

PATENT SPECIFICATION

(11) 1 306 402

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NO DRAWINGS

- (21) Application No. 15917/70 (22) Filed 3 April 1970
 (31) Convention Application No. 813 283 (32) Filed 3 April 1969 in
 (33) United States of America (US)
 (44) Complete Specification published 14 Feb. 1973
 (51) International Classification A23C 21/00
 (52) Index at acceptance A2B 1C 1H 1L



(54) A NEW HIGH PROTEIN, CALCIUM FORM OF DESALTED WHEY

(71) We, AMERICAN HOME PRODUCTS CORPORATION, a corporation organised and existing under the laws of the State of Delaware, United States of America, of 685 Third Avenue, New York 17, New York, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following statement:

This invention relates to a new high protein, calcium form of desalted whey product. In particular, it concerns a soluble, bland, palatable whey product which is high in soluble protein of high biological value, very low in the major minerals that are excreted in the urine and containing controlled amounts of calcium and phosphorus. Further, it is concerned with the new process for the preparation of this product. The product of the invention is useful in the preparation of foods for use in the dietary management of renal insufficiency.

The new process for the preparation of high

protein, calcium form of desalted whey product of this invention comprises

(a) desalting whey until the ash content is from 0.4 to 0.6 percent by weight of the total solids content;

(b) evaporating the desalted product under vacuum at a temperature up to 155°C. until the total solids content is from 40 to 60 percent of the total weight;

(c) cooling the evaporated product until at least 40 percent of the total solids content precipitates;

(d) separating the precipitate from the cooled product;

(e) adjusting the pH of the liquid portion with calcium hydroxide to a range of 6.0 to 7.5; and

(f) drying the adjusted liquid portion to a solid or concentrating the adjusted liquid portion and then drying it to a solid.

The starting material is whey, such as, whey produced from any cheese manufacturing process or for the conventional acid casein manufacturing process. The composition of a typical cheese whey is set forth in Tables A and B.

TABLE A

Composition of Cheese Whey

	High	Low	Average
Percent by weight Total Solids	6.5	6.3	6.41
pH	6.45	6.2	6.37
Titrateable acidity expressed as % w/w Lactic Acid	0.13	0.095	0.11

TABLE B

Composition of Cheese Whey on Total Solids Basis

	Percent		
	High	Low	Average
Protein	14.41	12.18	13.0
Fat	1.54	0.78	1.03
Ash	9.00	7.92	8.48
Calcium	0.703	0.595	0.639
Magnesium	0.154	0.101	0.131
Phosphorus	0.794	0.673	0.728
Sodium	0.971	0.730	0.827
Potassium	2.60	2.23	2.40
Chlorides	2.03	1.74	1.87
Citric Acid	3.19	1.42	2.61

5 The process of the invention will now be described in more detail. Desirably the starting material for the desalting step is first obtained by concentrating the whey under vacuum at a temperature not above about 165°F. until the total solids content is about 20 to about 30 percent of the total weight. Preferably, this concentration is conducted under vacuum at a temperature range from about 158° to about 161°F. until the total solids content is about 24 to about 28 percent of the total weight. Although this process could be conducted without this concentration, the subsequent dialysis becomes highly inefficient, in that, about four time the volume must be pumped and the dilute solution has a low electrical conductivity.

20 Secondly, the concentrated product is desalted by using any suitable technique, such as electrodialysis, ion exchange procedures, reverse osmosis and collidal gel fractionation. In this regard, the preferred techniques employed are electrodialysis and ion exchange procedures. The electrodialysis procedures which may be employed are exemplified by those described in U.S. Patents Nos. 2,631,100 and 2,671,055. Alternatively, the ion exchange procedures are exemplified by those described in U.S. Patents Nos. 2,477,558 and 2,584,158. The desalting is continued until the total ash of the whey is reduced to 0.4 to 0.6 percent of the total solids. Preferably, this desalting is continued until the total solids composition comprises from about 12 to about 16

percent protein, about 81 to about 85 percent lactose, about 0.8 to about 1.2 percent fat, about 0.4 to about 0.6 percent ash, about 0.12 to about 0.20 percent calcium, about 0.01 percent sodium, about 0.014 percent potassium and not more than about 0.01 percent chloride, all percentages being by weight.

Thirdly the desalted product is evaporated under vacuum at temperatures up to 155°F. until the total solids content is from 40 to 60 percent of the total weight. It has been noted that when temperatures substantially above 155°F. are employed the whey protein begins to coagulate. Although no minimum temperature limit is necessary, when temperatures below about 120°F. are employed excessive crystallisation of lactose takes place which may cause undue clogging of the evaporator.

To afford a final product that would contain a minimum of 20 percent by weight of protein, this evaporation would have to be continued until the total solids thereof constituted 40 percent of the total weight. If this evaporation is continued to a point substantially beyond a solids content of 60 percent total solids content, it would become difficult to separate the crystallised lactose from its liquor. Preferably this evaporation is conducted under vacuum at a temperature range from about 120° to about 140°F. until the total solids content is from about 49 to about 55 percent of the total weight.

Fourthly the evaporated product is gradually cooled with stirring over a period of seventeen

to thirty hours, preferably 22 to 24 hours, causing the crystallisation and precipitation of at least 40 percent of the total solids content. As easily recognised by one skilled in the art, considerable technique is involved in any fractional crystallisation of mixtures. In this regard, the crystallisation of lactose from the evaporated product has been found to be readily accomplished by holding the evaporated product at relatively higher temperatures, e.g. about 115°F. until the formation of relatively small lactose crystals commences. Thereafter, the cooling is continued at a rate in which these crystals grow and a minimum number of new crystals form. Once the lower temperatures, e.g. about 70°F. are achieved it is maintained at this temperature until crystallisation is complete. A preferred cooling schedule which is employed when the evaporated product contains a total solids content of from 49 to 60 percent is: maintaining the evaporated product for a period of about four to about five hours at a temperature range of about 105 to about 115°F, then cooling over about a thirteen to about a fifteen hour period to a temperature range of about 60° to 70°F, and thereafter holding the temperature in this latter range for an additional period of about five to about seven hours. It should be noted that these crystallisation temperatures and periods are not critical and may be readily altered by a skilled practitioner. However, it has been observed that if the crystallisation is continued for periods substantially beyond a total of thirty hours spoilage of the material begins to occur.

Fifthly, the separation of the crystallised lactose precipitate from the liquor may be accomplished by any available method for the separation of crystals from a liquid, such as, filtration, decantation and centrifugation. For example, the crystals may be allowed to settle for twenty-four hours and then the supernatant is decanted. Alternatively, the liquor is centrifuged either in a batch basket or in a continuous solid-liquid separator. The preferred technique is to employ a continuous solids-liquid separation centrifuge with a light water spray washing of the crystal layer in the centrifuge to increase the yield of liquor and obtain a purer lactose.

Sixthly, the pH and calcium content of the above-separated liquor is adjusted by the addition of a calcium hydroxide slurry until the pH of the liquor is in the range of 6.0 to 7.5 and the calcium content thereof is preferably from about 2.0 to about 2.5 percent of the product's protein content. Preferably the pH is adjusted to a range from about 6.5 to about 7.0 and the calcium content to a range of about 2.3. to about 2.4 percent of the protein content of the product.

Seventhly, this adjusted liquor may be concentrated before it is dried to a solid. Although

this concentration is not an essential step in the subject process, it is economical to so concentrate the adjusted liquor since an evaporator is more efficient than a spray drier in the amount of energy (BTU's) necessary to evaporate a unit of water. Normally this concentration is conducted under vacuum at a temperature not above 145°F. until the total solids content is about 45 percent of the total weight. At higher total solids the liquor becomes a gelatinous mass. Preferably, this concentration is effected under vacuum at a temperature range of about 130° to about 140° F. until the total solids content is from about 35 to about 40, or even 45 percent of the total weight. At these higher total solids contents it has been found that the dryer yields improve and the resulting solids is coarser.

Eighthly, the adjusted liquor or the concentrated adjusted liquor is dried to afford the high protein, calcium form of desalted whey product. Any drying process may be employed, such as, spray drying, foam drying or freeze drying. Preferably the product is dried in a spray drier until the moisture content is the range from about 1 to about 6 percent of the product weight. The designated moisture range is not an essential characteristic of the product but is advantageously utilised when it is desired to store the product for substantial periods before its use in the preparation of foods which are employed in the dietary management of renal insufficiency.

The high protein, calcium form of a desalted whey product prepared by the above-described process may contain from 20 to 45 percent milk serum proteins; 50 to 75 percent lactose and a mineral content of 2 to 5 percent of the milk serum proteins. The mineral content of the product comprises 55 to 65 percent calcium, about 35 percent phosphorus, not more than 4.5 percent sodium, not more than 4.5 percent potassium and not more than 2.5 percent chloride, all percentages being by weight. This product has a pH of about 6.0 to about 7.5 when reconstituted with water to form a suspension having about a 10 percent total solids content.

The novel high protein, calcium form of desalted whey product of the present invention may by routine procedures then be compounded into a food for use in the dietary management of renal insufficiency. For example, the whey product of this invention is dissolved in demineralised water and then admixed with fats, fat-soluble vitamins, sugars and trace minerals and warmed to 140—150°F. Thereafter, the mixture is pasturised, homogenised, cooled, admixed with water-soluble vitamins and spray dried to afford a dry form of a bland and palatable, water soluble food which, in a quantity that is easily ingested orally each day, provides a known and controlled quantity of protein of high biological value and adequate amounts of diet essentials with the ex-

ception of sodium, potassium and chloride. This food is useful in the dietary management of renal insufficiency.

Renal diseases resulting in renal insufficiency and failure of the kidneys to clear the normal dietary load of all substances excreted by the urine, are receiving more attention as methods of treatment are developed. In the dietary management of these conditions it is of particular importance that the intake of sodium, potassium, chloride, sulphate, to a degree phosphorus and other mineral constituents excreted in the urine be restricted. The protein intake should be adequate for maintenance and, in children, for some growth. Since protein metabolites accumulate in the body, the diet protein should be of high biological value to keep the quantity required as low as possible. The daily intake of water, soluble minerals, e.g. sodium, potassium, chloride and to a degree phosphorus should be balanced against the amounts excreted. Because calcium and magnesium are excreted about one-third in the urine and two-thirds in the feces, the levels of these two elements should also be controlled at no higher than the minimum required for a normal individual. Although trace mineral requirements have not been specifically established, intake should be at least adequate for the equivalent healthy individual. The vitamins should be supplied in normal amounts and caloric requirements are also normal for the amount of physical activity involved or permitted. In cases requiring severe restriction of choice of foods it is difficult to supply sufficient calories. This is most difficult with fats which often must be supplied in unpalatable pure form. Although pure sugars are more palatable, continued high intake thereof becomes repugnant to most individuals. The carbohydrate requirement is normal. However, as in the diet of a normal person too much lactose causes a chronic diarrhea. In this regard, although there is not specific limit, 50 to 60 grams of lactose per day is normally handled without resulting in diarrhea.

The optimum basic food for use in renal insufficiency is one which meets all of the above requirements so as to permit the greatest selection of other foods to provide the remainder of the calories and the diet roughage. The psychological aspects of management of the diet of those suffering from chronic renal insufficiency are also important. The patient must subsist on the product for long periods of time and failure to follow the prescribed diet is injurious. The product therefore must be as acceptable as possible to all patients. It should therefore be bland, lacking even in the normal sweetness associated with disaccharides, compatible with flavourings permitted in the diet, suitable for preparation in a number of familiar food forms, nonperishable and readily reconstituted. The high protein, calcium form of desalted whey product of this

invention is intended to meet and satisfy all of the above criteria.

The following examples are given by way of illustration:—

Example 1

A fresh cheddar cheese whey (Table I and II—A) is concentrated to 26% solids (Table I and II—B) in a triple effect falling vacuum evaporator at a maximum liquid temperature of 161°F. The concentrated whey is adjusted to pH 6.2. by the addition of 140 pounds of sodium hydroxide as a 30 percent aqueous solution and then passed through a solids-liquid separating milk clarifier. The clarified whey is desalted by the electrodialysis procedure as described in U.S. Patent No. 2,631,100, until the ash of the whey solids is 0.41 percent, the calcium content is 0.122 percent and total solids are 27.6 percent and total solids are 27.6 percent (Table I and II—C).

The desalted whey is concentrated in a single effect vacuum evaporator at a maximum liquid temperature of 138°F. until the total solids content of the liquid is fifty-four percent (Table I and II—D). This concentrated liquid, at a temperature of 113°F. is placed in a 3000 gallon vertical agitator equipped with a cold water jacketed tank and then agitated and allowed to cool to 110°F. over a four hour period. The jacket cooling water is turned on and the liquid-lactose crystal slurry cooled to 68°F. over fourteen hours and then held at this temperature for six hours. Continuous agitation is conducted throughout this twenty-four hour crystallisation period. This slurry is passed through a continuous centrifugal solids-liquid separator with a light spray of wash water placed on the lactose crystal mass at a point near the cake discharge end of the separator. A total of about 200 pounds of wash water are used. The discharged damp lactose cake representing 77% total solids (Table I and II—E) is tunnel dried to produce a crude lactose powder corresponding to 65.6% solids removed by centrifugation. The liquor (Table I and II—F) from the centrifugation step is placed in an agitator equipped tank and twenty-one pounds of calcium hydroxide are added thereto with agitation as a 6 percent suspension. When the addition is complete, agitation is continued for an additional hour. This neutralised liquid product (pH 6.6) is concentrated in a single effect vacuum evaporator at 135°F. maximum liquid temperature to 40 percent total solids (Table I and II—G) and then sprayed in a single stage pressure jet spray dryer using 330°F. inlet air and with 185°F. exhaust air temperature to afford a 3647 pounds of a high protein calcium form of a desalted whey product (Table I and II—H).

The composition of this product is as follows: 32.0 percent protein (TNx 6.38), 59.43 percent lactose, 3.61 percent moisture, 1.86

percent ash, 0.583 percent calcium, 0.33 percent phosphorous, 0.0125 percent sodium, 0.0245 percent potassium, less than 0.01 percent chloride having a pH of 6.3 when recon-
centrated in water to ten percent total solids, all percentages being by weight.

TABLE I

Materials and Constituent Quantities in Pounds

	Quantity	Water	Solids	Protein	Lactose	Calcium
A. Raw Whey (6.3% T.S.)	253,000	237,060	15,940	2,072	11,950	102
B. Conc. Whey (26% T.S.)	61,145	45,254	15,900	2,067	11,925	101.6
C. Desalted Whey (27.6% T.S.)	46,195	33,445	12,750	1,785	10,646	15.55
D. Desalted Whey (54% T.S.)	22,963	10,563	12,400	1,736	10,354	15.13
E. Damp Lactose Cake (77% T.S.)	10,558	2,428	8,130	333	7,642	3.25
F. Liquor (30.8% T.S.)	12,662	8,762	3,900	1,295	2,404	11.62
G. Conc. Liquor (40% T.S.)	9,250	5,550	3,700	1,229	2,281	23.38 (1)
H. Product Powder	3,647	132	3,515	1,167	2,167	21.26

(1) After addition of 21 lbs of calcium hydroxide containing 11.35 lbs of calcium.

TABLE II

Percent Composition of the Solids of Whey Through Processing

	Protein	Lactose	Calcium	Ash
Raw whey and concentrated whey (A,B)	13	75	0.639	8.48
Desalted whey and Conc. desalted whey (C,D)	14	83.5	0.122	0.41
Lactose Cake (E)	4.1	94	0.04	0.17
Liquor from centrifuge, conc. '40%' solids liquor and product solids powder (F,G,H)	33.21	61.65	0.298 native 0.307 added	1.5% native 0.43 added
			0.605 total	1.93 total

Example II

5 A sweet cheddar whey (Table III—A) is concentrated to 20% solids (Table III—B) and desalted by ion exchange. The cation column employed has an internal diameter of 16 cm. and 8050 ml. of a cationic resin in the hydrogen form having a capacity of 1.7 meq/ml of resin. The anion exchange column also has an internal diameter of 16 cm. and contains 14,800 ml. of an anionic resin in the hydroxyl form having a capacity of 1.33 meq/ml of resin. The columns are operated in series with a flow rate of 1.57 gal/min/sq.ft. of bed surface in the cation column and 0.55 gal/min/sq.ft. in the anion column. The void volume of the columns is 38 percent or a void value in both columns of 8664 ml. The first three void volumes of effluent from the co-

lums at the start of a run are discarded. At the end of the run three void volumes of water are put through the columns and the effluent combined with the main volume of whey. The resulting desalted whey (III—C) is collected from the columns and then concentrated under vacuum to fifty five percent total solids (Table III—D). The lactose is allowed to crystallise and then removed as in Example I. The respective compositions of the liquor and lactose are given in Table (III—E) and (III—F). The damp lactose cake contains 75% total solids representing removal of 64% total solids by centrifugation. The separated liquor which contains 34.3% total solids is adjusted by the addition of 14.8 gms. of calcium hydroxide (Table III—G) and then spray dried to afford a high protein, calcium form of a desalted whey product (Table III—H).

TABLE III

Materials and Constituent Quantities in Grams

	Quantity	Water	Solids	Protein	Lactose	Ash	P	Ca	Na	K	Cl
A. Raw whey (6.0% T.S.)	108,130	101,644	6466	840.6	5077	548.3	47.1	41.32	53.42	155.2	120.9
B. Con. whey (20% T.S.)	31,845	25,476	6369	828.0	5001	540.1	46.4	40.70	52.67	152.9	119.1
C. Desalted whey (14.6% T.S.)	31,845	27,282	4642	593.2	4022	27.00	8.28	2.03	0.16	0.22	0.15
D. Desalted whey (55% T.S.)	8,313	3,741	4573	584.3	3962	26.60	8.16	2.00	0.158	0.217	0.148
E. Liquor	4,781.9	3,138.5	1643.4	525.9	1091	26.5	8.12	1.98	0.157	0.197	0.128
F. Damp Lactose cake (75% T.S.)	3,906	976.5	2929.5	58.4	2871	0.1	0.04	0.02	0.01	0.02	0.02
G. Liquor pH Adjusted	4,796.1	983	1658.2	467.9	1060	37.7	8.12	9.98	0.156	0.195	0.126
H. Product Powder/100 gms.	100	3.5	96.5	27.2	61.7	1.61	0.47	0.581	0.009	0.011	0.007

Example III

12,000 Gms. of water are weighed into a jacketed tank and heated to 160°F. Thereafter, 2843 gms. of the high protein, calcium form of desalted whey product as prepared in Example I and 694 gms. of lactose are added with agitation. The resulting mixture is admixed with 5,477 gms. of corn syrup which has been preheated to 135°F. The fat blend (see Table IV) and the fat-soluble vitamins of Table V are then added to the mixture fo-

lowed by the addition thereto of the mineral salts listed in the latter Table. The mixture is heated to 165°F. held at this temperature for less than a minute and then cooled to 145°F. The cooled mixture is monogenised in a superhomogeniser, admixed with the water-soluble vitamins of Table V and then spray dried to afford an edible solid formulation having the measured properties hereinafter set forth in Table VI.

TABLE IV

Fat Blend Composition

Corn Oil	2311 gms
Coconut Oil	73 gms
Total Weight	3084 gms

TABLE V

Vitamin and Mineral Composition

		gms
Vitamin A Palmitate	1,599,000 USP units/mg	0.114
Beta Carotene	400,000 USP units/gm	0.087
Vitamin D ₃ (Cholecaliferol	1,000,000 USP units/gm)	0.015
Vitamin E (d alpha tocopheryl acetate	1360 IU/gm)	0.644
Ascorbic Acid		64.0
B ₆ Pyridoxine HCl		0.046
B ₁₂ Cyancobalamin 0.1% in Mannitol		0.007
Niacin		0.256
Folic Acid		0.00157
Ca Pantothenate		0.190
FeSO ₄ ·7H ₂ O		1.206
CuSO ₄ ·5H ₂ O		0.126
ZnSO ₄ ·7H ₂ O		0.462
KI		0.0016

Water is added to make 35% total solids composition.

TABLE VI

Measured Properties of the Finished Formulation

Determination	Assay
Moisture	0.92%
Ash	0.606%
Protein	9.99%
Fat	30.13%
pH (10% solution)	6.61
Ascorbic Acid	32 mg/100 gm
Phosphorus	121.8 mg/100 gm
Sodium	9.1 mg/100 gm
Potassium	6.75 mg/100 gm
Calcium	207 mg/100 gm
Iron	3.5 mg/100 gm
Zinc	1.26 mg/100 gm
Magnesium	17.1 mg/100 gm
Chloride (estimate)	<1. mg/100 gm

Similarly, the above procedure is repeated utilising the high protein, calcium form of desalted whey product of Example II to afford a comparable edible solid formulation.

5 WHAT WE CLAIM IS:—

1. A process for the preparation of a high protein, calcium form of a desalted whey product which comprises:

10 (a) desalting whey until the ash content is from 0.4 to 0.6 percent by weight of the total solids content;

15 (b) evaporating the desalted product under vacuum at a temperature up to 155°F. until the total solids content is from 40 to 60 percent of the total weight;

(c) cooling the evaporated product until at least 40 percent of the total solids content precipitates;

20 (d) separating the precipitate from the cooled product;

(e) adjusting the pH of the liquid portion with calcium hydroxide to a range of 6.0 to 7.5; and

25 (f) drying the adjusted liquid portion to a solid, or concentrating the adjusted liquid portion and then drying it to a solid.

2. A process as claimed in Claim 1, wherein

the whey which is desalted is a concentrate prepared by concentrating whey under vacuum at a temperature up to about 165°F. until the total solids content is from about 20 to 30 percent of the total weight.

3. A process as claimed in Claim 2, wherein the concentration step is conducted under vacuum at a temperature range from about 158° to about 161°F.

4. A process as claimed in Claim 2 or Claim 3, wherein the concentration step is carried out until the total solids content is about 24 to about 28 percent of the total weight.

5. A process as claimed in any one of the preceding claims, wherein the desalting step is carried out until the total solids composition comprises from about 12 to about 16 percent protein, about 81 to about 85 percent lactose, about 0.8 to about 1.2 percent fat, about 0.4 to about 0.6 percent ash, about 0.12 to about 0.20 percent calcium, about 0.01 percent sodium, about 0.014 percent potassium and not more than about 0.01 percent chloride, all percentages being by weight.

6. A process as claimed in any one of the preceding claims wherein the evaporation step is carried out at a temperature range from

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TABLE VI

Measured Properties of the Finished Formulation

Determination	Assay
Moisture	0.92%
Ash	0.606%
Protein	9.99%
Fat	30.13%
pH (10% solution)	6.61
Ascorbic Acid	32 mg/100 gm
Phosphorus	121.8 mg/100 gm
Sodium	9.1 mg/100 gm
Potassium	6.75 mg/100 gm
Calcium	207 mg/100 gm
Iron	3.5 mg/100 gm
Zinc	1.26 mg/100 gm
Magnesium	17.1 mg/100 gm
Chloride (estimate)	<1. mg/100 gm

Similarly, the above procedure is repeated utilising the high protein, calcium form of desalted whey product of Example II to afford a comparable edible solid formulation.

WHAT WE CLAIM IS:—

1. A process for the preparation of a high protein, calcium form of a desalted whey product which comprises:

(a) desalting whey until the ash content is from 0.4 to 0.6 percent by weight of the total solids content;

(b) evaporating the desalted product under vacuum at a temperature up to 155°F. until the total solids content is from 40 to 60 percent of the total weight;

(c) cooling the evaporated product until at least 40 percent of the total solids content precipitates;

(d) separating the precipitate from the cooled product;

(e) adjusting the pH of the liquid portion with calcium hydroxide to a range of 6.0 to 7.5; and

(f) drying the adjusted liquid portion to a solid, or concentrating the adjusted liquid portion and then drying it to a solid.

2. A process as claimed in Claim 1, wherein

the whey which is desalted is a concentrate prepared by concentrating whey under vacuum at a temperature up to about 165°F. until the total solids content is from about 20 to 30 percent of the total weight.

3. A process as claimed in Claim 2, wherein the concentration step is conducted under vacuum at a temperature range from about 158° to about 161°F.

4. A process as claimed in Claim 2 or Claim 3, wherein the concentration step is carried out until the total solids content is about 24 to about 28 percent of the total weight.

5. A process as claimed in any one of the preceding claims, wherein the desalting step is carried out until the total solids composition comprises from about 12 to about 16 percent protein, about 81 to about 85 percent lactose, about 0.8 to about 1.2 percent fat, about 0.4 to about 0.6 percent ash, about 0.12 to about 0.20 percent calcium, about 0.01 percent sodium, about 0.014 percent potassium and not more than about 0.01 percent chloride, all percentages being by weight.

6. A process as claimed in any one of the preceding claims wherein the evaporation step is carried out at a temperature range from

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about 120° to about 140° F. until the total solids content is from about 49 to about 55 percent of the total weight.

5 7. A process as claimed in Claim 6, wherein the cooling step is carried out by maintaining the evaporated product for about 4 to about 5 hours at a temperature range of from 105 to 115°F, then cooling for about 13 to about 15 hours to a temperature range of about 60°
10 to 70°F. and thereafter holding the temperature in this range for an additional period of from about five to seven hours.

8. A process as claimed in any one of the preceding claims wherein step (e) is carried
15 out by adjusting the pH and calcium content of the liquid portion with calcium hydroxide to a pH range of about 6.5 to about 7.0 and a calcium content from about 2.2 to about 2.4 percent of the protein content of the product.

20 9. A process as claimed in any one of the preceding claims wherein the adjusted liquid portion is concentrated under vacuum at a temperature range of about 130° to about 140°F. until the total solids content is from
25 about 35 to about 45 percent of the total weight; and then the concentrated liquid portion is dried to a solid.

30 10. A process as for the preparation of a high protein, calcium form of a desalted whey product which comprises:

(a) concentrating whey under vacuum at a temperature range of 158 to 161°F. until the total solids content is from 24 to 28 percent
35 of the total weight;

(b) desalting the concentrated product by electrodialysis until the ash content is from 0.4 to 0.6 percent of the total solids content;

40 (c) evaporating the electrodialysed product under vacuum at a temperature range from 120 to 155°F. until the total solids content is from 49 to 60 percent of the total weight—

45 (d) gradually cooling the evaporated product with stirring for a period of 17 to 30 hours, until at least 40% of the total solids content precipitates;

(e) separating the precipitate from the cooled product by centrifugation;

50 (f) adjusting the pH and calcium content of the liquid portion with calcium hydroxide to a pH range of 6.0 to 7.5 and a calcium content from 2.0 to 2.5 percent of the protein content of the product;

55 (g) concentrating the adjusted liquid portion at a temperature up to 145°F. until the total solids content is from 35 to 45 percent of the total weight; and

(h) spray drying said concentrated liquid portion.

60 11. A process for the preparation of a high protein, calcium form of a desalted whey product which comprises:

65 (a) concentrating sweet cheese whey under vacuum at a temperature range from 158 to 161°F. until the total solids content is from 24 to 28 percent of the total weight;

(b) desalting the concentrated product by electrodialysis until the total solid content comprises from 12 to 16 percent protein, 80 to 85 percent lactose, 0.8 to 1.2 percent fat, 0.4 to 0.6 percent ash, 0.12 to 0.20 percent calcium, about 0.01 percent sodium, about 0.014 percent potassium and about 0.01 percent chloride, all percentages being by weight;

70 (c) evaporating the electrodialysed product under vacuum at a temperature range from 120 to 155°F. until the total solids content is from 49 to 60 percent of the total weight;

75 (d) gradually cooling the evaporated product with stirring over a period of 22 to 24 hours, until at least 40% of the total solids content precipitates;

(e) separating the precipitate from the cooled product by centrifugation;

80 (f) adjusting the pH and calcium content of the liquid portion with calcium hydroxide to a pH range of 6.5 to 7.0 and a calcium content from 2.2 to 2.4 percent of the protein content of the product;

85 (g) concentrating the adjusted liquid portion at a temperature range from 130 to 140°F. until the total solids content is from 35 to 45 percent of the total weight; and

(h) spray drying the concentrated liquid portion.

90 12. A process for the preparation of a high protein, calcium form of a desalted whey product which comprises:

(a) concentrating sweet cheese whey under vacuum at a temperature range from 158 to 161°F. until the total solids content is from 24 to 28 percent of the total weight;

100 (b) desalting the concentrated product by electrodialysis until the total solids content comprises by weight 12 to 16 percent protein, 80 to 85 percent lactose, 0.8 to 1.2 percent fat, 0.4 to 0.6 percent ash, 0.12 to 0.20 percent calcium, about 0.01 percent sodium, about 0.014 percent potassium and about 0.01 percent chloride, all percentages being by weight;

105 (c) evaporating the electrodialysed product under vacuum at a temperature range from 120 to 155°F. until the total solids content is from 49 to 60 percent of the total weight;

110 (d) gradually cooling the evaporated product with stirring for a period of 4 to 5 hours to a temperature range of 105 to 115°F., then for a period of 13 to 15 hours to a temperature range of 60 to 70°F. and subsequently holding the temperature in this latter range for an additional period of 5 to 7 hours thereby causing precipitation of at least 40% of the total solids content;

115 (e) separating the resulting precipitate from the cooled product by centrifugation in a continuous solid-liquid separation centrifuge;

120 (f) adjusting the pH and calcium content of the liquid portion with calcium hydroxide to a pH range of 6.5 to 7.0 and a calcium content from 2.2 to 2.4 percent of the protein content of the product;

130



(g) concentrating the adjusted liquid portion at a temperature range from 130 to 140°F. until the total solids content is from 35 to 45 percent of the total weight; and

(h) spray drying the concentrated liquid portion.

13. A process for the preparation of a high protein, calcium form of a desalted whey product substantially as hereinbefore described with reference to Example I or Example II.

14. A high protein, calcium form of a desalted whey product whenever prepared by a process as claimed in any one of the preceding claims.

15. A high protein, calcium form of desalted whey product which comprises on a weight/weight basis:

(a) 20 to 45 percent milk serum proteins;

(b) 50 to 75 percent lactose;

(c) a mineral content of 2 to 5 percent of the milk serum proteins which comprises 55 to 65 percent calcium, about 35 percent phosphorous, not more than 4.5 percent sodium,

not more than 4.5 percent potassium and not more than 2.5 percent chloride; and which has a pH of 6.0 to 7.5 when reconstituted with water to about 10 percent total solids content.

16. A high protein, calcium form of a desalted whey product substantially as hereinbefore described with reference to Example I or Example II.

17. A food for use in the dietary management of renal insufficiency comprising a high protein, calcium form of desalted whey product as claimed in any one of Claims 14 to 16 and suitable conventional food ingredients.

18. A food as claimed in Claim 17 substantially as hereinbefore described with reference to Example III.

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