Electrolysis of a melt (Item No.: P1310500)

Curricular Relevance

Area of Expertise: Chemistry
Education Level: University
Topic: Physical Chemistry
Subtopic: Electro Chemistry
Experiment: Electrolysis of a melt

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Difficult	10 Minutes	20 Minutes	1 Student

Additional Requirements:

Experiment Variations:

Keywords:

Principle and equipment

Principle

Notes

To completely remove the salt melt from the V-tube, lay the tube in boiling water. Lead(II) chloride is very soluble in hot water. The V-tube can be filled with double or treble the stated amount of salt. The colour of chlorine and the quantity of lead deposited are then clearer for the students. This is not recommended, however, because of the problem of waste disposal - particularly with heavy metal waste. Sodium hydroxide can be used as an alternative to lead chloride. Oxygen and sodium are then produced. Whereas oxygen can be collected pneumatically and detected with a burning splint, elementary sodium is somewhat of a problem. It can be detected by the characteristic violent reaction with water, and the resulting alkaline pH value. But is difficult to remove the sodium from the V-tube without remains of sodium hydroxide. In all, the electrolysis of lead chloride melt is more illustrative. For the procedure with sodium hydroxide, the following are additionally required:

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Trough, AR-glass, d = 190 mm	34563.00	1
Shelf for trough	34567.00	1
Cylinder, coarsely ground rim, h = 400 mm	34217.00	1
Phenolphthalein solution, 1%, 100 ml	31714.10	1
Wood splints, pack of 100 (1 only)	39126.20	1
The following materials are then not required:		
Clamping holder, d = 18...25 mm	45520.00	2
Clamping holder, d = 8...10 mm, turnable	45522.00	1
Glass stopcock, T-shaped	36731.00	1
Test tube, DURAN, with hose connection, GL 25/8, 22 x 180 mm	36330.15	1
Glass tube, straight, l = 150 mm	36701.64	1
Gas washing bottle, 100 ml, with frit	36699.00	1
Glass tube, right-angled, l = 85 + 60 mm	36701.52	1
Evaporating dish, d = 100 mm	32518.00	1
Beaker, DURAN, tall form, 250 ml	36002.00	3
Wash bottle, 500 ml	33931.00	1
Funnel, glass, d = 80 mm	34459.00	1
Circular filters for qualitative work, 110 mm, pack of 100	32977.04	1
Stirring rod, BORO 3.3, l = 200 mm, d = 4 mm	40485.02	1
Lead chloride, 500 g	31117.25	1
Starch, soluble, 100 g	30227.10	1
Potasium iodide, 50 g	30104.05	1
Sodium thiosulphate pentahydrate, 250 g	30169.25	1

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Equipment

Position No.	Material	Order No.	Quantity
1	Multimeter ADM2, demo., analogue	13820-01	1
2	PHYWE power supply, universal DC: 0...18 V, 0...5 A / AC: 2/4/6/8/10/12/15 V, 5 A	13500-93	1
3	Frame for complete experiments	45500-00	2
4	Shelf with hanging device	45505-00	1
5	Lead-II chloride 500 g	31117-50	1
6	Wash tube with fritted disc	36699-00	1
7	Panel for complete experimental setups	45510-00	1
8	Apparatus carrier w. fix. magnet	45525-00	1
9	V-tube for electrolysis, PN19	37027-00	1
10	Clamping holder,turnable,8-10mm	45522-00	1
11	Teclu burner, DIN, natural gas	32171-05	1
12	Clamping holder, turnable, 18-25 mm	45521-00	2
13	Test tube GL25/8, w.hose connec.	36330-15	1
14	Rear-cover for compl.-exp. panel	45501-00	1
15	Clamping holder,18-25mm	45520-00	2
16	Stopcock,3-way,t-shaped, glass	36731-00	1
17	Glass tube,right-angled, 10 pcs.	36701-52	1
18	Starch,soluble 100 g	30227-10	1
19	Potassium iodide 50 g	30104-05	1
20	Spring plugs, 50 off	45530-00	1
21	Graphite electrode,d=7,l=150,6pc	44512-00	1
22	Sodium hydroxide, pellets, 500 g	30157-50	1
23	Sodium thiosulphate 5-hydr.250 g	30169-25	1
24	Contact socket f.bar electrodes	45283-00	2
25	Water, distilled 5 l	31246-81	1
26	G-clamp	02014-00	4
27	Safety gas tubing, DVGW, sold by metre	39281-10	2
28	Porcelain dish 140ml, d 100mm	32518-00	1
29	Connecting cord, 32 A, 1000 mm, red	07363-01	1
30	Rubber caps, pack of 20	02615-03	1
31	Glass tubes,straight, 150 mm, 10	36701-64	1
32	Spoon + spatula, steel, l=200mm	46948-00	1
33	Connecting cord, 32 A, 750 mm, red	07362-01	1
34	Connecting cord, 32 A, 750 mm, blue	07362-04	1
35	Glass beaker DURAN®, tall, 250 ml	36004-00	3
36	Circular filter,d 110 mm,100 pcs	32977-04	1
37	Lighter f.natural/liquified gases	38874-00	1
38	Funnel, glass, top dia. 80 mm	34459-00	1
39	Wash bottle, plastic, 500 ml	33931-00	1
40	Rubber tubing, i.d. 6 mm	39282-00	1
41	Hose clip f.12-20 diameter tube	40995-00	2
42	Rubber stopper, d = 22/17 mm, 1 hole	39255-01	2
43	Glass rod,boro 3.3,l=200mm, d=4mm	40485-02	1

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Safety information

Chlorine, a poisonous and corrosive gas, is generated in the experimental apparatus during this experiment. However, as it is captured at the end of the apparatus by an adsorption solution (caustic soda in a glass tube), the experiment can also be carried out outside of a fume cupboard, provided appropriate care is taken. Lead, a poisonous metal, is produced during electrolysis. It is harmful to health when swallowed, or when dust from it is inhaled, can harm unborn babies and possibly impair reproductive ability. There is the danger of a cumulative effect. Avoid exposure to it. Follow appropriate instructions before handling it. Lead chloride is harmful to health when inhaled of swallowed, can harm unborn babies and possibly impair reproductive ability. There is the danger of a cumulative effect. Avoid exposure to it. Follow appropriate instructions before handling it.

Sodium hydroxide is highly corrosive to skin, eyes and mucous membranes. Dust from it irritates respiratory organs. Do not inhale vapours or dusts. Avoid contact of the chemicals with skin and eyes.Wear suitable protective clothing, protective gloves and protective goggles when working with them! Observe the detailed information on safety measures in the appendix.

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Set-up and procedure

Set-up

A starch-iodide solution is to be prepared prior to the start of the experiment. Dissolve a heaped spoonful of starch in a beaker containing about 50 ml of water. Boil this briefly and filter it hot. After cooling, add a spatula tip of potassium iodide to the cooled solution. Should the starch dissolve almost completely in cold water, then boiling is not absolutely necessary. Prepare a somewhat more concentrated caustic soda solution by dissolving a sufficient amount in 50 ml of water.

Position the clamping holders on the panel for complete experiments as shown in Fig. 2. Then fill the V-tube with three spoonfuls of lead chloride and assemble the equipment as shown in Fig. 1. Fix it in position with the clamping holders. Fill the test tube with side arm two thirds full with the prepared starch-iodide solution, and the gas wash bottle about half-full with concentrated caustic soda. Remove the Velcro band from the apparatus carrier under the Vtube, so that this is not damaged by the heat from the burner. Secure the burner by pushing each side of its foot under the rubber band fixed to the bottom of the apparatus carrier. The burner must be tightly and safely held. Adjust the T-shaped stopcock so that gas can at first only flow through the test tube.

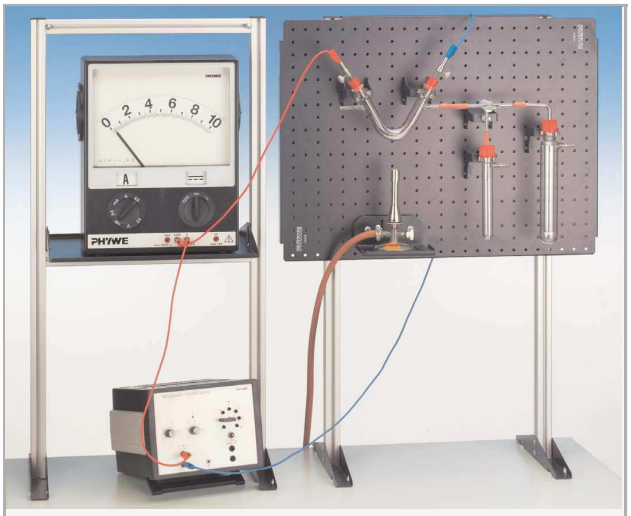

Fig. 1

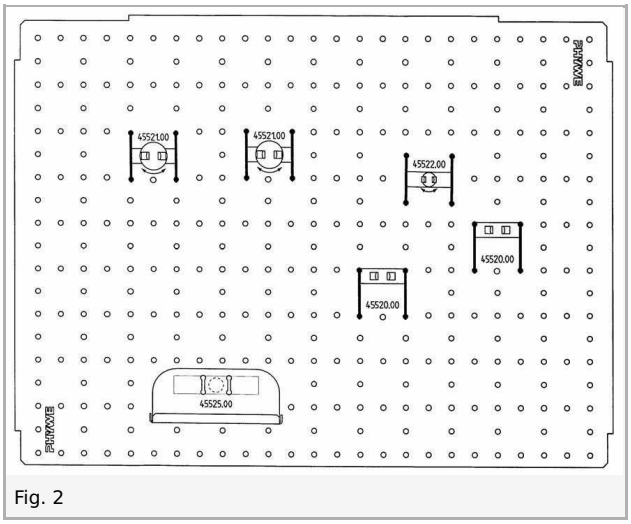

Fig. 2

Procedure

Close the left side of the V-tube with a closing cap, but do not insert the electrodes. Strongly heat the lead chloride until it melts. Following this, adjust the gas burner so that the melt just does not quite solidify, and add further lead chloride until the melt is about 1 cm higher than the bend. Now immerse the electrodes in the melt and carry out electrolysis at roughly 4 A. When the colour in the set tube has changed, set the Tshaped stopcock so that the gas that is generated can only flow through the caustic soda. After presentation of the results, remove the gas burner and transfer the hot lead chloride to an evaporating dish placed in readiness (otherwise the V-tube could crack because of the expansion of the salt on cooling).

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Observation and evaluation

Observation

When white lead chloride is heated, it melts to a yellow salt melt. When heated too much, part of the lead chloride sublimes, giving a white smoke that deposits in the V-tube further up. When electrolysis is started, a yellowish-green gas is evolved on the right side, and a liquid with a metallic sheen is produced on the left side. The gas colours the solution in the test tube blue to deep blue. The colour of the solution in the wash bottle does not change. The metal collects as drops in the bend of the V-tube.

Evaluation

The salt is decomposed to it constituent elements. Chlorine is formed at the anode, and lead at the cathode.

Oxidation (Anode): $2\,Cl^- \rightarrow Cl_2 + 2\,e^-$

Reduction (Cathode): $Pb^{2+} + 2\,e^- \rightarrow Pb$

Chlorine oxidises iodide ions to iodine, which forms a blue complex with starch, appearing deep blue to black at very high concentrations:

$$Cl_2 + 2\,I^- \rightarrow I_2 + 2\,Cl^-$$

The caustic soda serves to take up excess gaseous chlorine, that disproportions to hypochlorite and chloride. Reduce any remaining iodine to iodide with sodium thiosulphate and pour it to drain.

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