Organic chemistry

Organic compounds

Composition of organic compounds

Quantitative determination of carbon

Aims of the experiment

- To determine the composition of organic compounds
- To learn about quantitative analysis
- To analyse the proportion of carbon in a compound
- To determine the structure of organic compounds
- To learn an example of the application of a redox reaction

Principles

Organic chemistry is the chemistry of carbon compounds. The term was coined by Berzelius, as it was assumed in the 19th century that organic compounds can only be formed by living organisms. In this day and age, however, many organic compounds are produced synthetically, particularly ones which do not occur in nature.

Besides carbon atoms, organic compounds also contain hydrogen atoms. Compounds which only consist of these two atoms are referred to in simplified terms as hydrocarbons. There are further subdivisions within the hydrocarbons which are differentiated, for example, according to the bonds present. The carbon atom itself is always tetravalent.

The simplest of these families of substances are the so-called alkanes. They are referred to as saturated hydrocarbons, as each of the four possible bonds has a binding partner and is therefore saturated.

The general formula of saturated hydrocarbons present in the form of chains is:

$$C_nH_{2n+2}$$

A third element which occurs in many organic compounds is oxygen. An example of this are the alcohols which, based on the term alkane, are also called alkanoles with the general sum formula:

$$C_nH_{2n+1}OH$$

Hydrocarbons with more than three carbon atoms can be linked to each other in various ways. They can exist, for example, as long-chain or branched-chain molecules, or in a ring formation. Included in the chain-linked alkanes are the so-called constitutional isomers, which have the same sum formula but are linked in branches and therefore display a different structure.

Life today without hydrocarbons is unimaginable, as they represent indispensable products in everyday life. These include, for example, natural gas, heating oil, petrol and candle wax. However, they are also highly significant as a raw material for the production of plastics, such as polyethylene. At a proportion of about 29%, polyethylene is the most commonly produced plastic world-wide.

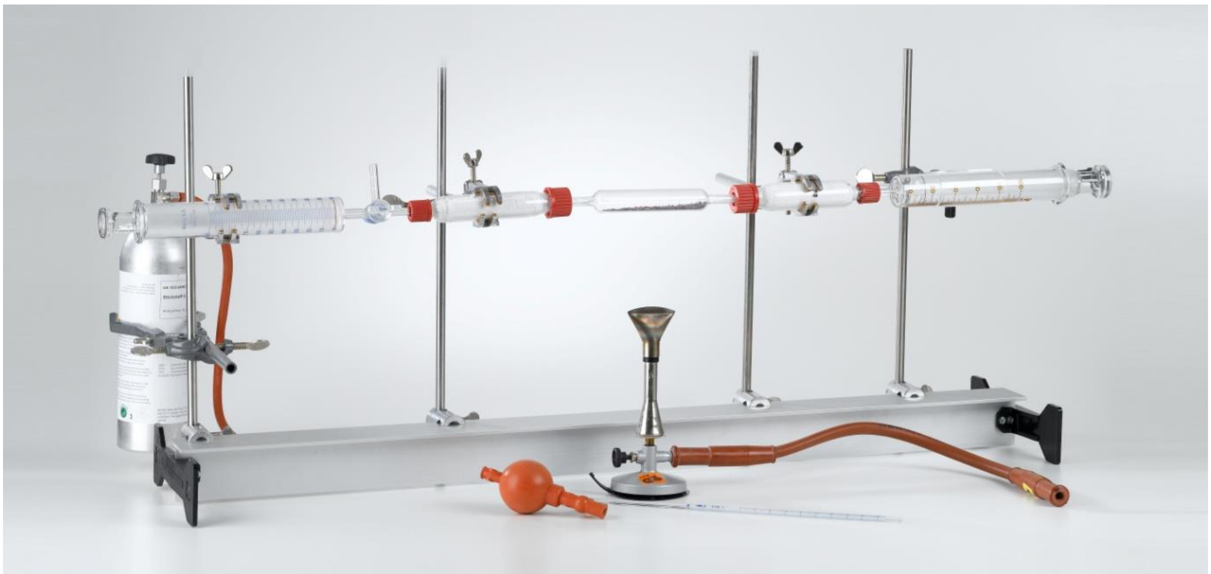

Fig. 1: Set-up of the experiment

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The quantitative analysis of a substance is a method by which the exact proportion of a substance contained in a sample is determined. Justus Liebig developed the method of elemental analysis for organic compounds. The exact proportions of carbon, hydrogen and oxygen can be determined based on this analysis. From this analysis, conclusions can then be drawn on the sum formula and to some extent on the structure of the sample.

In this experiment, the exact proportion of carbon in an alkane and in an alcohol is to be determined. To achieve this, the CO_2 that is liberated on combustion of the sample in an oxygen-enriched combustion tube is collected and the carbon content calculated based on the volume increase.

Risk assessment

N-butane and 1-propanol are both extremely flammable substances. It is imperative that they are kept away from external sources of ignition and protected from electrostatic discharges.

The apparatus should be set up behind a screen or in a fume cupboard to provide protection from flying pieces of glass in the case of a fire or an explosion.

Only extinguish a fire if the leak can be stopped safely, otherwise flammable gas will continue to leak out.

Copper(II) oxide	
Signal word: Caution	**Hazard statements** H302 Harmful if swallowed. H410 Very toxic to aquatic life with long-lasting effects. **Precautionary statements** P260 Do not breathe dust. P273 Avoid release to the environment.

n-butane	
Signal word: Hazard	**Hazard statements** H220 Extremely flammable gas. H280 Contains gas under pressure; may explode if heated. **Precautionary statements** P210 Keep away from heat/sparks/ open flames/hot surfaces. No smoking. P377 Leaking gas fire: Do not extinguish unless the leak can be stopped safely. P381 Eliminate all ignition sources if safe to do so. P403 Store in a well ventilated place.

1-Propanol	
Signal word: Hazard	**Hazard statements** H225 Highly flammable liquid and vapour. H318 Causes serious eye damage. H336 May cause drowsiness or dizziness. **Precautionary statements** P210 Keep away from heat/sparks/ open flames/hot surfaces. No smoking. P233 Keep container tightly closed. P280 Wear protective gloves/protective clothing/eye protection/face protection. P305+P351+P338 If in eyes: Rinse continuously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing. P313 Get medical advice/attention.

Calcium chloride	
Signal word: Caution	**Hazard statements** H319 Causes serious eye irritation. **Precautionary statements** P305+P351+P338 If in eyes: Rinse continuously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing.

Nitrogen	
Signal word: Caution	**Hazard statements** H280 Contains gas under pressure; may explode if heated. **Precautionary statements** P403 Store in a well ventilated place.

Equipment and chemicals

1	Reaction tube, quartz glass, 220 x 25 mm Ø	664 069
2	Drying tube, 1 x GL 25 + 1 x GL 18	665 374
2	Gas syringe, 100 ml with three-way stopcock	665 9148
1	Teclu burner, universal	656 017
1	Wide-flame attachment	666 724
1	Safety gas hose, 1 m	666 729
1	Base rail 95 cm	666 603
2	Stand tube 450 mm x 10 mm Ø, set of 2	666 609ET2
4	Universal bosshead	666 615
5	Bosshead S	301 094
4	Universal clamp 0...80 mm	666 555
1	Universal clamp 0...120 mm	301 72
1	Double-ended spatula, stainless steel, 150 mm	666 962
1	Graduated pipette 1 ml	665 994
1	Pipetting ball (Peleus ball)	666 003
1	Rubber tubing 7 mm Ø, 1 m	667 180
1	Rubber tubing 1 m x 4 mm Ø, DIN 12865	604 481
1	Tubing connector, PP, straight, 4/15 mm Ø	604 510
1	Funnel	665 004
1	Minican gas can, nitrogen	661 000
1	Minican gas can, n-butane	660 989
1	Fine regulating valve for Minican cans	660 980
1	Tweezers, blunt, 200 mm	667 034
1	Copper oxide, wire form, 250 g	672 9410
1	Calcium chloride, granulated, 250 g	671 2410
1	1-Propanol, 250 ml	674 4310
1	Glass wool, 100 g	672 1010

Set-up and preparation of the experiment

Set-up of the apparatus

1. The experiment is set up as shown in Fig. 1.

Preparation of the experiment

1. First prepare the two drying tubes. For this, push a little glass wool into one end of the tube and then fill it with calcium chloride. Then close also the other end with glass wool.

Note: The drying tubes are easier to fill with calcium chloride using a funnel.

2. Place the copper oxide in wire form into the quartz glass reaction tube using tweezers; just enough to cover the entire surface with the tube in a horizontal position.

3. Then set up the apparatus by clamping the stand tubes to the base rail using universal bossheads. Fasten the universal clamps to the stand tubes using S bossheads. Now the gas syringes, the filled drying tubes and the reaction tube can be fixed to the stand system and connected together in order.

4. Connect the nitrogen bottle to one of the gas syringes with a three-way stopcock by screwing the fine regulating valve onto the Minican gas can and connecting this to the three-way stopcock with 4 mm rubber tubing.

5. Now flush out the entire apparatus with nitrogen until it is free of air. To do this, lead the gas to the outside through the three-way stopcock of the second gas syringe. Following this, empty the apparatus by fully pushing in the pistons of the gas syringes.

Note: It is important that the apparatus is free of air, as the reaction would otherwise not proceed optimally owing to interference through oxygen from the air.

6. Then remove the Minican gas can and close the valve well, then fill the apparatus with 25 ml of n-butane gas in the same way.

Note: No air must enter the apparatus during the filling process. After this, remove the gas can well away from the experiment bench.

7. For the experiment with 1-propanol, measure out 0.12 ml of 1-propanol with a pipette and add it to the copper oxide wire in the reaction tube. Then flush out the apparatus with nitrogen.

Performing the experiment

1. Set the three-way stopcocks of the gas syringes such that they are only open to the reaction tube.

2. Position the universal Teclu burner under the reaction tube. Place the burner on a laboratory stand, if necessary.

3. Light the burner and heat the copper oxide until it glows red.

4. **Experiment with n-butane** Pass the n-butane several times over the copper oxide until no further increase in volume can be observed.

5. **Experiment with 1-propanol** Leave the burner under the reaction tube until no further increase in volume can be observed.

6. Turn the burner off and allow the apparatus to cool down.

7. After each experiment, set the volume in one gas syringe to 0 and read off the volume on the other gas syringe.

Observation

While pushing the pistons of the gas syringes backwards and forwards, it can be seen that the volume of gas that is formed on combustion of both substances increases in the apparatus. This procedure continues until a constant gas volume has been reached. From 25 ml of n-butane used, about 100 ml of gas results, and from 0.115 ml of 1-propanol, 107.5 ml of gas. In the reaction tube it can be seen that elemental copper is formed during the experiment (red colour).

Evaluation

By passing n-butane over the glowing copper oxide, or through the presence of 1-propanol in the reaction tube, the volume of gas in the apparatus increases. The gas formed is CO_2. In addition, elemental copper (red colour) is partly formed from the black copper oxide as well as water, which is bound by the calcium chloride. The reaction observed is a redox reaction.

The general reaction equation for this reaction for n-alkanes with the general sum formula C_nH_{2n+2} is:

$$C_nH_{2n+2} + 3n_{+1}CuO \rightarrow n\,CO_2 \; + n_{+1}H_2O + \; 3n_{+1}Cu$$

For alcohols such as 1-propanol, the reaction equation is:

$$C_nH_{2n+1}OH + 3n\,CuO \rightarrow n\,CO_2 \; + n_{+1}H_2O + 3n\,Cu$$

The resulting gas volume is read off for the evaluation. In the case of n-butane, the gas volume quadruples from 25 ml to almost 100 ml. In the experiment with 1-propanol, 107.5 ml of gas results. In both cases, the gas formed is CO_2.

The general reaction equations can be used for the evaluation.

If the volume of gas quadruples, as in the case of butane, then four volumes of CO_2 result from one volume of n-butane. This means that n-butane contains four carbon atoms.

The sum formula for n-butane is therefore C_4H_{10}.

To evaluate the experiment with 1-propanol, the number of C atoms must be calculated based on the resulting volume of CO_2 and compared with the information in the literature. For this, the following literature values are needed:

V(resulting CO_2) = 107.5 ml

$\rho(CO_2)$ = 1.98 g/l

M(CO_2) = 44.01 g/mol

M(C) = 12.01 g/mol

M(1-propanol) = 60.097 g/mol

Calculating the mass of the resulting CO_2

$m(CO_2) = V(CO_2) * \rho(CO_2)$

$= 0.1075\ l * 1.98\ g/l$

$= 0.21285\ g$

Calculating the amount of C in CO_2

$$m(C) = \frac{M(C) * \text{number of C-atoms} * m(CO_2)}{M(CO_2)}$$

$$= \frac{12{,}01\ g/mol * 1 * 0{,}21285\ g}{44{,}01\ g/mol}$$

$= 0.058\ g$

Calculating the mass of 1-propanol

$m(\text{1-propanol}) = V(\text{1-propanol}) * \rho(\text{1-propanol})$

$= 0.12\ ml * 0.786\ g/ml$

$= 0.094\ g$

Calculating the number of C atoms in 1-propanol

$$\text{number of c-atoms} = \frac{m(C) * M(\text{1-propanol})}{M(C) * m(\text{1-propanol})}$$

$$= \frac{0{,}058\ g * 60{,}097\ g/mol}{12{,}01\ g/mol * 0{,}094\ g}$$

$= 3.09 \approx 3$ C atoms

The sum formula for 1-propanol is therefore C_3H_7OH.

Results

In this experiment, carbon dioxide, water and elemental copper are formed in a redox reaction. With the help of the volume increase, conclusions can be drawn on the resulting volume of CO_2. With this again, the number of carbon atoms in the compound analysed can be calculated.

In this experiment, 3.09 C atoms were calculated for 1-propanol and 4 C atoms for n-butane. According to the literature, 1-propanol has the sum formula C_3H_8O and butane C_4H_{10}. With the help of this experiment, it was therefore possible to draw conclusions in each case on the number of C atoms present. The small discrepancies result from measurement inaccuracies.

The experiment can of course be performed with other compounds, such as methane and ethane.

Cleaning and disposal

The glass wool and the calcium chloride can be dried, stored in appropriately labelled containers and used again in similar experiments. The reduced copper(II) oxide rods can be reprocessed by oxidation to form copper(II) oxide again. Following this, store in a appropriately labelled container and use again in similar experiments. Otherwise they must be disposed of in the heavy metal waste container. The copper mirror can be removed from the reaction tube by treatment with nitric acid. *Note: Caution: nitrous gases are formed in the process. The cleaning must be carried out in the fume cupboard.*