longrightarrow CN^-$$

$$6\ CN^- + Fe^{2+} \longrightarrow [Fe(CN)_6]^{4-}$$

$$[Fe(CN)_6]^{4-} + Fe^{3+} \longrightarrow \{Fe[Fe(CN)_6]\}^-$$

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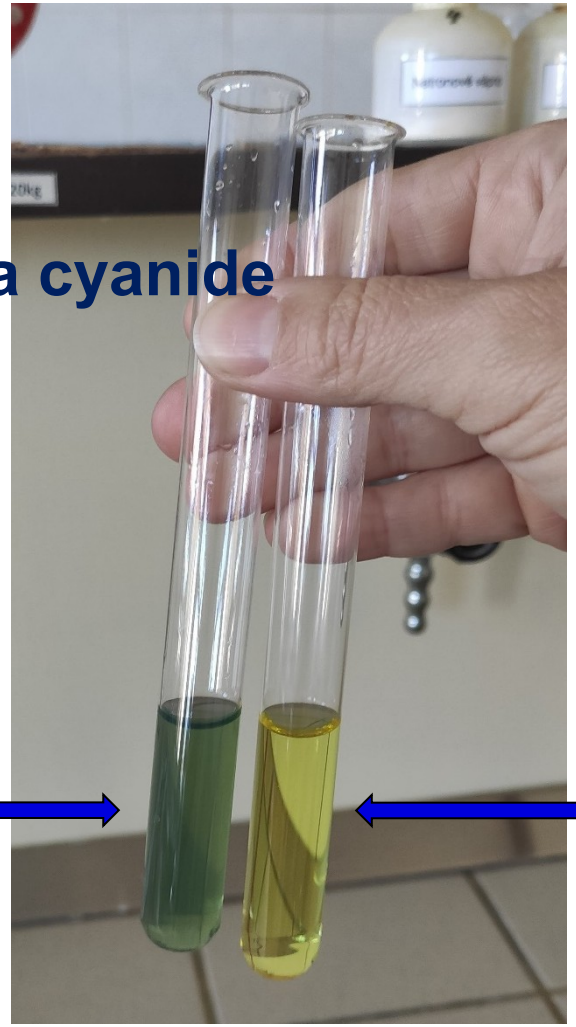
Lassaigne test

Determination of nitrogen as a cyanide



Positive reaction
- presence of nitrogen

Negative reaction
- nitrogen was not present

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Lassaigne test

Determination of nitrogen as a cyanide

- it can happen that just greenish solution is created, then filtrate this solution through dense filtration paper and Prussian blue is caught on it

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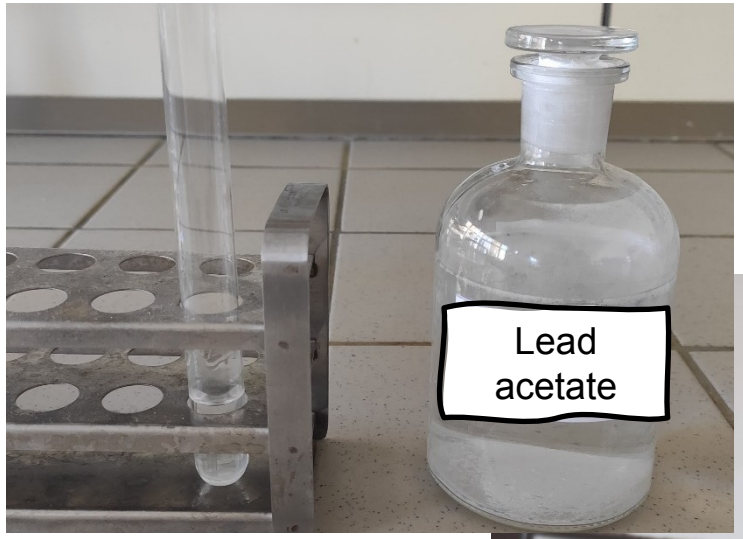
Lassaigne test

Determination of sulfur as sulfide



- add few drops of lead acetate to another 5 mL portion of the filtrate

$$S_{org} \longrightarrow S^{2-}$$

$$S^{2-} + Pb^{2+} \longrightarrow PbS$$

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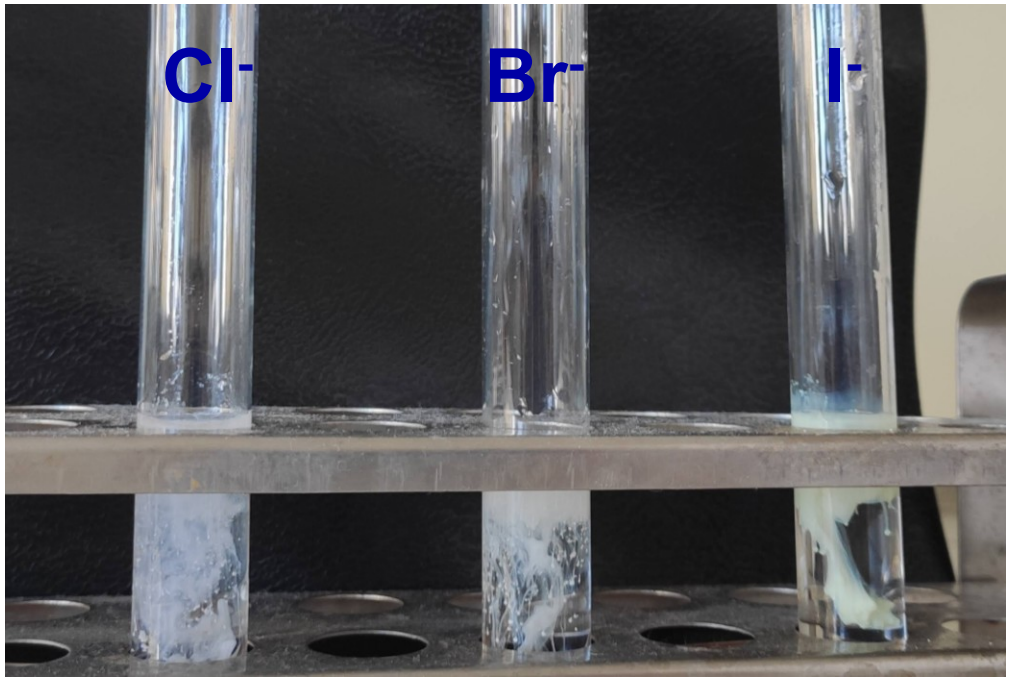
Lassaigne test

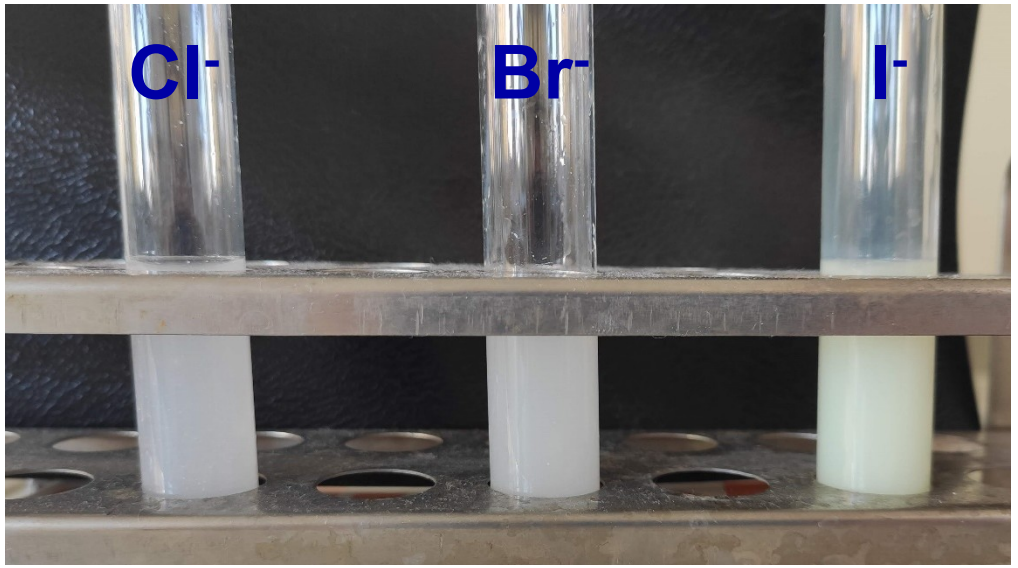
Determination of halogens as halides

- acidify another 5 mL portion with dilute nitric acid
- add silver nitrate

$$X_{org} \longrightarrow X^-$$

$$X^- + Ag^+ \longrightarrow AgX$$





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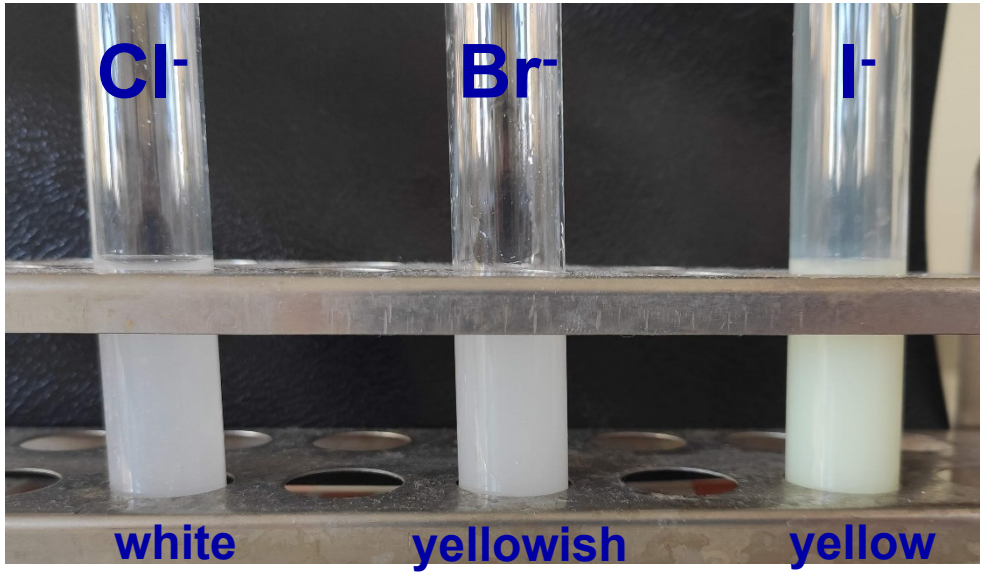
Differentiation of halides

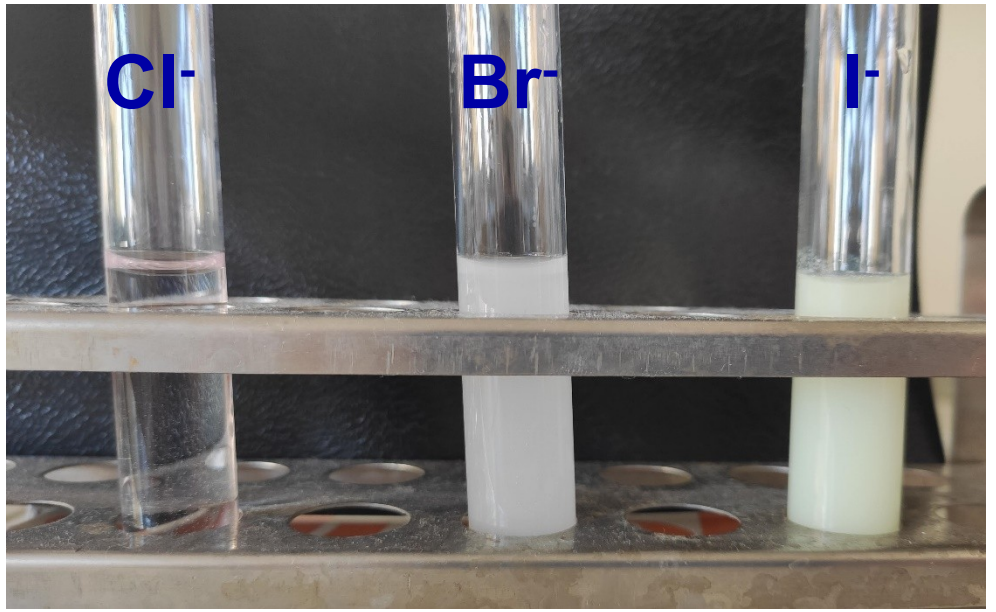
Color of the precipitate and its solubility in ammonia

- add dilute ammonia to the precipitate
- **easily** soluble precipitate => Cl^-
- precipitate soluble with **difficulties** => Br^-
- **insoluble** precipitate => I^-

$$AgCl + 2\ NH_3 \longrightarrow [Ag(NH_3)_2]^+ + Cl^-$$

Before addition of ammonia





After addition of ammonia

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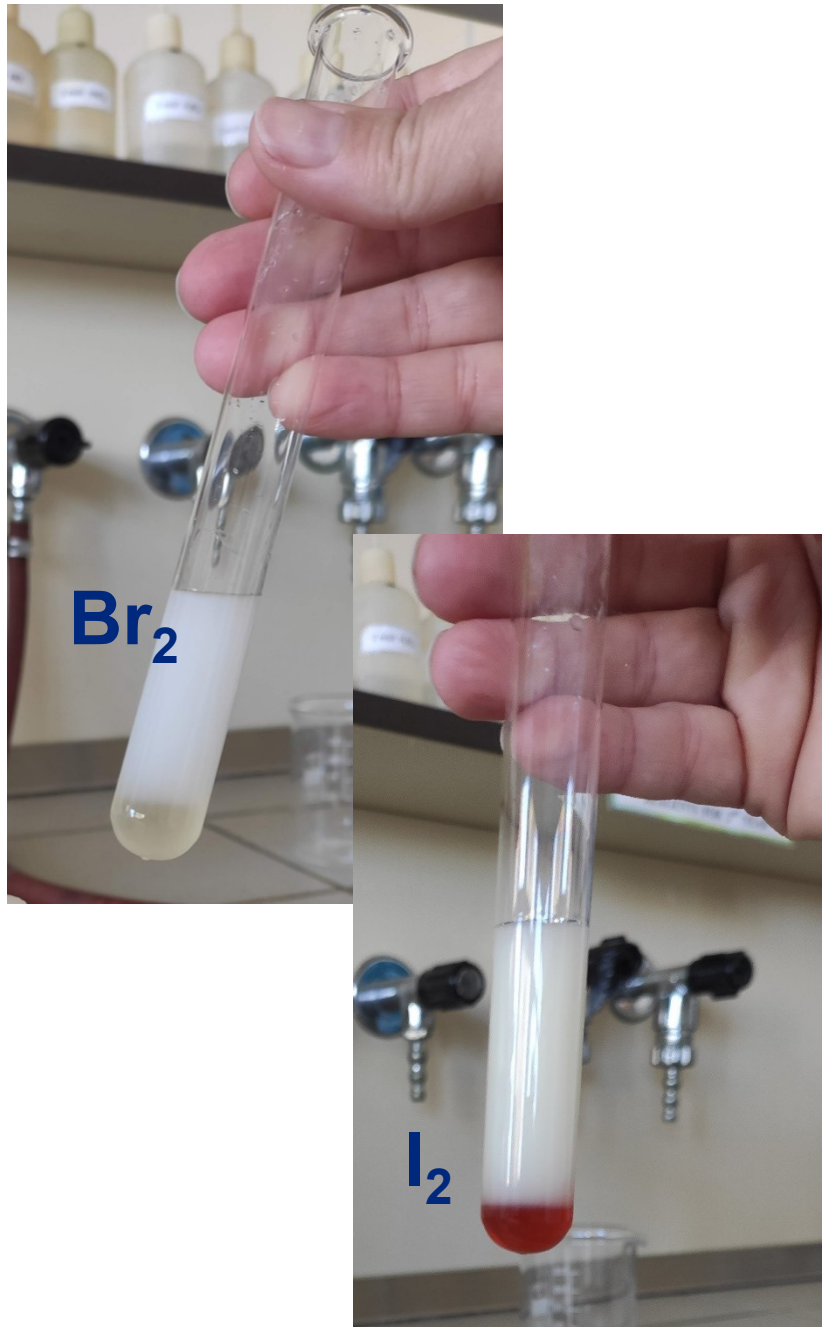
Differentiation of halides

Transformation to the elementary halogen

- add dilute sulfuric acid to 5 mL portion of Lassaigne filtrate, then add 1 mL of chloroform and 0.5 mL chloramine
- the lower layer is **colorless** => chlorine or fluorine
- the lower layer is **yellow to brown** => bromine
- the lower layer is **violet** => iodine



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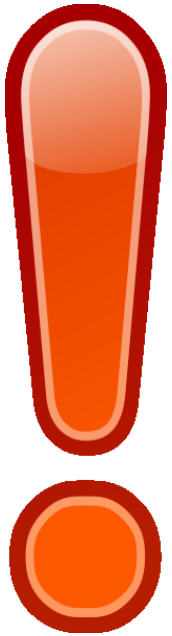
ATTENTION!

- if you found **nitrogen** or **sulfur**, you **COULD NOT** use the filtrate from Lassaigne fusion for evaluation of **halogens evidence**
- for orientation, prove **Belstein test** on the copper wire, in this case



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Belstein test

- heating of analyte on the copper wire
- creation of CuX_2
- the green color of the fire

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- if is the Belstein test positive let‘s do mineralization (combustion) with **calcium oxide!**

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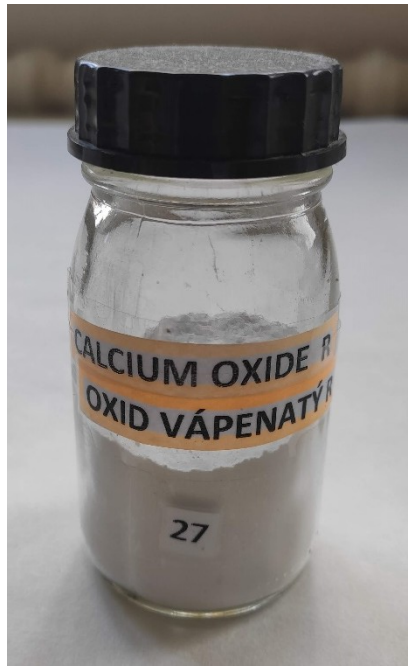
Mineralization with calcium oxide



- mix 0.01 g of your sample with 0.01 g of CaO
- put the mixture into the fusion tube
- cover the mixture with another portion of CaO

The space for the escape of vapors, which are created during the combustion

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Mineralization with calcium oxide

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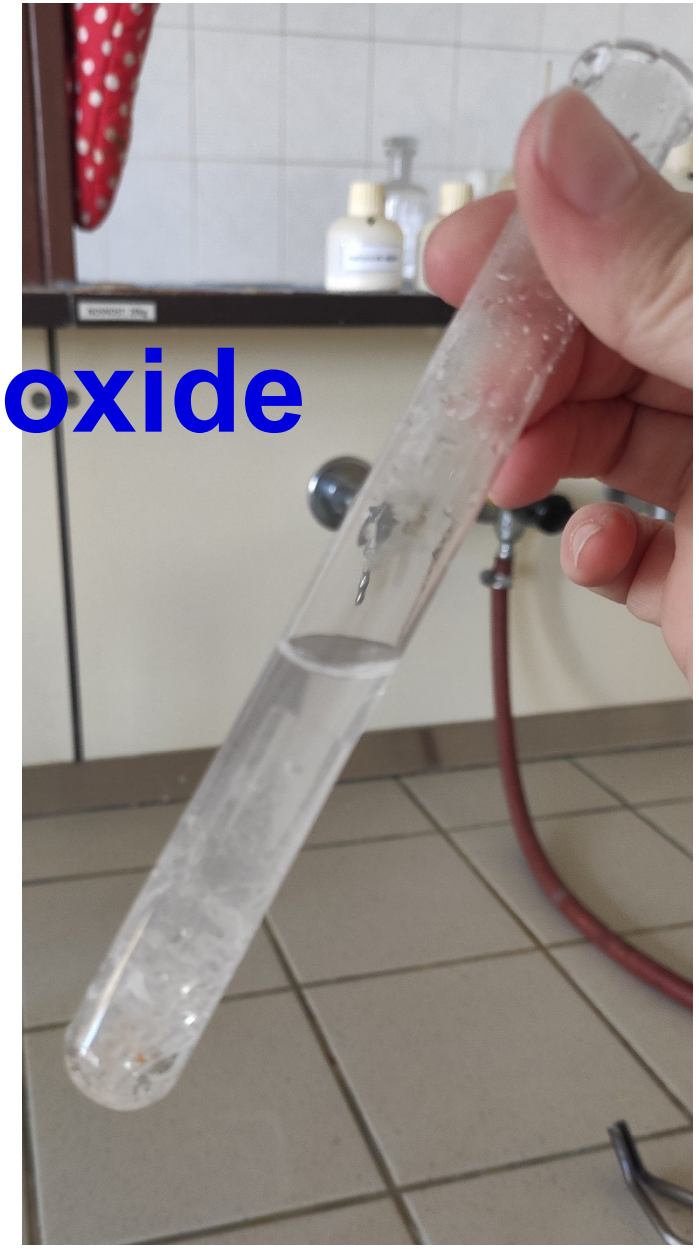
Mineralization with calcium oxide

- hot fusion tube is put into the test tube containing distilled water (about 15 mL)
- the content of the broken fusion tube is released into the water



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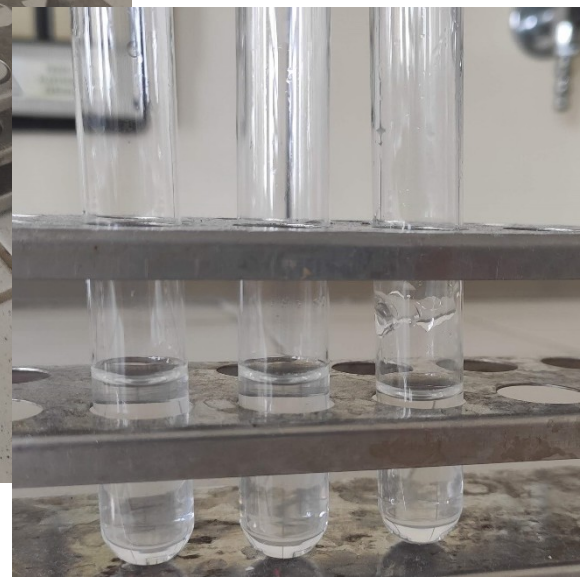
Mineralization with calcium oxide

- the content of the test tube is filtrated
- the filtrate is divided into 3 parts
- continue as is described on page no. 11



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Now you know which elements your sample contains and now you will continue by solubility testing

Solubility