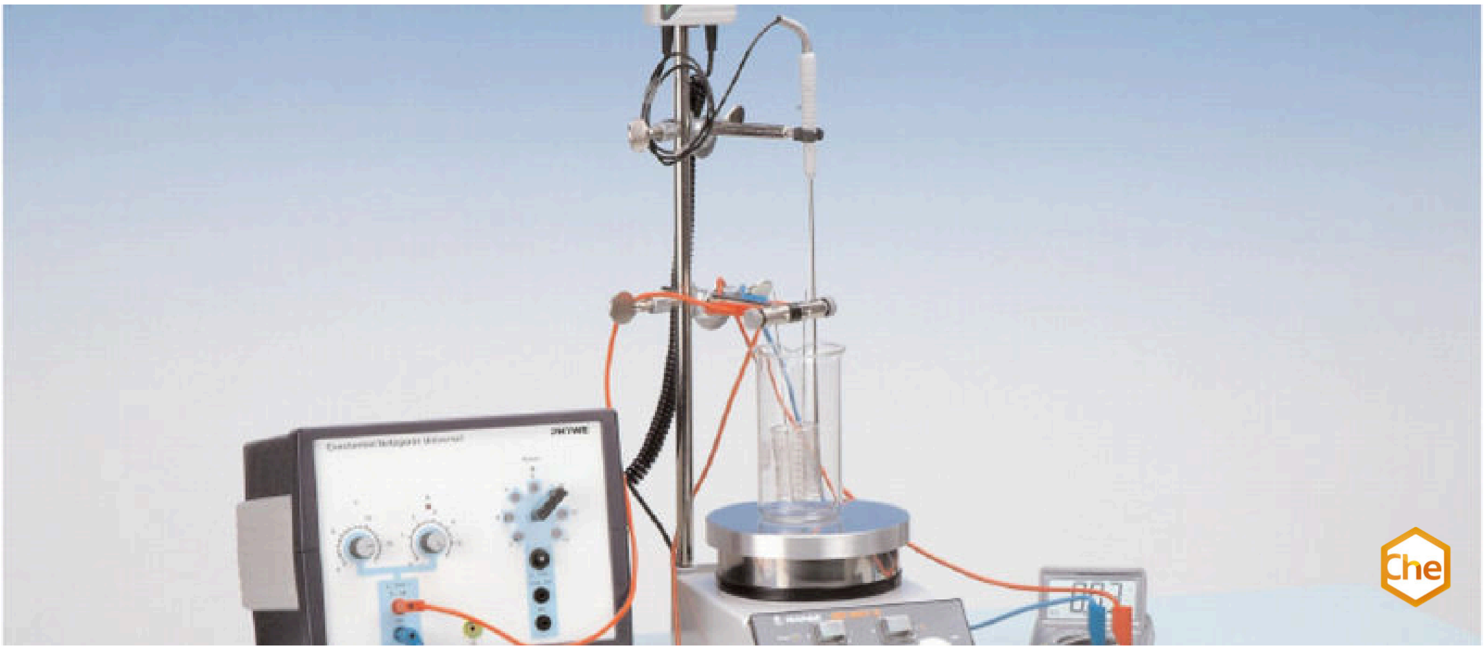
Electrogravimetric determination of copper



Chemistry → Physical chemistry → Electrochemistry → Electrolysis

Chemistry → Analytical Chemistry → Other chemical analysis methods

Difficulty level	Group size	Preparation time	Execution time
hard	2	10 minutes	20 minutes

This content can also be found online at:

http://localhost:1337/c/5f0ed171b6127b00030448d3

General information

Application

Experiment setup

Electrogravimetry is an analytical process that determines the concentration of a substance. Electrogravimetry initially reduces the solute to a solid.

As a rule, metal to be determined is deposited on the cathode as soon as direct voltage has been applied to the electrons. The cathode is precisely weighed before and after electrolysis and the mass is determined by determining the difference.

A platinum mesh is usually chosen as the cathode and a platinum wire as the anode. Electrogravimetric determinations are carried out in sulfuric acid solution in order to prevent oxidation at the anode

Other information (1/2)

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Prior knowledge

- Electrogravimetry serves predominately for the quantitative determination of cations that, on application of an electric current,are either reduced to metal at a cathode, or oxidized to a sparingly soluble oxide at an anode.
- The increase in weight at thecathode or anode at the end of electrolysis is determined by weighing, and from this the quantity of cations or anions can becalculated.

Scientific principle

- Faraday’s laws of electrolysis describe the correlation between the amounts of substances transformed in the reactions at theelectrodes and the charge applied (amount of electricity).
- Faraday’s constant, which appears as a proportionality factor, can bedetermined experimentally from this dependence

Other information (2/2)

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Learning objective

- Faraday's laws apply to the deposition of ions on electrodes.
- 1. Faraday's law The amounts of material deposited are proportional to the amount of current that has flowed through the conductor. The larger and outer electrode is generally chosen as the cathode. Due to the larger surface area, better separation of the copper is possible.

Tasks

- Electrogravimetry is primarily used to determine cations that are cathodically reduced to metal (e.g. Cu, Ag, Au, Ni, Co, Zn, Cd) by the electric current. In this experiment electrogravimetry is used to determine copper.
- The experimental set-up consists of two platmium electrodes. The cathode as a cylindrical wire mesh and the anode in the form of a spiral. The determination of copper is examined by electrogravimetric determination of the amount of copper in a given sample solution.

Safety instructions

Hazards

- Acids have a strong irritating effect!
- Ethanol is highly flammable
- Wear protective glasses!

Ethanol: Highly flammable liquid and vapour. Causes serious eye irritation

Sulphuric acid: May be corrosive to metals. Causes severe skin burns and eye damage

Nitric acid: May intensify fire; oxidizer. Causes severe skin burns and eye damage

Theory

Electrogravimetry is used for the quantitative determination of cations that are either reduced to metal at a cathode, or oxidized to a sparingly soluble oxide at an anode. The increase in weight at the cathode or anode at the end of electrolysis is determined by weighing. By the difference of weight the quantity of cations or anions can be calculated.

Noble metals and Cu, Ni, Co can be deposited in elementary form. For the electrolysis, a voltage is required for the continuous deposition of metals at the cathode, or of metal oxides at the anode. This value of this can be calculated by the difference between the anode potential and cathode potential, and the overvoltage (resulting fromreaction inhibitions) and the ohmic resistance of the electrolyte solution.

At higher temperatures (above 60 ° C) the conductivity can be increased significantly, in order to achieve higher current and faster deposition. Depositing the metals (e.g. Copper), the platinum electrode will be be copper-plated. For electrogravimetric separations the current density should be between 5-50 mA / cm²

Equipment

Position	Material	Item No.	Quantity
1	Magnetic stirrer with heater MRHei-Tec	35752-93	1
2	PHYWE Power supply, universal DC: 0...18 V, 0...5 A / AC: 2/4/6/8/10/12/15 V, 5 A	13504-93	1
3	Digital multimeter, 600V AC/DC, 10A AC/DC, 20 MΩ, 200 µF, 20 kHz, −20°C… 760°C	07122-00	2
4	Pt electrodes, electrogravimetry	45210-00	1
5	Connecting cord, 32 A, 500 mm, red	07361-01	1
6	Connecting cord, 32 A, 500 mm, blue	07361-04	1
7	Connecting cord, 32 A, 750 mm, red	07362-01	2
8	Connecting cord, 32 A, 750 mm, blue	07362-04	1
9	Magnetic stirring bar 30 mm, cylindrical	46299-02	1
10	Supp.rod stainl.st.,50cm,M10-thr.	02022-20	1
11	Spring balance holder	03065-20	1
12	Support rod with hole, stainless steel, 10 cm	02036-01	1
13	Holder for two electrodes	45284-01	1
14	Right angle boss-head clamp	37697-00	2
15	Beaker, Borosilicate, tall form, 250 ml	46027-00	3
16	Volumetric pipette, 50 ml	36581-00	1
17	Graduated pipette 10 ml	36600-00	1
18	Pipettor	36592-00	1
19	Pasteur pipettes, 250 pcs	36590-00	1
20	Rubber caps, 10 pcs	39275-03	1
21	Wash bottle, plastic, 500 ml	33931-00	1
22	Copper-II sulphate,cryst. 250 g	30126-25	1
23	Sulphuric acid, 95-97%, 500 ml	30219-50	1
24	Nitric acid , 65% 1000 ml	30213-70	1
25	Ethyl alcohol, absolute 500 ml	30008-50	1
26	Water, distilled 5 l	31246-81	1

Setup and procedure

Setup (1/2)

Experiment setup

For a quantitative electrogravimetric analysis, the platinum electrodes must be scrupulously clean, and chloride ions should be avoided in all solutions used, since the chlorine that would be produced from them at the anode would attack the platinum.

Clean the electrodes by dipping them into concentrated nitric acid solution (fume hood!), rinsing them thoroughly with distilled water, then drying them, either in an oven at 110°C or by dipping them into pure ethanol and allowing them to air-dry.

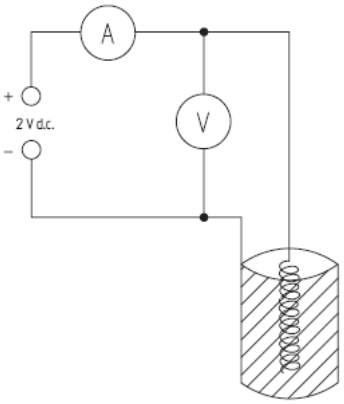
Setup (2/2)

Accurately weigh the cylindrical platinum gauze cathode. Clamp the cathode and the spiral platinum wire anode into the electrode holder and fasten it to the support rod of the magnetic stirrer.

It may be necessary to bend the anode wire slightly, so that it is positioned centrally within the cylindrical cathode. Lower the electrodes into the 250 ml glass beaker so that the magnetic stirrer bar can still freely rotate. Clamp the thermometer into position between the beaker and the cathode.

Platinelectrode

Procedure (1/2)



Electric circuit for electrolysis

Pipette exactly 50 ml of 0.1 M copper sulphate solution and 4 ml of concentrated sulphuric acid solution into the beaker, and add distilled water up to 5-10 mm below the top of the cylindrical wire gauze. As shown in Fig. 2, connect the multimeter used as ammeter in series, the one used as voltmeter in parallel. Adjust the magnetic stirrer to a medium stirring speed and heat the solution up to a temperature of 70-80 °C. Start electrolysis by applying a d.c. potential of +2 V at the anode relative to the cathode.

When the electrolysis current has decreased to approximately one tenth of its initial value (usually after about one hour) the solution should be colourless and electrolysis practically complete. To confirm this, dilute the solution with sufficient distilled water to cover the cathode completely. If, after electrolysing for a further 15 minutes, no fresh copper is deposited on the clean part of the cathode, then, without switching off the voltage supply, remove the thermometer and carefully raise the electrodes.

Procedure (2/2)

- Rinse the electrodes well with distilled water and dry them in an oven or by dipping them into pure ethanol and drying in air.
- Weigh the platinum gauze cathode again and determine the mass of metallic copper deposited from solution.
- The mass of copper is determined by differential weighing of the cathode before and after the deposition.

Evaluation (1/5)

Electrodes in set-up

When two platinum electrodes are dipped into the same CuSO4 solution and a small external voltage applied, however, thencopper deposits at the cathode and oxygen is formed at the anode.

Cathode:

$$Cu^{2+}_{(aq)} + 2\ e^{-} \longrightarrow \mathrm{Cu}_{(s)}$$

Anode:

$$H_2O_{(l)} \longrightarrow O_{2\ (g)} + 4\ H_3O^{+}_{(aq)} + 4\ e^{-}$$

Evaluation (2/5)

The cathode so now becomes a copper electrode and the anode an oxygen electrode, and these have different potentials. Theunattackable platinum electrodes are therefore polarizable. A voltage can be measured between them, the so-called polarizationvoltage. It is opposite to the extremely small voltage applied and prevents further electrolysis. When the external voltage isincreased, the current first hardly changes. From a certain value on, however, a break is to be seen in the current-voltage curveand there is a rapid, linear rise in the current. The value of the voltage here corresponds to the maximum polarization voltage,and is called the decomposition voltage

A high voltage is required to deposit the last weighable amount of copper. The decomposition voltage is so dependent on theconcentration.In practice, a more negative potential than that calculated using Nernst's equation is necessary for the deposition of oxygen, i.e.the development of oxygen is inhibited. This difference is called the overvoltage. Overvoltage is dependent on the electrodematerial, the current density, temperature and the nature of the electrode surface

Evaluation (3/5)

The minimum voltage required to ensure electrolysis may be estimated. An initial concentration of copper ions of 0.1 mol / l is assumed and the electrolysis is regarded as being complete when the concentration has fallen to 0.000001 mol / l. The pH ofthe acidic copper sulphate solution is approximately zero and the overpotential is taken to be +0.47 V. Substitution of thesevalues into equations 4 and 5 leads to a minimum calculated voltage range within which quantitative electrolytic deposition ofcopper at the cathode should occur of 1.39 to 1.54 V.

The calculation is approximate since, overpotentials exist at bothelectrodes, and are not always simple to quantify exactly, especially in stirred solutions. In addition, the solution between theelectrodes represents an ohmic resistance. A certain counter voltage, that is dependent on the type and concentration of theelectrolyte as well as on the temperature, must be employed to overcome it. Electrogravimetry is a very accurate means ofquantitatively determining copper, and it should be possible for the student to determine the copper content of the samplesolution to an accuracy of 1% or better.

Evaluation (4/5)

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Explain electrogravimetric determination

In the electrogravimetric determination of copper, the metal is ______ on a platinum network ______ and determined by ______ of the ______ before and after the deposition. This electrolytic deposition is typical used for the electrogravimetric determination of ______. The larger, outer electrode is chosen as the ______ because the ______ surface area means a better deposition.

- cathode
- metal ions
- larger
- deposited
- differential weighing
- cathode
- cathode

Check

Evaluation (5/5)

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What is the correct formula to calculate the potential of the copper electrolysis (how much voltage is required to deposit the amount of copper on the electrode)?

○ Formula A

○ Formula B

○ Formula C

Check

A)

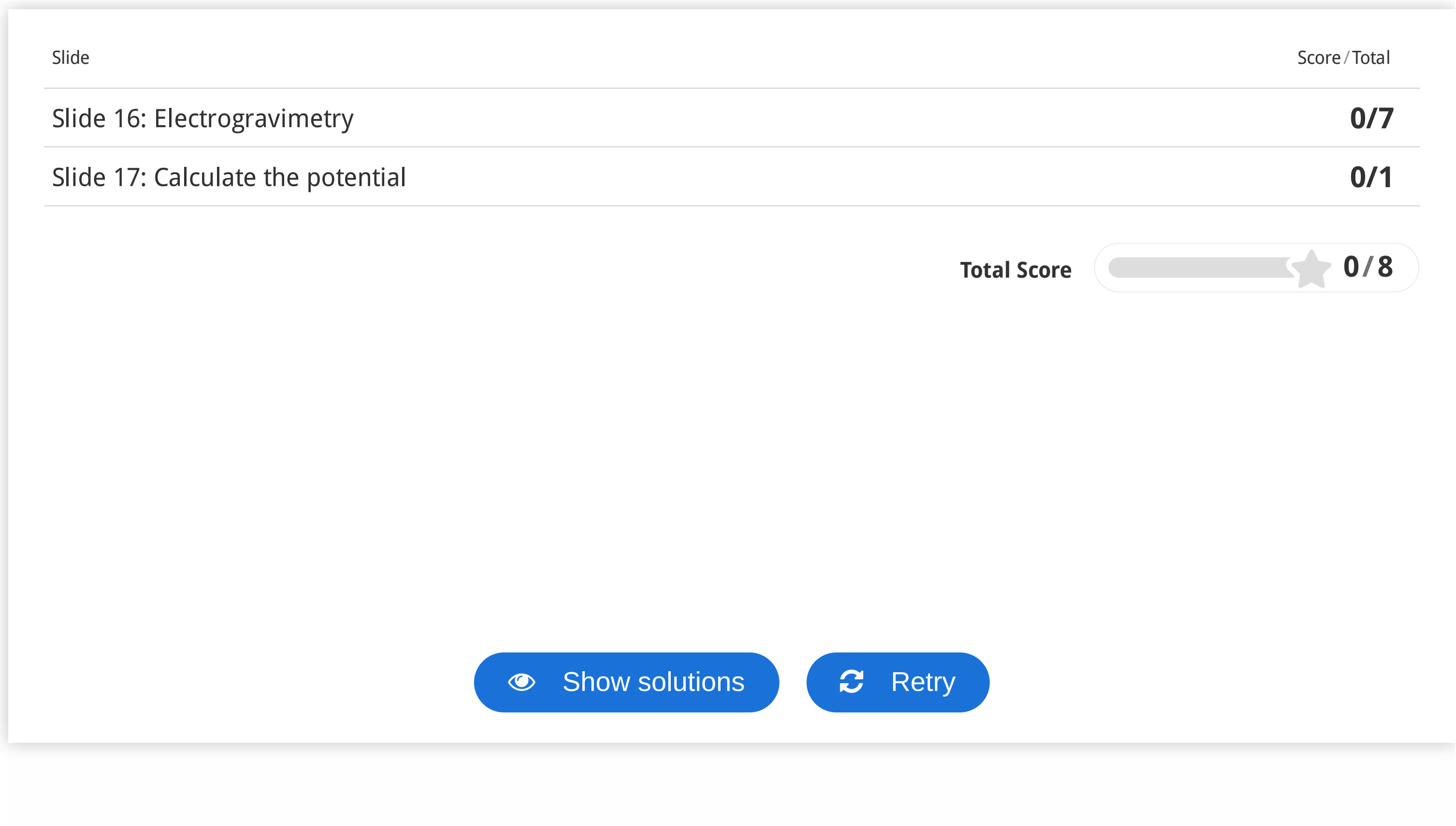
$$E_c = E_{Cu^{2+}|Cu} + \frac{RT}{nF} \ln \alpha_{Cu^{2+}}$$

B)

$$E_c = E_{Cu^{2+}|Cu} + \frac{RT}{F} \ln \alpha_{Cu^{2+}}$$

C)

$$E_c = E_{Cu^{2+}|Cu} + \frac{R}{n} \log \alpha_{Cu^{2+}}$$



Slide	Score / Total
Slide 16: Electrogravimetry	0/7
Slide 17: Calculate the potential	0/1

Total Score 0 / 8