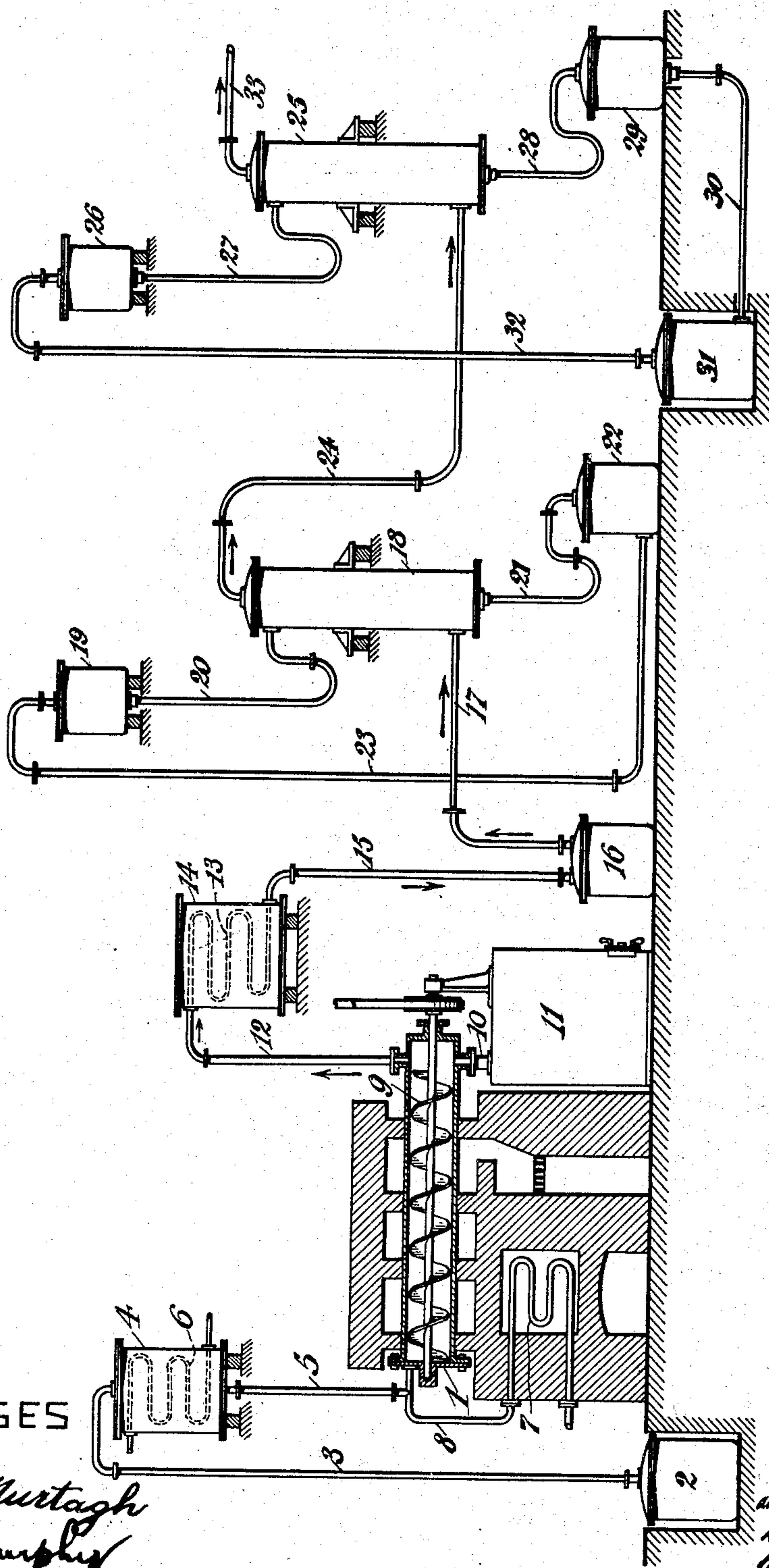


G. SCHILDHAUS & C. CONDREA.
 PROCESS OF OBTAINING SULFUROUS ACID FROM ACID SLUDGE.
 APPLICATION FILED APR. 16, 1909.

956,184.

Patented Apr. 26, 1910.



WITNESSES

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UNITED STATES PATENT OFFICE.

GUSTAV SCHILDHAUS AND CONSTANTIN CONDREA, OF CAMPINA, ROUMANIA.

PROCESS OF OBTAINING SULFUROUS ACID FROM ACID SLUDGE.

956,184.

Specification of Letters Patent.

Patented Apr. 26, 1910.

Application filed April 16, 1909. Serial No. 490,318.

To all whom it may concern:

Be it known that we, GUSTAV SCHILDHAUS, manager, a subject of the King of Roumania, residing at Campina, Strada Industriei, Casale, Stelei, Roumania, and CONSTANTIN CONDREA, chemist, a subject of the King of Roumania, residing at Campina, Strada I. C., Bratianu 68, Roumania, have invented certain new and useful Improvements in Processes of Obtaining Sulfurous Acid from Acid Sludge; and we do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

It has been proposed more than once to turn to account the acid tar produced in the purification of raw mineral oils, that is petroleum or naphtha and its distillation-products, by means of sulfuric acid by distilling or gasifying said tar and thereby obtaining from it sulfurous acid, liquid hydrocarbons and coke. None of the methods proposed have however found their way into practice because the sulfur contained in the tar was only in part obtained in the form of sulfurous acid. The sulfurous acid was in fact partly reduced to sulfureted hydrogen and even sulfur, and the other by-products have no great value owing to their indifferent quality.

It has now been found, that the acid tar may be distilled with good results by introducing heated air into the retort during distillation and thereby oxidizing the sulfur and its compounds. In this manner three products are obtained from the tar: 1. A mixture of gases mainly consisting of sulfurous acid, nitrogen and air and containing small quantities of carbonic acid and gaseous hydrocarbons. 2. Liquid hydrocarbons. 3. Coke. The tar is preferably vaporized at a comparatively low temperature of between 250° and 350° C. and the air introduced into the retort is preferably heated to the same temperature. The acid tar is run into the retort in a thin stream. The coke remaining in the retort is porous and brittle and is continuously evacuated by a mechanical device and transported to a closed chamber. The gases that issue from the retort are conducted through a cooler to condense the liquid hydrocarbons, and are then washed first by means of heavy hydrocarbon

oil and finally by means of concentrated sulfuric acid to remove the gaseous hydrocarbons and the last traces of the vapors of liquid hydrocarbons.

The figure is a diagrammatic side-view of the apparatus for carrying out the process, parts being shown in side-elevation and part in section.

The process is as follows: The tar is introduced in a thin stream into the retort 1 from the montejus 2 through the pipe 3, vessel 4 and pipe 5. It is heated within the vessel 4 by means of the heating coil 6. In the retort 1 the tar is vaporized, preferably at a comparatively low temperature of between 250 and 350° C. The air is preferably heated to the same temperature by means of the heating coil 7 and is introduced into the retort 1 through pipe 8. The coke remaining in the retort is porous and brittle, and is continuously evacuated by the feeding screw 9 through the pipe 10 into the closed chamber 11. The gases issuing from the retort 1 are conducted through the pipe 12 and cooling coil 13 which lies within the condenser 14. The larger part of the liquid hydrocarbons is condensed in the cooler and fed through pipe 15 into the vessel 16. The gases escaping from the vessel 16 are fed through pipe 17 into the scrubber 18 in which they are washed by means of heavy hydrocarbon oil which is run continuously from the vessel 19, through the pipe 20, through the scrubber 18 and the pipe 21 into the vessel 22 and back to the vessel 19 through the pipe 23. The washed gases escape from the scrubber 18 through the pipe 24 and enter the scrubber 25, where they are washed with sulfuric acid which is contained in the vessel 26 and flows through the pipe 27 into the scrubber 25, pipe 28 to vessel 29 and pipe 30 to the montejus 31 wherefrom it is fed back through the pipe 32 into the vessel 26. By washing the gases with heavy hydrocarbon in the scrubber 18 and afterward with sulfuric acid in the scrubber 25 they are freed from the last traces of the gaseous hydrocarbons. The purified gases consist in their essential part of sulfurous acid which escapes through the pipe 33, and they are then collected and may be utilized for known purposes, particularly for the production of sulfuric acid. The process described may be also applied

to the tar produced in the refining of mineral oils by means of fuming sulfuric acid or sulfuric anhydrid.

We claim:

5 1. Process of producing sulfurous acid, liquid hydrocarbon, and coke from acid tar or sludge which consists in distilling said acid tar and introducing a stream of air into the retort during distillation, condensing
10 the liquid hydrocarbons contained in the distillation products, washing the remaining sulfurous acid gas, and continuously removing the coke from the retort.

15 2. The process of producing sulfurous acid, liquid hydrocarbons and coke from acid tar or sludge which consists in distilling said acid tar, introducing a stream of air heated to from 200-300° C. into the retort during distillation, condensing the
20 liquid hydro-carbons contained in the distillation-products, washing the remaining

sulfurous acid gas, and continuously removing the coke from the retort.

3. The herein described process consisting in introducing acid tar or sludge into a re- 25 tort and heating the acid tar to a temperature of between 200° and 300° C., introducing air to substantially the same temperature into said retort, whereby gases are given off, condensing part of said gases to a liquid, 30 washing the remaining gases with heavy hydrocarbon oil, and finally washing said remaining gases with sulfuric acid.

In testimony, that we claim the foregoing as our invention, we have signed our names 35 in presence of two subscribing witnesses.

GUSTAV SCHILDHAUS.
CONSTANTIN CONDREA.

Witnesses:

GERHARD BERGER,
T. W. KRAFT.