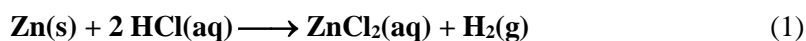


## Preparation of tetraacetato-diaqua-dichromium(II) complex

Tetraacetato-diaqua-dichromium(II) complex is a brick-red powdered substance with the structure depicted in Fig. 1. It is poorly soluble in cold water, but well soluble in diluted strong acids. Heating over 100 °C anhydrous chromium(II) acetate is formed.

Tetraacetato-diaqua-dichromium(II) complex will be prepared by a two-step reaction.

❶ In the first step dark-green chromium(III) chloride is reduced to blue chromium(II) chloride with hydrogen.



❷ In the second step the chromium(II) cation reacts with an excess of acetate anions in acidified solution. A brick-red precipitated product is immediately formed.

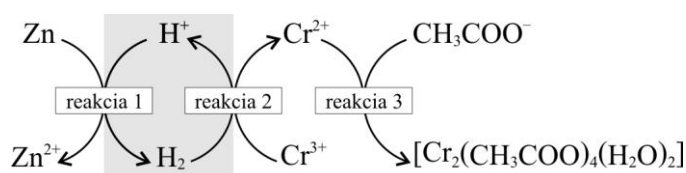
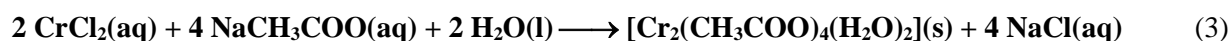


Fig. 2 The principle of the preparation.

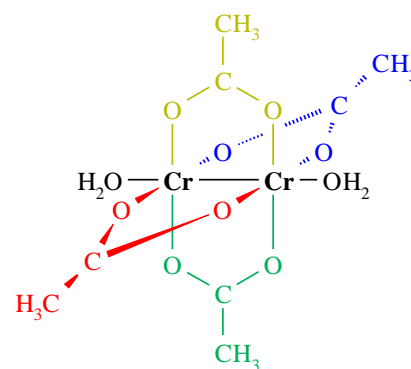


Fig. 1 The structure of tetraacetato-diaqua-dichromium(II) complex. The acetate ligands are color-coded.

### Work

Prepare tetraacetato-diaqua-dichromium(II) complex according to the following procedure.

### Chemikálie

- chromium(III) chloride hexahydrate,  $\text{CrCl}_3 \cdot 6 \text{H}_2\text{O}$ ,
- hydrochloric acid,  $\text{HCl}$ , concentrated,  $w(\text{HCl}) = 0,36$ ,
- zinc,  $\text{Zn}$ , granules,
- sodium acetate trihydrate,  $\text{NaCH}_3\text{COO} \cdot 3 \text{H}_2\text{O}$ ,
- ethanol,  $\text{CH}_3\text{CH}_2\text{OH}$ , denatured spirit,
- diethyl ether,  $(\text{C}_2\text{H}_5)_2\text{O}$ ,
- ice-cooled pre-boiled distilled water.

### Procedure

Built up an apparatus according to the Fig. 3. Fix spring tubing clamps or compressor-type tubing clamps on the rubber hoses. Open the left clamp and close the right one.

Put 9,0 g of zinc into a round-bottom flask. Add chromium(III) chloride solution prepared by mixing 5,0 g of chromium(III) chloride with 17,0 cm<sup>3</sup> of cooled pre-boiled distilled water. Water must be pre-boiled to remove all traces of dissolved oxygen. Add dropwise 16,0 cm<sup>3</sup> of concentrated hydrochloric acid from the dropping funnel depending on the intensity of fizzing of the reaction mixture. The reduction takes 15 to 45 minutes, and it is finished, when the reaction mixture has blue colour.

During the reduction prepare an oversaturated mixture of sodium acetate by mixing 30,0 g sodium acetate trihydrate with 27,0 cm<sup>3</sup> pre-boiled water. Stir the mixture until it is saturated. Pour

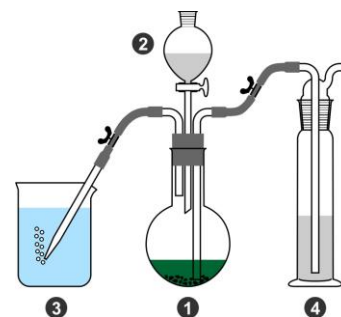


Fig. 3 Apparatus for the preparation of tetraacetato-diaqua-dichromium(II) complex.

1 – round-bottom flask with reaction mixture, 2 – dropping funnel with concentrated  $\text{HCl}$ , 3 – hydrogen detector – beaker with water, 4 – ground-glass gas-washing bottle with sodium acetate solution.

carefully the saturated solution to a gas-washing bottle. The crystals of non-dissolved acetate should remain in the beaker.

Once the reduction is finished, close the left clamp and open the right one. The blue mixture is transported by the hydrogen pressure to the gas-washing bottle with acetate. The brick-red precipitate is formed immediately. Detach the gas-washing bottle from the apparatus, close it with a stopper and shake it for a while rigorously. Then put the gas-washing bottle to an ice bath for around five minutes. Filter out the excluded precipitate on a filter funnel with glass frit. Wash it with ice-cool water, ethanol and finally with diethyl ether. Avoid a contact of the product with air by a layer of used solvents. After washing with diethyl ether, the product should be fully dry and it becomes stable on air. The product should be stored in a well-closed vessel.



chromium(III) chloride solution



chromium(II) chloride solution



dried tetraacetato-diaqua-dichromium(II) complex

### Zinc – Zn

- S7/8** Keep container tightly closed and dry.  
**S43** In case of fire use .. (indicate in the space the precise type of fire-fighting equipment. If water increases the risk add: Never use water).

### Hydrochloric acid – HCl

- R34** Causes burns.  
**R37** Irritating to respiratory system.  
**S2** Keep out of the reach of children  
**S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice

### Chromium(III) chloride hexahydrate – CrCl<sub>3</sub> · 6H<sub>2</sub>O

- R22** Harmful if swallowed.  
**R34** Causes burns.  
**R26/27** Very toxic by inhalation and in contact with skin.  
**S22** Do not breathe dust.  
**S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice  
**S45** In case of accident or if you feel unwell seek medical advice immediately (show the label where possible).  
**S36/37/39** Wear suitable protective clothing, gloves and eye/face protection.

### Sodium acetate trihydrate – NaCH<sub>3</sub>COO · 3H<sub>2</sub>O

–

### Ethyl alcohol – C<sub>2</sub>H<sub>5</sub>OH

- R11** Highly flammable.  
**S7** Keep container tightly closed.  
**S16** Keep away from sources of ignition – No smoking.

### Diethyl ether – (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>O

- R12** Extremely flammable.  
**R19** May form explosive peroxides.  
**S9** Keep container in a well-ventilated place.  
**S16** Keep away from sources of ignition – No smoking.  
**S29** Do not empty into drains.  
**S33** Take precautionary measures against static discharges.