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CARBOXYLIC ACID NITRILES

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It has been found that we can produce the nitriles of carboxylic acids in a simple way by treating carboxylic acid amides with trichloromethylchloroformate. The conversion is effected by warming the carboxylic acid amides preferably in the presence of a suitable volatile solvent or diluent such as benzol, toluol, benzine, chlorohydrocarbons, ethers, etc. together with the chloro-formiate of the trichloro-methyl alcohol. It is generally sufficient to use 1 mol of the formic acid ester for 2 mols of monocarboxylic acid amide. However, we may likewise use an excess of the ester.

The conversion is accompanied by an evolution of gas and it is continued until this evolution of gas practically stops. At first, in applying this method, we work at low temperatures, e. g. between 20 and 45° C. and finally we continue the heating for a while on a reflux condenser to finish the conversion.

We use as raw materials in our process mono- or polycarboxylic acid amides of the aliphatic, cycloaliphatic or aromatic series or their mixtures. They may contain straight-chained or branched-chained hydrocarbon residues. E. g. we may use aceticacid amide, butyric acid amide, capric acid amide, lauric acid amide, oleic acid amide, stearic acid amide, montanic acid amide, naphthenic acid amides, resinic acid amides, adipic acid diamide, benzamide and its substitution products, phthalic acid diamide, anthraquinone-2-carboxylic acid amide, tetrahydrophthalic acid diamide, hexahydrophthalic acid diamide, cyclohexane-diacetic acid diamide and the like. The hydrocarbon residues of these carboxylic acid amides may also contain hetero atoms or hetero atom groups such as oxygen, sulfur, nitrogen or halogen as well as ester groups and other acid amide groups. Mixed carboxylic acid amides may be converted with trichloromethylchloroformate also.

The carboxylic acid nitriles obtained according to the present method are valuable initial products for chemical conversions. For example, some may be used in the preparation of capillary active compounds, while others in the aromatic series—are valuable intermediate products for the production of dyestuffs.

The following examples serve to illustrate the described method for the manufacture of carboxylic acid nitriles, but they are not intended to limit it thereto; the parts being by weight:

Example 1

Two hundred and eighty-three parts of stearic acid amide (technically pure) and 110 parts of chloro-formiate of the trichloro-methyl alcohol are warmed in the presence of 1000 parts of benzene up to a temperature of 25–40° C., whereat an evolution of gas will be observed. The conversion is completed while slowly raising the temperature and finally by a short boiling on the reflux condenser. Now the solvent is distilled off after entirely expelling the developed hydrochloric acid gas and the residue to be free of chlorine is then distilled under reduced pressure. We thus obtain the stearic acid nitrile (boiling point 150 to 156° C. at 1.5 mm. pressure of mercury) with a yield of 95%.

In a similar way we obtain the benzonitrile from benzamide and the 1,2-dicyanobenzol from phthalic acid-diamide.

Example 2

To a suspension consisting of 36 parts of adipic acid amide in 200 parts of xylol, 55 parts of chloro-formic acid-trichloro-methyl ester are added. While warming up to 40–60° C. the evolution of gas will set in. The temperature is now slowly raised and regulated in proportion to the amount of gas evolved. By boiling on the reflux condenser the reaction is completed. When the solvent is distilled off the residue resulting at the distillation under reduced pressure is the adipic acid-dinitrile in the form of a colourless oil. Its boiling point is 150° C. at a mercury pressure of 11 mm.

What we claim is:

1. The process of producing a nitrile of a carboxylic acid comprising reacting a carboxylic acid amide with the chloro-formiate of trichloromethyl alcohol.

2. The process of producing a nitrile of a carboxylic acid comprising reacting a carboxylic acid amide with trichloromethylchloroformate in the presence of a volatile liquid.

3. The process of producing a nitrile of a carboxylic acid comprising reacting a dicarboxylic acid amide with trichloromethylchloroformate.

4. The process of producing a nitrile of a carboxylic acid which comprises heating a carboxylic acid amide with trichloromethylchloroformate until an evolution of gas has substantially ceased.

5. The process of producing a nitrile of a carboxylic acid which comprises reacting an aliphatic carboxylic acid amide with trichloromethylchloroformate.

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