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PROCESS FOR ISOLATING STEROLS

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This invention relates to the isolation of sterols which are found in such plants as soybean, cottonseed, corn and sugar cane.

The sterol-bearing oils, juices or saps are extracted from the plant. Conventionally, in the past these materials have been saponified with alkali and the unsaponifiable portion, which contains the sterols, is extracted from the soaps. In extracting the sterols from the soaps, the conventional method has been to dissolve the soap in alcohol, or to hydrolyze the oil with alcoholic alkali and to remove the sterols from the resulting soap solution with water-immiscible solvents such as ether, benzene, carbon tetrachloride, etc. However, an unsatisfactory condition exists in the fact that these reagents extract not only the sterols but most of the other unsaponifiable material. It is then necessary to separate the sterols from the undesirable, gummy unsaponifiable material which interferes with the crystallization of the sterols in the extract. As a remedy to this problem it has been suggested in the past that certain solvents, such as ethyl ether, isopropyl ether, and methyl ethyl ketone, be used to extract the sterols since the theory has been that such solvents extract substantially only the sterols and leave behind much of the objectionable, gummy unsaponifiable material.

Although this method is an improvement, none of these solvents have proved satisfactory in that they are not sufficiently selective, i. e., a substantial quantity of the undesirable unsaponifiable material is nevertheless extracted with the sterols and precipitates out along with the sterols. In an effort to eliminate these impurities, such teachings as those disclosed in U. S. Patents No. 2,273,045 and No. 2,273,046 recommend the use of a secondary or auxiliary solvent, glacial acetic acid. The extract is taken up by this solvent and the sterols caused to crystallize therefrom. This solvent, however, places the entire extract, the objectionable, gummy portion, as well as the sterols, in solution, thus materially damaging the recovery, both in quantity and purity. Furthermore, it is expedient following recovery, as the patents illustrate, that the sterol crystals be washed, conventionally with an alcohol to free the sterols from gummy material.

Now, in accordance with my invention, I have developed a process for extracting or isolating sterols, which novel process employs a specific solvent, the effect of which renders results heretofore unattained in the prior art in that only the sterol content of the unsaponifiable portion is placed in solution and no subsequent washing of

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the crystalline sterols is required. Furthermore, the sterol yields have proved to be quantitatively increased and yet purer in content.

This process for isolating the sterols from the unsaponifiable portion of plant extracts comprises combining methyl cyanide and a concentrate containing the sterols and unsaponifiable impurities and heating the mixture to form a hot methyl cyanide-sterol solution. The sterol-methyl cyanide solution which is thus formed is then separated from the insolube unsaponifiable impurities, the solution cooled to crystallize the sterols and the crystallized sterols removed from the methyl cyanide.

Now, having indicated in a general way the nature and purpose of this invention, the following examples will illustrate the invention. It is to be understood, however, that such examples are presented merely as illustrative of the invention and are not to be construed as limiting the same. In the examples the ingredients are given in parts by weight unless otherwise indicated.

Example 1

Five hundred grams of sugar cane oil was hydrolyzed and a yield of 23% of unsaponifiable extract containing sterols was rendered.

Ninety-two grams of this unsaponifiable fraction was admixed with one liter of methyl cyanide. The mixture was heated to a temperature of approximately the boiling point of the methyl cyanide or 82° C. This temperature was maintained for 10 to 15 minutes at which time it was visually evident that the insolube portion of the unsaponifiables, which comprises the undesirable gummy material, formed a layer at the bottom of the vessel whereas the sterols and methyl cyanide resolved in a clear solution. The sterol-methyl cyanide solution was then decanted off before the solution had an opportunity to substantially cool. The solution was then allowed to cool which resulted in the formation of white sterol crystals. When filtered from the solvent, the yield of sterols was approximately 16% by weight of the original sugar cane unsaponifiable fraction. The process was repeated and a yield of 26 grams, or approximately 24%, was realized.

Example 2

Four hundred grams of soya bean oil was hydrolyzed and the unsaponifiable portion extracted therefrom with ether. The extract which, after removal of the ether, weighed approximately 11 grams was treated in substantially the same manner as the unsaponifiable sugar cane portion

of Example 1. The soya bean sterol crystals were filtered off and displayed a melting point of 133–136° C. The yield of sterols was 2.25 grams or approximately 20% by weight of the total unsaponifiable portion.

Example 3

Four hundred grams of corn oil soap stock was hydrolyzed and the unsaponifiable fraction, which amounted to 12 grams, was extracted with ether. This fraction was then treated with methyl cyanide in substantially the same manner as the unsaponifiable portion of the sugar cane oil in Example. 1. A yield of 2.1 grams or approximately 17% of sterol crystals was rendered. These sterols had a melting point of 127–132° C.

It will be noted that methyl cyanide is the only solvent employed in the extraction of the sterol constituent from the entire unsaponifiable portion. I have experimented at length with solvents which are closely related to the methyl cyanide, such as propionitrile, acrylonitrile, and methacrylonitrile, and I have found that they are unsatisfactory in that a substantial portion of the undesirable gummy material, left as insoluble when employing methyl cyanide, is placed in solution by these solvents. Methyl cyanide, however, has proved to have rare selective solvent properties effecting only the sterol ingredient of the unsaponifiables and substantially little of the undesirable material.

As taught in Example 1, the extraction process may be repeated where the ultimate in purity and quantity is demanded. However, where reprocessing proves expensive, it should be appreciated that the singular performance of the extraction steps provides results in and of itself which are most satisfactory.

Most effective in purification of sterol crystals is the recrystallization of the extracted sterols in the hot methyl cyanide. However, this additional step is not required. Methyl cyanide, which is isolated from the sterol crystals either by filtering or centrifuging, may be re-used several times. This fact is additional evidence of its highly selective solvent power.

It should be appreciated that the temperatures employed in the process illustrated in the examples may be varied. However, it is necessary that the methyl cyanide-unsaponifiable material mixture be heated to a temperature of approximately the boiling point of the methyl cyanide or to a point where the sterols will dissolve in the methyl cyanide. It is also required, to obtain the best separation of the sterols, that the decanting or separating of the sterol-methyl cyanide solution from the undesirable insoluble portion be conducted at a temperature high enough to disallow the throwing off or crystallization of the sterols. This minimum temperature is preferably approximately 10° C. less than the boiling point of methyl cyanide.

The invention is not limited to the specific percentages of extracts rendered in the examples as free from other unsaponifiables. Furthermore, other modes applying the principles of the invention may be employed instead of those specifically set forth in the examples, changes being made as regards the method herein disclosed, providing the step or steps stated by any of the following claims or the equivalent of such stated step or steps be employed.

I claim:

1. A process for isolating sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to form a hot sterol-methyl cyanide solution, separating the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and removing said crystallized sterols from the methyl cyanide.

2. A process for isolating sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to form a hot stero-methyl cyanide solution, decanting the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and removing said crystallized sterols from the methyl cyanide.

3. A process for isolating sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to form a hot sterol-methyl cyanide solution, separating the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and filtering said crystallized sterols from the methyl cyanide.

4. A process for isolating sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to form a hot sterol-methyl cyanide solution, decanting the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and filtering said crystallized sterols from the methyl cyanide.

5. A process for isolating sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to a temperature of approximately 82° C., separating the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and removing said crystallized sterols from the methyl cyanide.

6. A process for isolating sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to a temperature of approximately 82° C., decanting the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and filtering said crystallized sterols from the methyl cyanide.

7. A process for isolating sugar cane sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to form a hot sterol-methyl cyanide solution, decanting the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and filtering said crystallized sterols from the methyl cyanide.

8. A process for isolating sugar cane sterols comprising combining methyl cyanide and a concentrate containing sterols and unsaponifiable impurities, heating the mixture to a temperature of approximately 82° C., decanting the sterol-methyl cyanide solution thus formed from the insoluble unsaponifiable impurities, cooling said solution to crystallize the sterols, and filtering said crystallized sterols from the methyl cyanide.

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No references cited.