

## Analysis of copper(I) oxide and copper(II) oxide

### Aims of the experiment

- To understand the law of multiple proportions.
- To recognise that elements can be present in compounds in different ratios.
- To learn about and understand stoichiometry.
- To determine the molar mass ratio of copper and oxygen, and from this the molar mass of copper.
- To learn about the redox reaction of copper oxide and hydrogen.

### Principles

When two elements react with each other, compounds with different ratios can result in the process. In this case, the masses of both elements always occur in integral multiples. Copper oxide can appear as red copper(I) oxide or black copper(II) oxide in this way. In copper(I) oxide ( $\text{Cu}_2\text{O}$ ), 2 parts of copper have reacted with 1 part of oxygen, in copper(II) oxide ( $\text{CuO}$ ) 1 part of copper with 1 part of oxygen.

This law, accepted today as a matter of course, was a milestone in the history of chemistry. It was described in 1808 by John Dalton in the law of multiple proportions. This is an extension of the law of constant proportions. The law of constant proportions states that the mass ratio of the elements present in a chemical compound is always the same. The extension to this law formulated by Dalton expresses the fact that elements can react with one another in various (= multiple) ratios, and also that these ratios are always in whole

numbers.

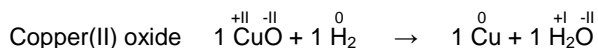
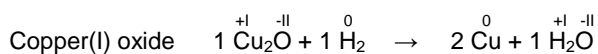
Both laws are the basis for the formation of chemical equations. They are used daily by chemists - mostly without realising it. Also, the quantitative calculation of chemical reactions, the so-called stoichiometry, is based on these laws. With stoichiometry calculations, a quantitative calculation can be derived from the qualitative knowledge about educts and products of a reaction.

Specifically, the masses of the educts used are converted into mole quantities of substance. The mole quantities of substance react in turn with each other in integer ratios as described in the chemical equation. The mole quantity of the resulting product can be calculated from the chemical equation. This can then in turn be converted into the mass of the product. The yield of a reaction, for example, can be determined in this way.

This experiment is intended to prove the validity of the law of



multiple proportions. For this purpose, two different copper oxides are reduced with hydrogen. The different mass ratios in the copper oxides can be experimentally determined by this quantitative reduction. The chemical equations of the redox reactions are as follows:









For evaluation of the experiment, the mass of the copper oxide weighed out is compared with the resulting mass of the copper, so the integer ratios are apparent on the one hand, and the multiple proportions of both copper oxides on the other hand. Furthermore, the relative molar mass of copper compared to oxygen can be calculated. If the molar mass of oxygen is already known, then the molar mass of copper can be determined from it.

### Risk assessment

When working with hydrogen, there is always the inherent risk of a detonating gas reaction. Therefore, only burn hydrogen if it is certain that there is no air in the apparatus, but that it is completely filled with hydrogen.

Under no circumstances must a reaction tube be used for this experiment which is not made from quartz glass. These melt on prolonged heating and leak. Oxygen could then enter the apparatus, resulting in a detonating gas reaction.

Hydrogen	
  <b>Signal word:</b> <b>Hazard</b>	<p><b>Hazard statements</b></p> <p>H220 Extremely flammable gas.</p> <p>H280 Contains gas under pressure; may explode if heated.</p> <p><b>Precautionary statements</b></p> <p>P210 Keep away from heat/sparks/open flames/hot surfaces. No smoking.</p> <p>P377 Leaking gas fire: Do not extinguish unless the leak can be stopped safely.</p> <p>P381 Eliminate all ignition sources if safe to do so.</p> <p>P403 Store in a well-ventilated place.</p>
Copper(I) oxide	
  <b>Signal word:</b> <b>Caution</b>	<p><b>Hazard statements</b></p> <p>H302 Harmful if swallowed. Avoid release to the environment.</p> <p>H410 Very toxic to aquatic life with long lasting effects</p> <p><b>Precautionary statements</b></p> <p>P261: Avoid breathing dust.</p> <p>P273: Avoid release to the environment.</p> <p>P301+312: IF SWALLOWED: Call a POISON CENTER or doctor/physician if you feel unwell</p>

Copper(II) oxide	
  <b>Signal word:</b> <b>Caution</b>	<p><b>Hazard statements</b></p> <p>H302 Harmful if swallowed. Avoid release to the environment.</p> <p>H410 Very toxic to aquatic life with long lasting effects</p> <p><b>Precautionary statements</b></p> <p>P261: Avoid breathing dust.</p> <p>P273: Avoid release to the environment.</p>

Equipment and chemicals	
1	Panel frame C100, two-level for CPS ..... 666 428
4	Adhesive magnetic board, 300 mm..... 666 4660
1	Adhesive magnetic board, 500 mm..... 666 4659
1	HydroFill PRO..... 666 4798
1	HydroStik PRO, CPS ..... 666 4795
1	Gas scrubber bottle, lower section ..... 664 800
1	Glass tube insert with filter ..... 664 806
1	Silicone tubing 4 mm diam., 1 m ..... 667 197
1	Silicone tubing 2 mm diam., 1 m ..... 667 198
1	Glass connector, 2 x GL 18 ..... 667 312
1	Magnetic holder, size 4, 27...29 mm..... 666 4664
1	U-tube, 160 x 22mm, 2 side Taps ..... 666 086
3	Magnetic holder, size 2, 11...14 mm..... 666 4662
2	Rubber stopper, solid, 16...21 mm. .... 667 255
1	Reaction tube, quartz, 300 x 20 mm ..... 666 077
2	Silicone stopper, 1 hole 7 mm..... 667 286
1	Glass nozzle, 90° angle ..... 665 238
2	Cartridge burner, DIN type ..... 666 714
2	Wide-flame attachment ..... 666 724
2	Laboratory stand II ..... 300 76
1	Drying oven UNB, 32 LI, with timer ..... 666 8032
1	Vacuum desiccator, 200 mm..... 664 925
1	Combustion boat, 80 x 12 mm ..... 666 988
1	Crucible tongs, 200 mm ..... 667 035
1	Analytical balance, 83: 0.0001g ..... 667 7988
1	Spoon-ended spatula, SS, 180 mm ..... 666 968
1	Double microspatula, steel, 130 mm..... 604 5671
1	Scissors, 200 mm, pointed..... 667 016
1	Tweezers, blunt, 200 mm..... 667 034
1	Glass stirring rod 200 mm., from set..... 665 212ET10
1	Copper(I) oxide, 25 g ..... 672 9300
1	Copper(II) oxide, powder, 100 g..... 672 9510
1	Silica gel with indicator, 500g..... 672 7781
1	Iron wool, 50 g ..... 671 8400
1	Glass wool, 10 g ..... 672 1000

### Set-up and preparation of the experiment

#### Preparation of the copper oxide

1. Dry the copper(I) and copper(II) oxide in the drying oven at 150°C for around 6 to 8 hours. In this way, any water that is present is removed and therefore cannot falsify the weighing. Also dry the silica gel drying agent.
2. After completing the drying process, store the substances in a desiccator over a drying agent (e.g. silica gel).

### Preparation of 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.

### Construction of the apparatus

1. The apparatus is constructed in the CPS panel frame on adhesive magnetic boards. Position all glassware on the board with magnetic holders.
2. Connect the regulating valve of the "HydroStik PRO" CPS module to a gas scrubbing bottle using silicone tubing. Fill the gas scrubbing bottle with some water. Attach it to the magnetic board with a magnetic holder to act as a bubble counter.
3. Using a glass connector, connect the gas scrubbing bottle to the side tap of a U-tube.
4. Clamp this U-tube with a magnetic holder and fill it alternately with glass wool and silica gel (with indicator) for drying the hydrogen. After filling, seal with two rubber stoppers.
5. Connect the U-tube to the quartz glass reaction tube. Attach this with magnetic holders at each end to the magnetic board. Both combustion boats will be placed into this reaction tube later.
6. Complete the construction of the apparatus using an angled glass nozzle filled with iron wool as a blow-back protection.
7. Position two Bunsen burners with wide-flame attachments, each on a Laborboy, under the reaction tube.

### Performing the experiment

1. Fire both combustion boats well over the Bunsen burner flame. After cooling, determine and note the exact mass of both combustion boats.
2. Then weigh exactly 1 g of copper(I) oxide and copper(II) oxide into each of the combustion boats, respectively. Place both combustion boats into the quartz glass tube in the apparatus (see construction photo).
3. Screw the HydroStik PRO so far into the regulating valve of the CPS module that bubbles are visible in the gas scrubbing bottle. Flush the apparatus out well with hydrogen.

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.

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

#### Caution!

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

9. Now carefully heat the combustion boats in the reaction tube with both Bunsen burners until they glow. Do not overheat, otherwise a copper mirror will form on the glass wall.

#### 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 Bunsen 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 detonating gas mixture that might be present.

10. Extinguish the Bunsen burners after a reaction time of about 10 minutes.

#### 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 detonating gas, which would then ignite.

11. Water will have formed on the edge of the reaction tube after cooling. Remove this with a glass rod wrapped in cotton wool. Only then, draw out the combustion boats with crucible tongs.

12. Determine the mass of both combustion boats after the reaction and calculate the mass of the copper which has formed.

### Observation

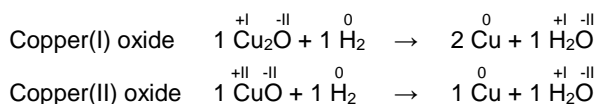
Elemental copper is formed in both combustion boats. This is easy to recognise from its reddish, shiny colour. Besides the elemental copper, water is also present as a reaction product. This is seen in the reaction tube in the form of small water droplets.

For the evaluation, first determine the mass difference of the quantities weighed in and the quantities weighed out for both copper oxides. This difference, that is the difference between the mass of the copper oxides and the mass of the pure copper, is the mass of the oxygen  $m_{\text{O}}$  in the copper oxides.

With the mass of the pure copper  $m_{Cu}$  and the mass of the oxygen  $m_O$  that has been released from the copper oxides, the ratio of both substances in the copper oxides can be calculated.

### Result of the experiment

Both copper(I) and copper(II) oxide are reduced on heating in the hydrogen stream. Water is also present, which can be detected with anhydrous copper(II) sulfate (blue colouration).



By comparing the quantities weighed in and the quantities weighed out of both samples, the mass of the oxygen bound to the copper and the corresponding mass ratios can be determined.

	Copper(I) oxide	Copper(II) oxide
Quantity weighed in: Mass of the copper oxide [g]	1.000	1.000
Quantity weighed out: Mass of the copper content $m_{Cu}$ [g]	0.888	0.799
Quantity weighed in - quantity weighed out: Mass of the oxygen content $m_O$ [g]	0.112	0.201
Ratio of copper to oxygen $m_{Cu}:m_O$	7.929: 1 $\approx 8: 1$	3.975: 1 $\approx 4: 1$

The mass ratio of copper to oxygen in the red copper(I) oxide is 8 to 1, in the black copper(II) oxide only 4 to 1. The following can be deduced from this:

1. Copper can react with oxygen in two different ratios.
2. Copper is about four times heavier than oxygen.
3. In red copper oxide, 1 part of copper and 2 parts of oxygen are bound. The formula is therefore  $\text{CuO}_2$ . In the black copper oxide, 1 part of copper and 1 part of oxygen are bound. The formula is  $\text{CuO}$ .

Copper can therefore react with oxygen in different ratios (multiple proportions). Therefore this law is confirmed.

The law of constant proportions can also be checked here. The ratios of the copper masses in both oxides are calculated, so the following is obtained:

$$m_{Cu(I)}: m_{Cu(II)} \approx 8: 4 \approx 2: 1.$$

The result from this is that copper reacts with oxygen in the ratio 2:1 or 1:1, therefore in the ratio of small whole numbers. It is useful to repeat this experiment to confirm the law.

The relative molar mass of copper can also be determined with this experiment. Given that the molar mass of oxygen is known ( $m_O = 15.999 \text{ g/mol}$ ), then the molar mass of copper can be calculated from these findings.

$$m_{Cu} = 3.975 \cdot m_O = 3.975 \cdot 15.999 \text{ g/mol} = 63.596 \text{ g/mol}$$

The theoretical value of the atomic mass of copper is 63.546 u.

### Disposal

Store the copper obtained by reduction in appropriately labelled vessels for further experiments.

### Remarks

The experiment can be repeated weighing in other amounts, e.g. 1.50 g or 2.00 g to reinforce understanding of the application. The averages of the mass ratios can then be determined.