

Coordination compounds II

1. Execution of the exercise

1.1. Complexes and acidity.

- 1.1.1. To a test tube containing 1–2 cm³ of distilled water add a few drops of the solution containing Fe²⁺ ions, a few drops of dimethylglyoxime (DMG) and app. 1 cm³ of **2 M NH₃ aq.** The red colour of the solution indicates the presence of the **Fe(II)-DMG-NH₃** complex (NH₃ is a base of pK_a = 9). Then add about 1 cm³ of **2 M H₂SO₄** to lower the alkalinity of the solution. Discoloration of the solution indicates the instability of the complex in an inert or acidic environment.
- 1.1.2. Add 1 cm³ of **2 M NaOH** and a few drops of **3% oxine** to a test tube containing a few drops of the **Al³⁺** ion solution. The presence of a yellow solid indicates the formation of **Al(III)-Ox** complex. Then add carefully (drop by drop) of **2 M H₂SO₄** and observe the solubility of the complex sediment as the environment is acidified, until the precipitate is completely dissolved. The pH of the solution should be evaluated using a universal pH-indicator paper.
- 1.1.3. Add a drop of Fe³⁺ solution ions to a test tube containing several drops of **NH₄SCN**. Then add a drop of **NH₄F** solution. Discoloration of the solution indicates the complexation of Fe³⁺ ions by fluorides. Then add cautiously, after drops, of the solution of concentrated **H₂SO₄**, until the solution becomes red again. It indicates the presence of the Fe³⁺ complex in the thiocyanate solution.
- 1.1.4. Add a few drops of **2 M NH₃** solution to the solution containing **Cu²⁺** ions. The blue colour of the solution indicates the presence of the copper-ammonia complex. Then carefully add **2 M HCl** dropwise and observing the colour change of the solution.

1.2. Stability of the complex.

- 1.2.1. Add after drops dilute solutions of Fe³⁺ and **NH₄SCN** to the porcelain plate with indentations. Then dilute the thiocyanate iron complex with a few drops of water and add one drop by dropper of **a/ 2 M HCl**, **b/ 2 M H₃PO₄**, **c/ 2 M H₂C₂O₄** to each of the wells. Discoloration of the thiocyanate complex indicates the formation of a permanent complex compound.
- 1.2.2. Perform the experiment on a porcelain plate with depressions by applying dropper solution after a drop of solution according to the following scheme:



2. Development of results

- Based on the observation of the intensity of colouring and on the basis of the literature, write chemical formulas of possible chemical structures of the complexes obtained in the experiment and determine their pK.
- Report in which reactions the masking process occurs.

3. Conclusion

For each of the above mentioned points, give a concise conclusion resulting from the experiment.

4. The scope of material

- Structure of the coordination compounds
- Role of ligands and metal ions in the formation of complexes
- Colour of ligands and complexes
- Masking reactions
- Nomenclature of chemical complexes
- Formation constant for complex

5. Literature

- G. Charlot *Qualitative Inorganic Analysis*, Wiley 2007 (<https://archive.org/details/in.ernet.dli.2015.151602>)
- Ulrich Müller *Inorganic Structural Chemistry, 2nd Edition*, Wiley 2006