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MANUFACTURE OF NITRILES

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1

The present invention relates to processes of making nitriles and particularly nitriles of high molecular weight.

Although not limited thereto, the present invention will be particularly described in its application to the manufacture of high molecular weight alkyl nitriles having preferably above 10 carbon atoms and desirably from 12 to 36 carbon atoms.

According to present processes of producing alkyl nitriles, it is frequently necessary to use complicated and expensive equipment or the processes will give poor yields.

It is among the objects of the present invention to prepare high alkyl nitriles with high yield from readily available raw materials, utilizing only the most simple type of equipment.

It is a further object to prepare high alkyl nitriles which are sufficiently pure as prepared for many technical purposes after simple filtration without requiring vacuum distillation.

Still further objects and advantages will appear in the more detailed description set forth below, it being understood, however, that this more detailed description is given by way of illustration and explanation only and not by way of limitation, since various changes therein may be made by those skilled in the art without departing from the scope and spirit of the present invention.

A feature of the present invention resides in the combination of more than one mol and less than 3 mols of cyanuric acid with one mol of a high boiling fatty acid at above 250° C. for less than 3 hours, to give substantially a 100% yield of a substantially pure fatty nitrile substantially free of amides, which only requires filtration for technical purposes.

The molar ratio should be above 1 mol and preferably not be above 2½ mols of cyanuric acid per mol of fatty acid and desirably the molar ratio is between 1 and 2 mols of the cyanuric acid to 1 mol of fatty acid.

Instead of using cyanuric acid, it is possible to use urea, which may be decomposed by rapid thermal decomposition into cyanuric acid in the presence of the fatty acid.

When urea is used, the reaction mixture should be stirred vigorously until the conversion to cyanuric acid is complete.

The molar proportion of the urea to the fatty acid should be preferably above 1 mol of urea and not above 3 mols of urea per mol of fatty acid. Larger quantities of urea are not preferred as this results in waste of urea and lower yields due to the nitrile being adsorbed by the excess cyanuric acid.

If desired, mixtures of urea and cyanuric acid may be employed. For example, mixtures containing 40% to 80% of cyanuric acid may readily be employed.

2

Besides saturated fatty acids having a boiling point over 250° C., unsaturated fatty acids of high molecular weight may be employed with production of light colored nitriles with substantially 100% yield. The preferred fatty acids are lauric, oleic, stearic, elaidic and palmitic, or mixtures of these various acids produced by hydrolysis of natural tri-glycerides, such as from tallow, cocoanut oil, palm oil, corn oil, peanut oil, rape seed oil and low grade olive oil.

Higher unsaturated fatty acids, such as drying oil fatty acids or hydroxylated fatty acids are not preferred, these acids being, for example, linoleic, linolenic, ricinoleic and eleostearic, since they tend to form polymerization products. However, if polymerized nitriles are desired, these more highly unsaturated acids may be employed.

Fatty acids, below lauric—say from 8 to 12 carbon atoms—which boil below 250° C., may be processed under pressure in an autoclave.

The reaction will usually be completed in about 3 hours or less at above 250° C. Higher temperatures in the range of 280° C. to 310° C. will shorten the reaction time. At temperatures below 250° C. the reaction not only takes much longer, but results in less desirable products, as the preferred conditions are departed from, without completion of the desired reaction.

After the reaction is completed, the reaction mixture may be directly filtered or given a short treatment with a decolorizing material, such as kieselguhr, diatomaceous earth, or the adsorbent aluminum silicate known as Filtrol. Generally, it is preferred to cool the reaction mixture to 125° C. to 175° C. before adding the filtering material.

The resulting nitriles, and particularly stearic, oleic, palmitic and lauric nitriles, are obtained in substantially 100% yield and of light color immediately useful for many technical purposes. No vacuum distillation is required.

Examples

1. 10 grams by weight of technical stearic acid (Hydrofol 150) and 5 grams of cyanuric acid (Eastman Kodak Co.) were refluxed at 280° C. to 300° C. for 3 hours. The excess cyanuric acid was removed by filtration. A light brown material was obtained, which had a low acid number, and was shown by analysis to be substantially stearonitrile.

2. 500 grams by weight of cocoanut fatty acids and 250 grams of urea were refluxed at 280° C. to 300° C. for 3 hours. The product was substantially a mixture of the corresponding fatty nitriles.

3. 500 grams by weight of a light yellow oleic acid and 250 grams of urea were heated at 285°

C. to 300° C. for 3 hours. The excess cyanuric acid was removed by filtration and a light orange oleonitrile obtained.

4. 500 grams by weight of stearic acid and 180 grams by weight of urea were heated at 280° C. to 290° C. for 4 hours. The mixture was then cooled to 150° C. and stirred with 25 grams of Filtrol for 20 minutes, and then filtered. After it had cooled to room temperature, the nitrile gradually crystallized to a light tan solid, which was substantially pure stearonitrile.

5. 15 lbs. of stearic acid and 7½ lbs. of urea were heated rapidly. The mixture was stirred until the molten urea was converted into solid cyanuric acid and stirring was then discontinued. After 2 hours at 290° C. the reaction mass was permitted to cool and was filtered to obtain stearonitrile.

In the above examples, in using oleic acid, it is possible to use crude or purified mixtures such as olive oil fatty acids, red oil or elaidic acid, as well as C. P. oleic acid.

In using lauric acid, it is possible to use either C. P. lauric acid or the crude mixture known as cocoanut oil fatty acids.

As many changes could be made in the above nitrile and methods of making the same, and many widely varying embodiments of this invention could be made without departing from the scope of the claims, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating cyanuric acid at above 250° C. and below approximately 310° C. with a fatty acid in the ratio of approximately 1–2 mols of cyanuric acid per mol of fatty acid.

2. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating cyanuric acid with a fatty acid having more than 10 carbon atoms at a temperature above approximately 250° C., and below approximately 310° C., in the ratio of approximately 1–2 mols of cyanuric acid per mol of fatty acid.

3. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating cyanuric acid at 250° C. to 310° C. with a fatty acid having 12 to 36 carbon atoms in the ratio of approximately 1–2 mols of cyanuric acid per mol of fatty acid.

4. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating 100 parts by weight of cyanuric acid with 200 to 300 parts by weight of a fatty acid having 12 to 18 carbon atoms at a temperature above approximately 250° C. and below approximately 310° C.

5. A process of making oleonitrile in substantially 100 per cent yield, which comprises heating oleic acid with cyanuric acid at above 250° C. and below approximately 310° C. in the ratio of 1–2 mols of cyanuric acid per mol of oleic acid.

6. A process of making stearonitrile in substantially 100 per cent yield, which comprises heating stearic acid with cyanuric acid at above 250° C. and below approximately 310° C. in the ratio of approximately 1–2 mols of cyanuric acid per mol of stearic acid.

7. A process of making cocoanut fatty acid nitriles in substantially 100 per cent yield, which comprises heating cocoanut fatty acids with

cyanuric acid at above 250° C. and below approximately 310° C. in the ratio of approximately 1–2 mols of cyanuric acid per mol of coconut fatty acid.

8. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating cyanuric acid at above 250° C. and below approximately 310° C. with a fatty acid in the ratio of approximately 1–2 mols of cyanuric acid per mol of fatty acid, cooling, adding a filter aid and filtering.

9. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating cyanuric acid at above 250° C. and below approximately 310° C. with a fatty acid in the ratio of approximately 1–2 mols of cyanuric acid per mol of fatty acid for about 2 to 3 hours.

10. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating urea at above 250° C. and below approximately 310° C. with a fatty acid in the ratio of approximately 1–2 mols of urea per mol of fatty acid.

11. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating urea with a fatty acid having more than 10 carbon atoms at a temperature above approximately 250° C. and below approximately 310° C. in the ratio of approximately 1–2 mols of urea per mol of fatty acid.

12. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating urea at 250° C. to 310° C. with a fatty acid having 12 to 36 carbon atoms in the ratio of approximately 1–2 mols of urea per mol of fatty acid.

13. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating 100 parts by weight of urea with 200 to 300 parts by weight of a fatty acid having 12 to 18 carbon atoms at a temperature above approximately 250° C. and below approximately 310° C.

14. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating urea at above 250° C. and below approximately 310° C. with a fatty acid in the ratio of approximately 1–2 mols of urea per mol of fatty acid, cooling, adding a filter aid and filtering.

15. A process of making high molecular weight fatty nitriles in substantially 100 per cent yield, which comprises heating urea at above 250° C. and below approximately 310° C. with a fatty acid in the ratio of approximately 1–2 mols of urea per mol of fatty acid for about 2 to 3 hours.

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