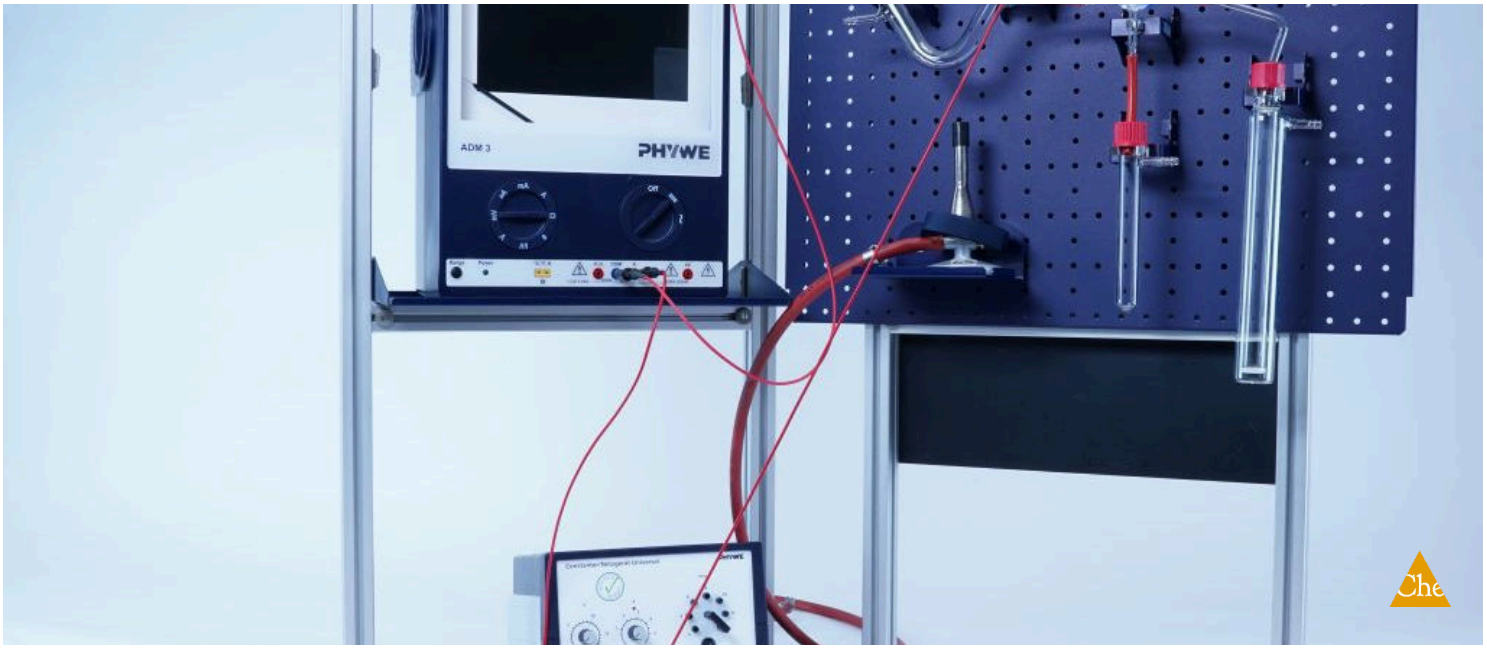
Fused-salt electrolysis



Students learn about fused-salt electrolysis in this experiment.

Chemistry > Inorganic chemistry > Chemistry of metals

Chemistry > Physical chemistry > Electrochemistry > Electrolysis

Difficulty level	Group size	Preparation time	Execution time
hard	1	10 minutes	20 minutes

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General information

Application

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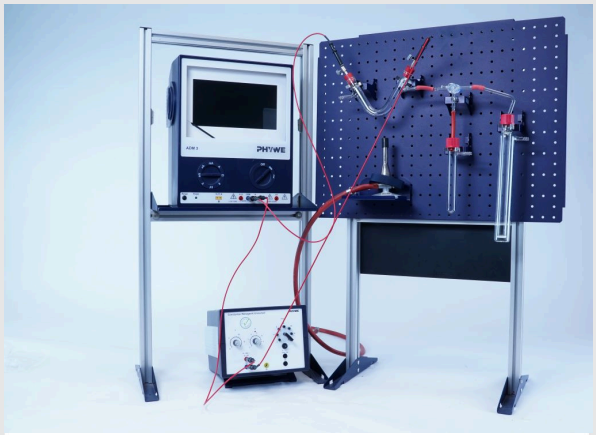

Test setup

In this experiment, the fused-salt electrolysis of lead(II) chloride is studied, with chlorine being deposited at the anode and lead at the cathode.

The liquid lead collects in the lower part of the V-tube, while the chlorine is fed into a wash bottle via the lateral preparation nozzle. There it is detected by decolorizing a potassium iodide starch solution located there.

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Other information (1/4)

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Prior

The students should already be familiar with electrolysis in theory and in particular with fused-salt electrolysis.

Principle

In fused-salt electrolysis, two electrodes are placed in a molten electrolyte and connected to a voltage source (direct current). The positively charged cations (of the melt) migrate to the negative pole (cathode) of the current source, which are reduced at the electrode. The positive pole (anode) attracts negatively charged ions (anions), which are oxidized. Therefore, reduction takes place at the cathode and oxidation at the anode.

Other information (2/4)

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Learning

Students learn about fused-salt electrolysis in this experiment.

Tasks

Students investigate the fused-salt electrolysis of lead(II) chloride.

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Other information (3/4)

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Notes

To completely remove the molten salt from the V-tube, place it in boiling water. Lead(II) chloride is readily soluble in hot water. You can also fill the V-tube with double to triple the amount of salt. This will make the color of the chlorine and the amount of lead deposited more obvious to students. However, in the context of waste prevention - especially of heavy metals - this is not recommended. As an alternative to lead chloride, sodium hydroxide can also be used. Oxygen and sodium are formed. While oxygen can be collected pneumatically and detected with a smoldering chip sample, elemental sodium causes some problems. It can be detected by the characteristically violent reaction with water and the resulting basic pH value. However, getting the sodium out of the V-tube without residual sodium hydroxide is problematic. Overall, fused-salt electrolysis with lead chloride is more descriptive. In addition to the sodium hydroxide, the following would be required:

Other information (4/4)

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Tub 34563-00 1	Evaporation tray, d = 100 mm 32518-00 1
Insert for tubs 34567-00 1	\Beaker, 250 mL, high form 36002-00 3
Stand cylinder 34217-00 1	Spray bottle, 500 mL 33931-00 1
Phenolphthalein solution, 1%, 100 mL 31714-10 1	Glass funnel, = 80 mm 34459-00 1
Wood chips 39126-20 1	Round filter, d = 110 mm, 1 from 32977-04 1
For this, the following positions would be eliminated:	Glass rod, d = 4 mm, l = 200 mm 40485-02 1
Clamp holder, d = 18...25 mm 45520-00 2	Lead(II) chloride, 250 g 31117-25 1
Clamp holder, d = 8...10 mm, rotatable 45522-00 1	Starch, soluble, 100 g 30227-10 1

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Safety instructions (1/2)

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- During the experiment, all persons in the room must wear protective goggles and gloves!
- During the experiment, the toxic and corrosive gas chlorine is generated in the experimental apparatus. However, since it is collected at one end of the apparatus by a glass tube filled with sodium hydroxide solution as an adsorption solution, the experiment can also be carried out outside a fume hood with due care.
- 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.

Safety instructions (2/2)

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- Electrolysis produces the toxic metal lead. It is also harmful to health when swallowed and inhaled from dust, can harm the child in the womb, and can potentially affect reproductive ability.
- Lead(II) chloride is harmful by inhalation and ingestion and may harm the unborn child and can potentially affect reproductive ability.
- There is a risk of cumulative effects. Avoid exposure. Obtain special instructions before use.

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Theory

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Molten salt electrolysis is a manufacturing process that is mainly used for the preparation of base metals or elements that are not stable in aqueous solution. Fused-salt electrolysis (like any electrolysis) is a redox reaction forced by electrical energy.

Fused-salt electrolysis is a special process for the preparation of base and very reactive substances. Unlike other (classical) electrolysis processes, fused-salt electrolysis does not use a solution containing water. Instead, a molten salt is used as the electrolyte.

In fused-salt electrolysis, two electrodes are inserted into a molten electrolyte and connected to a voltage source (direct current).

The positively charged cations (of the melt) migrate to the negative pole (cathode) of the current source, which are reduced at the electrode. The positive pole (anode) attracts negatively charged ions (anions), which are oxidized. Therefore, reduction takes place at the cathode and oxidation at the anode.

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Equipment

Position	Equipment	Item no.	Quantity
1	Frame for complete tests	45500-00	2
2	Back wall for complete tests	45501-00	1
3	Plate for complete tests	45510-00	1
4	Shelf with suspension	45505-00	1
5	Clamp holder, d = 18...25 mm	45520-00	2
6	Clamp holder, d = 18...25 mm, rotatable	45521-00	2
7	Clamp holder, d = 8...10 mm, rotatable	45522-00	1
8	Device carrier with holding magnets	45525-00	1
9	Spring cotter pin, 50 pieces	45530-00	1

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Structure and implementation

Structure (1/2)

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Preparation

A starch/iodide solution should be prepared before starting the experiment.

- To do this, dissolve a heaping spoonful of starch in a beaker with about 50 ml of water. Then boil the solution once briefly and filter it hot.
- Add a spatula tip of potassium iodide to the cooled solution. If the starch dissolves almost completely in the cold water, boiling is not absolutely necessary.
- A slightly more concentrated sodium hydroxide solution is prepared by dissolving a sufficient amount of sodium hydroxide in about 50 ml of water.

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Structure (2/2)

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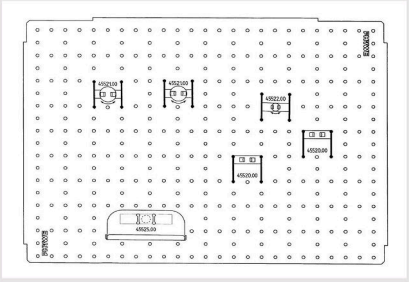
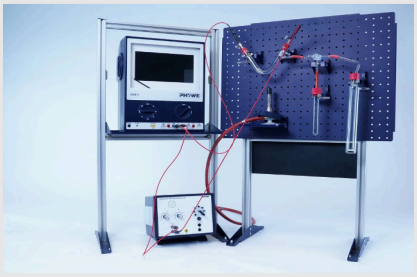
- The holders are attached to the top right of the plate for complete tests according to the illustration.
- Then fill the V-tube with three spoonfuls of bleach chloride and assemble the apparatus as shown in the figure below right. It is attached to the holders.
- The test tube with lateral preparation is filled to about two-thirds with the prepared starch/iodide solution and the gas wash bottle is filled about halfway with concentrated sodium hydroxide solution.





Procedure

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- The left side of the V-tube is closed with the dummy nozzle, the electrodes are not yet inserted. Now vigorously heat the lead chloride with the burner until it melts.
- The gas burner is then adjusted so that the melt just does not solidify, and further bleach chloride is poured into the V-tube until the melt is about 1 cm high at an angle (care must be taken here that the glass tube does not melt, as it may crack during cooling). Only now immerse the electrodes in the melt and electrolyze at about 4 A.
- After the color change in the test tube, the three-way stopcock is adjusted so that any gas produced can only escape through the sodium hydroxide solution. Now continue electrolyzing until sufficient products have been formed.
- After presenting the result, remove the gas burner and place the still hot bleach chloride in a provided heatproof evaporating dish (otherwise the V-tube may crack as the salt expands due to cooling).

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Evaluation

Evaluation (1/5)

Observation

When heated, the white lead chloride melts to form a yellow molten salt. If the heat is too high, part of the lead chloride sublimates and precipitates as white smoke further up the V-tube. After electrolysis begins, a yellow-green gas is formed on the right side and a shiny metallic liquid on the left. The gas colors the solution in the test tube blue to deep blue. The solution in the wash tube does not change color. The metal collects as drops in the knee of the V-tube.

Evaluation (1/2)

The salt is broken down into its components. Chlorine is formed at the anode and lead at the cathode:

$ Oxidation (anode): {2CL^-} \{CL_2} + {2e^-} $

$ Reduction (cathode): {Pb^{2+}} + {2e^-} \{Pb} $

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Evaluation (2/5)

Evaluation (2/2)

The chlorine oxidizes the iodide ions to iodine, which forms a blue complex with the starch that appears deep blue to black at very high concentrations:

$ {Cl_2} + {2I^-} \{I_2} + {2CL^-} $

The sodium hydroxide solution is used to absorb excess chlorine gas. In the process, chlorine disproportionates to form hypochlorite and chloride. The resulting iodine is reduced to iodide with sodium thiosulfate and the solution is poured into the sink.

Evaluation (3/5)

Drag the words into the correct boxes!

When heated, the _____ lead chloride melts to a _____ molten salt. If the heat is too high, some of the lead chloride sublimes and precipitates as white smoke further up the V-tube. After _____ begins, a yellow-green gas is formed on the right side and a shiny metallic liquid on the left. The gas colors the solution in the test tube blue to deep blue. The solution in the wash tube does _____ change color. The metal collects as drops in the knee of the V-tube.

not
yellow
electrolysis
white

Check

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Evaluation (4/5)

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What is created at the anode?

- [ ] Iodine is formed at the anode: $Cl_2 + 2I^- \longrightarrow I_2 + 2CL^-$
- [ ] Chlorine is formed at the anode: $2CL^- \longrightarrow CL_2 + 2e^-$
- [ ] None of the answers is correct. The salt is rebuilt at the anode, forming a yellow molten salt.
- [ ] Lead is formed at the anode: $Pb^{2+} + 2e^- \longrightarrow Pb$

Check

Evaluation (5/5)

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What is the sodium hydroxide solution used for?

- ○ The sodium hydroxide solution is used to deliver the required chlorine gas.
- ○ None of the answers is correct.
- ○ The sodium hydroxide solution is used to absorb excess chlorine gas.
- ○ Due to its strongly hygroscopic character, the caustic soda serves to introduce additional water.

Check

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Slide	Score/Total
Slide 18: Electrolysis	0/4
Slide 19: Anode	0/2
Slide 20: Caustic soda	0/1

Total 0/7

Solutions Repeat

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