Model experiment on the desulphurization of flue gas

(Item No.: P1310000)

Curricular Relevance

Area of Expertise: Chemie

Education Level: Klasse 10-13

Topic: Anorganische Chemie

Subtopic: Spezialtehmen: Brandbekämpfung, großtechnische Verfahren, Explosivstoffe , Kristalle, Baustoffe und Dünger

Experiment:

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Intermediate	10 Minutes	10 Minutes	1 Student

Additional Requirements:

Experiment Variations:

Keywords:

Principle and equipment

Principle

The model experiment presented here shows the process of desulphurization of flue gases, as nowadays used in power stations, in simplified form. The changes made, in comparison to the large scale desulphurization process, were necessary to make the experiment comprehensible. In modern power stations, the desulphurization of the flue gases produced are nowadays almost exclusively carried out by wet cleaning with an aqueous, weakly basic suspension of finely ground, natural limestone (calcium carbonate). The flue gases are led from the oven to the washing tower, where they are sprayed with this calcium carbonate wash liquid. As sulphur dioxide is present in excess here, calcium hydrogen sulphite (calcium bisulphite) is formed from the sulphur dioxide and calcium carbonate:

$$CaCO_3 + SO_2 \rightarrow CaSO_3 + CO_2 \uparrow$$
$$CaSO_3 + SO_2 + H_2O \rightarrow Ca(HSO_3)_2$$

The calcium hydrogen sulphite is oxidised to gypsum by the oxygen (air) blown in:

$$Ca(HSO_3)_2 + O_2 + 2\,H_2O \rightarrow CaSO_4 \cdot 2\,H_2O + H_2SO_4$$
$$H_2SO_4 + CaCO_3\ 2\,H_2O \rightarrow CaSO_4 \cdot 2\,H_2O + CO_2 \uparrow$$

The flue gas that has been so treated is now subjected to a secong wash with fresh limestone suspension. As the greater part of the sulphur dioxide has been removed in the first step, calcium carbonate is here in excess and forms calcium sulphite in the reaction:

$$CaCO_3 + SO_2 \rightarrow CaSO_3 + CO_2 \uparrow$$

The suspension from this second wash is subsequently fed back to the first wash, where sulphur dioxide is in excess, so that the calcium sulphite forms calcium bisulphite (see above). The flue gas freed from sulphur dioxide is led through the chimney into the artmosphere. The gypsum formed (calcium sulphate dihydrate) is filtered off from the wash liquid. The reactions above, that lead to the desulphurization of flue gases, take place spontaneously, without the need for any catalysts or additives. The gypsum obtained is very pure and in the form of a fine crystalline precipitate. When relatively pure limestone is used, the flue gas gypsum often has a purity of above 97%. On average, 1 ton of sulphur is contained in 100 tons of German coal. This produces about 5.4 tons of gypsum on desulphurization. A large power station with an output of 700 megawatts burns about 200 tons of coal per hour, and so produces about 260 tons of gypsum per day. With these quantities, it can truly be asserted, that a power station with a desulphurization plant is simultaneously a chemical factory.

Equipment

Student's Sheet

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Position No.	Material	Order No.	Quantity
1	Frame for complete experiments	45500-00	1
2	Secure bottle, 500 ml, 2 x Gl 18/8, 1 x 25/12	34170-01	1
3	Panel for complete experimental setups	45510-00	1
4	Apparatus carrier w. fix. magnet	45525-00	1
5	Combustion tube 200mm,quartz,PN19	33947-00	1
6	Spring manometer, 0...-1000 mbar	34170-02	1
7	Clamp on holder	02164-00	1
8	Teclu burner, DIN, natural gas	32171-05	1
9	Clamping holder, turnable, 18-25 mm	45521-00	2
10	Test tube GL25/8, w.hose connec.	36330-15	3
11	Water jet pump, plastic	02728-00	1
12	Quartz glass wool 10 g	31773-03	1
13	Rear-cover for compl.-exp. panel	45501-00	1
14	Liquid Indicator pH1-13 UNISOL113	47014-02	1
15	Clamping holder,18-25mm	45520-00	4
16	folded filter,qual.,185 mm,100pcs	47580-05	1
17	Stopcock,3-way,t-shaped, glass	36731-00	1
18	Glycerol, 250 ml	30084-25	1
19	Glass tubes,right-angled, 10	36701-59	1
20	Glass tube,right-angled, 10 pcs.	36701-52	1
21	Calcium hydroxide 500 g	30054-50	1
22	Hydrochloric acid,10%,tech.gr.,1l	31821-70	1
23	Spring plugs, 50 off	45530-00	1
24	Activated carbon, granular 250 g	30011-25	1
25	Test tube rack, wood, for 6 tubes d= 30 mm	40569-10	1
26	Sulphur, sublimated, 500 g	30216-50	1
27	Hydrogen peroxide, 30%, 250 ml	31710-25	1
28	Glass tubes,straight with tip, 10	36701-63	1
29	Universal clamp	37715-00	1
30	Iodine potassium iodide solution	30094-10	1
31	Barium chloride sol.,10%, 250 ml	30965-25	1
32	Pasteur pipettes, 250 pcs	36590-00	1
33	Sodium sulphite cryst. 250 g	30167-25	1
34	Water, distilled 5 l	31246-81	1
35	Safety gas tubing, DVGW, sold by metre	39281-10	3
36	Tweezers,straight,blunt, 200 mm	40955-00	1
37	Porcelain boats, 10 pcs	32471-03	1
38	Calcium chloride tube,str.150 mm	36941-00	1
39	Rubber tubing,vacuum,i.d.6mm	39286-00	1
40	Fixing bands,universal,100 pcs.	45535-00	1
41	Glass beaker DURAN®, tall, 250 ml	36004-00	2
42	Rubber caps, 10 pcs	39275-03	1
43	Lighter f.natural/liquified gases	38874-00	1
44	Hose clamp for 10-17 mm diameter	40998-00	2
45	Funnel, glass, top dia. 80 mm	34459-00	1
46	Spoon, special steel	33398-00	1
47	Pinchcock, width 10 mm	43631-10	1
48	Test tube,200x30 mm,DURAN	36304-01	4
49	Wash bottle, plastic, 500 ml	33931-00	1
50	Glass tube, right-angled	36701-07	1
51	Rubber tubing, i.d. 6 mm	39282-00	2
52	Glass rod,boro 3.3,l=300mm, d=7mm	40485-05	1
53	Rubber stopper, d = 22/17 mm, 1 hole	39255-01	2

Safety information

Sulphur dioxide, which is a poisonous gas, is generated in the experimental apparatus during this experiment. As this is captured by activated charcoal in a glass tube at one end of the apparatus, and by an adsorption solution at the other end, however, with appropriate care, the experiment can also be carried out outside of a fume cupboard.

Calcium hydroxide and hydrogen peroxide are corrosive. Dilute hydrochloric acid causes severe irritation of skin and eyes. Barium chloride is harmful to health when swallowed or inhaled. The univeral indicator is dissolved in ethanol. Ethanol is easily inflammable. Avoid contact of the chemicals with skin and eyes. Do not inhale gases, vapours or aerosols.Wear protective gloves and protective goggles! Observe the detailed information on safety measures in the appendix.

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Set-up and procedure

Set-up

A saturated calcium hydroxide solution is required for this experiment. Prepare it as follows:
Pour about 50 ml of distilled water into a 250 ml beaker and add portions of calcium hydroxide, under stirring, until added solid no longer dissolves. Filter the solution through a pleated filter into a second beaker.

Position the clamping holders on the panel for complete experiments as shown in Fig. 2. The glass apparatus is to be subsequently assembled and fixed to the clamping holders as shown in Fig. 1.

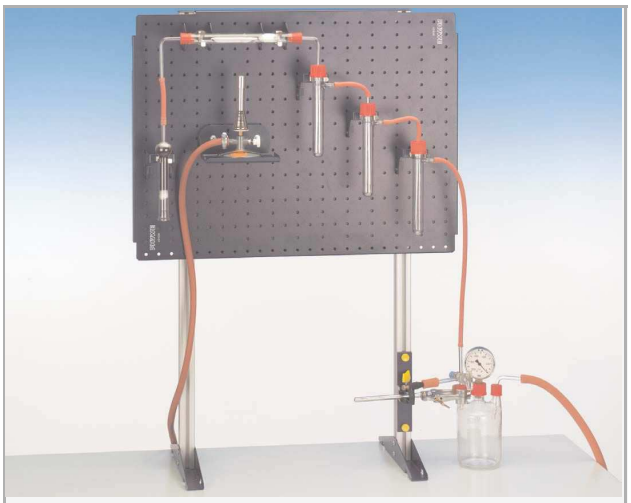

Fig. 1

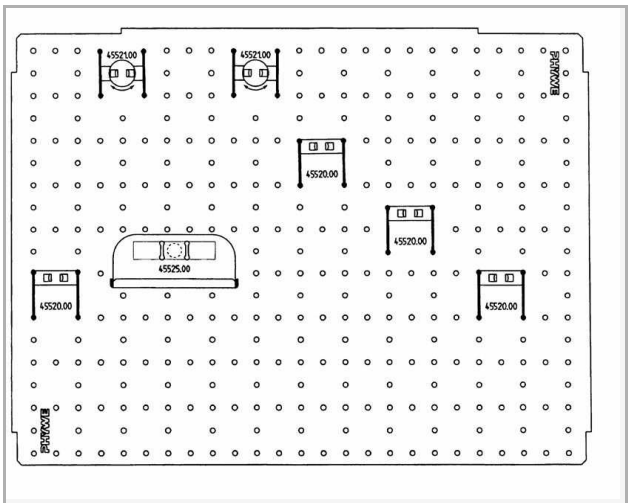

Fig. 2

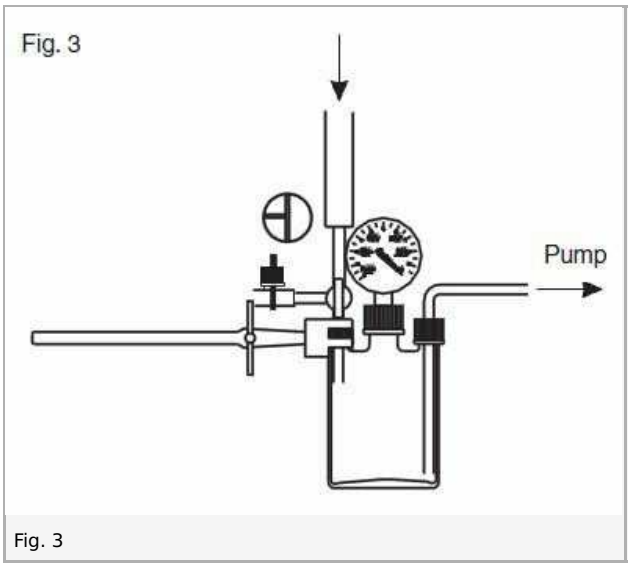

Fig. 3

Fill the drying tube with activated charcoal, using a plug of quartz glass wool at each end to prevent activated charcoal from falling out. The activated charcoal ensures that no sulphur dioxide can escape from the apparatus. Also loosely close the right end of the combustion tube with a plug of quartz glass wool, to prevent subliming sulphur from leaving the tube.

Fill the first and third test tubes about halfway up with iodine-potassium iodide solution. Half-fill the middle test tube with saturated calcium hydroxide solution, to which a few drops of universal indicator have been added. Ease a right-angled glass tube (85 + 60 mm) into a rubber stopper (previously lubricated with glycerol!) and connect its free end to the drying tube with a short length of tubing. Fit this stopper with glass tube into the left end of the combustion tube. Similarly ease a right-angled glass tube (230 + 55 mm) into a stopper, and fit the stopper into the right end of the combustion tube. Ease the long end of the glass tube through the connecting cap in the first test tube, until the end is just above the bottom of the test tube. Turn the screw cap to fix the glass tube air-tightly in this position. Insert a glass tube with tip into each of the other two test tubes, so that their tips are just above the bottoms of the test tubes. Connect the hose nipples of the test tubes to these glass tubes, in each case with a short length of tubing (Fig. 1).

Connect the hose nipple of the last test tube to the safety flask with a length of tubing (Fig. 3). Fit a short length of tubing to the third free end of the three-way tap on the safety flask, and fit a pinchcock onto it. Use vacuum tubing to connect the right-angled glass tube of the safety flask to the water jet pump. Secure the safety flask to the bottom end of the frame for complete

experiments with a universal clamp and the clamp on holder (Fig. 1).

Secure the teclu burner to the apparatus carrier with fixing magnets, by pushing its foot on each side under the rubber band that is fixed to the bottom of the carrier.

Procedure

Fill the porcelain boat with so much sulphur, that the bottom of it is covered to a depth of about 2 mm over its complete length. Open the combustion tube by taking off the left stopper, carefully slide the boat in until it reaches the middle of the tube, then close the tube again. Start the water jet pump and use the pinchcock on the safety flask to so adjust the flow of air, that it only slowly flows through the apparatus. Turn the three-way tap so that none of the three openings are closed (Fig. 3). Ignite the gas burner and adjust the flame so that it just burns colourless (not luminous), and use it to heat the sulphur in the porcelain boat.

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Observation and evaluation

Observation

After a short time, sulphur can be seen to precipitate on the walls as a result of sublimation. At the same time, the sulphur burns with a blue flame. Soon after this, the iodinepotassium iodide solution in the first test tube begins to lighten in colour and becomes completely colourless after a little while. Soon after this has occurred, the colour in the second test tube changes from blue to red. The solution in the third test tube does not change during this time.

Continuation:

When the colour in the second test tube has changed from blue to red, allow the reaction to proceed for about a further minute. The solution in the second test tube may become somewhat turbid. Now, at the latest, stop the reaction by extinguishing the gas burner and turning off the water jet pump. Should the reaction be stopped too late, then the adsorption capacity of the calcium hydroxide solution in the second test tube will be exhausted and the iodine-potassium iodide solution in the third test tube also become colourless. Carefully disconnect the tubings from the glass tube and hose nipple of the second test tube, and take it out of its holder. Distribute the contents evenly into two large test tubes. Add a little 10% hydrochloric acid to each of the large test tubes. To one of them, carefully add 2 to 3 ml of 30% hydrogen peroxide and shake for some time. Then add 2 to 3 ml of barium chloride solution to each of them.

After the addition of barium chloride solution, a white precipitate is to be seen in the test tube to which hydrogen peroxide was added.There is no precipitate in the test tube without hydrogen peroxide.

To illustrate the precipitation test with barium chloride solution, dissolve a spatula tip of sodium sulphite in each of two test tubes containing a little distilled water, acidify each with a little 10% hydrochloric acid. To one of them, carefully add 2 to 3 ml of 30% hydrogen peroxide, and after briefly shaking, add 2 to 3 ml of barium chloride solution to each of them.

After the addition of barium chloride solution, a white precipitate is to be seen in the test tube to which hydrogen peroxide was added.

Evaluation

Sulphur dioxide reduces iodine to iodide in the first test tube (with Lugol’s solution), and the solution is so decolourised:

$$I_2 + 6\,H_2O + SO_2 \rightarrow 2\,I^- + 4\,H_3O^+ + SO_4^{2-}$$

In the second test tube, sulphur dioxide from the flue gas is adsorbed by the calcium hydroxide solution, and reacts to calcium hydrogen sulphite (calcium bisulphite);

$$Ca^{2+} + 2\,OH^- + 2\,SO_2 \rightarrow Ca^{2+} + 2\,HSO_3^-$$

The initially alkaline solution slowly becomes acidic, as can be observed from the colour change of the universal indicator. In acidic solution, hydrogen peroxide oxidises hydrogen sulphite to sulphate:

$$2\,HSO_3^- + 2\,H_2O_2 \rightarrow 2\,SO_4^{2-} + 2\,H_3O^+$$

The sulphate formed is detected by the precipitation with barium chloride:

$$SO_4^{2-} + Ba^{2+} \rightarrow BaSO_4\downarrow$$