Name of the project:	**Digitization of chemistry experiments to improve the quality and support chemistry teaching in secondary schools**
Acronym:	**ChemIQSoc**
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Tittle: Argentometric determination of chlorides

Work instructions

Task: Determine the percentage chloride ion content of the sample by two methods:

1. according to Mohr,
2. according to Volhard.

Theory

Determination according to Mohr

The argentometric determination of Cl^- according to Mohr is based on the direct titration of the sample solution with a volumetric solution of $AgNO_3$ to form a white precipitate of AgCl.

$$\mathbf{Ag^+\,(aq) + Cl^-\,(aq) \rightarrow AgCl\,(s)} \quad (1)$$

It is titrated with the indicator K_2CrO_4, to forms a reddish-brown precipitate of Ag_2CrO_4 with an excess drop of $AgNO_3$.

$$\mathbf{2\ AgNO_3\,(aq) + K_2CrO_4\,(aq) \rightarrow Ag_2CrO_4\,(s) + 2\ KNO_3\,(aq)} \quad (2)$$

The yellow-coloured solution is titrated until the sharp yellow colour dulls to an ochre colour.

Determination according to Volhard

Argentometric determination of Cl^- by Volhard is an indirect determination based on back titration. A known excess of a volumetric solution of $AgNO_3$ is added to the chloride ion sample solution.

$$\mathbf{Ag^+\,(aq) + Cl^-\,(aq) \rightarrow AgCl\,(\downarrow\ white)} \quad (3)$$

The excess $AgNO_3$ is not consumed during chloride ion precipitation. Hence, it is determined by titration with a volumetric solution of NH_4SCN in HNO_3 using ferric ammonium sulfate as the indicator.

$$\mathbf{Ag^+\,(aq) + SCN^-\,(aq) \rightarrow AgSCN\,(\downarrow\ white)} \quad (4)$$

$$\mathbf{2\ Fe^{3+} + 6\ SCN^- \rightarrow Fe[Fe(SCN)_6]\ pink\ color} \quad (5)$$

Equipment: titration flasks, pipettes, scales, volumetric flask (250 ml), burette

Chemicals: silver nitrate, potassium chromate, nitric acid, ammonium thiocyanate, ferric ammonium sulfate (alum)

Procedures:

For both methods, preparation of a common stock solution were done by weighing 0.5 g of sample into a 250 ml volumetric flask.

Determination according to Mohr

1. Pipette 25 ml of the sample stock solution into the titration flask, dilute the solution with a small volume of distilled water and, after the addition of 2 ml of a 5% solution of K_2CrO_4, titrate the yellow-coloured solution with a volumetric solution of $AgNO_3$ until an ochre colour develops.

Determination according to Volhard

1. Using a burette, measure 40 ml of $AgNO_3$ solution into a clean titration flask (thoroughly rinsed with distilled water).
2. Add 25 ml of the sample stock solution to the solution and 10 ml of nitric acid solution at concentration of 4 mol/l.
3. Add 2 ml of 40% ferric ammonium sulfate solution and titrate the solution with NH_4SCN until the first pink colour of the solution appears above the white precipitate of AgCl and AgSCN.

Management of chemical substances

Chemicals	Form	H-statements	P-statements
$AgNO_3$	0,05 mol/l solution	H360d, H410	P202, P273, P280, P308 + P313, P391, P405, P501
NH_4SCN	0,05 mol/l solution	H318	P280, P305 + P351 + P338
K_2CrO_4	5 % solution, indicator	H315, H317, H319, H335, H340, H350i, H410	P202, P273, P280, P302 + P352, P305 + P351 + P338, P308 + P313
HNO_3	4 mol/l solution	H290, H314, H331	P234, P261, P280, P303 + P361 + P353, P304 + P340 + P310, P305 + P351 + P338
$NH_4Fe(SO_4)_2 \cdot 12\ H_2O$	40 % solution, indicator	H318	P280, P305 + P351 + P338

Sources of risk and assessment of risk severity

Silver nitrate: Causes tarnishing. Highly toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. Students should use protective equipment when working with this substance and work with dilute solutions. Acceptable risk.

Ammonium thiocyanate: Releases a highly toxic gas on contact with acids. Harmful by inhalation, skin contact and ingestion. Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment. Students should use protective equipment when working with this chemical substance, work only with dilute solutions, do not work with this chemical in the presence of acids. Acceptable risk.

Nitric acid: It causes fouling which has harmful effects on aquatic organisms and is a strong oxidizing agent. Students use protective equipment when working with this substance, the chemical is poured into an automatic dispenser, they do not come into direct contact with the chemical. Acceptable risk.

Alum indicator: It may irritate the skin, eyes, or respiratory system. The indicator is poured into an automatic dispenser, the student does not come into direct contact with this substance. Acceptable risk.

Potassium chromate: It can cause cancer if inhaled. May cause hereditary damage. Irritating to eyes, respiratory organs, and skin. May cause sensitisation by skin contact. Highly toxic to aquatic organisms, may cause long-term adverse effects in the aquatic environment. The student is not in direct contact with the chemical, which is placed in an automatic dispenser. Acceptable risk.

Waste management method

Under no circumstances should one pour silver nitrate, ammonium thiocyanate, nitric acid down the sink, but into a labelled container. Broken glass must be disposed of in a designated container. Do not return leftover solutions to storage bottles.

Risk reduction measures

Do not expose yourself to prolonged or repeated exposure. In the event of an accident or if you feel unwell, inform the teacher immediately. These substances or their containers must be disposed of as hazardous waste. Prevent the release of potassium chromate, ammonium thiocyanate, silver nitrate, and nitric acid into the environment. Do not eat, drink, or smoke while working, wash hands with warm water and soap after work or when work is interrupted or treat with reparative cream.

Worksheet

Experimental data

1. Write the concentration of silver nitrate from the bottle.
   $c(AgNO_3)$ = ... $mol.dm^{-3}$

2. Preparation of the sample stock solution (differentially 0.5 g in a 250 ml flask)

	weight [g]
Weighing boat with sample	
Weighing boat after emptying	
Sample weight	

3. Record the volume of the silver nitrate solution used in the direct determination according to Mohr.

Titration	**$V(AgNO_3)$ [ml]**
1	
2	
3	

4. Record the volume of the ammonium thiocyanate solution used in the Volhard determination.

Titration	**$V(NH_4SCN)$ [ml]**
1	
2	
3	

Calculations

1. Calculate the chloride content of the solution in % w/w in the Mohr determination.

2. Calculate the chloride content of the solution in wt. % in the Volhard determination.

Questions

1. Write the equation of the reaction that takes place in the determination of chloride according to Mohr.

2. Write the equations of the reactions that take place in the determination of chlorides according to Volhard.

3. Explain how we can indicate the equivalence point in argentometry.

4. Indicate the sources of errors in this method. How would you minimise these errors?

5. Is titration by Volhard direct or indirect method. Explain why?

6. Write the formula of ferric ammonium sulfate and explain why it is added to the solution in the Volhard titration.

Conclusion

Briefly summarize the objective of the experiment, the main results and compare them with the expected values.