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: Determination of ammoniacal nitrogen by distillation

Work instructions

Task: Determine the ammoniacal nitrogen content of the sample in % N by the distillation method.

Theory

The addition of a strong base and heating displaces NH_3 from the ammonium salt, which is quantitatively redistilled into the receiving flask with a known excess of the acid solution. Neutralization takes place:

$$\mathbf{NH_3 + HCl \rightarrow NH_4Cl} \quad (1)$$

The excess acid not consumed in neutralizing the ammonia is determined by back titration with a volumetric solution of NaOH to the indicator methyl red:

$$\mathbf{HCl + NaOH \rightarrow NaCl + H_2O} \quad (2)$$

It is titrated from red to onion colour.

Equipment: burner, tripod, wire gauze, iron ring, clamp, holder, matches, laboratory stand, volumetric flasks (250 ml), Erlenmeyer flask (500 ml), titration flask (250 ml), beakers (1000, 500, 250, 100 ml), pipette (50 ml), pipetting balloon, burette (25ml), heating mantle, funnel, graduated cylinder, syringe, boiling chips

Chemicals: hydrochloric acid (36 %), sodium hydroxide, methyl red

Procedures:

1. For the determination, prepare a stock solution of the sample by weighing 2.5 g of the sample made up in a 250 ml volumetric flask.
2. Pipette 25 ml of sample stock solution into a 500 ml Erlenmeyer flask, dilute with about 100 ml of distilled water and add a few boiling chips.
3. Pipette 50 ml of HCl solution into the titration flask, dilute with about 25 ml of distilled water and add 3 drops of methyl red.
4. The titration flask is placed in a one litre beaker in case it needs to be cooled with water.
5. The Erlenmeyer flask is connected by the transgressor so that the mouth of the transgressor is immersed in the acid solution.

6. Add 20 ml of 30% NaOH solution to the Erlenmeyer flask using a dispenser and immediately cap the flask.
7. A "water plug" is made around the stopper and the drilled hole.
8. The contents of the flask are brought to the boil and boiled for 20 minutes. After this time all the ammonia has been distilled off.
9. The distillation is terminated by lowering the receiving flask so that the end of the transgressor is not immersed in the solution. Only then is the flame switched off!
10. Rinse the end of the transgressor with distilled water and titrate the red-coloured solution with a volumetric NaOH solution until an onion colour develops.

Management of chemical substances

Chemicals	Form	H-statements	P-statements
HCl	0,2 mol/l solution	H315, H319, H335, H290	P261, P305 + P351 + P338
NaOH	0,2 mol/l solution	H315, H319	P280, P302 + P352
NaOH	30 % solution	H314, H290	P280, P310, P305 + P351 + P338
Methyl red	0,1 % solution, indicator	---	---

Sources of risk and assessment of risk severity

Methyl red is not a dangerous substance within the meaning of Act No.356/2003 Coll. The student works with a solution of methyl red with a concentration of 0.1%–indicator in an indicator bottle. Acceptable risk.

Sodium hydroxide, 30% solution–work with gloves and goggles. Acceptable risk.

The hydrochloric acid and sodium hydroxide concentrations used are 0.2 mol/dm^3, a very dilute solution. Acceptable risk.

Waste management method

Broken glass must be disposed of in a designated container. Do not return leftover solutions to storage bottles. Prevent contamination of surface and groundwater and soil. Formaldehyde, 30% sodium hydroxide must not be allowed to enter drains, risk of explosion. Dispose of remaining solutions as instructed by the teacher and teaching assistant.

Risk reduction measures

Avoid direct exposure to formaldehyde, use protective equipment. 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 chemicals into the environment. Do not eat, drink, or smoke while working, wash hands with warm water and soap after work or when interrupting work or treat with restorative cream. Use personal protective equipment, observe personal hygiene. Avoid contact with the substance, do not inhale fumes.

Worksheet

Experimental data

1. Study the working procedure and derive equation for calculating the mass % N for the distillation determination.

2. Preparation of sample stock solution (differentially 2.5 g into 250 ml flask)

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

3. Record the volume of the volumetric sodium hydroxide solution used in the titration.

Titration	**V(NaOH) [ml]**
1	
2	
3	

Calculations

1. Calculate the chemical amount of substance not consumed in the neutralization of ammonia.

2. Calculate the % ammoniacal N content of the sample.

Questions

1. Write the equations of the reactions that takes place in the distillation determination of ammoniacal nitrogen.

2. How the acid-base indicator works.

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

4. Is the distillation determination of ammonia nitrogen direct or indirect method. Explain why?

5. Why must the mouth of the transgressor between the Erlenmeyer flask and receiving flask extend below the level of the acid solution in the receiving flask?

Conclusion

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

Disclaimer

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