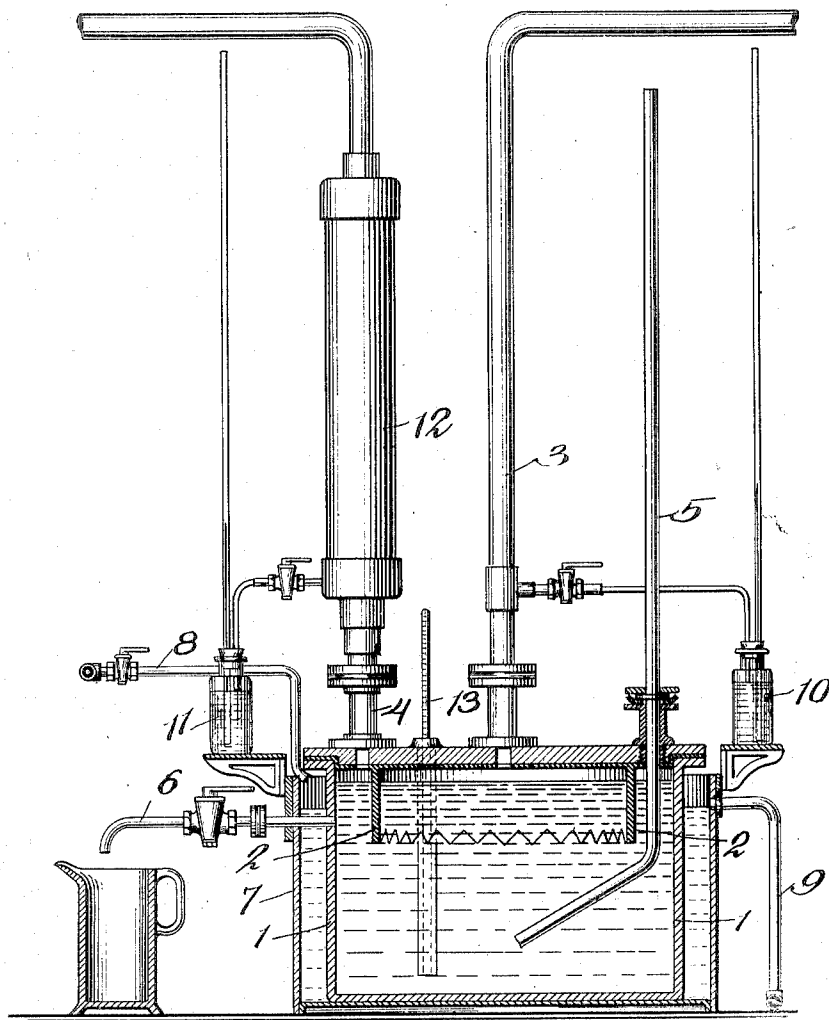


No. 816,918.

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R. KNIETSCH.
PROCESS OF MAKING SULFURIC ACID.
APPLICATION FILED JULY 16, 1901.



WITNESSES:

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PROCESS OF MAKING SULFURIC ACID.

No. 816,918.

Specification of Letters Patent.

Patented April 3, 1906.

Application filed July 16, 1901. Serial No. 68,498.

To all whom it may concern:

Be it known that I, RUDOLF KNIETSCH, doctor of philosophy and chemist, a subject of the King of Prussia, German Emperor, residing at Ludwigshafen-on-the-Rhine, in the Kingdom of Bavaria, Germany, have invented new and useful Improvements in the Manufacture of Sulfuric Acid, of which the following is a specification.

In the accompanying drawing I have shown an elevation of an absorber in section and of parts adjacent thereto adapted for carrying out my invention.

The method hitherto employed for absorbing sulfuric anhydrid (SO_3) manufactured on a commercial scale consists in passing the gas containing the sulfuric anhydrid through absorbers in communication with one another and containing sulfuric acid of various strengths. The concentration of the acid in the vessel nearest the source of the sulfuric anhydrid is greatest, while those farther removed contain weaker and weaker acids. The vessels are arranged in any convenient manner—for instance, one above the other in the form of terraces or steps—and weak acid—say chamber-acid or acid containing seventy-eight per cent. of H_2SO_4 —is allowed to flow into the uppermost vessel, while the gases containing SO_3 first enter the lowest vessel. The acid becomes gradually enriched in its downward passage, so that in the lowest vessel acid of high concentration, or even fuming sulfuric acid, is formed, and this is allowed to flow into receivers. The said process has, however, many defects. The plant is expensive to lay down and work, owing to the large number of single apparatus needed and the careful attendance which is thereby necessitated. Again, the absorbing apparatus are built of iron, which is practically the only material possible for this purpose—that is, when working on the large scale. It is consequently almost impossible to obtain in the manner described clear sulfuric acid technically free from iron, on account of the large surface of iron exposed to the action of sulfuric acid of such varying and different concentration. This want of constancy in strength of the acid also makes it exceedingly difficult, if not impossible, in practice to completely condense

the sulfuric anhydrid, so that not only is sulfuric acid liable to be lost, but also to escape as an injurious mist into the atmosphere.

The object of my present invention is to remedy the said defects by providing a method for completely absorbing sulfuric anhydrid by means of a considerably smaller plant than has hitherto been necessary and at the same time producing a highly-concentrated acid which for technical purposes may be considered free from iron.

I have discovered that sulfuric acid of a strength varying within narrow limits—namely, between ninety-seven and ninety-nine per cent.—and preferably containing ninety-eight and one-third per cent. of H_2SO_4 is capable of absorbing sulfuric anhydrid almost instantaneously and so efficiently that, if desired, the absorption can be carried out in a single absorbing apparatus. Acids of weaker concentration than ninety-seven per cent. absorb sulfuric anhydrid but slowly and incompletely, for in this case hydrate-fumes are formed which are then taken up by the acid with great difficulty, even when a long series of absorption vessels is employed. On the other hand, acid containing more than ninety-nine per cent. of H_2SO_4 (although no free SO_3 is present therein) allows fumes of sulfuric anhydrid to escape. Another advantage offered by the employment of acid of ninety-seven to ninety-nine per cent. for absorption purposes is to be found in the purity of the sulfuric acid that can thereby be obtained. I have mentioned above that the process hitherto in use for absorbing sulfuric anhydrid gives rise to an acid which usually contains iron compounds. I have also mentioned in the specification of application for Letters Patent Serial No. 68,499, of even date herewith, that iron is practically insoluble in fuming sulfuric acid containing twenty-seven per cent. or more of free SO_3 .

I have now further discovered that there is a second degree of concentration of sulfuric acid, at which the acid has but an exceedingly slight action on iron apparatus, and that this degree is approximately the same as that at which the power of the acid for absorbing sulfuric anhydrid is at its greatest. It is thus possible in one operation to effect a complete ab-

sorption of sulfuric anhydrid and to produce a highly-concentrated acid technically free from iron.

The process of manufacturing sulfuric acid which in view of the aforementioned discoveries I have worked out consists in bringing sulfuric acid containing from ninety-seven to ninety-nine per cent. of H_2SO_4 into an absorption apparatus. The gaseous sulfuric anhydrid or gas containing the sulfuric anhydrid is then passed through the same and the acid kept to the said degree of concentration by the uninterrupted introduction of a suitable quantity of steam, water, or pure dilute sulfuric acid. The acid as formed can be removed by suitable means, so that a constant level in the absorption apparatus is maintained.

The following example will serve to further illustrate the nature of my invention and the manner in which the same may best be carried into practical effect: but the invention is not confined to this example.

Example: Fill a vessel, preferably of cast-iron and made on the principle of a wash-bottle, with sulfuric acid containing ninety-seven to ninety-nine per cent. of H_2SO_4 , to a convenient depth. Fit the vessel with a tube (of material unacted on by sulfuric acid) whose mouth dips into the middle of the absorption liquid and which serves for the introduction of the steam or diluting liquid. The inflow of the latter is regulated by a valve or by other suitable means. Cause the gases containing the sulfuric anhydrid to pass either by suction or pressure through the absorption liquid. At the same time continuously introduce sufficient steam, water, or pure dilute sulfuric acid, so that the absorbing acid constantly contains ninety-seven to ninety-nine per cent. of H_2SO_4 . The amount of diluent to be employed is determined and controlled by the examination of the acid produced which is allowed to run off through an inverted siphon. Cool the absorbing vessel to remove the heat of absorption in any suitable manner.

Instead of directly absorbing the sulfuric anhydrid it can be first mixed with the calculated quantity of steam to form an acid containing ninety-seven to ninety-nine per cent. of H_2SO_4 , the procedure being otherwise as above described, and the absorption apparatus can be of any suitable form and is not limited to that of a wash-bottle.

The process hereinbefore described can also be used in combination with that set forth in the specification of the aforesaid application for Letters Patent, Serial No. 68,499, of even date herewith, thus making it possible in one operation to obtain fuming sulfuric acid and highly-concentrated sulfuric acid, as well as highly-concentrated acid alone, both tech-

nically free from iron, with the aid of a considerably-smaller plant than has hitherto been possible and without loss of sulfuric acid.

The absorber shown in the accompanying drawing may be described as follows: 1 is a cast-iron inclosure from the top of which is suspended a cast-iron hood 2, the lower edge of which is serrated. The sulfuric-anhydrid-supply pipe 3 is connected with the top of the hood. The exit-pipe 4 leads from the top of the inclosure 1. The inclosure 1 is supplied with sulfuric acid through the stand-pipe 5, extending to near the bottom of the inclosure, the level of the sulfuric acid within the inclosure 1 being kept above the teeth of the hood. 6 is a faucet through which the finished product is drawn off. 7 is a water-tank in which the inclosure 1 is immersed for cooling and through which a current of water is passed from the pipe 8 to the pipe 9. 10 and 11 are pressure-gages connected, respectively, with the inlet-pipe 3 and the outlet-pipe 4. The outlet-pipe 4 should be wholly or partially of larger diameter, as at 12, so that the unabsorbed gas passing away from the receptacle 1 will have a chance to deposit any liquid that it may contain, which will run back into the receptacle 1. 13 is a thermometer for determining the temperature in 1. In the operation of the apparatus the attendant charges the inclosure 1 with sulfuric acid of about the strength of the finished product, using acid of a lower strength or other diluent to replenish the inclosure 1 through the pipe 5.

Now what I claim is—

1. The improvement in the process of manufacturing sulfuric acid by the absorption of sulfuric anhydrid in sulfuric acid which consists in maintaining throughout the operation the strength of said absorbing acid between ninety-seven and ninety-nine per cent. of H_2SO_4 .

2. The process of manufacturing sulfuric acid, which consists in causing sulfuric anhydrid to be absorbed by a body of liquid acid and simultaneously supplying a diluting agent in sufficient quantity to maintain the liquid acid at its original degree of concentration, so that from the start of the operation to the discharge of the finished product, the strength of the sulfuric acid remains unaltered.

3. The herein-described process for manufacturing sulfuric acid, which consists in providing a body of liquid acid of substantially the same strength as the product it is desired to obtain, causing sulfuric anhydrid to be absorbed by said acid and supplying simultaneously with the sulfuric anhydrid a sufficient amount of a diluting agent to said body of liquid acid, to keep said liquid acid at a constant degree of concentration from the beginning of the operation to the end.

4. The process of manufacturing sulfuric acid, which consists in causing sulfuric anhydrid to be absorbed by liquid sulfuric acid, supplying simultaneously with the anhydrid
5 a diluting agent in sufficient quantity to keep the liquid acid at its original degree of concentration, and cooling the liquid acid where it absorbs the anhydrid.

In testimony whereof I have hereunto set my hand in the presence of two subscribing witnesses:

RUDOLF KNIETSCH.

Witnesses:

JACOB ADRIAN,
JOHN L. HEINKE.