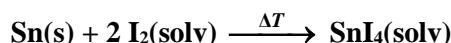


Preparation of tin(IV) iodide

Tin(IV) iodide is a shiny orange crystalline substance, well soluble in organic solvents, *e. g.* chloroform, carbon tetrachloride, benzene, cyclohexane, ethanol, acetone and diethyl ether. In solid and gaseous phases as well as in solutions tin(IV) iodide exists in form of tetrahedral molecules SnI_4 . It is a typical molecular halogenide. It has low melting point (144,5 °C) and at normal pressure sublimates at 180 °C. Tin(IV) iodide hydrolyses in water, where toward water (Lewis base) it acts as Lewis acid.

Tin(IV) iodide is commonly prepared by the reaction of tin with iodine dissolved in nonpolar organic solvents, *e. g.* chloroform, at higher temperature



We obtain the crystals of tin(IV) iodide by cooling and/or evaporating its chloroform solution.

Work

Prepare tin(IV) iodide by the reaction of 1,50 g of tin with 1,50 g of iodine in chloroform.

Chemicals

- tin, Sn, granules or thin wire,
- iodine, I_2 , dark-grey crystals with metallic shine,
- chloroform, CHCl_3 , colourless liquid,
- ethanol, $\text{CH}_3\text{CH}_2\text{OH}$, colourless liquid,
- acetone, CH_3COCH_3 , colourless liquid.

Procedure

Heat the reaction mixture (tin, iodine, and chloroform) in a water bath (b. p. of chloroform is 61 °C) in an absolutely dry apparatus consisting of an appropriate flask (100 mL) and reflux condenser (Fig. 1). Apparatus should be dried with ethanol or acetone.

Cut small pieces of tin from thin tin plate or wire to enlarge the reaction surface. Tin must be used in *cca* 200 % excess according to the reaction stoichiometry.

Be careful! You should work with iodine and chloroform in a tight apparatus or in a well-ventilated room. They are volatile substances and their fumes are very toxic. Iodine irritates skin and human respiratory system.

Put 1,50 g of tin finely cut into pieces into a ground glass flask. Add 1,50 g of iodine dissolved in 15 cm³ of chloroform and fix the reflux condenser. Slowly heat the dark-violet mixture in water bath. In a short time, the violet fumes of iodine in the condenser disappear as iodine reacts with tin. The reaction is finished when colourless drops of pure solvents condense in the condenser and reaction mixture is transparent and of light orange colour.

Filter the hot solution to remove the excess of tin directly to a dry crystallization dish. Cover the crystallization dish with a Petri dish and put it to ice bath. After cooling separate the crystals of tin(IV) iodide using Büchner funnel by vacuum filtration. Wait a while until entire chloroform evaporates from the filter cake and weight prepared dry product.

Warning! Collect all filtrates containing chloroform in a special vessel for further distillation.

Wash unreacted tin with small portion of ethanol and subsequently with acetone, dry it on air and give it back to your teacher. Wash glassware used during the synthesis in a similar way, with ethanol and acetone. Do not use water for cleaning, because tin(IV) iodide hydrolyses to form insoluble and badly cleanable tin(IV) oxide on the glassware surface.

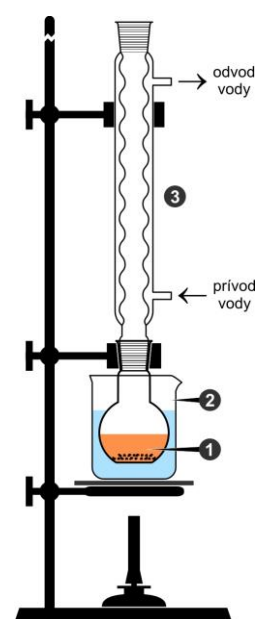


Fig. 1 Apparatus for preparation of SnI_4 .

- 1 – flask with reactants,
2 – water bath,
3 – reflux condenser.



boiling reaction mixture



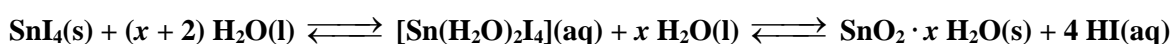
condensing fumes of solvent



dry solid tin(IV) iodide

Hydrolysis of tin(IV) iodide

Tin(IV) iodide hydrolyses in water, where it acts as Lewis acid. Diaqua-tetraiodido tin(IV) complex is temporarily formed, which decomposes forming insoluble white tin(IV) oxide and hydroiodic acid.



Hydroiodic acid is a very strong acid ($K_a = 10^{11}$), therefore it is totally ionized to oxonium cations H_3O^+ and iodide anions I^- . We determine the presence of hydroiodic acid by a universal litmus paper. Iodide anions may be detected by the precipitation reactions with silver and/or lead(II) cations.

Work

Prove that tin(IV) iodide hydrolyses in water.

Chemicals

- tin(IV) iodide, shiny orange crystalline substance,
- silver nitrate, AgNO_3 , 5 wt% aqueous solution,
- lead(II) nitrate, $\text{Pb}(\text{NO}_3)_2$, 5 wt% aqueous solution.

Procedure

Pour small amount of tin(IV) iodide into a test tube with 5 – 10 cm^3 of water. Shake the mixture until the orange iodide disappear. Cloudy white precipitation of tin(IV) oxide will be visible in a test tube. Check the pH with a universal pH paper. Split the solution in a test tube to two another test tubes and add few drops of silver nitrate solution and lead(II) nitrate solution. Explain the observed changes by the chemical reactions.

Safety instructions

Tin – Sn

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Iodine – I₂

- R20/21** Harmful by inhalation and in contact with skin.
- S23** Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer).
- S25** Avoid contact with eyes.

Tin(IV) iodide – SnI₄

- R34** Causes burns.
- R40** Limited evidence of a carcinogenic effect.
- R20/21/22** Harmful by inhalation, in contact with skin and if swallowed.
- R42/43** May cause sensitization by inhalation and skin contact.
- S22** Do not breathe dust.
- S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
- S28** After contact with skin, wash immediately with plenty of (to be specified by the manufacturer).
- S36/37/39** Wear suitable protective clothing, gloves and eye/face protection.

Chloroform – CHCl₃

- R22** Harmful if swallowed.
- R23** Toxic by inhalation.
- R38** Irritating to skin.
- R40** Limited evidence of a carcinogenic effect.
- R48/20/22** Harmful: danger of serious damage to health by prolonged exposure through inhalation and if swallowed.
- S36/37** Wear suitable protective clothing and gloves.