Physical Chemistry

Electrochemistry

Conductivity and ions

Separation of anions and cations

Time required: 60-80 min

Aims of the experiment

- To observe that ions migrate in an electric field.
- To observe the separation of cations and anions in solution.
- To recognise that the ions migrate toward oppositely-poled electrode.

Principles

Ions migrate in an electric field. In the case of coloured ions, the migration can be directly observed. The direction of migration and the speed (by order of magnitude) can be determined easily this way. For example, permanganate anions migrate toward the positively charged electrode and stains the electrolyte solution red/violet. Copper tetraammine cations migrate to the negatively charged electrode and stain the electrolyte solution blue.

The migration rate depends on two competing forces. Firstly, the force exerted by the electric field on the ions which accelerates them. Secondly, the friction that slows down the particles, which is due to interactions between ions of a specific radius and the solvent of a specific viscosity. This force is called *Stokes friction* in the literature.

As the ion radius increases, the migration rate decreases classically.

Fig. 1: Set-up of experiment and materials.

Both species are “complexes”. Complexes are usually inorganic compounds that have a metal atom in the centre. Around this metal atom are molecular ions, which are called ligands. Such complexes are also still in the solution, and are often stable in the gas phase and retain their structure.

In the preparation of the copper tetraammine solution, a ligand exchange takes place. First, there is the copper sulphate aqua complex. Sulphate and water molecules are not very strongly bound to copper.

The reaction is as follows.

$$Cu^{2+} + 4\ NH_3 \rightleftharpoons [Cu(NH_3)_4]^{2+}$$

In this experiment, anions and cations will be separated using an electric field. Here, the colouration of the electrolyte solution will provide a qualitative assessment.

Risk assessment

CAUTION! Potassium permanganate and hydrogen peroxide are strongly oxidising. Ammonia solution is corrosive and has a pungent odour. Avoid skin contact with copper sulphate. Wear safety goggles, lab coat and safety gloves! Do not eat or drink in the vicinity of the experiment. Avoid any sources of fire.

30% hydrogen peroxide	
 **Signal word: Hazard**	**Hazard warnings** H302 Harmful if swallowed. H318 Causes serious eye damage. **Safety information** P280 Wear protective gloves/protective clothing/eye protection/face protection. 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. P313 Get medical advice/attention.

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Potassium permanganate

Signal word: Hazard

Hazard warnings
H272 May intensify fire; oxidiser agent.

H302 Harmful if swallowed.

H410 Very toxic to aquatic life with long-lasting effects.

Safety information
P210 Keep away from heat sources.

P273 Avoid release into the environment.

Copper sulphate

Signal word: Caution

Hazard warnings
H302 Harmful if swallowed.

H319 Causes serious eye irritation.

H315 Causes skin irritation.

H410 Very toxic to aquatic life with long-lasting effects.

Safety information
P273 Avoid release into the environment.

P302+P352 IF ON SKIN: Wash with plenty of water and soap.

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.

Ammonia solution 2 mol/l

Signal word: Hazard

Hazard warnings
H314 Causes severe skin burns and eye damage.

H335 May cause respiratory irritation.

H400 Very poisonous to aquatic organisms.

Safety information
P280 Wear protective gloves and goggles/face protection.

P273 Avoid release into the environment.

P301+P330+P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.

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.

P309+P310 IF exposed or you feel unwell: Immediately call a POISON CENTER or doctor/physician.

Equipment and chemicals

1	Electrochemistry demonstration unit	664 4071
1	Panel frame C50	666 425
1	Electrochemistry table	666 472
1	Double U tube with 2 filters	664 091
1	Platinum leaf rod electrodes, set of 2	664 369
1	Clip plugs, small, set of 2	59002ET2
2	Connecting leads, 25 cm, pair	501 44
1	Erlenmeyer flask, 250 ml, narrow neck	664 243
1	Beaker, 100 ml	602 022
1	Beaker, 250 ml	664 103
1	Beaker, 1000 ml	664 133
1	Scales	ADACB501
1	Powder spatula	604 5682
3	Glass stirring rod	from 665 212ET10
1	Funnel	602 672
1	Folded filter	609 082
1	Filter stand	666 584
1	Ammonium sulfate, 250 g	670 4900
1	Potassium sulfate pentahydrate, 100 g	672 9600
1	Potassium permanganate, 100 g	672 7000
1	Ammonia solution, dilute 2 mol/l, 500 ml	670 3650
1	Hydrogen peroxide, 30%, 250 ml	675 3500
1	Water, pure, 1L	675 3400

Set-up and preparation of the experiment

Preparation of the electrolyte solution

The electrolyte solution is a 20% ammonium sulphate solution. To prepare it, add about 20 g of ammonium sulphate in an Erlenmeyer flask and dissolve in 80 ml of water (pure). Stir the solution until all precipitate has dissolved.

Preparing the separation solution

The solution to be separated consists of 3 parts copper tetraammine solution and one part potassium permanganate solution. Prepare a total of about 50 ml of the separation solution.

1. *Potassium permanganate solution* (c ≈ 0.1 M): Dissolve 0.2 g of $KMnO_4$ in 12.5 ml of water (pure) (use a 100 ml beaker). A deep violet solution will result.
2. *Copper tetraammine solution* (c ≈ 0.1 M): Dissolve 1 g of copper sulphate pentahydrate in 37.5 ml of water (pure) (use a 250 ml beaker). Add ammonia solution to this solution until a deep blue colour remains. The light blue precipitate that forms, copper hydroxide, will have completely dissolved.

Now add the potassium permanganate solution to the copper tetraammine solution and stir.

Set-up of the experiment

Set up the experiment as shown in Figure 1. Fasten the double U tube using the two clip plugs in front. Fasten the two platinum sheet electrodes to one shoulder, respectively. Fasten the electrodes to the outlet sockets (5) of the demonstration unit using the connecting lead (see Figure 2 and 3).

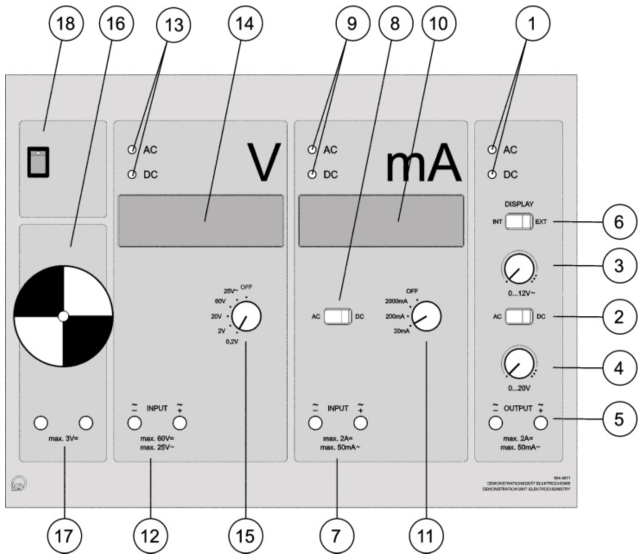

Fig. 2: Sketch of the demonstration unit.

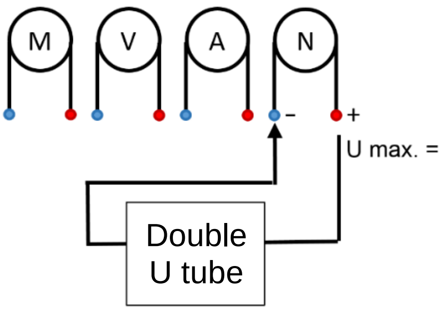

Fig.3: Circuit for the experiment.

Turn on the measurement display (10) and set the changeover switch (8) to direct current (DC).

Performing the experiment

Fill the ammonium sulphate electrolyte solution to about 2 cm below the opening of both outer shoulders. Immediately fill the separation solution in the centre shoulder up to about the same height and close lightly with a stopper. Suspend the sheet electrodes, not inserting the stopper too tightly to let developing gases escape.

Start the experiment. Place the control dial (4) to maximum voltage. Observe the change at the shoulders and respective filters every 10 - 20 minutes.

Observation

A current of 90 – 110 mA flows at a voltage of 11 – 12 V.

10 min: Slight pink colour at the positive filter.

20 min: On the positive side, a slight pink colour of the solution.

30 min: The negative filter becomes light blue, with a more intensive colour at the other side (red).

40 min: The electrolyte solution in the negative shoulder has a slightly blue colour. The positive shoulder is coloured up to about the first quarter.

50 min: The negative shoulder is a somewhat more intensive blue in the lower area. The other side is clearly coloured by permanganate up to just below halfway.

After 60 min, the observations are stopped.

Results

Under the effect of the applied electric field, permanganate and copper tetraammine ions migrate to the electrodes, which are oppositely charged:

$$MnO_4^- \longrightarrow \text{Positive pole}$$

$$[Cu(NH_3)_4]^+ \longrightarrow \text{Negative pole}$$

During migration, it can be seen that permanganate ions are faster than copper tetraammine ions: their mobility is higher.

Cleaning and disposal

The solution in the outer shoulders can be disposed of in the laboratory sink with plenty of water. Collect all residues that contain potassium permanganate in a beaker. Now, add portions of hydrogen peroxide to this solution.

CAUTION: Exothermic reaction. Reacts strongly during oxygen generation. Only carry out in a fume hood. Add hydrogen peroxide in portions, since otherwise foaming can occur.

Once the reaction with hydrogen peroxide has finished, the now brown solution can be separated through a filter and the filtration stand. Dispose of the liquid with plenty of water down the lab drain.

Dry the precipitate, which is manganese oxide MnO_2, in the filter overnight in the fume hood and then dispose of in the solid waste for inorganic solids. Soak the filters overnight with dilute hydrogen peroxide solution in order to remove the permanganate residues.

Disposal reaction:

$$MnO_4^- + H_2O_2 \longrightarrow MnO_2 + H_2O + O_2$$