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PROCESS OF MANUFACTURING ANHYDRIDS OF FATTY ACIDS.

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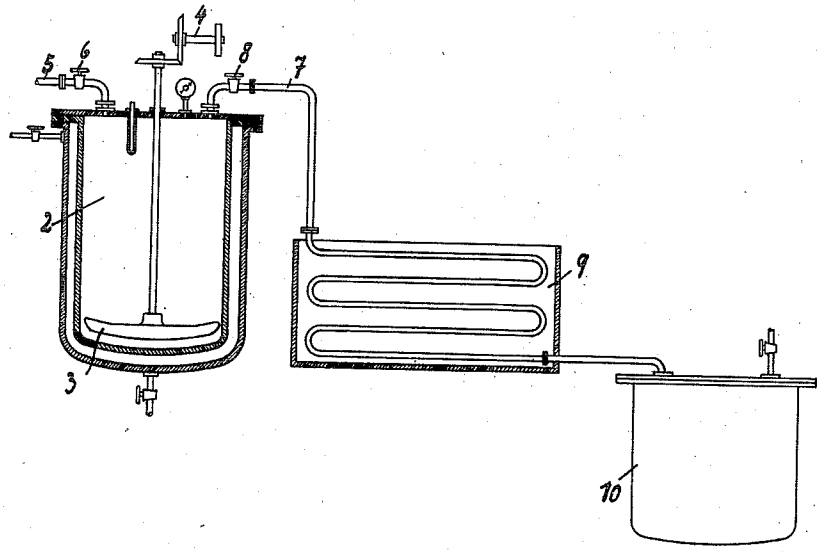

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UNITED STATES PATENT OFFICE.

JOSEF WEBER, OF ESSEN-ON-THE-RUHR, GERMANY.

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1,018,733. Specification of Letters Patent. Patented Feb. 27, 1912.

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To all whom it may concern:

Be it known that I, JOSEF WEBER, subject of the King of Prussia, German Emperor, resident of Essen-on-the-Ruhr, in the German Empire, have invented a new and useful Process of Manufacturing Anhydrids of Fatty Acids, of which the following is an exact specification.

This invention relates to an improved process of manufacturing anhydrids of fatty acids, especially acetic anhydrid, from a fatty acid salt or from a mixture of salts of the same fatty acid; the process being an improvement upon that disclosed in the prior application Serial No. 525,266, filed Oct. 29, 1909.

Prior to the aforesaid application the manufacture of fatty acid anhydrids from fatty acid salts by heating such salts with chlorid of sulfur had been proposed. The manufacture of this chlorid of sulfur had proved however, to be very troublesome and expensive. In the process disclosed in the aforesaid application the use of chlorid of sulfur was avoided and the process carried out with much less trouble by mixing a fatty acid salt or salts in a dry state with sulfur, usually in the form of powder of sulfur or flowers of sulfur, and acting on this mixture with dry chlorin, good results having been obtained by taking the materials in proportion, for example, of about 8 molecules of acetate (of a monobasic metal) to 1 atom of sulfur, these proportions, however, not being essential. In the process of the aforesaid application the reaction took place at a low temperature—say at —20° C. or less—and at that time the carrying out of the process at a low temperature seemed to be essential. At the end of the operation of carrying out the process of the aforesaid application the product obtained by the reaction was moderately heated for some time in order to transform any fatty acid chlorid still present into anhydrid, after which the anhydrid was eventually distilled *in vacuo* in a manner well understood. I have found, however, that in carrying out the process of the aforesaid application, especially with acetic anhydrid, the reaction on which said process is based takes place not only at the low temperatures above mentioned as set forth in the prior application, but that it also takes place, and that it is possible to work with good effect at considerably higher temperatures, if the mixture of the fatty acid salt or acetate with sulfur is acted upon by chlorin in the presence of a suitable diluting or distributing substance, such for example as acetic anhydrid. The temperature up to which such reaction may take place with good results may be said to be about 40°–50° C.

An important advantage resulting from the carrying out of this improved process is that it is not necessary in the present case, as it was in the process described in the aforesaid application, to maintain during the reaction temperatures which are only obtainable by the use of refrigerating machines. Thus in the present case, as these refrigerating machines are not required, both the process and the apparatus for carrying out the process are simplified in a high degree, and by this simplification the manufacture of fatty acid anhydrid, and particularly acetic anhydrid, on a large scale is facilitated and can be effected at much lower cost than in the case of the process described in the former application.

In the drawing apparatus suitable for carrying out the improved process is illustrated.

Referring to said drawing, 2 designates a tightly closed reaction chamber or vessel into which the mixture to be acted upon has been introduced. A stirrer or means for keeping in motion the substance forming the charge is illustrated at 3. It may be operated by any suitable mechanism, such as that shown at 4. The chlorin employed, which should be in a dry state, may be admitted at 5, and its flow regulated or varied as desired, as for example by means of a valve 6. The anhydrid resulting from the reaction will in this case leave the reaction vessel through a pipe 7, which may also have a valve 8 therein, by means of which communication with the reaction vessel may be made or cut off as required. The pipe 7 leads to the coils of a condenser 9, in which the anhydrid distilled over from the reaction vessel 2 is condensed and from which the liquid distilled passes to a receiving vessel 10.

In order to make clear the manner in which this process is practiced the following is given as one example: In the reaction vessel 328 kilograms of carefully dried sodium acetate are mixed with at least 16 kilograms of sulfur or flowers of sulfur and an amount

of acetic anhydrid equal to the quantity of sodium acetate used. The vessel 2 is then tightly closed and chlorin introduced into this mixture at a temperature of say 25° C., the mixture being at the same time thoroughly stirred. If desired the reaction vessel may be exhausted before introducing the chlorin. The introduction of chlorin is so regulated that in say 5 hours at least 106½ kilograms will have entered the reaction vessel. The chlorin admitted is quickly taken up by the mixture and the pressure prevailing in the chamber 2 at the end of the reaction usually exceeds that at the beginning by only one tenth of an atmosphere or a little more. It is believed to be unnecessary to consider whether in this reaction the acetic anhydrid present only acts as a diluent or distributing means, or whether its action is a catalytic one that results in advancing the reaction.

At the end of the reaction the resulting liquid anhydrid may be distilled substantially as in my aforesaid application. As in the former case it is advisable to hold a high vacuum during distillation, as it is only with the highest degree of vacuum that the output of anhydrid will be approximately the theoretical one, *i. e.*, 204 kilograms.

What I claim is:

1. A process for the production of fatty acid anhydrid, which consists in mixing a fatty acid salt with sulfur, both being in a dry state, and treating the mixture with chlorin at temperatures up to 40°–50° C. in the presence of a suitable diluent.

2. A process for the production of fatty acid anhydrid, which consists in mixing a fatty acid salt with sulfur, both being in a dry state, and treating the mixture with chlorin at temperatures up to 40°–50° C. in the presence of an anhydrid of a fatty acid.

3. A process for the production of acetic anhydrid which consists in mixing an acetate with sulfur, both being in a dry state, and treating the mixture with chlorin at temperatures up to 40°–50° C. in the presence of acetic anhydrid.

4. A process for the production of fatty acid anhydrid, which consists in mixing a plurality of salts of the same fatty acid with sulfur, all being in a dry state, and treating the mixture with chlorin at temperatures up to 40°–50° C. in the presence of a suitable diluent.

5. A process for the production of fatty acid anhydrid, which consists in mixing a plurality of salts of the same fatty acid with sulfur, all being in a dry state, and treating the mixture with chlorin at temperatures up to 40°–50° C. in the presence of an organic acid anhydrid.

6. A process for the production of fatty acid anhydrid, which consists in mixing a plurality of salts of the same fatty acid with sulfur, all being in a dry state, and treating the mixture with chlorin at temperatures up to 40°–50° C. in the presence of an anhydrid of the same fatty acid.

7. A process for the production of acetic anhydrid, which consists in mixing a plurality of acetates with sulfur, all being in a dry state, and treating the mixture with chlorin at temperatures up to 40°–50° C. in the presence of acetic anhydrid.

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