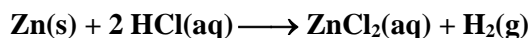


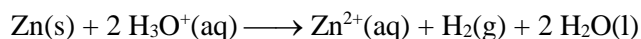
Preparation of hydrogen

Gaseous hydrogen H_2 is a colourless and odourless gas, poorly soluble in water. Mixed with oxygen forms an extremely explosive mixture.

In the laboratory, hydrogen is commonly prepared by the reaction non-noble metals such as zinc with non-oxidizing acids, such as hydrochloric acid or very diluted sulphuric acid.



The hydroxonium cation H_3O^+ formed by the ionization of hydrochloric acid acts as an oxidizer to zinc, according to their standard redox potentials.



$$E^0(Zn^{2+}|Zn) = -0,763 V \quad E^0(H_3O^+|H_2) = 0 V \text{ (exactly)}$$

Work

Prepare hydrogen by the reaction of zinc with hydrochloric acid.

Chemicals

- zinc Zn, grey metal,
- hydrochloric acid HCl, concentrated, $w(HCl) = 0,36$.

Procedure

At first, set up a hydrogen generator according to the Fig. 1 and fix it to a stand. Put the calculated quantity of zinc carefully into a fraction flask and connect an empty dropping funnel with rubber stopper. Do not pour hydrochloric acid to the funnel yet. If dry hydrogen is required, connect a gas-washing bottle filled with concentrated sulphuric acid (Fig. 2). Sulphuric acid also acts as a filter for insoluble impurities. For drying also solid desiccators such as silica gel, P_4O_{10} , NaOH, or $CaCl_2$, may be used.

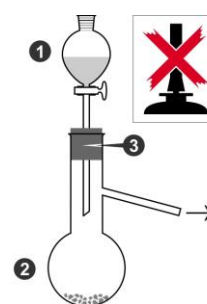
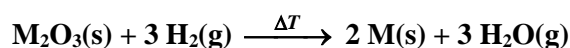
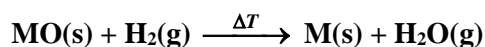


Fig. 1 The hydrogen generator. Do not heat!

- 1 – dropping funnel,
- 2 – fraction flask,
- 3 – rubber stopper.

Preparation of metals by the reduction of their oxides by hydrogen

Reductive properties of hydrogen may be exploited for the preparation of some metals from their oxides. The reduction occurs easily only in case of oxides with the enthalpy of formation lower than that of water. These oxides react with hydrogen at high temperature to form metal and water vapor.



The oxides decomposing to metal and oxygen (Ag_2O , HgO) at higher temperature must not be reduced by hydrogen. Similarly, the metals forming hydrides with hydrogen cannot be prepared in this way.

Work

Prepare 1,00 g of the selected metal from its oxide. Prepare 15-times more hydrogen than necessary.

Chemicals

- copper(II) oxide CuO , black powdered substance,
- iron(III) oxide Fe_2O_3 , dark red powdered substance.
- bismuth(III) oxide Bi_2O_3 , light yellow powdered substance.

Procedure

It has been proved experimentally that 15-times more hydrogen is necessary to prepare. Hydrogen is used not only for reduction itself, but also to displace air from the apparatus as well as to provide the inert atmosphere during the cooling. No oxygen must be present in the apparatus before we start heating!

The reduction occurs in a wider heat-resistant tube made of special glass (Fig. 2). It must be moderately pitched in order to prevent the condensed water from flowing back to the red-hot part of the tube. In the middle of the tube a porcelain boat with evenly spread powdered oxide is placed. At the end of the tube a capillary is attached, bent into a right angle. A test tube placed on the capillary upside-down serves as a collector of hydrogen leaving the apparatus.

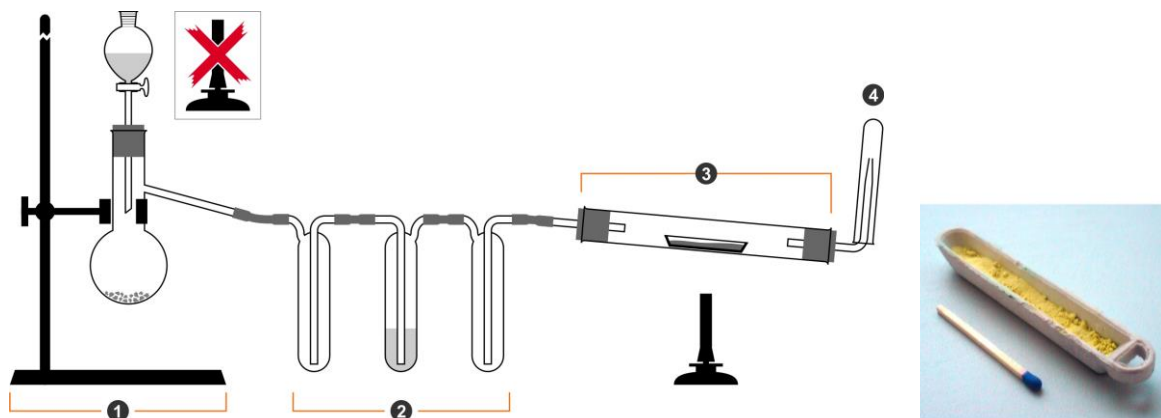


Fig. 2 Apparatus for the reduction by hydrogen.

1 – hydrogen generator (Fig. 1), 2 – filter/dryer = a gas-washing bottle with concentrated sulphuric acid,
3 – reactor = a porcelain boat with metal oxide in a heat-resistant tube,
4 – „detector“ = a test tube to collect hydrogen, photo – the porcelain boat with bismuth(III) oxide and a match.

Once the apparatus is built up, its tightness must be checked.

Open the cock of the dropping funnel. Remove the test tube placed on the capillary. Connect and activate the source of vacuum (water pump). Close the cock. If the apparatus is tight enough, bubbles in the gas-washing bottle must disappear in a while. The apparatus must not be heated until absolutely tight!

Then, open slowly the cock again, switch off the source of vacuum and detach it from the apparatus. Place the test tube on the capillary.

Close the cock. Pour the calculated volume of hydrochloric acid into the dropping funnel and add it dropwise to the fraction flask with zinc to produce hydrogen.

Regularly check the presence of oxygen in the apparatus.

Close the test tube with your finger and move it to the burner's flame. Open the test tube, but do not rotate it! Put it to the flame. If oxygen is still present, you should hear a loud sound of micro-explosion. No oxygen is present in apparatus, if only very quiet sound is hearable. Only now the apparatus can be heated.

At the beginning, heat the apparatus with mild flame. Then, set the strongest flame and anneal the glass tube from one corner of the porcelain boat to other one. The colour of powder in the boat changes as the oxide is reduced. When no original colour is visible, stop heating and let the apparatus cool down slowly to room temperature. Take out carefully the boat from the tube and weigh the reduced product.



CuO



Cu



Bi₂O₃



Bi

Safety instructions

Zinc – Zn

- S7/8** Keep container tightly closed and dry.
S43 In case of fire use .. (indicate in the space the precise type of fire-fighting equipment. If water increases the risk add: Never use water).

Hydrochloric acid – HCl

- R34** Causes burns.
R37 Irritating to respiratory system.
S2 Keep out of the reach of children.
S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.

Hydrogen – H₂

- R12** Extremely flammable.
S9 Keep container in a well-ventilated place.
S16 Keep away from sources of ignition – No smoking.
S33 Take precautionary measures against static discharges.

Copper(II) oxide – CuO

- R20/22** Harmful by inhalation and if swallowed.
R36/37/38 Irritating to eyes, respiratory system and skin.
S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S36 Wear suitable protective clothing.

Bismuth(III) oxide – Bi₂O₃

- R20/21/22** Harmful by inhalation, in contact with skin and if swallowed.
S36 Wear suitable protective clothing.

Copper – Cu

- R36/37/38** Irritating to eyes, respiratory system and skin.
S26 In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S37/39 Wear suitable gloves and eye/face protection.

Bismuth – Bi

- R20/21/22** Harmful by inhalation, in contact with skin and if swallowed.
S36 Wear suitable protective clothing.