

Thermal analysis of hydrocarbons

Aims of the experiment

- To learn about the composition of hydrocarbons
- To learn about the process of thermal analysis
- To learn about the homologous series of hydrocarbons
- To learn about the burning of gases

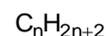
Principles

Carbon is present in countless compounds. The chemistry of hydrocarbon compounds is referred to as organic chemistry. This term was coined by Berzelius in the 19th century. It was assumed at that time that organic substances could only be formed from living organisms. In this day and age, however, many organic compounds are produced purely synthetically, particularly ones which do not occur in nature.

These 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, e.g. depending on the bonds present. Carbon itself is always tetravalent. The simplest of these compounds are alkanes, the saturated hydrocarbons. They are referred to as saturated as each of the four possible bonds has a binding partner.

Alkanes are present in many substances found in everyday

life. These include, for example, petrol, heating oil, natural gas as well as candle wax. Methane with the chemical formula CH_4 is the simplest compound in this group. Similarly constructed molecules which only differ in the number of C atoms form an homologous series, i.e. a series with the same molecular construction plan. The general formula of the chain-like alkanes is:



Hydrocarbons with more than 3 carbon atoms can be linked to each other in various ways. On the one hand, alkanes can be present in a non-branched form of long chains. These alkanes are referred to as normal or, in short, n-alkanes. There are so-called constitutional isomers of the chain-like alkanes, i.e. they have the same chemical formula, however they are linked to each other in a branched form. The number of possible isomers of an alkane increases with the number of carbon atoms. The isomers differ not only in their construction, but also in their physical and chemical characteristics.



Fig. 1: Construction of the experiment. The gas syringes can also be attached with one large and one small magnetic holder each. Then affix the tube with the small magnetic holders.

Ring-like alkanes are referred to as cycloalkanes.

In all, the alkanes are a group of virtually non-polar compounds. They therefore do not readily mix with polar compounds and form two-phase mixtures with water. Alkanes are very inert in terms of their reactions, but they are very inflammable.

In this experiment, the hydrogen content in various alkanes will be measured. For this purpose, a thermal analysis is carried out in an oxygen-free reaction tube. The gaseous alkanes burn up on a wire. Hydrogen is formed in the oxygen-free atmosphere, the volume of which can be measured. The general reaction equation for the thermal decomposition of alkanes is:



From n parts by volume of alkane, $n+1$ parts by volume of hydrogen are produced. If the initial volume of the alkane is known and the final volume of hydrogen has been measured in the experiment, the chemical formula of the investigated gases can be determined from this. An unknown gas can be identified in this way.

Risk assessment

Methane and ethane are extremely inflammable gases. 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 a leak can be stopped safely, otherwise inflammable gas will continue to leak out.

| Methane | |
|--|---|
|   Signal word: hazard | <p>Hazard statements</p> <p>H220 Extremely flammable gas.</p> <p>Precautionary statements</p> <p>P210 Keep away from heat/sparks/open flames/hot surfaces. No smoking.</p> <p>P377 Leaking gas fire: Do not extinguish unless the leak can be stopped safely.</p> <p>P381 Eliminate all ignition sources if safe to do so.</p> <p>P280 Wear protective gloves / eye protection.</p> <p>P410 + P403 Store away from sun exposure in a well-ventilated place.</p> |

| Ethane | |
|--|---|
|   Signal word: Hazard | <p>Hazard statements</p> <p>H220 Extremely flammable gas.</p> <p>Precautionary statements</p> <p>P210 Keep away from heat/sparks/open flames/hot surfaces. No smoking.</p> <p>P377 Leaking gas fire: Do not extinguish unless the leak can be stopped safely.</p> <p>P381 Eliminate all ignition sources if safe to do so.</p> <p>P280 Wear protective gloves / eye protection.</p> <p>P403 Store in a well-ventilated place.</p> |

| Equipment and chemicals | |
|-------------------------|---|
| 1 | Panel frame C100, two-level 666 428 |
| 2 | Adhesive magnetic board 300 mm 666 4660 |
| 1 | Blank panel, 200 mm, CPS 666 467 |
| 1 | Blank panel, 300 mm, CPS 666 468 |
| 2 | Holder, magnetic, size 1, 9...11 mm 666 4661 |
| 2 | Holder, magnetic, size 4, 27...29 mm 666 4664 |
| 2 | Gas syringe with three-way stopcock 665 914 |
| 1 | Combustion chamber with filament 666 460 |
| 1 | Connecting lead 19 A; pair 501 45 |
| 1 | High current power supply 521 55 |
| 1 | Equipment platform 350 mm 726 21 |
| 1 | Tubing connector, 4 x 15 mm 604 510 |
| 1 | Silicone tubing 4 mm diam., 1 m 667 197 |
| 1 | Silicone tubing 7 mm diam., 1 m 667 194 |
| 1 | Test tubes, Fiolax 664 042 |
| 1 | Test tube holder, wooden 667 052 |
| 1 | Bunsen burner, universal 656 016 |
| 1 | Safety gas hose, 1 m 666 729 |
| 1 | Safety screen 667 605 |
| 1 | Minican pressurised gas can, methane 660 987 |
| 1 | Minican pressurised gas can, ethane 660 988 |
| 1 | Fine regulating valve 660 980 |

Set-up and preparation of the experiment

Construction of the apparatus

1. Attach the adhesive magnetic boards, the CPS module "Combustion chamber with filament" and the blank panel to the CPS panel frame (see Fig. 1).
2. Attach both gas syringes, using two magnetic holders for each, onto the adhesive magnetic board. Here, fix the syringe piston with the large magnetic holders and the tubing with the small magnetic holders.
3. Connect the gas syringes to the combustion chamber. To do this, loosen the GL fittings on the combustion chamber to enable the glass tubes of the gas syringes to be easily inserted. The fittings must then be firmly tightened so that the apparatus is gas-tight and no air can get in. Finally, align the complete apparatus horizontally.
4. Now connect both connecting leads to the combustion chamber and plug in the power supply.

Preparation

The apparatus must first be flushed out with the gas being investigated in order to remove the air present. To do this, screw the fine regulating valve onto the Minican pressurised gas canister (methane or ethane). Connect the 4 mm diameter silicone tubing onto this and attach a further 7 mm diameter silicone tubing using a tubing connector. The second silicone tubing can now be connected onto the stopcock of the left gas syringe. To flush out the apparatus, fill it completely with gas. Lead the gas to the outside through the three-way stopcock of the second gas syringe. Repeat this procedure and then close the apparatus. It is now flushed out.

Caution! It is imperative that the room is well ventilated during the flushing procedure.

Performing the experiment

- For the experiment, now fill the left gas syringe with 20 mL of gas. Close the gas syringe. Ensure that with the second gas syringe there is only a connection to the combustion chamber and not to the surroundings. Close the Minican pressurised gas canister well after filling the gas syringe, unscrew the valve and remove both away from the site of the experiment.
- Supply current to the combustion chamber. For this, set a maximum voltage of 1.1 V on the power supply and a maximum current of 9 A.
- When the wire starts to glow, open up the filled gas syringe to the combustion chamber. Both gas syringes now only have one connection to the combustion chamber.
- Let the gas flow over the glowing wire. For this purpose, slowly move both syringe pistons back and forth.
- Repeat this procedure until the gas volume is constant. Note the volume. Then switch off the current at the power supply.
- To check the identity of the gases present, carry out the detonating gas test. The detonating gas test is performed as follows:
 - Hold a test tube upside down over the opening of one of the three-way stopcocks of the apparatus to collect the gas from the gas stream.
 - Place your thumb over the opening of the test tube and bring it close to a naked flame. At the same time, remove your thumb from the opening of the test tube.
 - If only a brief "plop" is heard, then the tube contains only pure hydrogen.

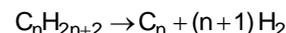
Observation

Whilst pushing both syringe pistons back and forth, it is possible to observe how the gas volume increases in the apparatus. Continue this procedure until a constant gas volume is reached. From a volume of 20 mL of gas deployed, 40 mL of gas are produced with methane and 60 mL of gas with ethane. Soot can be seen developing in the combustion chamber during the experiment, i.e., elemental carbon.

Evaluation

Two substances are produced from the thermal decomposition of methane or ethane. In the one case this is soot, i.e., elemental carbon, which collects in the combustion chamber. In the second case, the substance is a gas. This is analysed more closely using the detonating gas test. It is hydrogen.

From the volume ratio of deployed gas to the resulting gas, the number of hydrogen atoms in the initial compound can be calculated. The general reaction equation for the thermal decomposition of alkanes is



It can be deduced from this that from n parts by volume of the alkane, $n+1$ parts by volume of hydrogen are produced.

The volume of gas doubles during the thermal analysis of methane. From one part by volume of methane, two parts by volume of hydrogen are produced. The volume triples in the case of ethane. From one part by volume of ethane, three parts by volume of hydrogen are produced. From this, the chemical formulas of the compounds and the chemical equations can be deduced (see Tab. 1).

Tab. 1: Evaluation of the experiment for methane and ethane.

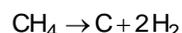
| | Methane | Ethane |
|------------------------------|---------------------------------------|--|
| Volume ratio (measured) | 1:2 | 1:3 |
| $(n+1) H_2$ | 2 | 3 |
| $n =$ number of C atoms | 1 | 2 |
| $(2n+2) =$ number of H atoms | 4 | 6 |
| Sum formula | CH ₄ | C ₂ H ₆ |
| Reaction equation | CH ₄ → C + 2H ₂ | C ₂ H ₆ → 2C + 3H ₂ |

The chemical formula of methane is therefore CH₄ and the chemical formula of ethane is C₂H₆.

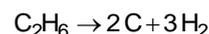
Result

Elemental carbon and elemental hydrogen form from the thermal decomposition of methane and ethane. From the volume ratio of deployed gas to the resulting gas, the number of hydrogen atoms in the initial compound can be calculated.

For methane, two parts by volume of hydrogen form from one part by volume of gas. As hydrogen is present in molecular form (H₂), 4 hydrogen atoms are therefore present in methane. The reaction equation is therefore:



In the case of ethane, the ratio is 1:3 for the hydrogen produced. It can be inferred from this that ethane contains 6 hydrogen atoms. The reaction equation can now be formulated as follows:

**Remarks**

The experiment can be carried out for substances other than methane and ethane. A thermal analysis of propane and butane is also possible. With these compounds, we are looking at numbers 3 and 4 in the homologous series. Accordingly, a quadrupling of the gas volume would result with propane and a quintupling with butane. The intensity of soot formation increases from methane through to butane, as the carbon content also increases.

If we do not know whether we are dealing with a chain-like alkane where the gaseous compound is concerned, then the general sum formula C_nH_{2n+2} cannot be applied. The proportion of C atoms in the compound must then be determined in another experiment. The carbon content can be determined by the Quantitative Analysis of Carbon (C2.1.1.1) experiment.

Cleaning and disposal

The incandescent wire in the combustion chamber should be carefully cleaned with a cotton bud. Be careful not to damage the delicate incandescent wire in the process. The cotton bud can then be disposed of in the normal waste.