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PROCESS FOR MAKING ALDOL

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There are known several processes for making aldol from acetaldehyde based on the knowledge that acetaldehyde in an alkaline medium is converted into aldol. With these processes either a strong alkali (e.g. sodium hydroxide) or some salt or compound of weaker alkalinity (sodium carbonate, strontium oxide, calcium oxide) have been used. The use of a strong alkali has, however, the disadvantage that the reaction starts quickly, the acetaldehyde easily and suddenly boils, during which a great part of it evaporises, while another part is converted into a thick, even hard aldol resin (poly-aldol) which is no more apt for further synthesis. Even if the sodium hydroxide is added with the greatest care and only with a very small surplus, even then we shall obtain a relatively great quantity of such poly-aldol which is of no use for further synthesis. If a weakly alkaline medium is used conversion will be very slow, but even then it may happen that the aldol formed at the start of the reaction polymerises into poly-aldol before the remaining acetaldehyde is converted in the course of the reaction.

Two problems must therefore be solved for making good quality aldol. On the one hand, we had by slowing down the reaction and by conducting it cautiously to prevent the boiling of the acetaldehyde before time on the other hand it had to be prevented to let the acetaldehyde once converted into monoaldol polymerise.

We have found that both the regulation of the course of the reaction and the prevention of the polymerisation of the aldol can be attained if we maintain in the weakly alkaline medium used for making the aldol a practically constant hydrogene ion concentration by obtaining alkalisation through a suitable buffer solution. An example of such a buffer solution is a mixture of sodium tetraborate and sodium hydroxide the pH of which is 11–12. The actual hydrogene ion concentration of the acetaldehyde made alkaline with this solution is much more constant than if either sodium hydroxide or some other weak alkali is used.

We can still more efficiently realise the regulation of the course of the reaction according to the invention and the prevention of the polymerisation of the aldol, if we add to the reaction mixture during the making of the aldol according to the above process according to our Application No. . . . . polyvalent phenoles, e. g. hydroquinon.

An example of the process is given as follows:

1 kg of possibly fresh acetaldehyde, distilling at 21–24 C° is cooled down to 0° and then carefully made neutral with a solution of n/2 or n/5 sodium hydroxide by using a phenolphtalein indicator. Care must be taken that during this procedure temperature should not increase above 10 C° and the addition of sodium hydroxide is stopped after the solution has kept its red colour for 4–5 minutes. Thereafter we slowly drop in during stirring or shaking the buffer solution which consists in a mixture of 50 cm^3 of n/10 sodium tetraborate and of 10 cm^3 sodium hydroxide. After having dropped in the buffer solution 10–15 gr of hydroquinon are added and exchanging continuously the outer cooling water for an always warmer one, we slowly heat the reaction mixture to 40 C°. If we proceed carefully we shall attain the 40 C° without the solution boiling at all. About 1½ to 2 hours are required for the operations described and for 7–8 hours from the end of these operations the mixture is kept at 40 C°. During this time neither stirring nor shaking are needed so that the maintenance of the inner temperature of 40 C° requires small supervision. After 9 hours from the start of the reaction the solution is neutralised by using n/2 or n/5 HCL in the presence of a BTB (brome thymol blue) indicator to a green colour and after one or two days standing it is distilled in vacuum.

Vacuum distillation is made under a slow, successive increasing of the temperature and successive decreasing of the pressure as in this way we shall obtain from the 1 kg of acetaldehyde mentioned as example—together with the aldol convertible from the pre-distillate according to the above process—about 700–750 gr of such mono-aldol the boiling point of which at a pressure of 2–3 mm is between 75–84 C° and which in further synthesis will behave according to the formula CH_3 CHOH CH_2—CHO.

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