Quantitative determination of hydrogen in organic compounds

Aims of the experiment

- Determine the composition of organic compounds.
- Learn about the process of thermal analysis.
- Analyse the content of hydrogen in a compound.
- Determine the structure of organic compounds.

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 substances could 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.

These organic compounds also contain hydrogen atoms besides carbon atoms. Compounds which only consist of these 2 atoms are referred to in simplified terms as hydrocarbons. There are further subdivisions within the hydrocarbons, these differing depending on the bonds present, for example. The carbon itself is always tetravalent.

The simplest of these families 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 for the saturated hydrocarbons, if they are present in chain form, is:

$$C_nH_{2n+2}$$

A third element that occurs in many organic compounds is oxygen. An example of this is the alcohols, which, based on the alkanes, are also called alkanols, with the following empirical formula:

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

Hydrocarbons with more than three carbon atoms can be linked to each other in various ways. They can be present as long-chain, branched or ring-shaped molecules. The chain-like alkanes include the so-called constitutional isomers, which have the same empirical formula but which are linked in branches, and hence exhibit a different structure.

Modern society would be unimaginable without hydrocarbons as they form indispensable products in everyday life. These typically include natural gas, heating oil, petroleum and candle wax. However, they are also very important as a starting material for the production of plastics such as polyethylene. Polyethylene is the most widely produced plastic in the world with a share of about 29 percent.

The quantitative analysis of a substance involves a method used to determine the precise percentage of a substance in a sample. Justus Liebig developed the method of elementary analysis for organic compounds. Based on this analysis, the precise percentage of carbon, hydrogen and oxygen can be determined. Conclusions can then be drawn from this in regard to the empirical formula and sometimes the structure of the sample.

The precise percentage of hydrogen alkane (butane C_4H_{10}) and an alcohol (1-propanol C_3H_7OH) is to be determined in this experiment. For this, the H_2O resulting during combustion of the sample in an oxygen-free reaction tube is absorbed by

Fig. 1: Set-up of the experiment.

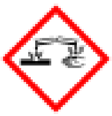
calcium chloride and calculated via the mass gain of the hydrogen component.

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 constructed 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 inflammable 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. **Safety statements** P260 Do not inhale 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. **Safety 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. **Safety 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. **Safety statements** P403 Store in a well ventilated place.

Equipment and chemicals

1	Reaction tube, quartz glass, 220 x 25 mm diam.	664 069
2	Drying tube, 1xGL 25 + 1xGL 18	665 374
2	Gas syringe 100 ml, with three-way stopcock	665 914
1	Gas syringe holder	665 918
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 tubes, 450 mm, 10 mm diam., set of 2	666 609ET2
4	Universal bosshead	666 615
5	Bosshead S	301 094
3	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 tube 7 mm diam., 1 m	667 180
1	Rubber tube 1 m x 4 mm diam., DIN 12865	604 481
1	Tube connector, PP, straight, 4/15 mm diam.	604 510
1	Funnel Boro 3.3, 80 mm diam.	665 004
1	Analytical balance ABS 220-4N	667 7990
1	Minican pressurised gas canister, nitrogen	661 000
1	Minican pressurised gas canister, n-butane.	660 989
1	Fine regulating valve for Minican canisters	660 980
1	Tweezers, blunt, 200 mm.	667 034
1	Copper oxide, wire form, 250 g	672 9410
1	Calcium chloride, encap., 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. The two drying tubes must be filled before the experiment. For this, add a little glass wool to one end and then fill with

calcium chloride. The other end is also closed with glass wool.

Note: The drying tubes can be filled more easily with calcium chloride if a funnel is used.

2. Weigh the two drying tubes before the experiment and note the weight.

3. Add the copper oxide in wire form to the quartz glass reaction tube using the tweezers; just enough so that the entire bottom area is covered when horizontal.

4. Then set up the apparatus by fastening the stand tubes on the base rail using the universal sockets. Fasten the universal clamps on the stand tubes using the bossheads S. The gas syringes, filled drying tubes and reaction tube can now be fastened in order on the stand system and connected to one another.

5. Connect the nitrogen gas canister with three-way stopcock to one of the gas syringes by screwing the fine regulating valve onto the Minican pressurised gas canister and connecting this to the three-way stopcock using a rubber tube, 4mm.

6. The complete apparatus is now flushed with nitrogen to free it of air. For this, lead the gas to the outside through the three-way stopcock of the second gas syringe. Then empty the apparatus by sliding the flask of the gas syringe in fully.

Note: It is important for the apparatus to be free of air, as otherwise the reaction will not progress optimally as the oxygen from the air would disrupt it.

7. Now remove the Minican pressurised gas canister and seal well, then fill the apparatus with 25 ml n-butane gas in the same way.

Note: Air must not enter the apparatus during filling. After this remove the gas cylinder from the experimentation site.

8. For the experiment with 1-propanol, measure off 1.15 ml 1-propanol with a pipette and add to the copper oxide wires in the reaction tube. Then flush with nitrogen.

Performance

1. Set the three-way stopcocks of the gas syringes so that these are only connected to the reaction tube.

2. Position the Bunsen burner below the reaction tube. If necessary, place the burner on a laboratory jack-stand.

3. Ignite the burner and heat the copper oxide until it becomes red hot.

Experiment with n-butane

4. Guide the n-butane several times over the glowing copper oxide. Do this until no further volume increase can be observed.

Experiment with 1-propanol

5. Leave the burner under the reaction tube until no further volume increase can be observed.

6. Extinguish the burner and allow the apparatus to cool.

7. After the relevant experiment, weigh the two drying tubes again and note the weights.

Observation

Whilst pushing both syringe pistons back and forth, it is possible to observe how the volume of gas increases in the apparatus during both experiments. Continue this procedure until a constant gas volume is reached. 25 ml applied n-butane result in almost 100 ml gas, while 1.15 ml liquid 1-propanol produce 30 ml gas. It can be seen in the reaction tube that elementary copper forms during the experiment (red colouration). The two drying tubes have also increased in mass.

Evaluation

Experiment with n-butane

Guiding the n-butane over the flowing copper oxide or the presence of 1-propanol results in a volume increase by the gas present in the apparatus. The resultant gas is CO_2. The black copper oxide also partially yields elementary copper (red colouration) and water, which is bound by the calcium chloride. The reaction observed is a redox reaction.

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

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

The reaction equation for alcohols such as 1-propanol is:

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

The mass increase of the drying tubes is required for the evaluation for both experiments, so that conclusions can be drawn regarding the volume of H_2O resulting. The measured values determined in the experiment with n-butane are summarised in Tab.1.

Tab. 1: Measured values determined in the experiment with n-butane.

	Mass / Volume
m (drying tube 1_{before})	110.013 g
m (drying tube 2_{before})	107.000 g
m (drying tube 1_{after})	110.034 g
m (drying tube 2_{after})	107.090 g
V (n-butane)	25 ml

Calculation of *m* (n-butane)

The applied mass m of n-butane must first be calculated. The density ρ is required for this; this can be taken from the literature (ρ = 2.67 g/L).

The mass is calculated according to the following formula:

m (n-butane) = ρ (n-butane) * V (n-butane)

m (n-butane) = 2.67 g/L * 0.025 L

m (n-butane) = 0.0668 g

Calculation of the resultant *m* (H_2O)

As the resultant H_2O is bound in the calcium chloride, m (H_2O) can be calculated via the mass increase in the drying tubes.

m (H_2O) = (m (drying tube 1_{after}) - m (drying tube 1_{before}))

+ (m (drying tube 2_{after}) - m (drying tube 2_{before}))

m (H_2O) = (110.034 g - 110.013 g) + (107.090 g - 107.000 g)

m (H_2O) = 0.021 g + 0.090 g

m (H_2O) = 0.111 g

Calculation of the *m* (H)

The mass of hydrogen released during the experiment can be calculated via the mass of H_2O now known, taking into account the molar mass M of hydrogen and oxygen. The molar masses can be taken from the literature for this.

The following applies: M (H_2) = 2.0156 g/mol

M (H_2O) = 18.015 g/mol

m (hydrogen) = m (H_2O) * M (H_2) / M (H_2O)

m (hydrogen) = 0.111 g * 2.0156 g/mol / 18.015 g/mol

m (hydrogen) = 0.0124 g

Calculation of the percentage of hydrogen in n-butane

The following formula is used to calculate the percentage:

$$\text{Proportion H (\%)} = \frac{m\text{ (H)}}{m\text{ (n-butane)}}$$

0.0124 g / 0.0668 g = 18 % H are contained in n-butane.

Calculation of the theoretical percentage of hydrogen in n-butane

To classify the measuring results, the percentage of hydrogen determined practically is compared with the theoretical values. The molar mass M of n-butane must first be calculated for this:

$$M\text{ }(C_4H_{10}) = 4 * M\text{ (C)} + 10 * M\text{ (H)}$$

$$= 4 * 12.011\text{ g/mol} + 10 * 1.008\text{ g/mol}$$

$$= 58.124\text{ g/mol}$$

$$\frac{\text{Number H atoms * M (H)}}{\text{M (n-butane)}} = \frac{10 * 1{,}008\text{ g/mol}}{58{,}124\text{ g/mol}} = 17\ \%$$

Calculation of the number of hydrogen atoms in n-butane

$$\text{Number H atoms} = \frac{\text{\% Proportion} * M\text{ (n-butane)}}{100}$$

$$= \frac{18 * 58{,}124\text{ g/mol}}{100}$$

Number of H-atoms = 10.4 ≈ 10

Experiment with 1-propanol

Table 2 summarises all data required for the experiment with 1-propanol.

Tab. 2: Measured values determined in the experiment with n-propanol.

Required data	Values
m (drying tube 1 before)	110.034 g
m (drying tube 2 before)	107.090 g
m (drying tube 1 after)	110.074 g
m (drying tube 2 after)	107.147 g
V (1-propanol)	0.115 ml

Calculation of *m* (1-propanol)

The applied mass m of 1-propanol must first be calculated again; the density is required for this. This can be taken from the literature (ρ = 0.83 g/ml).

m (1-propanol) = ρ (1-propanol) * V (1-propanol)

m (1-propanol) = 0.83 g/ml * 0.115 ml

m (1-propanol) = 0.096 g

Calculation of the resultant *m* (H_2O)

The resultant mass of water is then calculated again.

$$m\text{ }(H_2O) = (m\text{ (drying tube }1_{after}) - m\text{ (drying tube }1_{before}))$$
$$+ (m\text{ (drying tube }2_{after}) - m\text{ (drying tube }2_{before}))$$

m (H_2O) = (110.074 g - 110.034 g) + (107.147 g - 107.090 g)

m (H_2O) = 0.040 g + 0.057g

m (H_2O) = 0.097 g

Calculation of the *m* (H)

In the next step the volume of hydrogen is calculated.

m (hydrogen) = m (H_2O) * M (H_2) / M (H_2O)

m (hydrogen) = 0.097 g * 2.0156 g/mol / 18.015 g/mol

m (hydrogen) = 0.0109 g

Calculation of the % of H in 1-propanol

The following formula is used to calculate the percentage:

$$\text{Proportion H (\%)} = \frac{m\text{ (H)}}{m\text{ (1-propanol)}}$$

0.0109 g / 0.096 g = 11 % H are contained in n-butane.

Calculation of the theoretical percentage of H in 1-propanol

To classify the measuring results, the percentage of hydrogen determined practically is again compared with the theoretical values. The molar mass M of 1-propanol must first be calculated for this:

$$M\text{ }(C_3H_8O) = 3 * M\text{ (C)} + 8 * M\text{ (H)} + 1 * M\text{ (O)}$$

$$= 3 * 12.011\text{ g/mol} + 8 * 1.008\text{ g/mol} + 1 * 16\text{ g/mol}$$

$$= 60.097\text{ g/mol}$$

$$\frac{\text{Number (H)} * M\text{ (H)}}{M\text{ (1-Propanol)}}$$

$$\frac{8 * 1{,}008\text{ g/mol}}{60{,}097\text{ g/mol}} = 13\ \%$$

Calculation of the percentage of H in 1-propanol

$$\text{Number H atoms} = \frac{\text{\% Proportion} * M\text{ (1-propanol)}}{100}$$

$$= \frac{13 * 60{,}097\text{ g/mol}}{100} = 7{,}8 \approx 8$$

Result

This experiment results in carbon dioxide, water and elementary copper in a redox reaction. The mass increase in the drying tubes allows us to draw conclusions in regard to the resultant mass of water during the experiment. This in turn allows us to calculate the number of hydrogen atoms in the initial compound.

A number of 10 hydrogen atoms was determined in the initial compound for n-butane. This also corresponds precisely with the empirical formula of n-butane, C_4H_{10}.

A number of 8 hydrogen atoms was calculated for 1-propanol. This also corresponds precisely with the empirical formula of 1-propanol, C_3H_8O.

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

Cleaning and disposal

The glass wool and calcium chloride can be dried then stored in vessels labelled for this and reused for similar experiments. The reduced copper (II) oxide rods can reprocessed to copper (II) oxide through oxidation. Then store in an appropriately labelled vessel and reuse for similar experiments. Otherwise they must be disposed of in heavy metal waste. The copper mirror can be removed by etching with nitric acid.

Note: Caution: this produces nitrous gases. Cleaning may only be carried out under a fume cupboard.