curricuLAB[®] PHYWE

Sublimation and solubility of iodine



The students learn the sublimation and solubility of iodine in practice.

Chemistry	Physical chemistry Phaseequilibrium		ilibrium
Difficulty level	RR Group size	O Preparation time	Execution time
hard	2	10 minutes	20 minutes





General information

Application





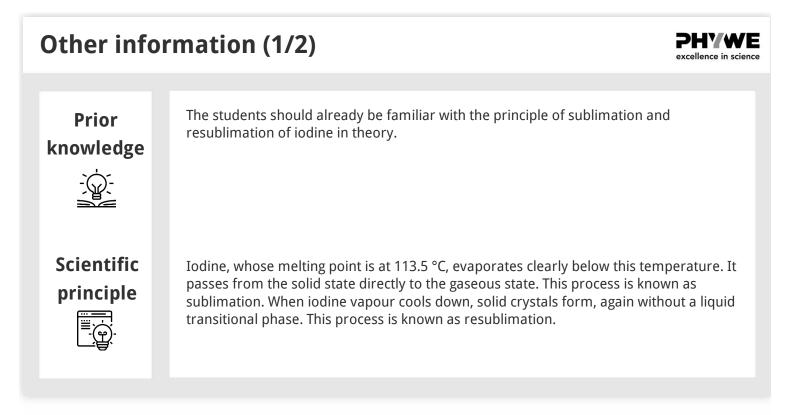
Experimental setup

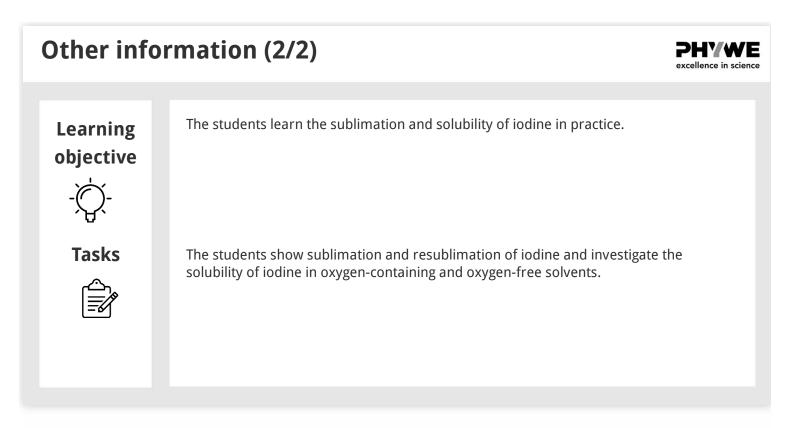
Iodine, whose melting point is at 113.5 $^{\circ}\text{C},$ evaporates clearly below this temperature.

It passes from the solid state directly to the gaseous state. This process is known as sublimation. When iodine vapour cools down, solid crystals form, again without a liquid transitional phase. This process is known as resublimation.

The students will have to deal with these two processes in this experiment.







Safety instructions





- Iodine vapours should not be inhaled! n-pentane and cyclohexane are colourless, highly flammable liquids that cannot be mixed with water. In combination with air, their vapours may form explosive mixtures. Prolonged contact with the skin leads to its degreasing.
- Diethyl ether is a volatile, highly flammable liquid. In combination with air, its heavy vapours may form explosive mixtures. Risk of peroxide formation. Narcotic effect.
- Ethyl alcohol is a highly flammable liquid that can be mixed with water. In combination with air, its vapours may form explosive mixtures.
- $\circ~$ For the H- and P-phrases please refer to the corresponding safety data sheets.
- The general instructions for safe experimentation in science education apply to this experiment.

Theory



Sublimation is the transition of a substance from the solid to the gaseous state without first becoming liquid. Resublimation is the opposite of this.

Iodine, whose melting point is at 113.5 °C, evaporates clearly below this temperature. It passes from the solid state directly to the gaseous state. This process is known as sublimation. When iodine vapour cools down, solid crystals form, again without a liquid transitional phase. This process is known as resublimation.

All of the solvents used during this experiment dissolve iodine considerably better than water. This is why they are also able to extract iodine from aqueous solutions. In oxygen-containing solvents, iodine dissolves with a brown colour (e.g. water, ether, ethyl alcohol), whereas it dissolves with a red-violet colour in oxygen-free solvents (e.g. n-pentane, cyclohexane).



Equipment

Position	Material	Item No.	Quantity
1	Retort stand, h = 750 mm	37694-00	1
2	Right angle boss-head clamp	37697-00	1
3	Universal clamp	37715-01	1
4	Round bottom flask, 250 ml, 2-neck, GL25/12, GL18/8	35843-15	1
5	Condenser, reflux, with 2GI conn.	35900-02	1
6	Closure caps,10, GL18	41220-03	1
7	Crystallizing dish, boro3.3, d = 150 mm	46245-00	1
8	Mortar with pestle, 150 ml, porcelain	32604-00	1
9	Rubber stopper,d=18/14mm,w/o hole	39254-00	5
10	Lab thermometer,-10+150C	38058-00	1
11	Test tube, 160 x 16 mm, 100 pcs	37656-10	1
12	Test tube rack for 12 tubes, holes d= 22 mm, wood	37686-10	1
13	Spoon, special steel	33398-00	1
14	Wash bottle, plastic, 500 ml	33931-00	1
15	Rubber tubing, i.d. 6 mm	39282-00	2
16	Hose clip, diam. 8-16 mm, 1 pc.	40996-02	2
17	lodine resublimed 25 g	30093-04	1
18	Ethyl alcohol, absolute 500 ml	30008-50	1
19	Diethyl ether 250 ml	30007-25	1
20	n-pentane 250 ml	31707-25	1
21	Cyclohexane 1000ml	31223-70	1
22	Water, distilled 5 I	31246-81	1



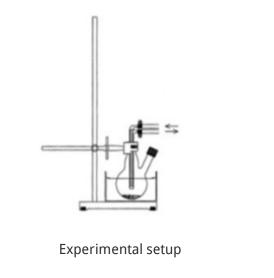
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Setup and procedure

Setup and procedure (1/2)

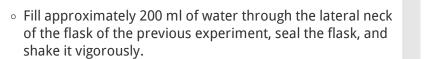
- Grind a small amount of crystalline iodine (covering the tip of a spatula) slightly in a mortar and fill it into a double-neck round bottom flask (250 ml). Insert a condenser for GL screw connectors into the upper neck of this iodine-filled flask and seal the lateral neck with a sealing cap.
- Place the prepared flask as shown in Fig. right into a crystallising dish that is filled with hot water (80-90 °C). Secure the flask in position by way of a clamp. In order to ensure cooling, let cooling water continuously flow through the condenser during the sublimation (secure the hoses with hose clips).



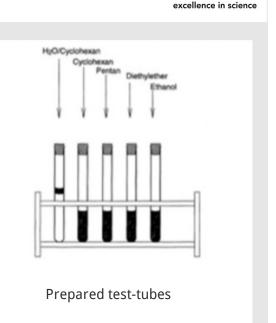


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Setup and procedure (2/2)



- Then fill approximately 10 ml of the aqueous solutions thus prepared into a test tube and add approximately 1 ml of cyclohexane. Seal the test tube with a rubber stopper and shake the mixture in the test tube vigorously.
- Then fill a small amount of iodine into four additional test tubes. Fill some millilitres of cyclohexane into the first test tube, the same amount of n-pentane into the second test tube, diethyl ether into the third test tube, and ethyl alcohol into the fourth test tube. Seal the test tubes with rubber stoppers and shake them vigorously.







Evaluation



Robert-Bosch-Breite 10 37079 Göttingen Tel.: 0551 604 - 0 Fax: 0551 604 - 107

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Evaluation (1/6)

1. Sublimation and resublimation

Observation

The iodine evaporates slowly without passing through the liquid state beforehand. The iodine vapour is violet. After a few minutes, small, rapidly growing iodine crystals can be observed on the cooled wall of the condenser.

Result

Iodine, whose melting point is at 113.5 °C, evaporates clearly below this temperature. It passes from the solid state directly to the gaseous state. This process is known as sublimation. When iodine vapour cools down, solid crystals form, again without a liquid transitional phase. This process is known as resublimation.

Evaluation (2/6)



2. Solubility of iodine in oxygen-containing and oxygen-free solvents

Observation

After step 1: Only a little iodine dissolves in the water. The solution is slightly yellow-brown in colour. The largest part of the iodine remains in an undissolved state on the bottom of the flask. (Note: The solubility of iodine in water is 0.29 g/l at 20 °C.)

After step 2: The aqueous solution is completely discoloured. However, the layer of cyclohexane that is on top of the water is now violet.

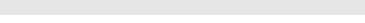
After step 3: In all of the cases, the resulting solutions clearly change their colour due to the uptake of iodine. Diethyl ether and ethyl alcohol have become brown, whereas cyclohexane and n-pentane have become red to violet.



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Evaluation (3/6)



2. Solubility of iodine in oxygen-containing and oxygen-free solvents

Result

All of the solvents used during this experiment dissolve iodine considerably better than water. This is why they are also able to extract iodine from aqueous solutions. In oxygen-containing solvents, iodine dissolves with a brown colour (e.g. water, ether, ethyl alcohol), whereas it dissolves with a red-violet colour in oxygen-free solvents (e.g. n-pentane, cyclohexane).

Notes

The deep brown solution that is based on ethyl alcohol is known as iodine tincture in medicine. However, the medical importance of iodine tincture for the treatment of wounds is rather low since it may trigger intolerances.

Evaluation (4/6)



What process is known as sublimation?

O If a substance passes from the gaseous state directly to the solid state without a liquid transitional phase

- O If a substance passes from the solid state directly to the gaseous state without a liquid transitional phase.
- O If a substance is build up from its smallest parts.

✓ Überprüfen



Evaluation (5/6)	PHYWE excellence in science
What process is known as resublimation?	
O If a substance passes from the solid state directly to the gaseous state without a liquid transphase.	nsitional
O If a substance passes from the gaseous state directly to the solid state without a liquid tran	nsitional phase
O If a substance is split up into its molecules.	
♥ Überprüfen	

Evaluation (6/6)

Summary of the experiment		
All of the	used during this experiment dissolve iodine	solvents
considerably	than water. This is why they are also able to	aqueous solutions
extract iodine from	. In oxygen-containing solvents, iodine	red-violet
dissolves with a	colour (e.g. water, ether, ethyl alcohol),	better
whereas it dissolves with a	colour in oxygen-free solvents	
(e.g. n-pentane, cyclohexane).		brown

Überprüfen

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Folie		Punktzahl/Summe
Folie 15: Sublimation		0/1
Folie 16: Resublimation		0/1
Folie 17: Dissolving iodine		0/5
	Gesamtsumme	0/7