Analysis of waste gases

Aims of the experiment

- To learn about exhaust gas analysis
- To work with testing tubes
- To learn about detection reactions
- To learn about the composition of exhaust gases from combustion engines

Principles

Hydrocarbons are sources of energy whose energy is released when they are burned. In the ideal case, only carbon dioxide (CO_2) and water are formed when hydrocarbons are burned. However, when fossil fuels such as coal and mineral oil, and products manufactured from these such as petrol and Diesel, are burned, then other substances are also formed. Very many of these substances are highly toxic both for humans and for the environment. It is therefore important to monitor the content of harmful substances and to maintain the release of these to as low a level as possible.

For example, sulphur is contained in fossil fuels. This burns in the air to produce sulphur dioxide (SO_2). This is a strong respiratory poison and is mainly responsible for the production of acid rain. If the combustion is incomplete then, instead of CO_2, carbon monoxide (CO) is created which is also toxic to humans and prevents oxygen from being taken up in the blood. A third group of substances that form at high combustion temperatures from the nitrogen contained in the air are the nitrogen oxides (NO_x). Nitrogen oxides are involved in the formation of the atmospheric poison ozone.

In this experiment, the three compounds described, or mixtures of these (CO, NO_x and SO_2) are to be detected in automobile exhaust gas. Testing tubes, in which the presence of the individual analyte causes a colour reaction to take place, will be used for detection. The individual reactions that take place in this case are:

CO testing tube $5\ CO + I_2O_5 \xrightarrow{H_2SO_4} I_2 + 5\ CO_2$

(white → brown)

NO_x testing tube $3\ NO + 2\ CrO_3 \xrightarrow{H_2SO_4} 3\ NO_2 + Cr_2O_3$

$2\ NO_2 + 2\ R\text{-}NH \rightarrow R\text{-}NNO + R\text{-}NH{:}HNO_3$

(white → dark blue)

SO_2 testing tube $SO_2 + I_2 + 2\ H_2O \rightarrow SO_4^{2-} + 2\ I^- + 4\ H^+$

(purple → white)

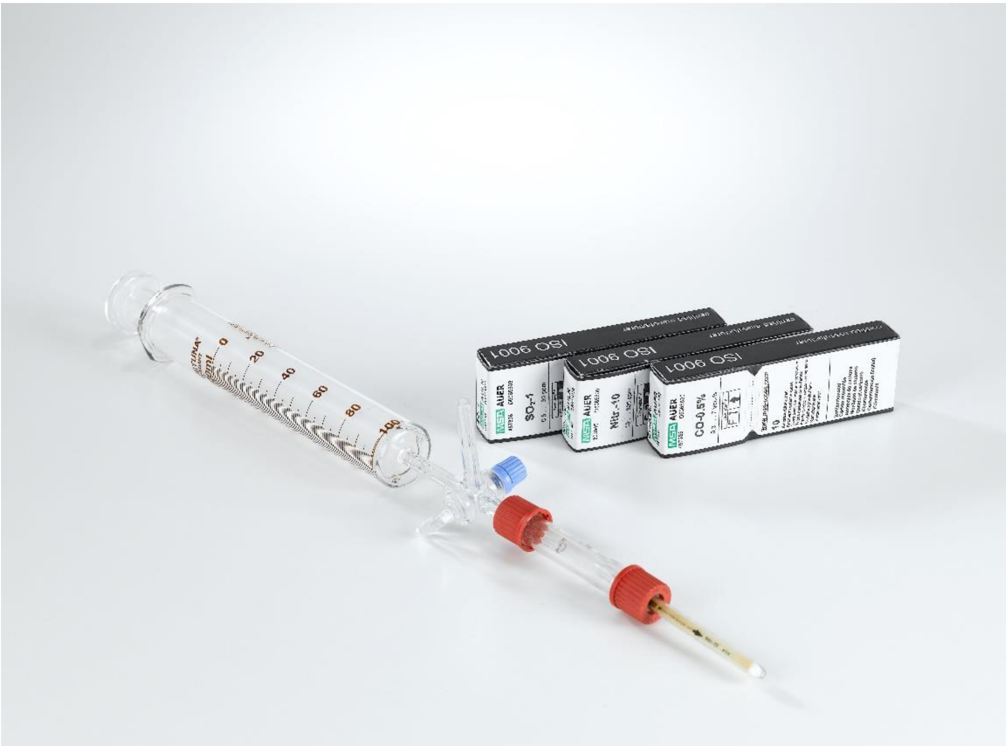

Fig. 1 Set-up of the experiment

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Risk assessment

The substances investigated in the experiment, carbon monoxide, nitrogen oxides and sulphur dioxide, form in such small amounts that they do not represent a hazard. Nevertheless, the experiment should be performed in the fume cupboard owing to this exhaust gas.

CO testing tube	
	Hazard statements R20/21/22 Harmful on inhalation, in contact with skin and if swallowed. R35 Causes severe burns. R37 Irritating to respiratory system. **Precautionary statements** P102 Keep out of reach of children. P260 Do not breathe dust/fume/gas/ mist/vapours/spray. P262 Do not get in eyes, on skin, or on clothing. 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. P302+P352 IF ON SKIN: Wash with soap and water.
NO_x testing tube	
	Hazard statements R21/22 Harmful in contact with skin and if swallowed. R34 Causes burns. R43 May cause sensitisation by skin contact. **Precautionary statements** P102 Keep out of reach of children. P262 Do not get in eyes, on skin, or on clothing. 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. P302+P352 IF ON SKIN: Wash with soap and water.
SO_2 testing tube	
	Hazard statements R20/21/22 Harmful on inhalation, in contact with skin and if swallowed. R35 Causes severe burns. **Precautionary statements** P102 Keep out of reach of children. P260 Do not breathe dust/fume/gas/ mist/vapours/spray. P262 Do not get in eyes, on skin, or on clothing.

Equipment and chemicals

3 Gas syringe, 100 ml with three-way stopcock 665 9148
3 Glass connector, 2 x GL 18 .... 667 312
3 Screw cap, GL 18, with hole 11 .... 667 305
3 Silicone gasket, GL 18, ... 667 296
1 Testing tube NO_x 0.5 … 50 ppm, set of 10 .... 666 313
1 Testing tube CO 0.5 … 7.0%, set of 10 .... 666 319
1 Testing tube SO_2 1 … 25 ppm, set of 10 .... 666 314
1 Glass file 667 015
1 Hand-held stopwatch.................................... 313 07
1 Air bag, set of 30 .... 662 302
1 Funnel, PP. 75 mm Ø.................................... 665 009
Also required:
Exhaust gas sample, e.g. car exhaust, possibly a rubber band

Set-up and preparation of the experiment

Set-up of the apparatus

1. Connect the gas syringe and the glass connector as shown in Fig. 1.

Preparation of the experiment

1. A screw cap with a small silicone gasket are needed in each case for the other end of the glass connectors.
2. The exhaust gas from a vehicle will be used for the analysis.
3. An air bag and a funnel will be used to collect the exhaust gas. The tube on the air bag should be cut down to a length of about 5 cm.
4. Insert the testing tubes into the screw caps containing the small silicone gasket. With the help of the glass file, break off both tips of the tube and smoothen the sharp edges.
5. Connect the testing tubes such that the arrow points in the direction of the gas flow.

Note: It is necessary to cut the tube down, as the air bag would otherwise be difficult to fill.

6. Insert the funnel into the tube of the air bag and hold it against the exhaust pipe of a vehicle in order to collect the gas.

Note: The funnel must not be held too closely to the exhaust pipe and only briefly after starting. Also, the exhaust gas sample should be taken within 5 minutes of starting the vehicle, as the vehicle's catalytic converter is activated after this time. Press the accelerator to produce a higher pressure, if necessary.

7. When the air bag is filled, remove the funnel and seal the bag tightly.

Performing the experiment

1. Now connect the air bag to the first gas syringe. To do this, untie the knot in the tube and push the tube over the gas syringe. To ensure gas-tightness, hold the tube firmly in the fingers or fix with a rubber band.
2. Now set the three-way stopcock such that the air bag is connected to the gas syringe.
3. For the analysis, 100 ml of exhaust gas are required. To obtain this, slowly withdraw the piston of the gas syringe and press lightly on the air bag.
4. Once the gas syringe is filled with 100 ml of exhaust gas, close the three-way stopcock such that no connection can

result between the gas syringe and the atmosphere, remove the air bag and seal it again.

5. Connect the glass connector with the first testing tube to the outlet of the gas syringe.

6. For the analysis, set the three-way stopcock to produce a connection between the gas syringe and the glass connector.

7. Empty the gas syringe slowly and without exerting much pressure so that the exhaust gas passes through the testing tube.

8. Start the stopwatch and read off the result on the testing tube after two minutes.

9. In the meantime, proceed in the same way with the other two testing tubes. To do this, first attach the air bag to the gas syringe and fill it with exhaust gas.

Note: Testing tubes in which a reaction turns out to be negative can be used again on the same day.

Observation

The gas syringe is filled by drawing in exhaust gas and the air bag empties at the same time. By passing the exhaust gas through the testing tube, a reaction takes place between the relevant analyte, when it is present, and the material in the testing tube. Colour changes take place in the testing tube which have already been described under Principles.

The result for the individual analysis can be read directly on the testing tube. It is worth noting that the test was positive for CO and NO_x. In contrast, the test for SO_2 was negative.

Evaluation

In this experiment, the exhaust gas from a Diesel vehicle was investigated without the catalytic converter being activated. The content of CO, NO_x and SO_2 in the exhaust gas was tested. The analyses were evaluated using the calibrated scale on the testing tubes. The values need to be read off and noted and can be compared with the values in the literature.

Results

The results of the analyses of CO, NO_x and SO_2 in the exhaust gas of a Diesel vehicle are shown in Table 1.

Tab.1 Results of the individual analyses.

Analyte	Results
CO (n = 1)	0.3%
NO_x (n = 5)	3 ppm
SO_2 (n =3)	-

Depending on the information on the testing tube, the concentration value read off (e.g. in % or ppm) must be multiplied by the factor *n* stated in the package insert in order to arrive at the exact concentration of the analyte. This is necessary in this case only for nitrogen oxides, which can thus be detected at a concentration of 15 ppm.

In Table 2, the literature values for the analytes in the exhaust gas of a petrol engine are shown.

Tab.2 Literature values for analytes in the exhaust gas of petrol engines.

Analyte	Vol %
CO	0.2 - 5 (Diesel engine lower)
NO_x	0.005 - 0.4 (Diesel engine higher)
SO_2	0.006 (Diesel engine higher)

In order to make a comparison with the literature values, the result for NO_x needs to be converted to percent by volume. Note in this case that 1% = 10,000 ppm.

So in our case the NO_x content is 0.0015%. To obtain percent by volume, one must now refer back to the volume of exhaust gas used for the analysis. The volume in the experiment was 100 ml. The NO_x content in percent by volume was therefore 0.015%.

CO is present in the exhaust gas of Diesel engines only in small amounts (see Table 2). In this experiment, the percentage by volume is somewhat higher than expected. This is because Diesel engines actually emit a lower proportion of CO than petrol engines. However the value of 0.3% is at the lower end of the range given in the literature for CO in the exhaust gas of petrol engines (see Table 2).

Nitrogen oxides are formed from the nitrogen and oxygen contained in the air through the high temperatures reached during the combustion of petrol and Diesel fuels. In the case of Diesel engines, the proportion is even higher compared with petrol engines. The reason for this is the higher operating temperature and the higher air excess in Diesel engines. In this experiment, a value for NO_x of 0.015% was determined. This lies also in the range in which petrol engines emit NO_x. The reason for the lower NO_x content in the exhaust gas sample is probably due to the fact that it was taken with the engine in a cold condition, and therefore the high operating temperature could not be developed at which NO_x is formed.

SO_2 could not be detected as the concentration in the exhaust gas is too low. The sulphur contained in crude oil is removed to a very large extent during the manufacture of petrol. This is necessary as the SO_2 resulting from the combustion would act as a poison for the catalytic converter.

Cleaning and disposal

The testing tubes must on no account be disposed of in the normal waste, as they contain small amounts of chemicals. The laws on waste disposal and on protection against hazardous substances must be observed.

The testing tubes for CO and SO_2 contain inorganic substance, they must therefore be disposed of in the waste for inorganic solids. In the testing tube for NO_x there is an organic amine as well as chromium(VI) oxide, which is highly poisonous. For this reason, these testing tubes must be collected in a specially labelled container for disposal.

The residual exhaust gas in the air bag can be emptied in the fume cupboard.