General and Inorganic Chemistry

Air and other gases
Reactions with gases

LD Chemistry Leaflets

C1.4.3.1

Hydrogen as a reducing agent

Aims of the experiment

- To learn about a reaction with gases.
- To investigate a redox reaction.
- To learn that the reaction of hydrogen to produce water is an oxidation.
- To investigate the reduction of copper(II) oxide to copper.
- To characterise water as a reaction product.

Principles

Hydrogen is a colourless, odourless and tasteless gas. It is combustible and can be used in numerous reactions. One of the best-known reactions is the oxy-hydrogen or detonating gas reaction, in which hydrogen gas reacts with oxygen gas when energy is supplied in the form of heat.

$$2\ H_2 + O_2 \rightarrow H_2O$$

Under the same reaction conditions, hydrogen is able to remove oxygen from many metal oxides. Hydrogen therefore acts on many metal oxides as a reducing agent when heated. This is one of the cleanest reduction reactions, as the water produced can be distilled off and the metal is then present in its purest form.

If one heats black copper oxide powder (CuO) and allows hydrogen to pass over it, glowing red copper powder is formed together with small water droplets. In this way, a chemical reaction takes place in which new substances with new chemical properties are formed.

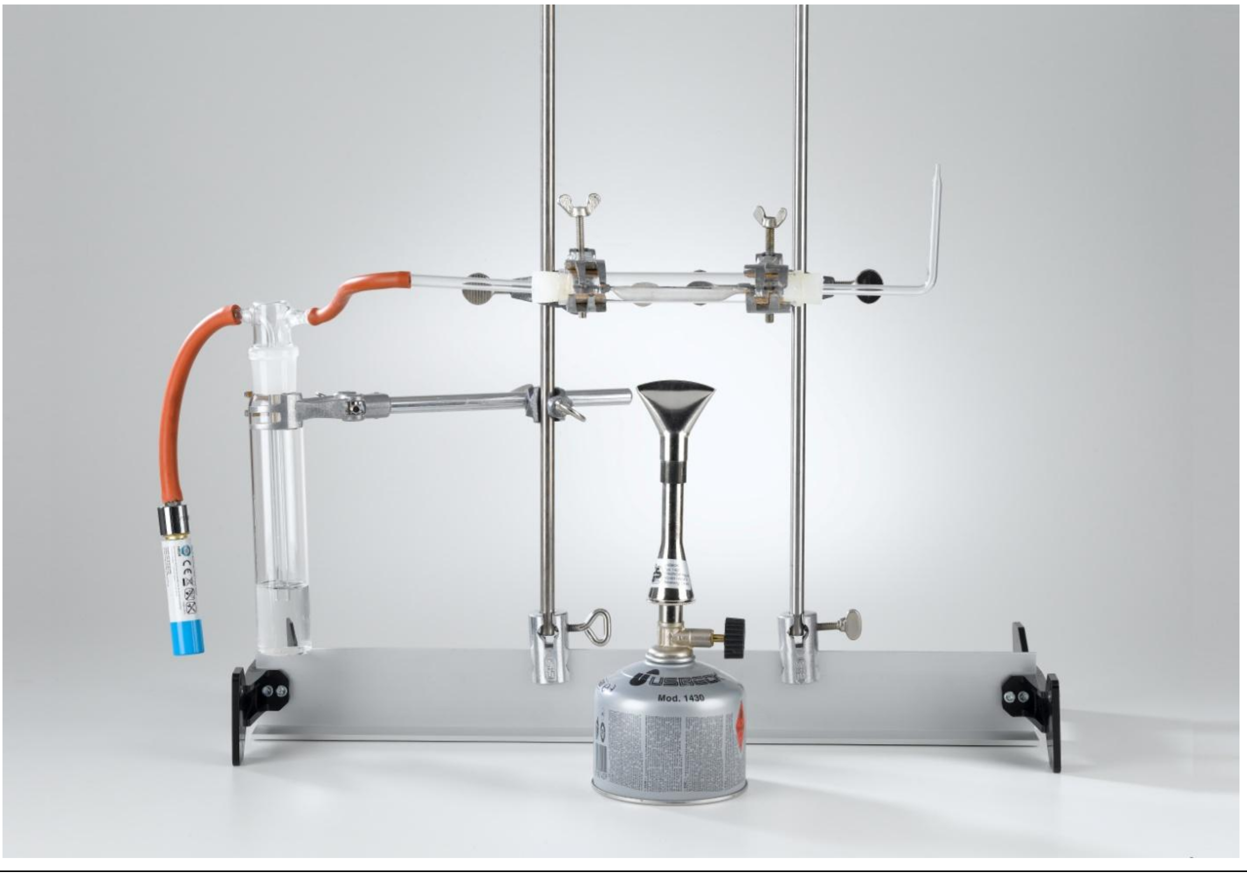
The equation for the reaction described is as follows:

$$CuO + H_2 \rightarrow Cu + H_2O$$

In this case, we are dealing with a redox reaction. This means that one substance is reduced and the other is oxidised. The term oxidation denotes primarily all reactions with oxygen. If one expands this definition, then it is apparent that electrons are generally emitted in the case of oxidation.

Accordingly, the removal of chemically bound oxygen is called reduction. If one also expands this definition, the term reduction denotes the acceptance of electrons. Redox reactions (electron transfer reactions) always involve the combination of a reduction (electron acceptance) and an oxidation (electron emission) process.

In the case of the reaction of hydrogen with copper oxide, oxygen is removed from the copper oxide and with that it is reduced. On the other hand, the hydrogen is oxidised.



MS-2014-02

Reduction:	$\overset{+II}{Cu}\overset{-II}{O} + 2\,e^-$	→	$\overset{0}{Cu} + \overset{-II}{O}{}^{2-}$
Oxidation:	$\overset{0}{H_2} + \overset{-II}{O}{}^{2-}$	→	$\overset{+I}{H_2}\overset{-II}{O} + 2\,e^-$
Redox reaction:	$CuO + H_2$	→	$Cu + H_2O$

Hydrogen serves in this reaction as the reducing agent. This means that the hydrogen is acting as an electron donor. It reduces its reaction partner CuO by donating an electron to it. CuO, on the other hand, is the oxidising agent in this case. It oxidises the hydrogen and accepts its electrons. In this way, it acts as an electron acceptor.

Risk assessment

The use of hydrogen is unproblematic as long as the apparatus is completely filled with hydrogen and the combustion is carried out under controlled conditions.

Protective clothing must be worn (gloves, lab coat, protective glasses) when using sulphuric acid to avoid acid burns.

Copper(II) oxide	
 	**Hazard statements** H302 Harmful if swallowed. H410 Very toxic to aquatic life with long lasting effects **Precautionary statements** P260 Do not breathe dust/fume/gas/mist/vapors/spray P273 Avoid release to the environment
Hydrogen	
 	**Hazard statements** H220 Extremely flammable gas H280 Contains gas under pressure; may explode if heated **Precautionary statements** P210 Keep away from heat/sparks/open flames/hot surfaces – No smoking P377 Leaking gas fire – do not extinguish unless leak can be stopped safely P381 Eliminate all ignition sources if safe to do so P403 Store in a well ventilated place
Sulphuric acid	
	**Hazard statements** H314 Causes severe skin burns and eye damage H290 May be corrosive to metals **Precautionary statements** P280 Wear protective gloves/protective clothing/eye protection/face protection P301+330+331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting P309+310 IF exposed or you feel unwell: Immediately call a POISON CENTER or doctor/physician P305+351+338 IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do – continue rinsing

Equipment and chemicals

1	Base rail 55 cm	666 602
3	Universal bosshead	666 615
2	Stand tube 50 mm x 13 mm diam.	666 605
2	Universal clamp 0...80 mm	666 555
3	Bosshead S	301 09
1	Gas-washing bottle 250 ml	602 421
1	Reaction tube, quartz, 16 cm	664 0772
2	Silicone stopper, 16 x 21 x 25 mm, 1 hole	667 286
1	Glass tube 80 mm x 8 mm diam.	665 201
1	Rubber tubing 8 mm diam.	667 183
1	Glass nozzle 90°, 80 x 80 mm, 8 mm diam.	665 238
1	HydroStik PRO	666 4796
1	Regulating valve	666 4797
1	HydroFill PRO	666 4798
1	Silicone tubing 2 mm diam.	667 198
1	Silicone tubing 4 mm diam.	667 194
1	Combustion boat, 80 x 12 mm	666 988
1	Analytical balance 83 : 0.0001 g	667 7988
1	Double microspatula, steel, 150 mm	604 5672
1	Cartridge burner, DIN type	666 714
1	Wide-flame attachment	666 724
1	Copper(II) oxide, powder, 50 g	672 9500
1	Sulphuric acid, 95-98%, 500 ml	674 7860
1	Iron wool, 50 g	671 8400

Setup and preparation of the experiment

Setup of the apparatus

1. Attach two stand tubes to the base rail using two universal bossheads.
2. Attach one universal clamp to one of the stand tubes using a bosshead S coupling, and attach two universal clamps to the other stand tube.
3. Now fill a gas-washing bottle with about 40 ml of concentrated sulphuric acid as a washing solution. This is used to dry the hydrogen gas.
4. Fasten the washing bottle to the lower of the two universal clamps on the stand tube with two clamps.
5. Clamp a quartz reaction tube between the other two universal clamps and close it with two silicone stoppers.
6. Connect the reaction tube on the one side to the washing bottle using a glass tube and a piece of rubber tubing (8 mm diameter). On the other side, fit a right-angled glass nozzle which has previously been filled with iron wool as a blow-back protection.
7. Finally, connect the hydrogen storage cartridge (HydroStik PRO) to the other side of the gas-washing bottle using rubber tubing.

The hydrogen source

The hydrogen is provided from a HydroStik PRO metal hydride storage cartridge. This is filled with hydrogen which has previously been produced by electrolysis. The electrolysis takes place in a HydroFill PRO system, from which the HydroStik PRO is also filled at the same time.

1. Open the cover of the water tank and carefully pour in distilled or deionised water up to the inner ridge. Close the cover.
2. Connect the AC-DC adaptor and plug it into an AC mains socket. The status indicator will now flash green.
3. Screw the HydroStik PRO into the HydroFill PRO. The status indicator will now change from green to red to show that the connection has been made. Firmly screw in the HydroStik PRO.
4. The HydroStik PRO is filled automatically, which is shown by the red status indicator. Charging takes about 4 to 6 hours.

An occasional hissing sound indicates that the system is rinsing. The oxygen produced can be seen in the form of bubbles.

5. When the status indicator changes to green, the HydroStik PRO is fully charged and can be removed. A brief hissing sound will occur at this time.

6. A further HydroStik PRO can now be charged. For this, repeat the instructions from step 3.

7. When charging is complete, disconnect the HydroFill PRO from the mains socket and empty the water tank.

Preparation of the experiment

1. Weigh approximately 100 mg of copper(II) oxide into the combustion boat.

2. After weighing, insert the combustion boat into the reaction tube and close the tube again.

Performing the experiment

1. Connect the HydroStik PRO metal hydride storage cartridge to the apparatus. For this purpose, the regulating valve is connected to the gas-washing bottle using tubing. In this case, screw the 2 mm diameter silicone tubing onto the regulating valve. Attach this to the 4 mm diameter tubing, which can then be attached to the tube connector of the gas-washing bottle.

2. The HydroStik PRO opens when it is screwed into the regulating valve. The HydroStik PRO closes automatically when it is unscrewed again.

3. Allow the hydrogen to flow into the apparatus and flush the apparatus for about 1 minute. There must be no naked flame near the apparatus during this time.

4. In order to ensure that the apparatus does not contain an explosive mixture, but is filled only with hydrogen, a detonating gas test must be performed. The detonating gas test is performed as follows:

   a. Hold a test tube upside down over the glass nozzle at the exit from the apparatus to collect the gas from the gas stream.

   b. Place your thumb over the opening of the test tube and bring it close to a naked flame. At the same time, remove your thumb from the opening of the test tube.

   c. If only a brief plop is heard, then the tube contains only pure hydrogen. However, if a whistle and a detonation are heard, then there is still oxygen in the apparatus.

5. When the result of the detonation gas test is negative several times, the hydrogen can be ignited at the end of the right-angled glass nozzle.

Caution!

The detonation gas test must in all events be negative, as the apparatus could otherwise explode.

6. The copper(II) oxide can now be heated with the burner.

Caution!

If during heating of the copper oxide no further bubbles can be seen in the gas-washing bottle – i.e., the hydrogen supply is too low – or if the flame at the glass nozzle should extinguish, the burner flame must be turned off immediately. It is best in this case to cover the apparatus with a previously prepared wet towel. This serves to quickly cool down the apparatus so as to remove energy which could ignite any detonation gas mixture that might be present.

7. When all of the copper oxide has glowed away, turn the burner off.

8. The experiment can be terminated when a complete change in colour of the black copper oxide can be seen.

Caution!

Do not turn off the supply of hydrogen until the entire apparatus has cooled down. If the hydrogen supply is turned off before this, oxygen from the air can be drawn into the hot apparatus. This would result in the formation of detonation gas, which would then ignite.

Observations

The reaction continues autonomously after the burner has been removed. The black copper oxide changes from black to a glossy reddish colour during the reaction. Small water droplets can be identified on the wall of the tube on closer observation.

Result of the experiment

The black copper(II) oxide changes during the reaction. A solid substance with a glossy reddish colour results. Elemental copper is formed which can be clearly identified by its colour. The reaction which takes place here is as follows:

$$CuO + H_2 \rightarrow Cu + H_2O$$

If the hydrogen supply is turned off too soon, then copper oxide will be reformed with heat and with the help of oxygen from the air. This is mainly copper(I) oxide, but partially also copper(II) oxide.

$$4\ Cu + O_2 \rightarrow 2\ Cu_2O$$

$$2\ Cu + O_2 \rightarrow 2\ CuO$$

Cleaning and disposal

The copper obtained can be collected in an appropriately labelled container and used in other experiments.

Disposal

Copper(II) oxide: G4 inorganic waste with heavy metals

Sulphuric acid: Dilute carefully with water (add acid to water, never water to acid!). It can then be emptied into a drain or can also be neutralised beforehand using an alkaline solution.