Reduction - reducing agent - redox process (Item No.: P3100300)

Curricular Relevance

Area of Expertise:	Education Level:	Topic:	Subtopic:	Experiment:
Chemistry	University	General Chemistry	Redox Reactions	Reduction - reducing agent - redox process

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Intermediate	1 Hour	1 Hour	2 Students

Additional Requirements:

Experiment Variations:

Keywords:

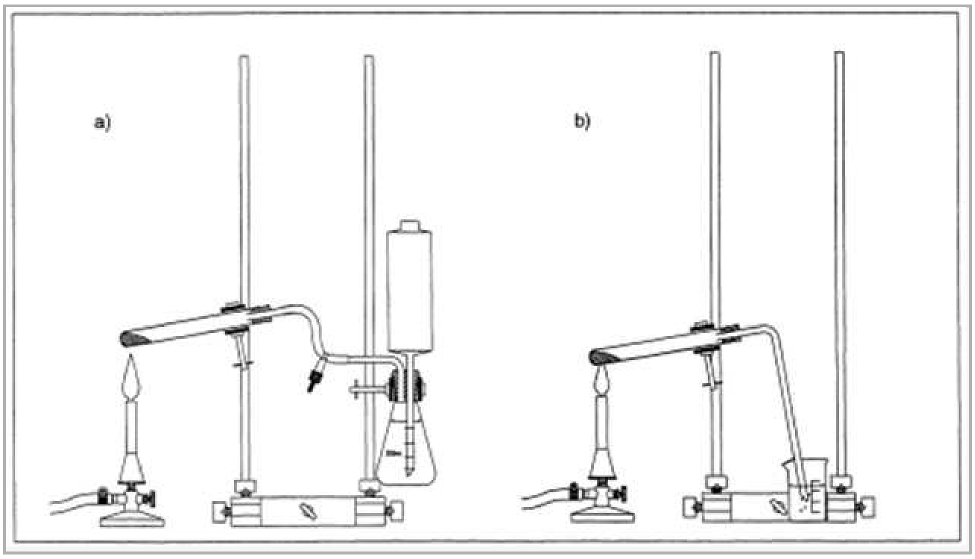
Reduction, Oxidation, Redox reaction, Lead, Iron, Thermolysis

Task and equipment

Introduction

Principle:
The reduction, as the reversal of the oxidation, can be achieved thermally or with the aid of a reducing agent.
Some metal oxides can be decomposed into the metal and oxygen under the influence of thermal energy. In the case of less noble metals, a reducing agent is required for obtaining the elements. The redox processes during the preparation of lead demonstrate the relationship between oxidation and reduction. By way of this experiment it can be shown that during the reduction of an oxide the reducing agent itself is oxidised: hydrogen to water, carbon to carbon dioxide. A reduction process is always coupled with an oxidation process, which is why this type of reaction is referred to as a redox reaction.

Safety information:
Hydrogen is a colourless, flammable gas that forms explosive mixtures with air. In the case of reactions in apparatuses, these must be tested for the presence of oxygen (oxyhydrogen test). Disposal: Collect any solutions that contain heavy-metal ions in a collecting vessel for heavy-metal salt solutions. Solid residues that contain heavy metals or their ions must also be collected in this vessel. Me-tal powders that are produced by reduction are self-igniting! Do not dispose of them in the normal waste. Instead dissolve them in hydrochloric acid!
When fed into lime water: Risk of blowback!



Safety instructions

Hydrogen is a colourless, flammable gas that forms explosive mixtures with air. In the case of reactions in apparatuses, these must be tested for the presence of oxygen (oxyhydrogen test).
Pyrophoric iron is self-igniting! Do not dispose of it in the normal waste! Dissolve any residues in hydrochloric acid!
Disposal: Collect any solutions that contain heavy-metal ions in a collecting vessel for heavy-metal salt solutions. Solid residues that contain heavy metals or their ions must also be collected in this vessel. Me-tal powders that are produced by reduction are self-igniting! Do not dispose of them in the normal waste. Instead dissolve them in hydrochloric acid!
When fed into lime water: Risk of blowback!

Calcium hydroxide
H315: Causes skin irritation.
H318: Causes serious eye damage.
H335: May cause respiratory irritation.
P261: Avoid breathing dust/fumes/gas/mist/vapours/spray.
P280: Wear protective gloves/protective clothing/eye protection/face protection.

Lead(IV) oxide
H272: May intensify fire; oxidizer.
H360: May damage fertility or the unborn child.
P201: Obtain special instructions before use.
P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.
P261: Avoid breathing dust/fumes/gas/mist/vapours/spray.

Lead(II) oxide
H332: Harmful if inhaled.
H360: May damage fertility or the unborn child.
P264: Wash … thoroughly after handling.
P280: Wear protective gloves/protective clothing/eye protection/face protection.

Lead
H332: Harmful if inhaled.
H360: May damage fertility or the unborn child.
P201: Obtain special instructions before use.
P273: Avoid release to the environment.

Hydrogen
H220: Extremely flammable gas.
H280: Contains gas under pressure; may explode if heated.
P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.
P381: Eliminate all ignition sources if safe to do so.

Pyrophoric iron
H228: Flammable solid.

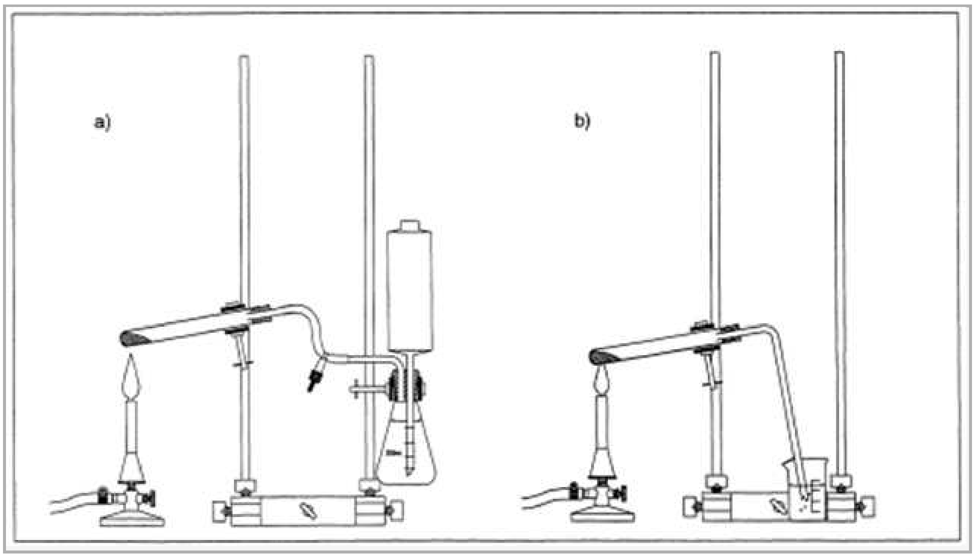
H251: Self-heating; may catch fire.
P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.

Equipment

Position No.	Material	Order No.	Quantity
1	Support base DEMO	02007-55	1
2	Support rod, stainless steel, l = 600 mm, d = 10 mm	02037-00	2
3	Right angle boss-head clamp	37697-00	2
4	Universal clamp	37715-00	2
5	Test tube,180x20 mm, PN19	36293-00	1
6	Rubber stopper, d = 22/17 mm, 1 hole	39255-01	2
7	Glass tube, straight, l=80 mm, 10/pkg.	36701-65	1
8	Glass tubes,right-angled, 10	36701-59	1
9	Gas bar	40466-00	1
10	Pinchcock, width 15 mm	43631-15	1
11	Beaker, high, BORO 3.3, 100 ml	46026-00	1
12	Combustion tube, 300 mm, quartz, ns	33948-01	1
13	Connecting tube IGJ 19/26-GL 18/8	35678-01	2
14	Glass tube,right-angled w.tip,10	36701-53	1
15	Clamp f.ground joint,plastic,NS19	43614-00	2
16	Teflon sleeve IGJ 19, 10 pcs	43616-00	1
17	Porcelain boats, 10 pcs	32471-03	1
18	Magnet, d = 10 mm, l = 200 mm	06311-00	1
19	Rubber tubing, i.d. 6 mm	39282-00	2
20	Porcelain dish 140ml, d 100mm	32518-00	1
21	Teclu burner, DIN, natural gas	32171-05	1
22	Safety gas tubing, DVGW, sold by metre	39281-10	1
23	Hose clip f.12-20 diameter tube	40995-00	2
24	Lighter f.natural/liquified gases	38874-00	1
25	Steel cylinder hydrogen, 2 l, full	41775-00	1
26	Reducing valve for hydrogen	33484-00	1
27	Table stand for 2 l steel cylinders	41774-00	1
28	Wrench for steel cylinders	40322-00	1
29	Protective desk plate 40 x 40 cm	39180-10	1
30	Test tube, 160 x 16 mm, 100 pcs	37656-10	1
31	Wood splints, package of 100	39126-10	1
32	Tweezers,straight,blunt, 200 mm	40955-00	1
33	Spoon, special steel	33398-00	1
34	Circular filter,d 125 mm,100 pcs	32977-05	1
35	Beaker, high, BORO 3.3, 600 ml	46029-00	2
36	Scissors, straight,180 mm	64798-00	1
37	Iron-III oxide, red 500 g	48114-50	1
38	Calcium hydroxide 500 g	30054-50	1
39	Lead-IV oxide -lead diox.- 250 g	31122-25	1
40	Charcoal powder 250 g	30087-25	1
41	Water, distilled 5 l	31246-81	1

Task

Redox process - a chemical reactions in which atoms have their oxidation number changed.
By way of this experiment it can be shown that during the reduction of an oxide the reducing agent itself is oxidised.



Set-up and procedure

Hydrogen is a colourless, flammable gas that forms explosive mixtures with air. In the case of reactions in apparatuses, these must be tested for the presence of oxygen (oxyhydrogen test).
When fed into lime water: Risk of blowback!

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1. Reduction of lead(IV) oxide to lead(II) oxide by thermolysis

Fasten a test tube with a spoonful of lead(IV) oxide in a slightly inclined position to a support stand as shown in Fig. 1a. Connect it to the small gasometer (taken from the gas bar, order no. 40466-00). Heat the lead(IV) with a strong, non-luminous flame. A gas develops. It is collected in the small gasometer. When the gas deve-lopment stops, stop heating the test tube, block the tubing with the pinchcock, and pull it immediately off the glass tube of the test tube. Test the gas with a glowing splint. To do so, fill a test tube with the collected gas by inserting the tubing into the test tube and by opening the pinchcock. Then, insert a glowing wood splint into the gas-filled test tube.

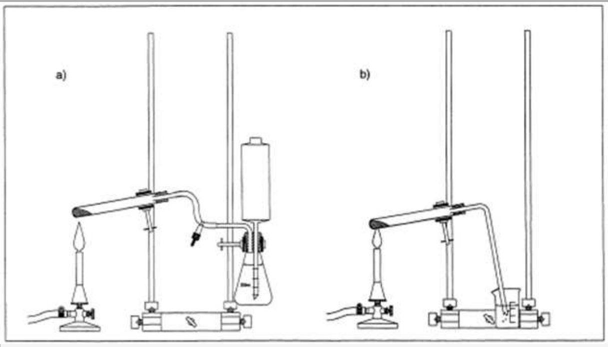

Fig. 1

2. Reduction of lead(II) oxide by way of charcoal to obtain elementary lead

Add half a spoon of charcoal powder to the oxide melt of the first experiment and seal the test tube with a perforated stopper and a right-angled glass tube (Fig. 1b). Immediately continue to heat this lead oxi-de/charcoal mixture and guide the resulting gas in accordance with Fig. 1b into a small beaker containing a calcium hydroxide solution.

3. Reduction of iron oxide including the formation of hydrogen based on pyrophoric iron

Attention! Hydrogen forms explosive mixtures with air! Apparatuses must be tested for the presence of oxygen (oxyhydrogen test). Pyrophoric iron is self-igniting! Do not dispose of it in the normal waste! Dissolve any residues in hydrochloric acid! In accordance with Fig. 2, place a porcelain boat containing iron(III) oxide into a quartz glass tube that is fastened to a support stand in a horizontal position. Connect the supply line and the outlet nozzle (use quartz glass wool to ensure protection against blowback) and seal it with clamps for ground joints. Flush the apparatus with hydrogen and perform an oxyhydrogen test to ensure that the apparatus is free from oxygen. This is absolutely essential prior to igniting the hydrogen at the outlet nozzle. Adjust the hydrogen flow so that the burning flame is approximately 3 cm high. Then, heat the iron(III) oxide strongly in the non-luminous gas flame.

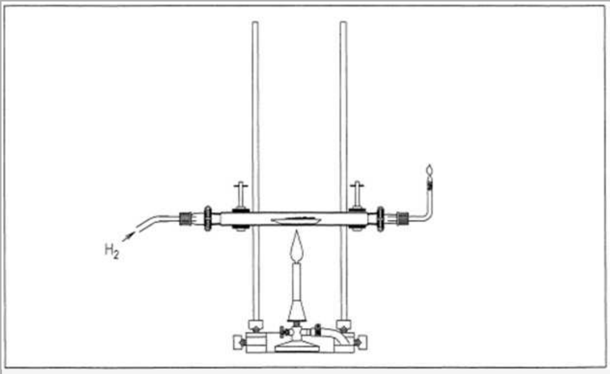

Fig. 2

Results and evaluation

1. Reduction of lead(IV) oxide to lead(II) oxide by thermolysis
Observation:
When lead(IV) oxide is heated, a gas develops. This gas is collected in the small gasometer. When a glowing wood splint is brought into this gas, the glowing ember flares and produces a flame.

Interpretation:
When lead(IV) oxide is heated, a gas develops. The glowing splint test identifies it as oxygen. The lead(IV) oxide, however, is not transformed into elementary lead. Instead, it becomes lead(II) oxide:

$$2\,\mathrm{Pb}^{+6}\mathrm{O}_2^{-2} \xrightarrow{\text{heat}} 2\,\mathrm{Pb}^{+2}\mathrm{O}^{-2} \;+\; \mathrm{O}_2^{\pm 0}$$

Note:
If silver oxide is used instead of lead(IV) oxide, the thermolysis can continue until elementary silver results.

2. Reduction of lead(II) oxide by way of charcoal to obtain elementary lead
Observation:
The calcium hydroxide solution starts to cloud due to the formation of a white calcium carbonate precipitate. This means that carbon dioxide was formed. After some time, elementary lead can be seen in the test tube. When a drop is big enough for demonstration, stop the reaction and pour the content of the test tube onto a tile or into an evaporating dish for chilling it. The lead drop can be further examined by the students.

Interpretation:
Under the influence of heat, lead(IV) oxide is transformed into lead(II) oxide and oxygen. The resulting lead(II) oxygen is reduced to elementary lead by way of carbon.

$$2\,\mathrm{Pb}^{+2}\mathrm{O}^{-2} \;+\; \mathrm{C}^{\pm 0} \xrightarrow{\text{heat}} 2\,\mathrm{Pb}^{\pm 0} \;+\; \mathrm{C}^{+4}\mathrm{O}_2^{-2}$$

3. Reduction of iron oxide including the formation of hydrogen based on pyrophoric iron
Observation:
The iron oxide is transformed into iron while glowing (evidence concerning the presence of iron can be provided by holding a magnet against the tube). In the cooler sections of the quartz tube, water condenses. After the experiment, evidence concerning the presence of water can be provided by way of cobalt chloride paper (see the appendix). The iron that is produced is a powder with a very large surface (pyrophoric iron = self-igniting iron). It would immediately oxidise in the air. This is why it is left to cool down in a slight hydrogen flow. When the hydrogen flow is stopped, remove the cool porcelain boat. The pyrophoric nature can be demonstrated in an impressive manner by letting the powder trickle through the air and onto a fire-proof plate. The iron starts to glow due to the spontaneous oxidation.

Interpretation:
The reduction, as the reversal of the oxidation, can be achieved thermally or with the aid of a reducing agent.
Some metal oxides can be decomposed into the metal and oxygen under the influence of thermal energy. In the case of less noble metals, a reducing agent is required for obtaining the elements. The redox processes during the preparation of lead demonstrate the relationship between oxidation and reduction. By way of experiments 2 and 3, it can be shown that during the reduction of an oxide the reducing agent itself is oxidised: hydrogen to water, carbon to carbon dioxide. A reduction process is always coupled with an oxidation process, which is why this type of reaction is referred to as a redox reaction. The reaction equations with the added oxidation numbers (formal charge distribution) show that charge units (electrons) are added to the substance that is reduced, while charge units (electrons) are withdrawn from the substance that is oxidised.

$$\mathrm{Fe}_2^{+3}\mathrm{O}_3^{-2} \;+\; 3\,\mathrm{H}_2^{\pm 0} \xrightarrow{\text{heat}} 2\,\mathrm{Fe}^{\pm 0} \;+\; 3\,\mathrm{H}_2^{+1}\mathrm{O}^{-2}$$