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PROCESS FOR SEPARATION OF ACRYLIC ACID NITRILE

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Specification No. 343,269 describes a process of preparing acrylic acid nitrile which comprises bringing acetylene together with hydro-cyanic acid into contact with a suitable catalyst. This process is based on the perception that catalysts for bringing about those additionary reactions of acetylene which result in vinyl derivatives thereof also effect the addition of hydro-cyanic acids on acetylene. The process consists in bringing acetylene together with hydrocyanic acid into contact with an acid reacting solution of cuprous chloride as a catalyst.

In this process acetaldehyde, monovinylacetylene, divinylacetylene, and higher boiling compounds are obtained as by-products which leave the reaction vessel together with the acrylic acid nitrile, the excess acetylene, the nonconverted hydrocyanic acid, and water vapours. The separation of the acrylic acid nitrile may be accomplished by condensing it together with the by-products—with the exception of acetylene which is recycled—and fractionally distilling. The separation of acrylic acid nitrile from monovinyl acetylene, acetaldehyd, and hydrocyanic acid causes no difficulties and with respect to those by-products a fractionation would be operable. Difficulties however arise from the divinyl acetylene due to the similar boiling points of this substance and acrylic acid nitrile and practically speaking separation from divinyl acetylene is impossible by a distilling operation. A further difficulty consists in the sensitiveness of divinyl acetylene against heat and therefore a heating process must be avoided.

Now we have found that the acrylic acid nitrile may be separated in a simple manner from the reaction mixture obtained according to the process of specification 343,269. Our process consists in a washing operation of the reaction mixture with a suited solvent especially water.

The gas mixture leaving the reaction vessel is continuously washed in a washing column preferably under atmospheric pressure and at low temperatures (0–20°C). Only small quantities of acetylene and monovinyl acetylene but almost the total quantity of acetaldehyd, hydrocyanic acid and acrylic acid nitrile dissolve in the washing medium. By using for example for the washing of 125 parts by volume of gas mixture, 1 part by volume of water of a temperature of 15–20°, 96% of the acrylic acid nitrile are absorbed, the water containing only 0,4% of dissolved acetylene and traces of monovinylacetylene. Divinyl acetylene is not dissolved in the washing water. The loss of acetylene which takes place in the washing process, is therefore very low, what is especially to be pointed out. By increasing the ratio gas mixture to washing water it is possible to wash out the acrylic acid nitrile in a still more complete way but then the quantity of the dissolved acetylene is also increased. Generally speaking all conditions must be chosen in such a way that a high yield of dissolved acrylic acid nitrile corresponds to a small loss of acetylene only. If necessary however it is possible to recover and recycle the acetylene dissolved in the washing water.

The aqueous solution obtained is distilled in order to get the acrylic acid nitrile.

The acetylene which leaves the washing column contains mono- and divinyl acetylene. It may be freed from these products by low cooling and recycled.

Example

Through 2,5 liters of a catalyzer composed of 300 parts of cuprous chloride, 100 parts of ammonium chloride, 5 parts of concentrated hydrochloride acid, 10 parts of copper powder and 400 parts of water, 0,25 liters of acetylene together with 26 g of hydrocyanic acid per hour are passed in at a temperature of 80° C. The escaping gas mixture is washed in a usual washing column filled with Raschig-rings with 2 liters of water per hour. The aqueous solution leaving the column is continuously distilled. An azeotropic mixture consisting of acrylic acid nitrile and water passes over which is somewhat contaminated with hydrocyanic acid and acetaldehyd. The raw acrylic acid nitrile which forms an oily layer is separated, dried, for example with calcium chloride and then fractionated. After a small fraction at a low temperature which consists chiefly of acetaldehyd and hydrocyanic acid the pure acrylic acid nitrile distills at 76–78°C (760 mm).

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