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PREPARATION OF ETHYLENE OXIDE

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The present invention regards an improvement in methods for the preparation of ethylene oxide.

We have found that the reaction between beta-chloroethylacetate and an alkali metal hydroxide produces either ethylene oxide or glycol, or both in varying proportions. The proportions of these two compounds in the product from the foregoing reaction varies widely, depending upon the conditions in which the reaction is carried out. Moreover, the reaction may be very rapid, particularly in the presence of an excess of the hydroxide, and the high reaction velocity makes it very difficult to control the course of the reaction so as to produce a good yield of ethylene oxide. The yield of ethylene oxide is also affected by the concentration of the metal hydroxide.

We have now found that, under certain conditions, the formation of glycol is materially suppressed and the reaction proceeds chiefly to the formation of ethylene oxide. It is, therefore, an object of this invention to provide suitable conditions under which the reaction between beta-chloroethylacetate and a base, such as an alkali metal or alkaline earth metal hydroxide or carbonate, can be carried out to produce a maximum yield of ethylene oxide while avoiding substantially the formation of glycol.

To the accomplishment of the foregoing and related ends, we have now found that the difficulties and uncertainties formerly encountered in producing a good yield of ethylene oxide by the aforesaid reaction are avoided if the base is gradually added to the beta-chloroethylacetate, while vigorously agitating the reaction mixture, and maintaining it at a temperature between about 40° and about 150° C. In this manner we at all times avoid the presence of an excess of unreacted base in the reaction mixture. Then, by such procedure the reaction is made practically independent of the concentration of base employed. Thus, the concentration of the aqueous base may be varied between wide limits, e. g. from as low as 10 per cent thereof up to 70 or 80 per cent by weight, the choice of concentration being largely a matter of convenience and economy. The following examples show the results obtained by carrying out the reaction between beta-chloroethylacetate and an alkali metal or alkaline earth metal base at varying temperatures and with different concentrations thereof.

Example 1

In each run 1.0 mole of beta-chloroethylacetate was placed in a round-bottom flask provided with a stirrer and a reflux condenser. The flask was then heated to and maintained at the various temperatures shown in the following Table I while 2.2 moles of sodium hydroxide in a 20 per cent aqueous solution was gradually added thereto over the periods of time shown. The reaction mixture was vigorously stirred throughout the reaction period. The yield of ethylene oxide is in per cent of theoretical based on the beta-chloroethylacetate reacted, which was in all cases above 95 per cent.

Table I

Run No.	Reaction temperature in ° C.	Time in minutes	Percent yield of ethylene oxide
1	45°	45	54.4
2	60°	60	63.3
3	75°	60	68.3
4	85°	60	69.8
5	100°	90	81.6

Example 2

The following Table II shows the yield of ethylene oxide obtained during a series of runs carried out as above wherein the temperature was held constant at 100° C., but the concentration of sodium hydroxide was varied.

Table II

Run No.	Concentration of sodium hydroxide	Time in minutes	Percent yield of ethylene oxide
6	10	65	64.3
7	20	90	81.6
8	40	60	83.3
9	60	50	83.4

Example 3

We have carried out the preparation of ethylene oxide using 1.1 moles of calcium hydroxide as the base, obtaining a yield of 74.6 per cent of theoretical based on 97.2 per cent of the beta-chloroethylacetate reacted.

Example 4

We have also carried out the preparation of ethylene oxide at a temperature of 100° C., using 1.1 moles of sodium carbonate in a 25 per cent aqueous solution as the base, to obtain a yield of 60.4 per cent of theoretical based on the beta-chloroethylacetate reacted.

It is a feature of our improved method that the presence of a material excess of unreacted base in the reaction mixture is prevented at all times, which condition we have found essential for producing the highest yield of ethylene oxide. In fact, when the foregoing procedure is reversed, that is, when the beta-chloroethylacetate is added to the base, whereby the base is always in excess, the formation of ethylene oxide is largely, if not entirely, suppressed. For example, when beta-chloroethylacetate was gradually added to a 40 per cent aqueous sodium hydroxide solution at a temperature of about 100° C., the yield of ethylene oxide was only 2.7 per cent, the chief product being ethylene glycol.

It is readily apparent from the foregoing description of our invention that our improved method for the preparation of ethylene oxide provides a procedure capable of producing high yields of the desired compound without the formation of large proportions of undesirable products, particularly glycol.

Other modes of applying the principle of our invention may be employed instead of those explained, change being made as regards the process herein disclosed, provided the step or steps stated by any of the following claims or the equivalent of such stated step or steps be employed.

We therefore particularly point out and distinctly claim as our invention:—

1. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with a base, the step which consists in gradually adding the base to the beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature between about 40° and about 150° C.

2. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with a hydroxide of an alkali forming metal, the step which consists in gradually adding the hydroxide to the beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature between about 40° and about 150° C.

3. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with an alkali metal hydroxide, the step which consists in gradually adding the hydroxide to the beta-chloroethylacetate, in the molar ratio of about two moles of hydroxide per mole of beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature between about 40° and about 150° C.

4. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with an alkali earth metal hydroxide, the step which consists in gradually adding the hydroxide to the beta-chloroethylacetate, in the molar ratio of about one mole of hydroxide per mole of beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature between about 40° and about 150° C.

5. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with an alkali metal hydroxide, the step which consists in gradually adding the hydroxide to the beta-chloroethylacetate, in the molar ratio of about two moles of hydroxide per mole of beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature of about 100° C.

6. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with sodium hydroxide, the step which consists in gradually adding the hydroxide to the beta-chloroethylacetate, in the molar ratio of about two moles of hydroxide per mole of beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature between about 40° and 150° C.

7. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with sodium hydroxide, the step which consists in gradually adding the hydroxide to the beta-chloroethylacetate, in the molar ratio of about two moles of hydroxide per mole of beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature of about 100° C.

8. In carrying out the preparation of ethylene oxide from beta-chloroethylacetate by reaction thereof with an aqueous solution of a base, the step which consists in gradually adding the aqueous solution of the base to the beta-chloroethylacetate, while agitating the reaction mixture and maintaining it at a temperature between about 40° and about 150° C.

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