Patented Apr. 10, 1951

UNITED STATES PATENT OFFICE

2,548,369

2,548,369

TREATING FATTY ACID NITRILES TO IMPROVE THE ODOR THEREOF

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No Drawing. Application December 10, 1948, Serial No. 64,694

4 Claims. (Cl. 202—57)

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This invention relates to a process for treating high molecular weight fatty acid nitriles to remove objectionable odors.

The high molecular weight fatty acid nitriles having 12 or more carbon atoms, which are commercially produced by the reaction of fatty acids and ammonia, are known to possess a characteristic odor which is quite pungent. This odor is found only in connection with these nitrile products and its presence has always indicated the presence of either the fatty acid nitriles or derivatives thereof. The odor usually carries through at least to some extent to the fatty acids, amines, amides or other derivatives of the nitriles.

The fatty acid nitriles and their derivatives are used industrially for many and varied purposes. For example, they are used as plasticizers, as emulsifiers, and as modifiers in many different processes. Their usefulness for such purposes is many times restricted because of their characteristic pungent odor. It would be a great advantage to be able to prepare nitriles which have no such odor.

Although it has heretofore been suggested that the odor of the fatty acid nitriles may be somewhat improved by washing with certain acid agents, such as hydrochloric or sulfuric acid, none of the suggested agents has been effective in bringing about a complete removal of the objectionable odor, and even after treatment with these agents, the nitriles remain unsuitable for many of the above uses.

We have now discovered that a nitrile product can be prepared which has none of the usual nitrile odor by subjecting the odoriferous nitriles to treatment with phosphoric acid so as to form a precipitate. In carrying out our process we contact the odoriferious nitriles with phosphoric acid in any convenient way. We may simply place the nitriles in a vat or tank and stir in a quantity of phosphoric acid. The acid acts to form a flocculate which if left to settle will form in a bottom layer. The main body of nitriles may then be decanted or drawn off to physically separate them from the precipitate layer which is discarded.

Not only does our discoverey make possible the preparation of fatty acid nitriles of improved odor, but we find that the improvement carries through to the nitrile derivative products such as amines, amides, quaternary ammonium compounds, etc. so that these derivative products need no longer contain the certain objectionable odor which has heretofore been identified with these compounds as nitrile derivatives.

Our process is applicable to all the high molecular weight fatty acid nitriles containing 12 or more carbon atoms. Examples are lauric acid nitrile, palmitonitrile, oleonitrile and stearonitrile. The high molecular weight acids from which such nitriles are prepared are commercially obtained principally by hydrolysis of natural oils or fats such as lard, tallow, soya bean oil, cottonseed oil, coconut oil, etc., and nitriles prepared from the fatty mixtures obtained through hydrolysis of such oils are greatly benefited by treatment according to our process.

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Although the phosphoric acid may be added at full strength, we prefer in most cases to add it in more or less dilute form. For example, we have found that an 85% solution of phosphoric acid in water works very satisfactorily when added to a quantity of odoriferous nitriles.

The amount of the acid solution to be added varies, depending on the concentration of the acid, the type of nitrile being treated, etc. A preferred amount of an 85% phosphoric acid solution to be added is from 1 to 5% by weight of the amount of nitriles present. More phosphoric acid than the 5% above referred to may be used, but without substantial additional benefit.

After the phosphoric acid has been added we prefer to apply heat and agitate to produce good dispersion and hasten the effect of the acid on the nitriles. We may heat the mixture to from 25° C. to 100° C. for from 2 to 6 hours, the higher temperature going with the shorter periods of time. At the end of this treatment the mixture may be allowed to settle and the precipitate separated as already explained.

Any suitable means of separating the precipitate from the nitriles may be employed. Suitably, separation may be accomplished without waiting for stratification by the use of a centrifuge; or a substantial removal of the objectionable nitrile odor may be effected by simply adding phosphoric acid to the nitriles and then distilling the mixture.

Specific examples of the practice of our invention are given as follows:

Example 1

100 parts of crude tallow nitriles were treated with 5 parts of 85% phosphoric acid. The mixture was stirred and heated at 90° C. for 6 hours. Upon standing a black sludge separated and was removed. The nitriles was washed with water to remove traces of phosphoric acid and was found to be free of objectionable color.

Example 2

100 parts of crude coconut nitriles were treated with 5 parts of 85% phosphoric acid and the mixture was heated at 60° C. to 70° C. and stirred for 4 hours. The black sludge which formed was separated and the nitriles washed with water to remove traces of phosphoric acid. The resulting nitrile product was found to be free of objectionable odor.

Example 3

100 parts of crude cottonseed nitriles were treated with 5 parts of 85% phosphoric acid. The mixture was stirred at 25° to 30° C. for 6 hours. The black sludge which formed was separated and the material was washed with water as in Examples 1 and 2. The product was free of any objectionable odor.

Example 4

To 100 parts of crude tallow nitrile were added 5 parts of 85% phosphoric acid. The resulting mixture was distilled at a pressure of 1 mm., the temperature reaching a maximum of 160° C. 95 parts of distilled nitrile were obtained which were found to be substantially free of objectionable odor.

In any process which involves improving the odor of a substance, the effectiveness of the process must be tested by smelling the final product, and, since the sensitivity of the olfactory lobes is quite apt to vary from person to person, we have devised an additional test for the effectiveness of our process. This test, which is described and explained below, is much more sensitive than merely smelling the final nitrile product and involves smelling the concentrated odor producing substance after it has been removed from the nitrile.

When, in our process, odoriferous nitriles are treated with phosphoric acid, the latter combines with the odor-producing substance to form a complex which precipitates and is separated as a sludge. In our additional test, we then hydrolyze or split this complex by treating with NaOH. This frees the odor-forming part of the complex, and the characteristic undesirable odor immediately becomes noticeable. Thus, if any odor-producing is removed from the nitrile, its presence in the sludge can readily be detected.

The utility of this additional test may be illustrated by the following examples:

Example 5

The black sludge (phosphoric acid) layer, which was separated and removed in Example 1, was treated with NaOH. The typpical disagreeable odor usually associated with nitriles immediately became noticeable.

The nitrile layer, which was separated in Example 1, was once again treated with 5% of 85% phosphoric acid. The mixture was stirred and heated at 90° C. for 6 hours. The phosphoric acid layer was then treated with NaOH. No typical disagreeable odor was given off, thus indicating that the first treatment with phosphoric acid had removed the odor-producing substance completely.

Example 6

150 g. tallow nitriles were treated with 5% of 5% HCl. The mixture was stirred and heated at 90° C. for 6 hours. The HCl layer was separated and treated with NaOH. A slight odor was given off.

The nitrile layer was washed to remove HCl and was then treated with 5% of 85% phosphoric acid. The mixture was stirred and heated at 90° C. for 6 hours. The phosphoric acid layer was then separated and treated with NaOH. A typical disagreeable odor was immediately given off, indicating that all the odor-producing substance had not been removed from the nitriles by the HCl treatment.

Example 7

150 g. of tallow nitriles were treated with 5% of 5% H_2SO_4. The mixture was stirred and heated at 90° C. for 6 hours. The H_2SO_4 layer was treated with NaOH. A slight odor was given off.

The nitrile layer was washed to remove H_2SO_4 and was then treated with 5% of 85% phosphoric acid. The mixture was stirred and heated at 90° C. for 6 hours. The phosphoric acid layer was then separated and treated with NaOH. A typical disagreeable odor was immediately given off, thus indicating that all the odor-producing substance had not been removed from the nitriles by the H_2SO_4 treatment.

The foregoing detailed description and examples have been given for explanation only and it is to be expected that the methods and procedures above given may be widely varied, all within the spirit of our invention.

This is a continuation in part of our application Serial No. 731,862, filed March 1, 1947, now abandoned.

We claim:

1. A process for treating fatty acid nitriles containing at least 12 carbon atoms to remove objectionable odor therefrom, comprising admixing with said nitriles phosphoric acid, heating the mixture to between 25° and 100° C. for a period of from 2 to 6 hours, and separating from said nitriles a precipitate formed through the action of said mineral acid.

2. A process for treating fatty acid nitriles containing at least 12 carbon atoms to remove objectionable odor, comprising admixing with said nitriles phosphoric acid, heating the mixture to between 25° and 100° C. for a period of from 2 to 6 hours, permitting the resulting precipitate to form in a layer apart from the main body of said nitriles, and separating said layer from the remaining nitriles.

3. In a process for the treatment of nitriles containing at least 12 carbon atoms to remove objectionable odor therefrom, the steps of contacting with said nitriles phosphoric acid in a proportion of about 5% of the nitriles present, heating the mixture to between 25° and 100° C. for a period of from 2 to 6 hours, and distilling to separate said nitriles from phosphoric acid and the product formed as a result of said contact.

4. In a process for the treatment of nitriles containing at least 12 carbon atoms to remove objectionable odor therefrom, the step of distilling said nitriles in the presence of phosphoric acid.

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