

Copper refining

Time required: 40 – 50 min

Aims of the experiment

- Introduction to metallurgy.
- To understand refining as a purification process.
- To purify copper through refining.
- To use gravimetry as a quantitative verification technique.

Principles

After metals are recovered, most of them contain contaminants and interfering factors. The removal of these is called refining.

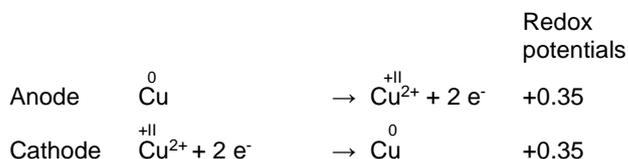
For different metals, there are also different methods of refining. Moreover, refining can affect the subsequent properties of the metal. For example, through refining not only can contaminants be removed, but also targeted additives can be introduced which affect the metal's properties.

In *electrolytic refining*, there is a series of metals (copper, chromium, nickel, silver, gold, zinc, and lead) that can be purified. In the process, the impure/contaminated metal serves as the anode. As the electrolyte, a salt solution of the metal to be purified is used.

The pure metal precipitates in a finely-distributed fashion at the cathode. In the refining of copper, a copper(II) sulfate solution is usually used.

However, what happens during this electrolysis?

At the anode, in addition to the impure metal, copper is also oxidised and thus enters the solution. At the cathode, this is then reduced and precipitates. The impure metals in the anode (e.g. zinc) are oxidised and go into solution. They are not precipitated at the cathode and remain in the solution. This supernatant is called the anode slime. The following equation shows the electrode reactions.



In this experiment a brass plate, which serves as a substitute for crude copper, will be refined and the amount of precipitated copper will be gravimetrically examined.

Risk assessment

CAUTION! Sulfuric acid and sodium hydroxide are corrosive; wear laboratory coat and goggles. If necessary use safety gloves.

Copper sulfate is an environmental hazard. Do not dispose of in the laboratory drain.



Fig. 1: Set-up and materials for the experiment.

Copper (II) sulfate-5-hydrate	
  Signal word: Caution	<p>Hazard warnings</p> <p>H302 Harmful if swallowed.</p> <p>H319 Causes serious eye irritation.</p> <p>H315 Causes skin irritation.</p> <p>H410 Very toxic to aquatic life with long-lasting effects.</p> <p>Safety information</p> <p>P273 Avoid release into the environment.</p> <p>P302+P352 IF ON SKIN: Wash with plenty of water and soap.</p> <p>P305+P351+P338 IF IN EYES: Rinse carefully with water for several minutes. Remove contact lenses if present and if possible to do so. Continue rinsing.</p>
Sulfuric acid, 95-98 %	
 Signal word: Hazard	<p>Hazard warnings</p> <p>H302 Causes severe skin burns and severe eye damage.</p> <p>H290 May be corrosive to metals.</p> <p>Safety information</p> <p>P280 Wear protective gloves/protective clothing/eye protection.</p> <p>P301+P330+P331 IF SWALLOWED Rinse mouth. Do NOT induce vomiting.</p> <p>P309 + P310 IF exposed or you feel unwell: Immediately call a POISON CENTER or doctor/physician.</p> <p>P305+P351+P338 IF IN EYES: Rinse carefully with water for several minutes. Remove contact lenses if present and if possible to do so. Continue rinsing.</p>
Sodium hydroxide solution, diluted, approx. 2 N (8%)	
 Signal word: Hazard	<p>Hazard warnings</p> <p>H314 Causes severe skin burns and eye damage.</p> <p>H290 May be corrosive to metals.</p> <p>Safety information</p> <p>P280 Wear protective gloves/protective clothing/eye protection.</p> <p>P303+P361+P353 IF CONTACT WITH SKIN OCCURS (or hair): Remove/take off all contaminated, soaked clothing immediately. Rinse skin with water/shower.</p> <p>P305+P351+P338 IF IN EYES: Rinse carefully with water for several minutes. Remove contact lenses if present and if possible to do so. Continue rinsing.</p> <p>P310 Call a POISON CENTER or doctor/physician immediately.</p> <p>P301 + P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.</p> <p>P406 Keep in corrosion-resistant containers with corrosion-resistant linings</p>

Equipment and chemicals

1	Electrochemistry demonstration unit, CPS....	664 4071
1	Panel frame C50, two-level, for CPS.....	666 425
1	Electrochemistry table, CPS.....	666 472
1	Electrochemistry accessories set	664 401
1	Electrolysis cell.....	from 664 401
1	Grindstone.....	from 664 401
1	Tweezers.....	from 664 401
1	Drip pan.....	from 664 401
2	Connecting leads.....	from 664 401
2	Crocodile clips	from 664 401
1	Copper electrode.....	from 664 401
1	Brass electrode	from 664 401
1	Beaker, 250 ml	664 130
1	Beaker, 400 ml	664 131
1	Measuring cylinder, 100 ml	665 754
1	Precision balance	ADAHCB123
1	Cartridge burner	666 714
1	Glass stirring rod	602 783
1	Dropping pipette	665 9531
1	Rubber bulbs.....	665 9541
1	Filter stand	666 584
1	Glass funnel	665 005
1	Folded filter	from 609 081
1	Copper(II) sulfate-5-hydrate, 100 g	672 9600
1	Sulfuric acid, conc., 250 ml	674 7850
1	Sodium hydroxide solution, approx. 2 N.....	673 8400
1	Water, pure, 1l.....	675 3400

Additionally:
Paper towel

Set-up and preparation of the experiment

Set-up of the experiment

Suspend the demonstration unit (Fig. 2) and the electrochemistry table in the panel frame. Place the drip pan in the centre of the electrochemistry table. Fix both half cells in the centre of the electrolysis cell with the screws. The electrolysis cell should now be sealed.

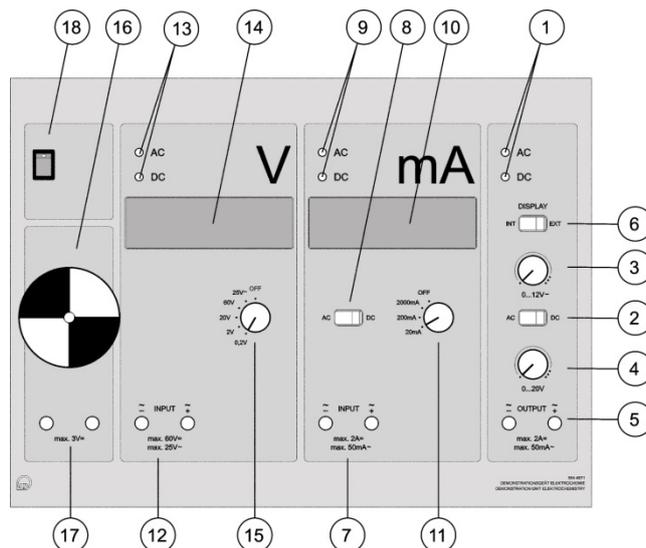


Fig. 2: Sketch of the demonstration unit.

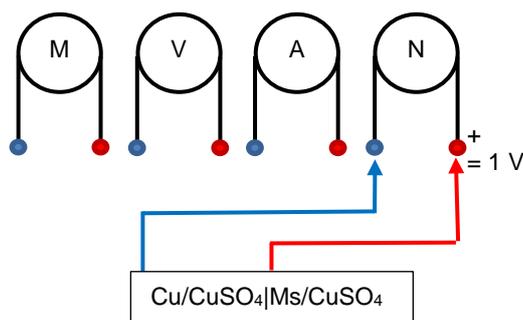


Fig. 3: Circuit for the experiment.

Preparation of the experiment

For a 0.5 molar copper sulfate solution, 19 g (76 mmol) of copper (II) sulfate-5-hydrate are dissolved in 150 ml of distilled water in a beaker (250 ml). 15 g (about 8 – 9 ml) of conc. sulfuric acid is weighed into this solution using a dropping pipette. The solution is then homogenised using a glass stirring rod. A copper and a brass electrode are cleaned with the grindstone and tared on an analytical scale.

The previously tared copper and brass electrodes are suspended in the third outermost groove of the electrolysis cell. Place the electrolysis cell prepared as described in the centre of the electrochemistry table.

Connect the electrodes using a connecting lead and gripping clamp to the power supply (5) of the demonstration unit (see Fig. 2). In the process, connect the positive outlet jack to the brass electrode and the negative outlet to the copper electrode.

Set the changeover switch (2) to direct current (DC) at the power supply (5). Also set the changeover switch (8) and rotary controller (15) to direct current (DC). Place the selector switch (6) to internal power source.

Performing the experiment

Transfer the electrolyte solution to the electrolysis cell. In the process, raise the electrodes somewhat so that the solution also gets behind the electrodes.

At the beginning of the electrolysis, turn on the demonstration unit using the switch (18). On the controller (4), set a voltage of 1 V. Concentration differences that occur during electrolysis can be compensated by lightly shaking the electrolysis cell or by stirring it with a glass stirring rod.

The electrolysis can be ended after 30 minutes by turning off the demonstration unit (switch (18)). Remove the electrodes and dry them over a roaring burner flame. Then, place the electrodes onto a paper towel to let them cool down. Examine the electrodes for any external changes and then determine their weight. What happens?

Observation

The electrodes appear differently on the outside. At the copper electrode, there is a finely distributed precipitate. The brass electrode has irregularities on its surface.

Evaluation

The electrodes were tared prior to the beginning of the experiment. The voltage during the experiment was $U = 1.07$ V and the current was $I = 0.735$ A.

Tab. 1: Experimentally-determined Cu precipitate amount and theoretical comparison.

Electrode	Tare	Δ Tare	Theoret. amount
Brass	25.832 g	- 0.471 g	-
Copper	26.422 g	+ 0.517 g	0.453 g

Deviation: + 14.1 %

The theoretical precipitate amount can be calculated using Faraday's law.

$$m = \frac{I \cdot t \cdot M}{F \cdot z} = \frac{0.735 \text{ A} \cdot 1800 \text{ s} \cdot 63.456 \frac{\text{g}}{\text{mol}}}{F \cdot 2} = 453 \text{ mg}$$

Results

At the copper electrode, finely-distributed copper has precipitated (perform verification reaction IN THE FUME HOOD ONLY: Dissolve in conc. nitric acid). The brass electrode has an uneven surface, due to the dissolution of elemental copper. The additional taring of the two electrodes before and after the electrolysis confirms this observation. The brass electrode has become lighter, and, in contrast, the copper electrode has become heavier.

As can be seen, the electrolytic refining of copper can also be done at laboratory scale. Up or down scaling does not lead to a worsening of the results.

Cleaning and disposal

Transfer the electrolysis solution to a beaker (400 ml). To this solution, add ice-cold sodium hydroxide solution, 2N, in portions. A light blue precipitate will form. Filter this using a folded filter and a glass funnel and filter stand. Add ice-cold sodium hydroxide solution to the filtrate again. If no further precipitation occurs, the solution can be disposed of down the laboratory drain with plenty of water. Dry the filter in the fume hood; it can then be disposed of in the waste for inorganic solids.