

# UNITED STATES PATENT OFFICE

2,302,321

## PROCESS FOR THE PRODUCTION OF THE DILACTONE OF $\gamma$ KETOPIMELIC ACID

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No Drawing. Application June 12, 1941, Serial No. 397,740. In Germany July 8, 1940

6 Claims. (Cl. 260—344)

The present invention relates to a process for the production of the dilactone of  $\gamma$ -ketopimelic acid.

$\gamma$ -ketopimelic acid dilactone, as is known, can be prepared by decarboxylating dry succinic acid at high temperatures. The yield of  $\gamma$ -ketopimelic acid dilactone thus obtained, however, does not exceed from 40 to 45 per cent of the theory, because of considerable quantities of unavailable resins and tarry materials being formed simultaneously.

We have now found that  $\gamma$ -ketopimelic acid dilactone can be produced with a far better yield by heating succinic acid or its anhydride to a temperature of between 240 and 250° C. in the presence of an inert organic solvent boiling at or above the reacting temperature, i. e. at least at 240° C. A yield of  $\gamma$ -ketopimelic acid dilactone amounting to 70 per cent and more is obtained in this manner.

Suitable inert solvents are, for example, the alkylnaphthalenes, the chloronaphthalenes, fluorene, dibenzyl or high-boiling mineral oils.

The reaction is carried out by heating the succinic acid or its anhydride, together with an equal or larger or smaller amount of the inert solvent, to a temperature of from 240 to 250° C., if necessary while stirring. When larger quantities of succinic acid are employed, the quantity of the solvent need not be proportionately increased, since the dilactone formed will act as a solvent. The dilactone of the  $\gamma$ -ketopimelic acid formed is isolated by blowing off the inert solvent, for example, by means of steam or by distillation at normal or reduced pressure, and subsequently subjecting the remaining dilactone to a high vacuum distillation. It boils at 170° C. at a pressure of 2 millimeters (mercury gauge).

The following examples serve to illustrate how the present invention may be carried out in practice, but the invention is not restricted to the said examples. The parts are by weight.

### Example 1

90 parts of succinic acid and 150 parts of  $\alpha$ -methyl-naphthalene are heated at from 240° to 250° C., while stirring, until carbon dioxide is no longer evolved. After blowing off the  $\alpha$ -methyl-naphthalene by means of steam, the residue is subjected to a high vacuum distillation. There

are obtained 32 parts of  $\gamma$ -ketopimelic acid dilactone in the form of an almost colorless crystal mass which, being recrystallized from alcohol, melts at 71° C. 17 parts of succinic anhydride are recovered.

### Example 2

90 parts of succinic acid and 30 parts of  $\alpha$ -chloronaphthalene are heated at from 240° to 250° C. until the evolution of carbon dioxide is finished. The  $\alpha$ -chloronaphthalene and the unconverted succinic anhydride are then removed by distillation and the dilactone remaining behind is purified by high vacuum distillation. There are obtained 28 parts of  $\gamma$ -ketopimelic acid dilactone, while from 15 to 20 parts of succinic anhydride are recovered.

### Example 3

60 parts of succinic acid and 40 parts of dibenzyl are heated at from 240 to 250° C., until carbon dioxide is no longer evolved. By working up the reaction product in the manner described in Example 2 there are obtained more than 50 per cent (of the theory) of  $\gamma$ -ketopimelic acid dilactone. Instead of dibenzyl, a mineral oil fraction boiling between 250° and 270° C. may be employed.

What we claim is:

1. A process for producing  $\gamma$ -ketopimelic acid dilactone which comprises heating a member of the group consisting of succinic acid and its anhydride to a temperature of between 240 and 250° C. in the presence of an inert organic solvent having a boiling point of at least 240° C.

2. The process as described in claim 1, wherein the inert organic solvent as defined therein is an alkylnaphthalene.

3. The process as described in claim 1, wherein the inert organic solvent is  $\alpha$ -methyl-naphthalene.

4. The process as described in claim 1, wherein the inert organic solvent as defined therein is a chloronaphthalene.

5. The process as described in claim 1, wherein the inert organic solvent is  $\alpha$ -chloronaphthalene.

6. The process as described in claim 1, wherein the inert organic solvent is dibenzyl.

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