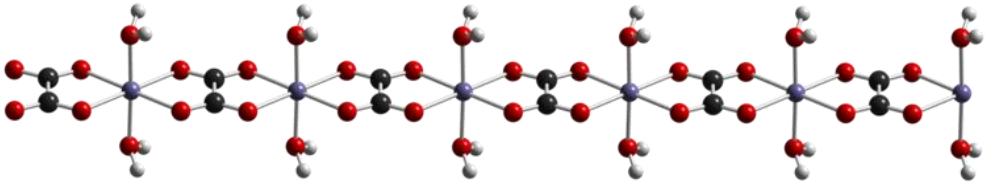
Preparation of iron(II) oxalate dihydrate.

Iron(II) oxalate dihydrate is yellow microcrystalline, poorly soluble in water and insoluble in common organic solvents. It can be prepared by the precipitation reaction of soluble iron(II) salt with soluble oxalate or oxalic acid.

$$\mathbf{Fe^{2+}(aq) + C_2O_4^{2-}(aq) + 2\ H_2O(l) \longrightarrow FeC_2O_4 \cdot 2H_2O(s)}$$



Structure of solid iron(II) oxalate dihydrate.

Work

Prepare 0,0400 moles of iron(II) oxalate dihydrate.

Chemicals

- ammonium iron(II) sulphate hexahydrate, Mohr's salt, $Fe(NH_4)_2(SO_4)_2 \cdot 6\ H_2O$
- oxalic acid dihydrate, $(COOH)_2 \cdot 2\ H_2O$

Procedure

To the well stirred 20% aqueous solution of ammonium iron(II) sulphate acidified with few drops of 2% sulphuric acid we add 150 % of stoichiometric amount of 10% aqueous solution of oxalic acid. A beautiful yellow precipitation will form. Bring the resulting mixture carefully to a boil, then stop heating and let the precipitation settle. Repeat decantation twice using hot water. Filter out the pure product on the Büchner funnel and rinse it thoroughly on the filter with hot water and finally with ethanol. Dry the product in the open air.

Pyrophoric iron – Test tube experiment

Gentle heating of yellow iron(II) oxalate dihydrate in a test tube leads to four oxides:

$$FeC_2O_4 \cdot 2H_2O(s) \xrightarrow{\Delta T} FeO(s) + CO(g) + CO_2(g) + 2\ H_2O(g)$$

Heated mixture changes its colour to black because of black iron(II) oxide.
Intensive heating of iron(II) oxalate dihydrate leads to formation of very fine powdered iron.

$$FeC_2O_4(s) \xrightarrow{\Delta T} Fe(s) + 2\ CO_2(g)$$

Very great surface of the fine iron makes it very reactive. In the test tube, where the formed carbon dioxide creates inert atmosphere, iron has no contact with air. Carbon dioxide ($M \approx 44$ g mol^{-1}) is heavier than air ($M \approx 29$ g mol^{-1}). When we pour the content of the test tube on a non-flammable pad the hot powdered iron starts to react with air violently. The great amount of energy released during the reaction, forms small sparks in the darkness.

$$4\ Fe(s) + 3\ O_2(g) \longrightarrow 2\ Fe_2O_3(s) \qquad \Delta_r H^{\varnothing} = -1648{,}4\ \text{kJ mol}^{-1}$$

The hot finely powdered iron is sometimes called pyrophoric, derived from Greek words *πυρ (pyr)* = fire, *φέρω (féro)* = to bear).

Safety instructions

***Ammonium iron(II) sulfate hexahydrate* – $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$**

R36 Irritating to eyes.
S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
S37/39 Wear suitable gloves and eye/face protection.

***Iron(II) sulfate heptahydrate* – $FeSO_4 \cdot 7H_2O$**

R22 Harmful if swallowed.

***Oxalic acid dihydrate = Ethanedioic acid dihydrate* – $(COOH)_2 \cdot 2H_2O$**

R21/22 Harmful in contact with skin and if swallowed.
S2 Keep out of the reach of children
S24/25 Avoid contact with skin and eyes.

***Sulfuric acid* – H_2SO_4**

R23 Toxic by inhalation.
R34 Causes burns.
R49 May cause cancer by inhalation.
S23 Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer)
S45 In case of accident or if you feel unwell seek medical advice immediately (show the label where possible)
S36/37/39 Wear suitable protective clothing, gloves and eye/face protection.

***Iron(II) oxalate dihydrate* – $Fe(COO)_2 \cdot 2H_2O$**

R21/22 Harmful in contact with skin and if swallowed.
S24/25 Avoid contact with skin and eyes.