Patented Nov. 10, 1942

2,301,827

UNITED STATES PATENT OFFICE

2,301,827

PROCESS OF MAKING HIGHER ALIPHATIC LACTONES

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No Drawing. Original application May 25, 1934, Serial No. 727,583. Divided and this application April 29, 1940, Serial No. 332,395. In Switzerland July 17, 1933

2 Claims. (Cl. 260—344)

This application is a division of application Serial Number 727,583 filed May 25, 1934.

It is well known that higher lactones can be obtained by suitably oxidising higher carbocyclic ketones. It is also known that higher lactones are obtained if ω-halogen fatty acids are heated with metal oxides or the salts of ω-halogen fatty acids in indifferent solvents (German patent specification No. 449,217). As is apparent from the examples in the said German patent specification this prior process always involves lactonisation in the heterogeneous phase, that is to say, with undissolved salts, such as for example, copper and silver salts.

In contradistinction thereto it has been found that the higher lactones can be obtained with much better yields if the lactonisation (inner esterification) of the corresponding hydroxy acids or functional derivatives thereof is carried out in the vapor phase under conditions producing a low partial pressure. It is preferable to operate under conditions of reduced pressure so that the reactions taking part can proceed in the mono-molecular form.

Under these new conditions, contrary to what is said in German specification No. 449,217, the lactonisation of free ω-hydroxycarboxylic acids can be successfully effected. Of course, corresponding to the esterification reactions chosen for the lactonisation, derivatives of the hydroxycarboxylic acids may be used in place of the free acids, such as, for example, their esters, halides, acid chlorides and so forth.

The present method of carrying out the lactonisation is limited to those hydroxycarboxylic acids which possess a chain of eleven or more carbon atoms between their functional groups. It is immaterial whether the chain is branched or normal, whether it is saturated or unsaturated, or whether it still contains other substituted groups.

The operations may be conducted in the presence of esterifying catalysts. Only those derivatives of the ω-hydroxycarboxylic acids can be used which possess a certain stability at about 200° C., such as, for example, the formates or the carbonates of the hydroxycarboxylic acids. In order that the vaporisation takes place at the lowest possible temperature operations are carried out in the best possible vacuum i. e. with a low molecular concentration of the hydroxycarboxylic acid or derivative. It is then preferable to dilute the vapours with other inert gases or vapours.

The present invention enables higher lactones to be prepared in a manner which is very simple technically and in outstanding yields. As compared with the methods heretofore known, therefore, which either had to commence with costly higher ketones or, in the case of direct lactonisation of the ω-hydroxycarboxylic acids, afforded very small yields, the process according to the present invention represents an important technical advance.

The lactones made according to this invention are intended for use in the perfume industry.

Example

The formic acid ester of 14-hydroxytetradecane-1-carboxylic acid is very slowly led over a titanium dioxide or other acid-splitting catalyst heated to 200° to 300° C. in a vacuum, i. e. at a reduced partial pressure. The distillate collected in the cooled receiver is freed from admixed acid by means of soda solution and is purified by vacuum distillation.

What I claim is:

1. A process for making monomeric lactones from the formates of hydroxycarboxylic acids of the form HCOOR.COOH, where R is an aliphatic residue containing a chain of at least ten carbon atoms, consisting in removing the elements of formic acid from the acid in the presence of a catalyst consisting of titanium dioxide at elevated temperature, the formate of the acid being in the vapor phase and under a reduced pressure.

2. A process for making monomeric lactones from esters of hydroxy carboxylic acids of the formula XCO.OR.COOH in which XCO is an acyl group, where R is an aliphatic residue containing a chain of at least ten carbon atoms, consisting in removing the elements of the acid XCOOH from the acids in the presence of a solid phase catalyst at elevated temperature, the ester of the acid being in the vapor phase and under a reduced pressure.

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