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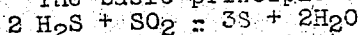
Sulfur Extracting Process of the Sachtleben A.G.
Szombathy process, Ger. pat. appl. S117,702

1. History of the process:

The process was invented by Dr. von Szombathy, but the original method (as described in Ger. pat. app. S 117, 702) was a failure when applied to the H₂S-Removal from lignite gas.

2. Procedure

The basic principle of the process is the well known reaction:



whereby the reaction is carried out in a thiosulfate solution. The process claims to recover a sulfur which can be easily filtered. The attached sketch represents a flow sheet of the process. The SO₂ can be obtained either by the combustion of sulfur or of 1/3 of the H₂S which is present in the gas after 2/3 of the H₂S have been extracted by a preceding process.

A combustion of the H₂S is advisable only if waste gases are available, chiefly waste gases which leave the claus kiln. Tests of such type were carried out at Gelsenkirchen.

The proper proportion of H₂S:SO₂ must always be maintained otherwise the sulfur content of the processed gas would be increased correspondingly. But insignificant variations are compensated by the solution. With a low sulfur content of the gas, variations of 100-150% with respect to the stoichiometric proportion are compensated, but with a higher sulfur content even variations of 10% might give rise to difficulties. Since the volume of the scrubbing solution, a small part of which is accumulated in the scrubber, is responsible for the compensating effect, it is possible to increase the latter by increasing the total volume of the solution. The concentration of the solution, which mostly was 10% during the test runs, is of no great importance.

A solution of sodium thiosulfate, which by introduction of SO₂ followed by a vigorous stirring accompanied by an introduction of SO₂ + H₂S, was activated and applied as scrubbing agent. The activating process as described before is disputable. While Dr. von Szombathy believes, that the presence of H₂S is not required for the activation and that an additive compound of oxygen and thiosulfate is formed with a simultaneous liberation of sulfur from thiosulfate and oxygen under the influence of a vigorous stirring, the experts of Sachtleben, who confirmed that H₂S is required for the activation, are of the opinion that an intermediate formation of tri- and tetra-thionates may occur. Nevertheless the fact is still unexplained that the proportion of sulfur to alkali does not change during the activating procedure while the iodine consumption decreases and may even drop to zero. No explanation can be given for that phenomenon. When the Pruckdorf tests were run, the activation process was carried out in such a manner that the iodine consumption (expressed in ccm N iodine solution consumed by 2 ccm of the scrubbing solution) was reduced from 20 to 16, whereby the value 16 indicates a "strong activation". But comparing the attached flow sheet iodine, consumption of 12.0 or 13.2 respectively can be observed.

Should lignite carbonization gas have to be extracted, the correct maintenance of a pH value of 3.8 (permissible limits 3.5-4.0, determined by "Lyphan-pa per"). With the pH value below the mentioned figures the purification effect deteriorates and the solution is spoiled by a

formation of sulfate. Operation with a higher pH value must be prevented only if low temperature carbonization gases are to be purified. By such an improper operation an elemental sulfur will be obtained, which is hard to filter due to its adhesive properties. The reason is supposed to be a formation of mercaptanes due to unsaturated hydrocarbons which are present in the low temperature carbonization gas. Should low temperature carbonization gases have to be treated, the upper limit of the admissible temperature is 45°C . and no excess of H_2S should be present during the activating process.

If gases are scrubbed which do not contain such unsaturated organic hydrocarbons, the pH value can be increased to 6. It is then possible to operate at higher temperatures whereby an excess of H_2S must be used. But it must be borne in mind that working under those conditions the excess H_2S is carried away with the treated gas.

Sometimes it is difficult to maintain the proper temperatures. One must differentiate between the following types of gases:

1. Gases which contain high concentrations of H_2S .
2. Gases rich in H_2S -content
3. Gases with low H_2S concentration

Only if gases which are rich in H_2S -content are treated, the heat of reaction is just high enough to vaporize the reaction-water during the activating process without exceeding a temperature of 45°C . The water vapors are condensed in a following condenser. But it will be difficult to dispose of the reaction heat if highly concentrated gases are scrubbed, while treating gases low in H_2S content heat must be fed in in order to vaporize the reaction water. Difficulties arise especially if temperatures above 45°C . are required for the vaporization of the reaction water. As mentioned before such a procedure can only be used if the gas to be scrubbed does not contain unsaturated hydrocarbons and if H_2S -losses are permissible.

3. Details of the process

The process is controlled by determining the pH value, the iodine consumption and the density of the solution. The adjustment of the proper pH value of the scrubbing solution is performed by controlling the ratio of the introduced H_2S and SO_2 , whereby the volume of the latter is regulated by the quantity of the produced SO_2 .

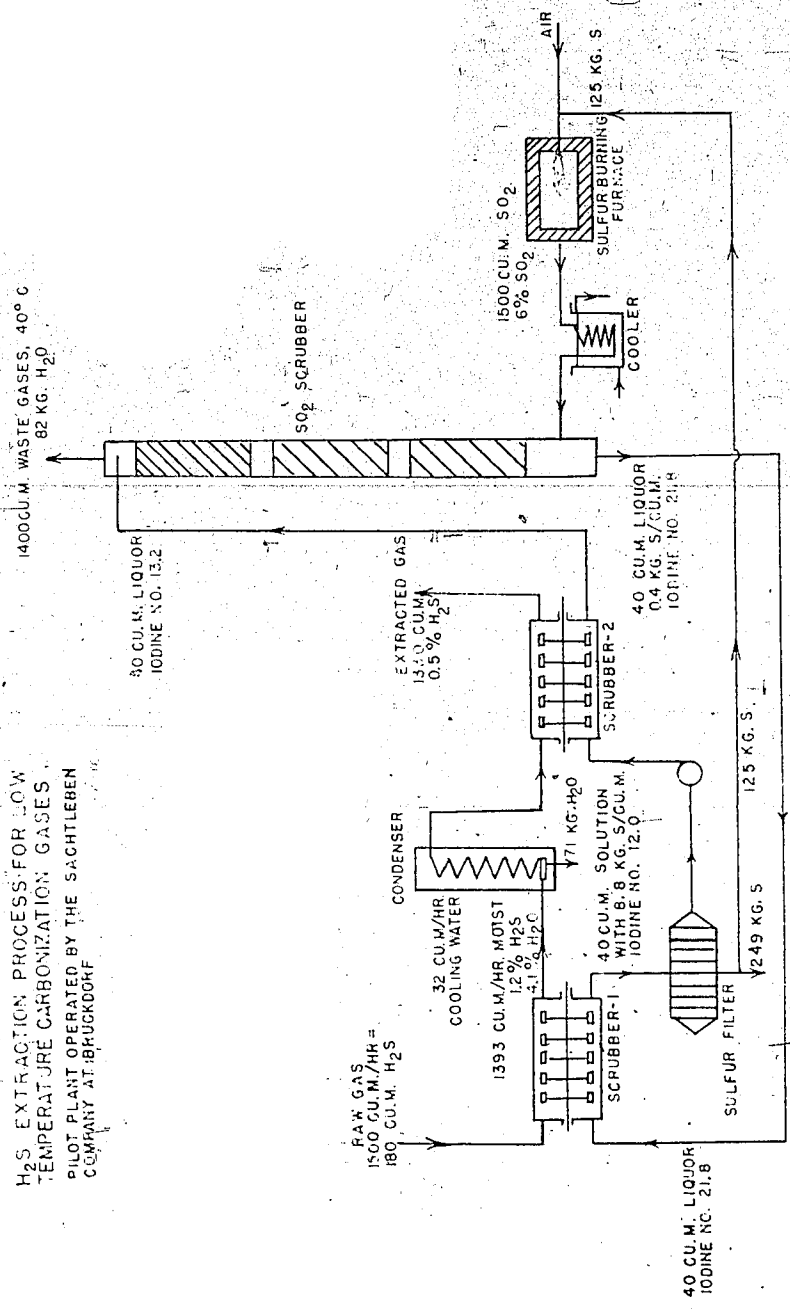
Operating the Bruckdorf plant a H_2S -removal of 90% was obtained. Oil mist which was present in the gas was not objectionable to the process.

The contact time of the gas was 1.2 minutes. Stroeder washers are therefore not as suitable as Raschig-ring-packed towers. A new scrubber was designed at the Bruckdorf plant which was similar to a Stroeder scrubber but contained numerous paddles instead of the rotating discs. One third of the horizontal section is filled with the scrubbing solution, which passes through the scrubber in a parallel flow to the gases. The energy consumption is 1 kwh per 1 cu. m. volu of the scrubber.

Performing the Bruckdorf tests a gas volume of $3 \text{ m}^3/\text{hr}$ was processed. The output of the laboratory scrubber was 600 liter per hour its volume was 12 liter.

The solution can not be evaporated or, if it must be done, vacuum should be applied. Since the activated solution is unstable, it should not be allowed to remain in a tank for any length of time, in order to prevent decomposition. The fouled solution however which leaves the scrubber is completely stable. Thiosulfate losses based on the recov-

H₂S EXTRACTION PROCESS FOR LOW TEMPERATURE CARBONIZATION GASES
PILOT PLANT OPERATED BY THE SACHTLEBEN COMPANY AT BRÜCKDORF



elemental sulfur are about 3%, calculated as sodium carbonate. Approx 2% is lost during the filtering procedure of the elemental sulfur and 1% is due to SO_3 -formation during the SO_2 -production. The Lurgi company confirmed that no higher losses are to be expected. The thiosulfate losses can be compensated in the form of sodium carbonate.

The recovered sulfur contains approx. 60% moisture. The first parts of the washing liquor are returned to the recirculating solution. Rubber or lead coated steel is suitable as material of construction of the scrubber whereas the containers which handle the liquor can be made out of wood or brick lined steel.

4. Costs of the process

The costs of the process are not yet determined. It is only known that 150 kwh and 30 kg of sodium carbonate are required for the production of 1 metric ton of sulfur.

5. Performance of additional test runs

The laboratory apparatus will be operated for a short period of time at the Bruckdorf plant in order to be able to supply some more details of the process.

6. Questions concerning the patent situation

The patent office of I.G. Farben, after having read the patent application S-107, 702 doubts whether it will be possible to obtain a patent.