Preparation of lead(II) iodide

Lead(II) iodide forms intensive orange crystals. It is at least soluble of all lead(II) halogenides. In 100 g of water dissolves 0,44 g at 100 °C and only 0,04 g at 0 °C. The crystals forming during the cooling of a saturated solution and falling slowly down to the bottom of a beaker are sometimes called the „golden rain."

reaction $\mathbf{Pb(NO_3)_2(aq) + 2\,KI(aq) \longrightarrow 2\,KNO_3(aq) + PbI_2(aq)}$

crystallization $\mathbf{PbI_2(aq) \xrightarrow{cooling} PbI_2(s)}$

Work

Prepare lead(II) iodide by non-isothermal crystallization.

Chemicals

- lead(II) nitrate, $Pb(NO_3)_2$, white crystalline
- potassium iodide, KI, white crystalline
- nitric acid, HNO_3, water solution, $w(HNO_3) = 0{,}65$

Procedure

- Dissolve 0,33 g of lead(II) nitrate in a beaker with 70 cm^3 of distilled water. (Fig. 1).
- If a white turbidity appears add some drops of concentrated nitric acid until the turbidity disappears.
- In another beaker dissolve 0,33 g of potassium iodide in 70 cm^3 of distilled water (Fig. 1). Be careful to add iodide in a small excess to provide participation of dissolved poisonous lead(II) ions in form of lead(II) iodide.
- Filter both prepared solutions separately (Fig. 2).
- Bring both solutions to boil (Fig. 3) and mix them together in a bigger beaker (Fig. 4). Place the beaker in a cool part of laboratory (Fig. 5).
- In a while small golden crystals appear in solution.
- After cooling to room temperature filter out the shiny yellow crystals at diminished pressure on a Büchner funnel with a suction flask connected to a vacuum pump. (Fig. 6).
- Filtrate (saturated solution of lead(II) iodide at room temperature) pour from a suction flask into a special collecting flask (ask the teacher). Spread the filter cake (solid yellow product on a filter) with a spatula in a Petri dish where it dries out in short time. Scrape off the dry product from the Petri dish to a watch glass and weigh it.
- Calculate a theoretical yield of lead(II) iodide and compare it with experimental weight. Try to explain the differences between both values.

Warning! Pay special attention to a safety vessel (Fig. 6), which must always be connected between a suction flask and a vacuum pump. If a water exhauster is used instead of a vacuum pump then a safety vessel must be connected in the opposite direction.

Fig. 1 Preparation of solutions

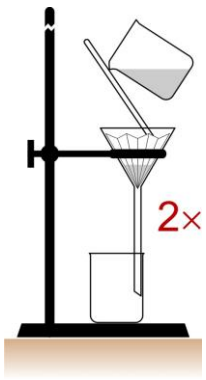

Fig. 2 Filtration at normal pressure

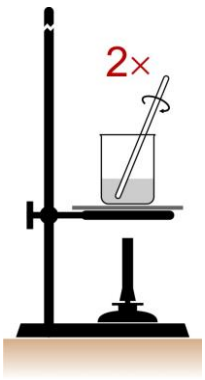

Fig. 3 Heating of solutions

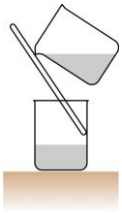

Fig. 4 Mixing (reaction)

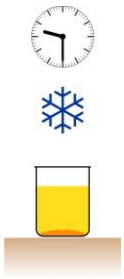

Fig. 5 Cooling (crystallization)

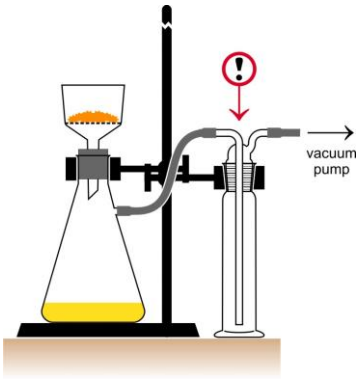

Fig. 6 Filtration at diminished pressure. A safety vessel must be used to protect a pump.

Safety instructions

***Lead(II) nitrate* – $Pb(NO_3)_2$**

R8	Contact with combustible material may cause fire.
R33	Danger of cumulative effects.
R23/25	Toxic by inhalation and if swallowed.
S13	Keep away from food, drink and animal feedingstuffs
S17	Keep away from combustible material
S20/21	When using do not eat, drink or smoke.

***Nitric acid* – HNO_3**

R8	Contact with combustible material may cause fire.
R14	Reacts violently with water.
R34	Causes burns.
R23/24/25	Toxic by inhalation, in contact with skin and if swallowed.
S17	Keep away from combustible material
S26	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
S27	Take off immediately all contaminated clothing
S36/37/39	Wear suitable protective clothing, gloves and eye/face protection.

***Potassium iodide* – KI**

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***Potassium nitrate* – KNO_3**

R8	Contact with combustible material may cause fire.
S16	Keep away from sources of ignition – No smoking
S41	In case of fire and/or explosion do not breathe fumes

***Lead(II) iodide* – PbI_2**

R33	Danger of cumulative effects.
R61	May cause harm to the unborn child.
R62	Possible risk of impaired fertility.
R20/22	Harmful by inhalation and if swallowed.
R50/53	Very toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
S45	In case of accident or if you feel unwell seek medical advice immediately (show the label where possible)
S53	Avoid exposure – Obtain special instructions before use
S60	This material and its container must be disposed of as hazardous waste
S61	Avoid release to the environment. Refer to special instructions/safety data sheet