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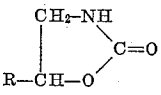
PRODUCTION OF 2-OXAZOLIDONES

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The present invention relates to a simple and economical process for the production of 2-oxazolidones.

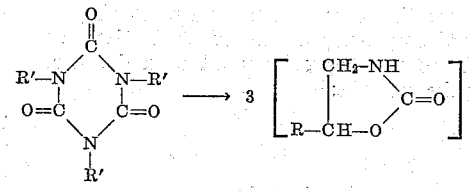
The 2-oxazolidones obtained by the process of this invention may be represented graphically by the following formula:



wherein R is a member of the group consisting of hydrogen and alkyl radicals. Of the alkyl radicals, methyl and ethyl are preferred.

The process is carried out by heating a tris-2-hydroxyalkyl isocyanurate in which the hydroxyalkyl radical contains at least 2 carbon atoms, preferably 2 to 4 carbon atoms, at temperature of about 150° to 250° C., preferably about 170° to 190° C., thereby forming a reaction mixture comprising a 2-oxazolidone compound, and recovering said 2-oxazolidone compound from the reaction mixture.

The reaction involved in the process of the present invention may be represented by the following equation:



tris-2-hydroxyalkyl isocyanurate — 2-oxazolidone compound

wherein R has the same significance as above and R′ is a hydroxyalkyl radical containing at least 2 carbon atoms, preferably 2 to 4 carbon atoms. When the alkyl component in the isocyanurate is ethyl, the resultant product is 2-oxazolidone. When the isocyanurate contains a higher alkyl component, the 2-oxazolidone compound formed is substituted in its 5-position with the alkyl radical containing 2 less carbon atoms than the alkyl component. For example, tris-2-hydroxypropyl isocyanurate forms 5-methyl-2-oxazolidone, and tris-2-hydroxybutyl isocyanurate forms 5-ethyl-2-oxazolidone.

High product yields and purities are favored by carrying out the reaction at low pressure, preferably at pressure not above about 3 mm. of mercury. At such low pressure, the 2-oxazolidone compound distills from the reaction zone and is recovered by condensation. When atmospheric pressure is employed, the 2-oxazolidone compound is retained in the reaction zone.

The tris-2-hydroxyalkyl isocyanurates may be prepared by the process described and claimed in our copending application Ser. No. 848,214, now Patent No. 3,088,948, of even date, wherein cyanuric acid is reacted with an

alkylene oxide, preferably containing 2 to 4 carbon atoms, at temperature of about 25° to 140° C., preferably about 100° to 140° C., in the presence of an inert solvent for the cyanuric acid and alkylene oxide and an alkaline catalyst. The inert solvent is preferably a dialkylformamide in which each of the alkyl radicals contains from one to four carbon atoms, and the alkaline catalyst is preferably a strongly alkaline material such as an alkali metal hydroxide.

The present invention may be carried out in two steps, involving as the first step the production and isolation of the tris-2-hydroxyalkyl isocyanurate, and as the second step the heat treatment of the intermediate isocyanurate to form the desired 2-oxazolidone compound. Alternatively, the reaction mixture from the first step may be heated, without isolating the intermediate isocyanurate, to form the 2-oxazolidone compound. At the reaction temperatures of this invention, conversion of the isocyanurate to the 2-oxazolidone compound by either procedure is relatively fast, generally taking place in a matter of seconds at temperature of about 170° C. or higher.

Exceptionally high yields of 2-oxazolidones are obtained by carrying out the reaction at the preferred temperature range of about 170° to 190° C. at temperatures below about 150° C., the reaction tends to become too slow to be practical. Moreover, at temperatures above about 250° C., the product tends to decompose resulting in unacceptably low yields.

The reaction is desirably effected at pressures ranging from 0.15 mm. to 3 mm. of mercury, and preferably from about 1 to 3 mm. of mercury. As indicated above, at these low pressures, the 2-oxazolidone compound distills from the reaction zone. The distilled 2-oxazolidone compound is then condensed to form the desired product.

Additional purification of 2-oxazolidone, which is obtained as a crystalline solid, may be accomplished by recrystallizing the solid from inert organic liquids, such as chloroform, carbon tetrachloride, methylene chloride, methanol and ethanol.

The following examples of the invention are given for purposes of illustration only. It will be understood that the invention is not limited to these examples. In these examples, parts are by weight.

EXAMPLE 1

(A) *Preparation of Tris-2-Hydroxyethyl Isocyanurate*

A solution of 600 parts of cyanuric acid and about 2800 parts of dimethylformamide was heated to a temperature of 130° to 140° C. in a reaction vessel equipped with a stirrer, thermometer and Dry Ice reflux condenser. When the solution attained the desired temperature, 40 parts of potassium hydroxide were added. 600 parts of gaseous ethylene oxide were then gradually introduced below the surface of the liquid at atmospheric pressure and at rate sufficient to maintain the temperature of 130° to 140° C. The ethylene oxide addition was completed in 3 hours. The reaction mass was cooled. Excess solid potassium hydroxide and by-product potassium salt of cyanuric acid were removed from the product solution by filtration. Excess dimethylformamide was then removed by vacuum distillation at pot temperature of about 85° C. and pressure of about 12 to 18 mm. of mercury to leave a viscous residue which crystallized upon cooling. This crystalline residue was recrystallized from ethanol. 779.5 parts of

recrystallized material comprising tris-2-hydroxyethyl isocyanurate having a melting point of 133° to 135° C. were obtained. This represented a yield of about 64% of theory (based on the cyanuric acid fed).

(B) *Preparation of 2-Oxazolidone*

127 parts of tris-2-hydroxyethyl isocyanurate were placed in the pot of a glass still arranged for distillation under reduced pressure. The pot was gradually heated to 180° C., while maintaining the pressure at 1 to 2 mm. of mercury. 2-oxazolidone distilled at an overhead temperature between 130° and 140° C. The reaction mass reached a temperature of 190° C. at the end of the distillation period. The distilled 2-oxazolidone was then condensed to a liquid which solidified to form crystallized 2-oxazolidone. 114 parts of 2-oxazolidone product, representing a yield of about 90% of theory (based on the tris-2-hydroxyethyl isocyanurate fed) were obtained. The product had a melting point of about 85° to 87° C.

EXAMPLE 2

A solution of 14.9 parts of cyanuric acid and 84.8 parts of dimethylformamide was heated to a temperature of 130° to 140° C. in a reaction vessel equipped with a stirrer, thermometer and Dry Ice reflux condenser. When the solution attained the desired temperature, 0.3 part of sodium hydroxide was added. Next, 14.9 parts of gaseous ethylene oxide were gradually introduced at atmospheric pressure below the surface of the liquid, maintaining the temperature of 130° to 140° C. The resultant reaction mass wass cooled and filtered to remove solid sodium compounds. The filtrate containing tris-2-hydroxyethyl isocyanurate was then heated in a film evaporator operating at 170° C. at atmospheric pressure, the bulk of the dimethylformamide being removed as distillate. The liquid residue comprising 2-oxazolidone was returned to the reaction vessel and heated at pot temperature of 115° C. and pressure of 30 mm. of mercury until the remaining dimethylformamide distilled off. The 2-oxazolidone residue was then cooled and recrystallized twice from methanol. The yield of 2-oxazolidone was approximately 50% of theory (based on the cyanuric acid fed). The product had a melting point of about 86.5° to 87° C. and a nitrogen analysis of 15.9% (theory—16.1%).

EXAMPLE 3

53 parts of tris-2-hydroxypropyl isocyanurate were placed in the pot of a glass still arranged for distillation under reduced pressure. The pot was gradually heated to 150° C., while maintaining the pressure at 3 mm. of mercury. 5-methyl-2-oxazolidone distilled at an overhead temperature of 113° to 118° C. The distilled 5-methyl-2-oxazolidone was then condensed to form a liquid constituting 35.2 parts. This represented a yield of about 61% of theory (based on the tris-2-hydroxypropyl isocyanurate fed). The product had $n_D^{25°\ C.}$ of 1.461 (literature—1.465) and molecular weight of 104 (theory —101).

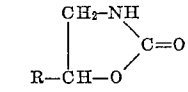
By means of the process of this invention, exceptionally high yields of relatively pure 2-oxazolidones are obtained. In particular, use of the preferred temperature and pressure conditions set forth above enables the obtainment of relatively pure 2-oxazolidone in yields of about 90% of theory or higher (based on the tris-2-hydroxyethyl isocyanurate fed).

2-oxazolidone possesses outstanding utility as a defoliating agent, especially of cotton plants. It can also be used as a source of ethylene imine and as a solvent for nylon-type polyamides. Moreover, 2-oxazolidone, as well as its 5-alkyl derivatives, are physiologically active.

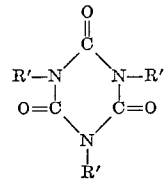
Since various changes and modifications may be made in the invention without departing from the spirit thereof, the invention is deemed to be limited only by the scope of the appended claims.

We claim:

1. A process for producing a 2-oxazolidone compound having the formula:

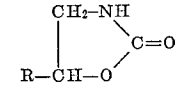


wherein R is a member of the group consisting of hydrogen and alkyl radicals, which comprises heating a tris-2-hydroxyalkyl isocyanurate having the formula:

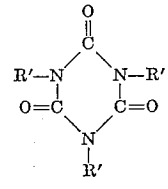


wherein R′ is a hydroxyalkyl radical containing at least 2 carbon atoms, at temperature in the range of about 150° to 250° C., thereby forming a reaction mixture comprising the 2-oxazolidone compound, and recovering said 2-oxazolidone compound from the reaction mixture.

2. A process for producing a 2-oxazolidone compound having the formula:

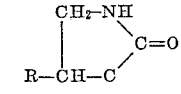


wherein R is a member of the group consisting of hydrogen, methyl and ethyl, which comprises heating a tris-2-hydroxyalkyl isocyanurate having the formula:

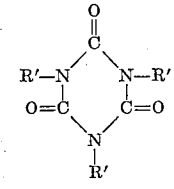


wherein R′ is a hydroxyalkyl radical containing 2 to 4 carbon atoms, at temperature in the range of about 150° to 250° C., thereby forming a reaction mixture comprising the 2-oxazolidone compound, and recovering said 2-oxazolidone compound from the reaction mixture.

3. A process for producing a 2-oxazolidone compound having the formula:



wherein R is a member of the group consisting of hydrogen, methyl and ethyl, which comprises heating a tris-2-hydroxyalkyl isocyanurate having the formula:



wherein R′ is a hydroxyalkyl radical containing 2 to 4 carbon atoms, at temperature in the range of about 150° to 250° C. and at low pressure, whereby the 2-oxazolidone compound is produced in vapor form, and condensing and recovering said 2-oxazolidone compound.

4. A process for producing 2-oxazolidone which comprises heating tris-2-hydroxyethyl isocyanurate at temperature in the range of about 150° to 250° C. and at low pressure, whereby 2-oxazolidone is produced in vapor form, and condensing and recovering said 2-oxazolidone.

5. A process for producing 2-oxazolidone which comprises heating tris-2-hydroxyethyl isocyanurate at tem-

perature in the range of about 170° to 190° C. and at low pressure, whereby 2-oxazolidone is produced in vapor form, and condensing and recovering said 2-oxazolidone.

6. A process for producing 2-oxazolidone which comprises heating tris-2-hydroxyethyl isocyanurate at temperature in the range of about 170° to 190° C. and at pressure not above about 3 mm. of mercury, whereby 2-oxazolidone is produced in vapor form, and condensing and recovering said 2-oxazolidone.

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