Production of gases with a Kipp's apparatus

Aims of the experiment

- To learn a method for producing gases.
- To work with the Kipp's apparatus.
- To follow a reaction.
- To observe the various aggregate states of substances.

Principles

The gaseous state is one of the three states of aggregation in addition to the solid and liquid state. The particles contained in a gas move freely at a large distance from one another. Thus, there is no solid structure in gases. They use the entire space made available to them and are limited only by the walls of a storage container, for example.

Many gases are generated through the reaction of various substances, such as a liquid and a solid substance. This includes hydrogen, oxygen, chlorine gas, nitrogen dioxide or carbon monoxide, for example.

In this test, hydrogen gas is produced using the Maey gas generator. This generator is similar to the Kipp's apparatus for hydrogen generation. In this generator, zinc as a solid and hydrochloric acid as a reaction liquid react with one another. In the process, zinc is added to the insert tube of the gas generator. The insert tube contains a glass frit. Liquid can pass through the frit, but not solids. A dilute hydrochloric acid solution is added to the flask. When the insert tube with the frit and the zinc is dipped into the flask, and when the hydrochloric acid passes through to the zinc, both substances can react with one another and hydrogen is generated.

The following reaction occurs:

$$Zn + H^+ \rightarrow Zn^{2+} + H_2 \uparrow$$

The special feature of the Kipp's apparatus is that the reaction can be stopped by closing the stopcock, and it can be restarted by reopening it. The reason for this is that when the stopcock is closed, gas being generated can no longer escape. This causes the pressure to increase inside the apparatus and the liquid is pushed back through the insert tube with the frit into the flask. The reaction stops as soon as the liquid, in this case hydrochloric acid, can no longer reach the zinc granules. When the stopcock is opened again, the pressure inside the apparatus drops. The falling pressure allows the liquid to rise again into the insert tube and the reaction begins again.

After the reaction, the synthesised gases are scrubbed by washing with concentrated sulphuric acid. The purpose of this procedure is to remove water vapour from the gas. In general, scrubbing gases removes undesirable contaminants. Depend-

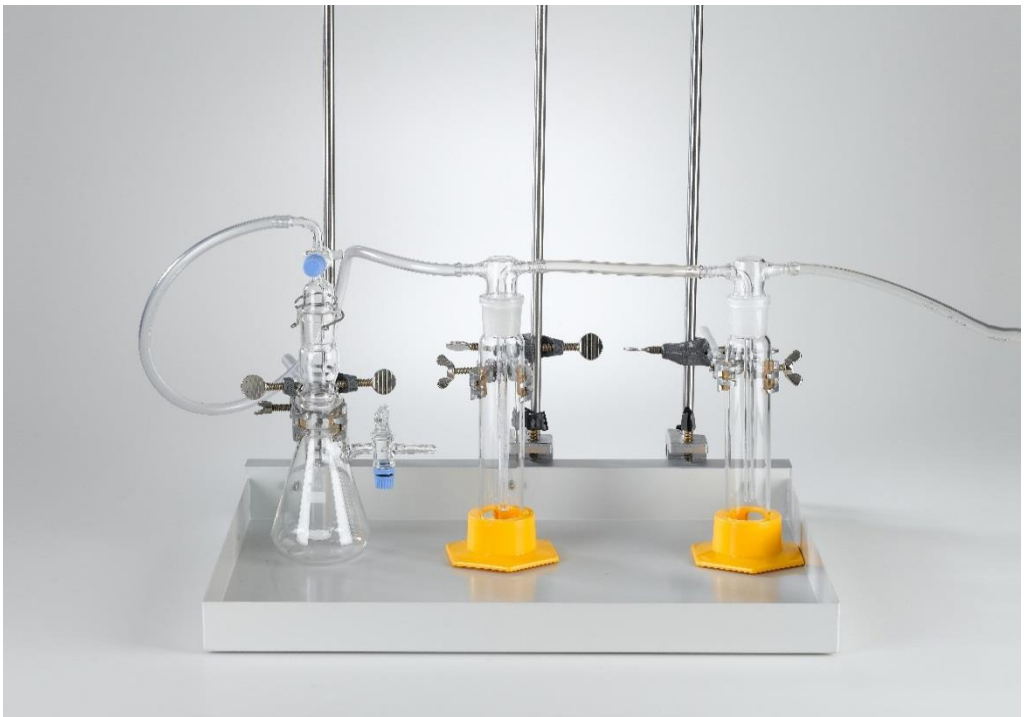

Fig. 1: Set-up of the experiment with stand material.

ing on the scrubbing liquid, other contaminants can also be removed. For example, solid particles can be removed with water and traces of acid can be removed with alkaline solutions.

Risk assessment

When handling hydrochloric acid, gloves, goggles and a lab apron should be worn in order to avoid possible burns. This also applies when filling the sulphuric acid into wash bottles.

Hydrochloric acid, 2 mol/l

Signal word: Caution

Hazard statements

H290 Can corrode metals

Safety statements

P390 Absorb spillage to prevent material damage.

Sulphuric acid, 95-98 %

Signal word: Hazard

Hazard statements

H290 Can corrode metals

H314 Causes severe skin burns and severe eye damage.

Safety statements

P280 Wear protective gloves/protective clothing/eye protection/face protection.

P305+P351+P338 IF IN EYES:

Rinse carefully with water for several minutes. Remove contact lenses if present and easy to do so. Continue rinsing.

P309 IF exposed or you feel unwell:

P310 Immediately call a POISON CENTER or doctor/physician.

P301+P330+P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.

Equipment and chemicals

For set-up in the CPS

1	Maey gas generator	665 648
1	Panel frame C50, two-level, for CPS	666 425
2	Adhesive magnetic board, 500 mm	666 4659
1	Holder, magnetic, size 3, 18...22 mm	666 4663
2	Holder, magnetic, size 4, 27...29 mm	666 4664
4	Laboratory stand 16 cm x 13 cm	300 76
2	Glass connector, 2 x GL 18	667 312

For set-up with stand material

1	Maey gas generator	665 647
1	Metal experiment tray	666 6221
1	Angle strip	666 623
3	Leybold multiclamp	301 01
3	Stand rod 50 cm, 10 mm diam.	301 27
4	Bosshead S	301 09
4	Universal clamp 0...80 mm	666 555

For both variations

1	PVC tube 7 mm diam., 1 m	604 501
2	Gas scrubber bottles - lower section	664 800
2	Glass tube insert	664 805
1	Gas syringe, 100 ml with 3-way stopcock	665 914
1	Set of 10 test tubes, 16 x 160 mm	664 043
1	Zinc, granulated, 100 g	675 4800
1	Hydrochloric acid, approx. 2 mol/l, 500 ml	674 6920
1	Sulphuric acid, 95-98 %, 500 ml	674 7860

Additionally required:
Bunsen burner

Set-up and preparation of the experiment

Construction of the CPS apparatus

1. Insert both adhesive magnetic boards into the CPS panel frame.

2. The Maey gas generator consists of an Erlenmeyer flask with a GL-32 screwed connection. The insert tube with glass frit is attached through the GL-32 cap and the seal ring is pushed on and screwed tight at the Erlenmeyer flask. The insert tube also has a GL-24 screwed connection in which a 90 degree one-way cock is placed.

3. The Maey gas generator is fastened to the lower adhesive magnetic board using a magnet holder (size 3).

4. Fasten both gas scrubber bottles to the adhesive magnetic board using a holder (size 4) for each. In the process, the two openings with the straight handle must face one another.

5. Connect the one-way cock of the gas generator to the first gas scrubber bottle using a glass connector.

6. Connect the two gas scrubber bottles together using a glass connector.

7. A piece of PVC hose is attached to the rear gas scrubber bottle and then to a gas syringe.

For set-up with stand material

1. The Maey gas generator consists of an Erlenmeyer flask with a ground glass joint. The inlet tube with the glass frit is connected to the ground glass joint and fastened with a joint clamp.

2. The gas outlet cock is placed on the inlet tube and also fastened with a joint clamp.

3. Screw the angle strip onto the experiment pan. Fasten three Leybold clamps to the angle strip, with the three stand rods inserted into the clamps.

4. Fasten a bosshead S clamp to each of the stand rods, and then place a universal clamp into the bosshead S clamp.

5. Fasten the gas generator to the first stand rod using the universal clamp.

6. Make sure the gas scrubber bottles are fastened to the other two stand rods so that the openings with the straight handles point to one another.

7. Connect the gas generator to the first gas scrubber bottle by way of the gas outlet cock using a piece of PVC tube.

8. Likewise, connect the gas scrubber bottles together using a piece of PVC tube.

9. Attach a piece of PVC tube that is attached to a gas syringe to the rear gas scrubber bottle.

Preparation of the experiment

H_2 gas is generated in this experiment. To dry the generated gas, sulphuric acid as a desiccant is filled into the second gas scrubber bottle downstream of the gas generator.

Note: The first gas scrubber bottle is only used as a safety wash bottle between the gas generator and the desiccant.

1. The Erlenmeyer flask is filled with about 200 ml of the reaction liquid, in this case hydrochloric acid.

2. The solid is added to the inlet tube with the glass frit; in this experiment the solid is zinc.

3. Fill enough sulphuric acid into the second gas scrubber bottle so that the handle of the glass tube insert dips into the liquid.

Performing the experiment

1. With the stopcock closed, slowly lower the inlet tube into the reaction liquid.

Note: Make absolutely certain that the one-way stopcock for gas intake and the side opening of the Erlenmeyer flask are not closed during the experiment. This can cause pressure to build up.

2. The generated gas can now be collected through the connected gas syringe. Open the stopcock for this purpose.

When a sufficient amount of gas has been collected in the gas syringe, the experiment can be ended by closing the gas intake stopcock. Gas is then collected in the Erlenmeyer flask and the inlet tube. This causes the liquid in the inlet tube to displace and the reaction to stop.

Observation

As soon as the inlet tube with the glass frit dips into the liquid and hydrochloric acid enters, small gas bubbles can be observed. Bubbles occur at the zinc surface and rise.

Evaluation

To check the identity of the gases present, carry out the detonating gas test. The detonating gas test is performed as follows:

a. Hold a test tube upside down over the opening of one of the 3-way stopcocks of the syringe to collect the out-flowing gas.

b. 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.

Result

In this test, hydrogen gas is produced using the Maey gas generator. Hydrogen is generated through the reaction of solid zinc with hydrochloric acid as a reaction liquid. This can be collected in a gas syringe. With the help of the detonating gas test, the identity of the resultant gas can be checked at the end of the experiment. A short "plop" sound clearly verifies that the generated gas is pure hydrogen.

Other gases can also be generated using the gas generator, see Table 1. Alternatively, gases can also be generated using an apparatus with a dropping funnel (see VC 1.4.2.2)

Cleaning and disposal

Hydrochloric acid can be neutralised and then disposed of in the waste stream.

Zinc should be collected in a container for this purpose. If it has not been completely used up, it can be re-used for the test.

Tab. 1 Gases that can be generated using the Kipp's apparatus.

Generated gas	Solid reaction partner	Reaction liquid	Desiccant for the gas	Remarks
H_2	Zinc (granulates)	HCl (dilute)	e.g. H_2SO_4	Detonating gas test!
CO_2	Marble (pieces)	HCl (dilute)	e.g. H_2SO_4	-
H_2S	FeS (pieces)	HCl (semi-concentrated)	e.g. H_2SO_4	poisonous, work in the fume cupboard!
NO	Copper (chips)	HNO_3 (semi-concentrated)	e.g. H_2SO_4	poisonous, work in the fume cupboard!