

ALIEN PROPERTY CUSTODIAN

PROCESS FOR EXTRACTING DURABLE CONCENTRATES OF FAT-SOLUBLE VITAMINES FREE FROM FATTY ACID

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The subject matter of this invention is a process for extracting durable concentrates of fat-soluble vitamins free from fatty acid, e. g. from substances of vitamin content which owing to their high percentage of free fatty-acid, chromatogenous matter, unpleasant odour and flavour, also other impurities are unserviceable for the vitaminisation of foodstuffs, pharmaceutical preparations and for direct internal therapeutic injection.

In the extraction of fat-soluble concentrated vitamins from natural fats containing vitamins or from adipose substances by means of the usual concentration methods of distillation (molecular distillation amongst others), perforation with solvents and chromatography of the neutral fat portion, not only an over concentration of the active material (vitamins) takes place, but furthermore a reduction in the content of undesirable chromatogenous, odiferous and flavouring matter, and above all the free fatty-acids concomitant with natural fats results.

It is also known to refine fats with ethyl or methyl alcohol respectively, or to refine them by dissolving same in solvents, e. g. benzene, and then neutralising them with ammoniacal alcohol. All these processes aim however, only the total refining of raw fat with the object extracting a practically neutral edible fat as regards appearance, flavour and odour, whilst the vitamin content of the final product is disregarded.

The process under discussion on the other hand concerns itself with the refinement of fat-soluble vitamin concentrates.

Conforming with the invention durable, acid-free fat-soluble vitamin concentrates, incontestable chromatically, adiferously and in flavour can be acquired, when vitamin concentrates, as for example obtained through the process of extraction from vitaminised and stearinated fats, or other similar substances obtained by means of monovalent alcohol, are dissolved in an organic solvent, subsequently extracted with diluted monovalent alcohol, e. g. through shaking or perforation.

The hypothesis is that both solvents do not, or to a minimum degree mix in the presence of vitamin concentrates to be refined. As organic solvents all fat-dissolvents come into consideration having little or no solutiveness for diluted monovalent alcohol, e. g. benzene or carbon tetrachloride, and monovalent alcohols which have been approved in practice are methyl alcohol and isopropyl alcohol. It is immaterial accord-

ing to which method the two solvents are allowed to influence or permeate, since all methods of fluid extraction such as shaking, perforation in suitable apparatus amongst others, have the same aim.

After exhaustive extraction of the acid vitamin concentrates with diluted monovalent alcohol, after severance of both phases and after separate processing by means of distillation one retains a fatty-acid-free concentrate of fat-soluble vitamins having good odour and flavour, and an alcohol extraction residue of the collective free fatty-acids, a large proportion of the chromatogenous, flavour and odoriferous substances besides small quantities of fat-soluble vitamins, which can be greater or lesser according to the concentration of the monovalent alcohols.

Examples

(1) 100 kgs cod liver oil with an acid value 2, a vitamin-A-content of 2000 I. E. per gramme and a vitamin-D-content of 300 I. E. per gramme are exhaustively extracted with 20 kgs of concentrated anhydrous methyl alcohol. The resultant extract (10 kgs) after liberation of methanol shows an acid value 21, a vitamin-A-content 18000 I. E. per gramme and a vitamin-D-content of 2000 I. E. per gramme. This acid vitamin concentrate is dissolved in 10 litres carbon tetrachloride and perforated with 5 litres 90% methanol in the usual manner, six hours being necessary for completion of the process. The alcohol extract and the carbon tetrachloride quintessence are vapourised separately. The alcohol extract (approx. 1 kg) shows an acid value 185 and contains small quantities of vitamins-A and D, flavour and odoriferous substances and stearine, whilst the vitamin concentrate liberated of carbon tetrachloride has an acid value 2, 5, showing a vitamin-A-content of 19000 I. E. per gramme and a vitamin-E-content of 2200 I. E. per gramme.

(2) 1 kg vitamin-E-concentrate with an acid value 50 and a vitamin-E-content 2.5% $\alpha + \beta$ tocopherol is dissolved in 1 litre benzene and thrice shaken with each 300 ccm 88% ethylic alcohol; these ethylic alcohol quintessences are then combined and vapourised. The residue, approximately 300 grammes is very acid and contains 0.5% $\alpha + \beta$ tocopherol; and the vitamin-D-concentrate, liberated of benzene has an acid value 5.6 and contains 2.3% $\alpha + \beta$ tocopherol.

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