(Item No.: P3110500)

Curricular Relevance

Area of Expertise: Chemistry
Education Level: University
Topic: Industrial Chemistry
Subtopic: Metallurgy
Experiment: Preparation of iron from oxidic ores (blast furnace process)

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Intermediate	10 Minutes	10 Minutes	2 Students

Additional Requirements:

- Precision balance, 620 g / 0.001 g

Experiment Variations:

Keywords:

Iron, Blast furnace process, Slug, Production of iron, Reduction, Oxidation

Task and equipment

Introduction

Principle
This is a model experiment to show the industrial blast furnace process to produce iron from iron(III) oxide. During the experiment a furnace gas flame that is approximately 10 to 20 cm high can be ignited at the stack outlet. Cavities form in the burning carbon layer. These cavities collapse over time. Apart from ash and carbon residues, metallic lumps can also be found in the frame after the end of the experiment.
Samples of these lumps lead to the formation of hydrogen when they are treated with hydrochloric acid.

Safety instructions











For the reduction with hydrogen, the right-angled glass tube must be filled with quartz glass wool as blowback protection. Prior to igniting the hydrogen, perform an oxyhydrogen test until the result is negative!

During the second experiment (blast furnace model), there is a risk of a large amount of flying sparks. This is why the experiment should be performed under closed exhaust hood or outdoors and with a sufficient safety distance. It is absolutely essential to take suitable fire protection measures and to ensure that observers cannot be injured by the hot sparks. In addition, the reaction leads to the formation of carbon monoxide. It can be burnt at the blast furnace stack. Carbon monoxide is a colourless, odourless, flavourless, toxic, and highly flammable gas. If mixed with air, there is a risk of explosion (lower flammability limit: 12.5%, upper flammability limit: 74%). As a very strong haemotoxin, it leads to oxygen depletion in the organism. In the event of strong poisoning, this lack of oxygen may cause long-term damage.
First aid: Immediately fresh air, artificial respiration (mouth-to-mouth resuscitation) if necessary. Keep the respiratory tract free. Position the affected person in a warm and comfortable position. If there is a risk of unconsciousness, position and transport the affected person in the recovery position. Experiments involving carbon monoxide must be performed under a strong exhaust hood.

Concentrated acids are highly caustic. They burn the skin and destroy textile fabrics. For diluting, first add the water, then the acid (protective glasses, laboratory coat, gloves).
First aid: Rinse the affected skin areas and eyes with the lid gap wide open thoroughly with plenty of water.
Disposal: Solutions must be diluted with water, neutralised (pH 6-8), and flushed away.

Hydrochloric acid, 37%
H290: May be corrosive to metals.
H314: Causes severe skin burns and eye damage.
H335: May cause respiratory irritation.
P234: Keep only in original container.
P260: Do not breathe dust/fumes/gas/mist/vapours/spray.

Sulphuric acid, 95%
H290: May be corrosive to metals.
H314: Causes severe skin burns and eye damage.
P280: Wear protective gloves/protective clothing/eye protection/face protection.

Quartz glass wool
H332: Harmful if inhaled
H335: May cause respiratory irritation
P261: Avoid breathing dust/fumes/gas/mist/vapours/spray.

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.
P377: Leaking gas fire – do not extinguish unless leak can be stopped safely.

Iron(III) oxide
H302: Harmful if swallowed.
H315: Causes skin irritation.
H318: Causes serious eye damage.
P280: Wear protective gloves/protective clothing/eye protection/face protection.

Equipment

Position No.	Material	Order No.	Quantity
1	Retort stand, h = 750 mm	37694-00	2
2	Support base DEMO	02007-55	1
3	Support rod, stainless steel, l = 600 mm, d = 10 mm	02037-00	2
4	Right angle boss-head clamp	37697-00	3
5	Universal clamp	37715-00	3
6	Protective desk plate 40 x 40 cm	39180-10	1
7	Combustion tube, l 300mm	37023-01	1
8	Gas washing bottle, 100 ml	36691-00	1
9	Clamp for ground joint, plastic, IGJ29	43615-00	1
10	Teflon sleeve IGJ 29, 10 pcs	43617-00	1
11	Rubber stopper, d = 22/17 mm, 1 hole	39255-01	2
12	Glass tube, straight, l=80 mm, 10/pkg.	36701-65	1
13	Glass tube,right-angled w.tip,10	36701-53	1
14	Test tube, 160 x 16 mm, 100 pcs	37656-10	1
15	Test tube rack for 12 tubes, holes d= 22 mm, wood	37686-10	1
16	Porcelain boats, 10 pcs	32471-03	1
17	Steel cylinder hydrogen, 2 l, full	41775-00	1
18	Reducing valve for hydrogen	33484-00	1
19	Table stand for 2 l steel cylinders	41774-00	1
20	Wrench for steel cylinders	40322-00	1
21	Support,w.closed-circuit pipeline	36688-01	1
22	Rings, ceramic fibre, 5 pcs	36688-08	1
23	Blast furnace stack	36688-09	1
24	Hot air blower with adaptor	36688-93	1
25	Pinchcock, width 15 mm	43631-15	1
26	Mortar with pestle, 150 ml, porcelain	32604-00	1
27	Bar magnet, l 150mm	06310-00	1
28	Teclu burner, DIN, natural gas	32171-05	1
29	Safety gas tubing, DVGW, sold by metre	39281-10	1
30	Lighter f.natural/liquified gases	38874-00	1
31	Hose clip f.12-20 diameter tube	40995-00	2
32	Rubber tubing, i.d. 6 mm	39282-00	1
33	Rubber tubing, i.d. 8 mm	39283-00	1
34	Tweezers,straight,blunt, 200 mm	40955-00	1
35	Crucible tongs, 200 mm, stainless steel	33600-00	1
36	Spoon, special steel	33398-00	1
37	Funnel, glass, top dia. 55 mm	34457-00	1
38	Wash bottle, plastic, 500 ml	33931-00	1
39	Beaker, high, BORO 3.3, 100 ml	46026-00	1
40	Pasteur pipettes, 250 pcs	36590-00	1
41	Rubber caps, 10 pcs	39275-03	1
42	Quartz glass wool 10 g	31773-03	1
43	Hydrochloric acid 37 %, 1000 ml	30214-70	1
44	Sulphuric acid, 95-98% 500 ml	30219-50	1
45	Iron ore, 500 g	36688-05	1
46	Activated carbon, granular 500 g	30011-50	1
47	Charcoal,small pieces 300 g	30088-30	1
48	Iron-III oxide, red 500 g	48114-50	1
49	Water, distilled 5 l	31246-81	1

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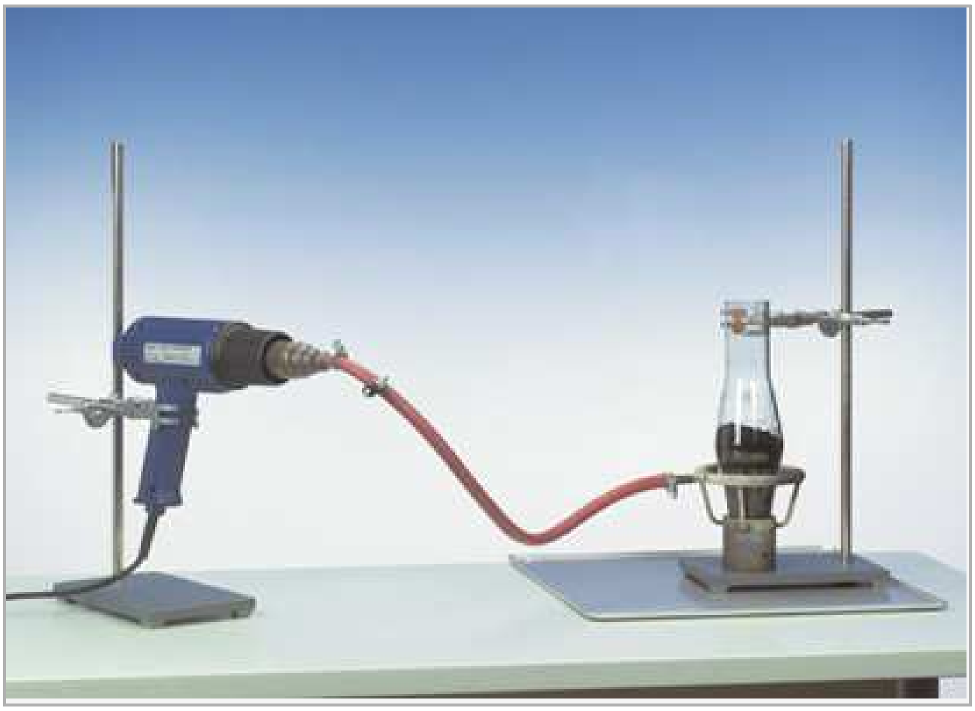
Task

Preparation of iron from oxidic ores (blast furnace process) Reduction of iron(III) oxide and blast furnance model.



Set-up and procedure

1. Reduction of iron(III) oxide

- Fill some iron(III) oxide into a porcelain boat and push it into the combustion tube that is secured horizontally on a support stand as shown in Fig. 1.
- Connect the gas wash bottle, which is filled up to 4 cm high with concentrated sulphuric acid, to one end of the combustion tube. Seal the other end of the tube with a rubber stopper and a right-angled glass tube with a tip. Quartz glass wool in the glass tube ensures protection against blowback.
- Let a slow flow of hydrogen flow through the apparatus. When this flow is free from oxygen (oxyhdrogen test), ignite the hydrogen that escapes from the tip of the glass tube and heat the iron oxide in the porcelain boat with a burner. The hydrogen flame should not be higher than two centimetres.
- After the reaction is over, let everything cool down in a hydrogen flow. Test the cold product by way of a magnet that must be held against the combustion tube. Fill some of the product into a test tube and add a few millilitres of diluted hydrochloric acid (approximately 10%, made from 10 g of concentrated hydrochloric acid and 27 g of water).

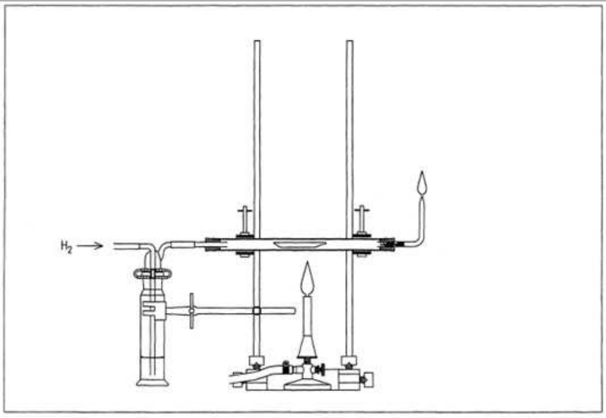


Fig. 1

2. Blast furnace model

- The blast furnace model consists of a metal "frame" with closed-circuit air pipes and the "stack" made of glass that is difficult to melt. The glass cylinder is positioned on the frame on a ring of ceramic fibres. The air supply is ensured via an 8 mm rubber tubing (pinchcock for flow control) and by way of a hot/cold air blower that is fastened to a support stand.
- Place the blast furnace model with the second support stand on a safety desk plate as shown in Fig. 2.
- Remove the glass stack and ignite a piece of charcoal on the burner. When the charcoal glows sufficiently, place it into the frame and switch the air supply on. Control the air flow so that the charcoal continues to glow.
- Then, pile the activated carbon up around the glowing charcoal until it reaches the rim of the frame. Ensure that the embers are not smothered.
- After that, place the stack onto the frame and secure it with a universal clamp at its upper end.

Caution! Hot gases will rise in the stack. Risk of burns. Then, fill some activated carbon carefully into the stack until it is well compacted.
Caution! During this process, carbon dust may be blown out of the stack. Add a layer of 1 to 2 cm of iron ore on top of the activated carbon and cover it with another layer of activated carbon of the same thickness. During the reaction, test from time to time as to whether the gases at the stack outlet can be ignited (use the burner). Ensure a uniform combustion by controlling the air supply accordingly. After the reaction, let the entire system cool down, but leave the stack on the frame. Remove the product with the frame, grind it in a mortar, fill some of it into a test tube and add some diluted hydrochloric acid (see above).

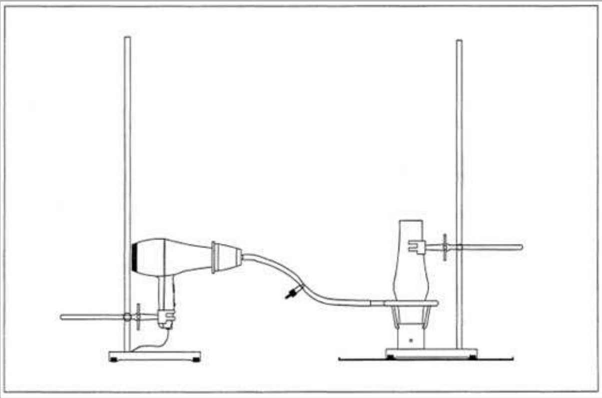


Fig. 2

Results and evaluation

1. Reduction of iron(III) oxide

After a short period, water condensates at the end of the combustion tube and the hydrogen flame shrinks slightly. The red-brown iron oxide turns black. Unlike the initial substance, the product is magnetic and reacts with diluted hydrochloric acid under the formation of gas.

Iron(II) oxide is reduced to iron by hydrogen. The iron reacts with hydrochloric acid while hydrogen is formed.

$$Fe_2O_3 + 3\,H_2 \longrightarrow 2\,Fe + 3\,H_2O$$
$$2\,Fe + 6\,HCl \longrightarrow 2\,FeCl_3 + 3\,H_2\uparrow$$

2. Blast furnace model

If the air supply is adjusted correctly, a furnace gas flame that is approximately 10 to 20 cm high can be ignited at the stack outlet. During the blast furnace process, cavities form in the burning carbon layer.
These cavities collapse over time. Apart from ash and carbon residues, metallic lumps can also be found in the frame after the end of the experiment. Samples of these lumps lead to the formation of hydrogen when they are treated with hydrochloric acid.

The following main chemical reactions take place in a blast furnace:

$$C + O_2 \longrightarrow CO_2$$
$$CO_2 + C \longrightarrow 2\,CO$$
$$Fe_2O_3 + 3\,CO \longrightarrow 2\,Fe + 3\,CO_2$$

Due to caking and conglutination in the upper layers that are caused by thermal expansion, the carbon and ore layers cannot always sink down regularly. This leads to the formation of cavities that can collapse and disturb the reaction. In order to avoid this to the largest possible extent, blast furnaces have a typical double-cone shape. It enables the sinking material to expand without caking too much. In the model, this effect is less distinct. However, cavities can be caused to collapse by briefly interrupting the air supply (by pinching the tubing). The temperatures that can be reached with the model are not sufficient for collecting any liquid iron and slag in the frame. This is also why they cannot be tapped off. As a result, the product is always iron mixed with slag.

Note: This experiment can only lead to satisfactory results if low-melting iron ores are used, e.g. Mount Nimba iron ore or bog iron.