UNITED STATES PATENT OFFICE.

ROLAND L. ANDREAU, OF WILMINGTON, DELAWARE, ASSIGNOR TO E. I. DU PONT DE NEMOURS AND COMPANY, OF WILMINGTON, DELAWARE, A CORPORATION OF DELAWARE.

PROCESS FOR THE PRODUCTION OF SYNTHETIC CAMPHOR.

1,326,248. Specification of Letters Patent. Patented Dec. 30, 1919.

No Drawing. Application filed February 27, 1917. Serial No. 151,172.

To all whom it may concern:

Be it known that I, ROLAND L. ANDREAU, of Wilmington, in the county of New Castle, and in the State of Delaware, have invented a certain new and useful Improvement in Processes for the Production of Synthetic Camphor, and do hereby declare that the following is a full, clear, and exact description thereof.

My invention relates particularly to the production of camphor synthetically, but it relates especially to the production of the same from isoborneol.

Hitherto it has been found that while borneol could be changed to camphor at relatively low temperatures by treatment with pure nitric acid, isoborneol when subjected to a similar treatment only gave a yellow oil of uncertain composition. Again, when treated with pure boiling nitric acid isoborneol would produce explosive products. Furthermore, under no circumstances was it possible to obtain any large yield of camphor from isoborneol by the application of pure nitric acid.

The object of my invention is to produce camphor by the treatment of isoborneol with pure nitric acid in such a manner as to readily produce large yields of camphor without the formation of explosive compounds.

I have discovered that this can be accomplished by heating the acid to a temperature of from 75 to 86° C. in the presence of the isoborneol and that the reaction may be readily carried out by locally heating a portion of the body of acid to the desired temperature, whereupon the reaction proceeds throughout the whole body of the isoborneol and acid, thus producing a nearly theoretical yield without the formation of explosive products. By proceeding in this manner it is possible after the initial heating to carry on the reaction at a temperature of about 40° C. or less despite the fact that the nitric acid is initially substantially free from substances capable of inducing a vigorous oxidation of isoborneol by the nitric acid at a temperature of 40° C.

While my invention is capable of being carried out in many different ways for the purpose of illustration I have only described certain forms of my invention hereinafter.

For example I may introduce 100 kilograms of nitric acid having a specific gravity of from 1.40 to 1.42 in an acid proof container and heat the same to 40° C. Thereupon 1 kilogram of isoborneol is sprinkled on top of the acid. A heating tube about one inch in diameter and eighteen inches long made of glass closed at one end and heated inside with an electric resistance or a current of steam is plunged into the mass so as to raise its temperature locally to from 75 to 86° C., whereupon the reaction begins. The heating tube is then withdrawn and isoborneol is added in small quantities until 25 kilograms thereof have been introduced, the temperature of the mass being maintained meantime between 40 and 45° C. The reaction then continues until the entire quantity of the isoborneol has been acted upon. The supernatant liquid is then decanted and allowed to run slowly into a body of water with constant stirring. The camphor will then separate as a flocculent precipitate, which is subsequently drained, washed and dried.

As another example of my invention I may proceed in the same manner as in the example just described, except instead of using the heating tube I may provide on the container for the acids an outside tube opening into the main body of acid, which may be arranged to contain approximately 500 grams of acid and 10 grams of isoborneol. This tube is heated from the outside to from 75 to 86° C., whereupon the reaction begins and is propagated throughout the entire mass of material.

While I have described my invention above in detail I wish it to be understood that many changes may be made therein without departing from the spirit of my invention as for example the temperatures and the quantities of the materials may vary to a considerable extent.

I claim:

1. The process of producing camphor from isoborneol by oxidation which comprises heating isoborneol to a temperature of from about 75 to 86° C. in the presence of nitric acid which is substantially free from substances capable of inducing a vigorous oxidation of the isoborneol by nitric acid at a temperature of 40° C., and then lowering the temperature of the heated mass materially below 75° C. after the reaction is well started.

2. The process of producing camphor from isoborneol by oxidation which comprises heating isoborneol to a temperature of from about 75 to 86° C. in the presence of nitric acid which is substantially free from substances capable of inducing a vigorous oxidation of the isoborneol by nitric acid at a temperature of 40° C., and then lowering the temperature of the heated mass to from about 40 to 45° C. after the reaction is well started.

3. The process of producing camphor from isoborneol by oxidation which comprises locally heating to a temperature between 75 and 86° C. a relatively small unseparated portion of a mixture of isoborneol and nitric acid, said nitric acid being substantially free from substances capable of inducing a vigorous oxidation of isoborneol by nitric acid at a temperature of 40° C., and then maintaining the temperature of the mixture materially below 75° C. after oxidation of the isoborneol is well started.

4. The process of producing camphor from isoborneol by oxidation which comprises locally heating to a temperature between 75 and 86° C. a relatively small unseparated portion of a mixture of isoborneol and nitric acid, said nitric acid being substantially free from substances capable of inducing a vigorous oxidation of isoborneol by nitric acid at a temperature of 40° C., and then maintaining the temperature of the mixture at from about 40 to 45° C. after oxidation of the isoborneol is well started.

In testimony that I claim the foregoing I have hereunto set my hand.

ROLAND L. ANDREAU.

Witnesses:
RALPH S. GIFFORD,
RALPH B. SEGAR.