

# UNITED STATES PATENT OFFICE

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## METHOD OF PRODUCING VITAMIN D PRODUCT

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This invention relates to the preparation of materials rich in vitamin D and more particularly to the preparation and use of a vitamin D-protein simplex suitable for use in food or medicinal products.

Very considerable amounts of clinical and biological research work have been conducted in connection with the effect of vitamin D upon animals and human beings and particularly in the effect of this vitamin in the dietary of infants as a prevention or cure for rickets. It has been previously found that vitamin D may be synthesized by treatment of food products and medicinal preparations with ultra-violet ray or they may be prepared in other ways. For example, a product of high antirachitic value may be prepared by irradiating cows' milk or by using the milk from cows fed irradiated yeast, or by irradiation of crude sterols (as in preparation of viosterol). Also, cod liver oil has been found to contain this vitamin. It has been the common view that the vitamin D in irradiated milk is entirely associated with the oil or fat and that the increased effectiveness in the milk is attributable to the calcium and phosphorous content of the milk.

An important feature of the present invention is the discovery that vitamin D may be caused to become associated with proteinaceous matter, for example, lactalbumin, in milk, and that such combination or association of the vitamin D and proteinaceous matter (as lactalbumin) has greater antirachitic effectiveness than the same amount of vitamin D not so associated or combined with proteinaceous matter. Another important feature is the discovery that such a combination or association of vitamin D and proteinaceous matter can be separated from the aqueous medium, dried and redispersed as a colloidal solution with full or substantially full retention of the vitamin effectiveness of the vitamin D-lactalbumin combination in its original aqueous medium.

I have also found that certain protein constituents separated from milk, as for example lactalbumin, with prosthetically bound lipid material, can be activated with vitamin D by irradiation with ultra-violet rays; also that such proteinaceous materials, with or without prosthetically bound lipid material, can be activated by contacting a water suspension of it with suitable substances rich in vitamin D, for instance, in a solution which is miscible with water and which permits a high degree of diffusion of the vitamin in contact with the dispersed proteinaceous matter; appropriate solvents for vitamin D miscible with water are, for example, propylene glycol, ethyl alcohol or certain other alcohols.

Reference is made herein to the product as a simplex since the proteinaceous matter, lactalbumin, for example, acts as the high molec-

ular carrier and the vitamin D as the prosthetic group, and the biological data clearly demonstrate that the combination of the lactalbumin with vitamin D has more biological activity than vitamin D without the high molecular carrier. However, there may be still some question as to the distinction between simplex and complex and it is not intended to restrict the invention by the particular terminology used or theoretical explanation given or to exclude equivalent materials or procedures because they may be considered to relate to complexes rather than to simplexes.

It is an object of the present invention to provide a new method for preparing products suitable for use as, or in the preparation of foods or medicinal products, which are rich in vitamin D and of high antirachitic value. Another object is to provide a method of preparing a vitamin D containing product in which the effectiveness and the antirachitic value of the vitamin D are enhanced. It is also an object to produce a new product of enhanced vitamin D effectiveness, including the preparation of a water soluble antirachitically active substance combined with or associated with protein material, in the absence or substantial absence of fats or oils. It is also an object to provide a new method for increasing the vitamin D or antirachitic value of milk or milk products. Other objects will become apparent.

As a specific example of the preparation of a vitamin D-protein simplex in accordance with the present invention, one or more of the purified or impure soluble albumins prepared as described in Patent No. 2,023,014, granted upon the copending application Serial No. 650,499 of Supplee and Flanigan, may be used. A predetermined quantity of the albumin, which may or may not have been previously desiccated, is added to water, the amount being such as to permit complete or substantially complete solution or colloidal dispersion of the albumin used. If by virtue of the selection of a product of relatively impure grade, there is any substantial or objectionable degree of turbidity or agglomeration of protein or extraneous products, such may be removed, if desired, by mechanical means as, for example, by filtration or sedimentation, or centrifuging. A satisfactory concentration of the solubilized albumin is between .02% and .5%. Concentrations on either side of these limits may, however, be used if practical considerations so dictate. It is desirable, however, to have the albumin in a high degree of colloidal dispersion in order that the maximum surface of contact between the dispersed molecules or colloidal particles of the protein, and the similarly highly dispersed or diffused molecules of the vitamin D