Major Thesis in Dairy Chemistry,

Submitted by

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to

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Subject:

A Study of Milk Made From Condensed and Powdered Milks.

materials and the sediments collected from them. The determinations made were for solids, fat, protein, ash and some of the ash constituents. In analysing the sediments, the great difficulty encountered was that of obtaining representative samples, and in order to secure these, much time was spent in trying to separate out the sediments

Methods used for obtaining sediments:

1. A quantity of the sample was diluted with distilled water in the proportion of four to one, placed in separatory funnels and allowed to stand for several hours. The lower portion was then drawn off and whirled in a centrifuge at 2000 revolutions per minute for ten minutes. About half of the centrifuged liquid was then decanted off and the remainder diluted with distilled water and whirled as before. This process was repeated until the fluid was about clear. The amount of dilution necessary to clear the sediments of almost all soluble material was about 1 to 4000 parts. The sediments were then collected, dried in a water oven and weighed.

Per cent of Sediment:

The first sediments from evaporated milk were very granular and only an average of 0.29 per cent of the original material was recovered as sediment. For this reason the method of collecting sediments was changed, and the sample was taken directly from the can, weighed, diluted, and placed in the centrifuging tubes and completed as before.

Per cent of Sediment.

Table I.

Van Camps Brand, Can No. 2 ---	0.55
" " " " " 3 ---	0.21
Pet Brand " " 4 ---	0.56
Van Camps " " " 5 ---	1.63
" " " " " 6 ---	0.44
" " " " " 8 ---	0.44

These results are variable but are more uniform than the analytical résutls secured for the different constituents. This may be explained by an inspection of the analyses which shows that much of the material must have been lost in the process of dilution and centrifuging. This method was considered unsatisfactory after the work of collecting sediments and analysing had progressed far enough to secure a comparative survey of the results, for with some brands that contained granular sediments, notably "Pet" and "Van Camps" evaporated milks, the granular substance preponderated greatly in the washed sediment; the ash content rose as high as 38.67 per cent with a CaO content of 70.2 per cent.

Table I.

Ash Constituents of Original Milks and Their Sediments.

	Original Milk			Sediments.					
Sample No.	3	5	8	1	2	3	4	5	8
Per Cent Sediment				0.29	0.55	0.21	0.56	1.63	.44
Per Cent Ash	1.68	1.66	1.59		11.21	38.67	29.19	17.80	
Per Cent CaO	20.38	17.37	13.84		31.82	*70.20	*63.60	*53.20	
Per Cent MgO	2.77	2.50			1.22	0.87	0.94	1.44	
Per Cent P_2O_5	20.87	28.35	29.00			1.90		10.40	
Per Cent SO_3									
Per Cent K_2O	25.52	28.43	26.00						
Per Cent Na_2O	6.20	9.74	9.76						

*Large granular particles in the sediment

Sample No. 1 was taken by the separatory funnel method and the per cent of sediment determined. Because of the granular appearance of the sediment a further analysis was not made.

A great variation exists in the results obtained in Samples 2, 3, 4, and 5. The per cent of ash in the sediments varied from 11.21 to 38.67. Sediments 3, 4, and 5 were very coarse in appearance, and these coarse particles were evidently calcareous in nature since the per cent of CaO was extremely high while the other constituents were relatively low.[1]

Samples for the per cent of sediment were taken directly from the can for Numbers 4, 5 and 8, and in these particular determinations greater uniformity was secured. But even with this method the objection noted in the previous method was evident. The lighter flocculent particles of the precipitate were lost in decantation for it seemed that these became so finely divided when being washed and centrifuged that they would not settle by gravity in the centrifugal tubes after whirling.

Table II.

Calcium Determination in Sediment.

Grams Dried Material	0.1345
Grams CaO	0.0403
Grams Equivalent $CaCO_3$	0.0718
Per Cent $CaCO_3$	53.4

1. See analysis of granular sediment

The above sediment after washing was dried on a steam bath and weighed. It was then treated with HCl and effervescence took place showing that the lime was partly in the form of carbonates. A gelatinous precipitate also appeared. This was filtered out, washed, ignited, and the ash added to the HCl solution.

The foregoing results were not satisfactory enough to warrant a continuation of this method of extracting sediments and for this reason the complete analyses were not made.

Separation of Sediment by other Methods:

The method next tried was as follows: 100 to 200 gms. of the sample were diluted to about 1000 c c with distilled water, thoroughly shaken for at least five minutes, placed in a 2000 c c graduated cylinder and made up to a definite volume. To the solution was added a few drops of formaldehyde to prevent bacterial action. The sediment was allowed to settle by gravity, and after four hours the upper portion of the solution was drawn off by means of a siphon bent so that the suction end was turned upward. At first, only about one-third was siphoned off and the remainder diluted to the original volume, but later more than two-thirds were siphoned off each time. Dilution and drawing off were repeated until the washings were fairly clear, when the sediment was dried and weighed.

Table III.

Amount Dilution and Sediments Obtained.

A.	Water used in wahing	10200 c c to 369 gms sample.
	Amount of Sediment	0.015 per cent
B.	Water used	8000 c c to 133 gms.
	Amount of sediment	0.02 per cent

<u>Washing Sediments</u>:

Before proceeding further it seemed desirable to ascertain if this large amount of washing actually eliminated the solutble constituents so that they would not interfere with the sediment analyses. Earlier in the work, qulitative tests showed sugars in the sediments when all these should have been eliminated from a properly washed sediment. In order to trace the amount and nature of the sugars in washings and sediments the following procedure was followed: Sweetened condensed milk was being used at this time.

a. Determine total and reducing sugars in the original condensed milk.

b. Dilute 100 c c of sample to 1000 c c, centrifuge, and analyze washings for sugar. Make up sediments to 1000 c c and analyze for sugar.

c. Thoroughly wash and dry sediments and analyze for sugar.

All sediments were washed by dilution, centrifuging and decanting in 1 to 1000 parts water.

The results from this procedure were as follows:

In 100 c c (128.9 gms.) condensed milk were		16.77 gms. Lactose	
97.5% of the washings contained		16.70 "	
2.5% " and sed. contained		0.09 "	
Weight of Sediment		0.32 "	
Per cent Lactose	Actual	28.1 %	
" " "	Theoretical	21.6 %	

This result corresponds fairly well with a previous analysis which showed 21.6% Lactose in the sediment when the copper reduction method was used.

Hence it is apparent that although the amount of lactose left in the washings was small, it was very large when evaporated and dried with the sediment. Much more washing would be required to eliminate it completely. It follows that all the previous analyses of sediments must have been influenced by this residue and also by residues of other soluble constituents that should have been eliminated.

The following table gives the per cent of the sugars found in the original material, serum and sediment.

The last method for separating sediments as outlined was adhered to, but on account of the foregoing results with sugars more washing was given, being 1 to 1000 parts of water. The final wahhings gave practically no indication of sugars when tested with Fehling's solution.

Sweetened condensed milk was used in this part of the work. There were no heavy, grainy sediments in these cans, this being due probably to the greater density of the sweetened product, which prevented the recrystalization or coalescence of the particles and held them in a state of fine dividion or suspension. The sediment was present, however, apparently in as great proportion as before, as can be seen by Fig. I, which shows the centrifugal tubes with different proportions of milk.

Difficulty in separating all of this finely divided sediment was to be expected, and it is shown in the results of the quantitative determination of sediment that the amount of sediment was very variable, so much so that the whole method of procedure had to be changed after this trial.

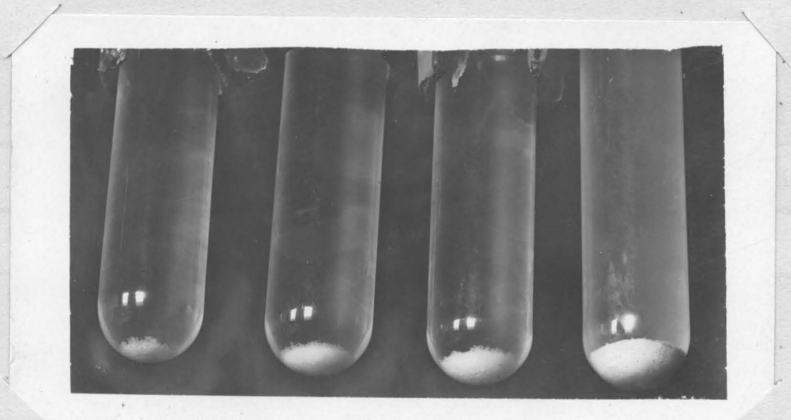
Table V.

Per cent of Sediment Can 11	0.32	a
" 12	0.02	b
" 13	0.022	b
" 12	0.005	c
" 13	0.008	c

a. Centrifuged small portion of original milk
b. Made up to 1 - 10000 parts water, allowed to settle by gravity, and decanted.
c. Centrifuge method with whole can.

Fig. I.

Sweetened Condensed Milk.



1 2 3 4

1. Dilution one to four of water.
2. Dilution one to three of water.
3. Dilution one to two of water.
4. Dilution one to one of water.

Before finally deciding to abandon the method, much work had been done in analyzing the original milk and sediments, and these results are given in Table VI.

Sediments from cans 12 and 13 were combined and the analysis shows that the calcium content of the ash is high.

Sediment No. 10 was analyzed but No. 11 was used for sugar determination.

Table VI.

Ash Constituents of Sweetened Condensed Milk and Sediments.

	Original Milk		Sediment	
SAMPLE NO.	10	12 & 13.	10	12 & 13
Per Cent Ash.	1.93	1.90	14.59	19.00
" " CaO	14.7	18.81	51.74	40.58
" " MgO	2.92	4.00	2.42	2.17
" " P_2O_5	22.79	58.77	3.74	32.51
" " SO_3	4.50		2.17	
" " Na_2O	11.39	5.83	2.40	1.28
" " K_2O	28.39	24.08	5.44	14.00

The results shown in Table VI indicate a great variation with respect to the phosphorus and potash. The ash, and lime content of the ash are high, while magnesium is present in normal quantity. (?)

Sediment from Milk Powder.

The characteristic sediment previously mentioned in this paper referred to that obtained from milk powder. It was found that the milk powder went into solution with great difficulty and that a large per cent of the material would not go into solution, but remained in a very finely divided condition and was separated by decantation with difficulty. The sediment did not become floculent but persisted as finely divided but well defined particles, and had the consistency of a fine silt.

All the samples analyzed were mixed with distilled water, shaken continuously for fifteen minutes and then at intervals afterwards. A few drops of formalin were added and the material allowed to stand over night. They were shaken again thoroughly before analysis next day, and the centrifuging was conducted as for condensed milk. Apparently there was a much larger amount of sediment in the tubes than in the condensed milks, and quantitative determination showed this to be so. See Table VII, also Fig. 2.

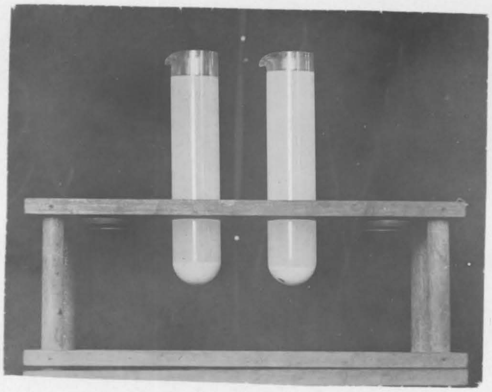
Milk Powder Sediment.

An analysis of the extracted sediment is given in Table VII. From this table it will be seen that the amount of sediment recovered was higher than that obtained from the evaporated and condensed milks.

From an inspection of the centrifugal tubes the amount seemed to be more than was actually recovered after washing.

Fig. 2.

Milk Powder.



Dilution one of powder to ten of water.

Table VII.

Milk Powder and Sediment.

Per Cent	Milk Powder	Sediment.
Sediment		0.77
Solids	93.21	100.00
Ash	7.92	12.10
" CaO	16.47	38.01
" MgO	3.06	2.53
" P_2O_5	26.11	18.96
" K_2O	28.28	9.81
" Na_2O	10.25	8.04

In the sediment the calcium oxid is high, the phosphorus somewhat lower than in the original material; the alkal s very low, while the ash as a whole is high.

PART II.

New Method Used For All Determinations.

All the previous work done up to this point was not perfectly satisfactory in analytical results or quantitative determinations, due mainly to the difficulties encountered in separating out the sediments, in washing these sediments, and in retaining all of the constituents in insoluble form when being washed. When dealing with the quantitative amount of sediment in milk powders, the amount visible in the sedimentation tubes was unquestionably far greater than that obtained after complete washing and decantation.

The difficulties encountered in securing a sediment that was free from all soluble constituent (see work done on sugar determinations) and also the fact that certain constituents seemed to floculate when treated with repeated washings of water have been noted previously.

These difficulties suggested the use of a method in which it would not be necessary to separate the sediment to obtain the desired results, hence analysis of the serum was tried to see if the results would not be more definite.

This new method naturally divided itself into two parts as did that previously mentioned, but instead of endeavoring to separate out the sediment, the serum, which contained the soluble constituents and the fat, was analysed and the difference between the amount of total consitutents found in the original substance and that in the serum, was taken as representing the amount that remained insoluble.

The sugar, being entirely soluble, was not determined, but the total solids, fat, total protein, total ash, and the individual ash constituent were all determined, since it was necessary to find out what rearrangement, if any, had taken place between these constituents.

Analysis of Original Substance.

The analysis of the original material was carried out for all the constituents. This was regular in every respect, except that the sample for total solids had to be quite small so that evaporation would not be delayed. With large samples of sweetened condensed milk, it is almost impossible to obtain constant weight, due probably to the early oxidation of the sugars. The fats were determined by the Roesse-Gottleib method, two extractions. In dealing with the problem, the fat had to be eliminated from the calculation, for it could hardly be held in the sediment in any appreciable quantity. The solids, not fat, therefore formed a convenient basis on which to work, and was used as such for making up solutions.

Analysis of the Serum:

A variation in the density of all the solutions would cause a variation in the ability of a definite force, either gravitational or centrifugal, to separate out the sediment from the serous portions, and since the accuracy of the results depended on a complete separation, all solutions were made up with as near the same percentage of solids, not fat, as possible. The amount of dilution necessary to give the standard of normal milk containing 9% SnF was used.

One other standardizing condition must be noted also in connection with the length of time samples were taken after solution was effected. This time was about 24 hours afterward and was limited, because of the floculating effect noted previously.

<u>Kinds of Milks Analyzed</u>:

There were three kinds of milk products used in the former work, namely, unsweetened condensed milk, sweetened milk, and milk powder, and these were used in the second part also. The brands of unsweetened condensed milk used were "Pet" and "Van Camps"; the sweetened "Bordens" and a so called skim milk powder.

At the very beginning of the new method of procedure several difficulties were encountered, which may be explained as follow:

First, the material was weighed and made up to a definite volume (V) with distilled water. Some of this material did not go into solution, and the volume of the serum minus the volume of the insoluble portion had to be determined, so that from a portion of the serum the total constituents contained in it could be found. If V represents the volume of the sediment then the volume of the serum (S_V) would be expressed by the formula

$$S_V = V - V_1$$

But the derivation of any formula for a case of this kind, dealing with volumes, is extremely difficult, seeing that the unknown specific gravity of the sediment must be determined and that the relationship between the volumes of the soluble and insoluble parts of complex mistures in partial solution is indeterminate.

That the problem had to be solved was certain, for the error introduced by taking the full volume V as S_v was so great that V_1 would, in some cases be zero, thus leaving no sediment whatever by calculation.

It was found that by substituting weight, throughout the whole procedure, for volume, the difficulty admitted of solution by easy mathematical methods.

The formulae may be derived as follows:

Let W = Total weight of mixture in grams.

W_1 = Weight of solids put in mixture.

W_{11} = Weight of solids in 1 gram of serum.

X = Weight of serum in grams.

Then $W - X$ = Weight of sediment in grams.

And $W_1 - W_{11}X$ = " " " " "

Therefore $W - X = W_1 - W_{11}X$

$$X = \frac{W - W_1}{1 - W_{11}}$$

The second difficulty that arises is that fat forms part of the original solids. On diluting and centrifuging, part of the fat rises to the top, and for reasons that are evident, cannot be included wholly in the serum analysis, and part is left in the serum. So $W - X$ (2) cannot be all sediment, but is sediment plus the fat that rises. So also is $W_{\prime} - W_{\prime\prime}X$ (3). From this it is apparent that the amount of fat that rises must be determined and subtracted from $W_{\prime} - W_{\prime\prime}X$ to get the grams of true sediment.

Let p = per cent fat in the solids of original material.

$p_{\prime}$ = per cent fat in the serum

y = grams of fat risen

S = grams of true sediment.

Then $$\frac{pW_{\prime}}{100} = \text{total grams fat} \qquad (4)$$

And $$\frac{p_{\prime}X}{100} + y = \text{total grams fat}$$

$$\frac{pW}{100} = \frac{p_{\prime}X}{100} + y.$$

$$y = \frac{pW_{\prime} - p_{\prime}W}{100}$$

Therefore $$S = W_{\prime} - W_{\prime\prime}\frac{W - W_{\prime}}{1 - W_{\prime\prime}} - \frac{pW_{\prime} - p_{\prime}\frac{W - W_{\prime}}{1 - W_{\prime\prime}}}{100}$$

Or $$S = W - \frac{W - W_{\prime}}{1 - W_{\prime\prime}} - \text{(ditto)}$$

The third problem that arose in connection with the calculation of results was in the determination of W, the total weight of the mixture in grams. This could have been done by direct weighing but much of the analytical work had been completed before it was found that weight had to be substituted for volume. The mixture had been made up to definite volumes and aliquot portions taken for analysis. To find W from V, all that is necessary is to obtain the specific gravity of the solids put in, then from their weight calculate their volume, subtract this from V, and thus secure the c c of water in V, which may be taken as the grams of water. The grams of water plus the grams of the solids put in, gives the weight of V, or W. The specific gravity of milk solids may be determined when the specific gravity and the per cent solids of the milk are known.

s = specific gravity of milk
t = per cent solids
S = specific gravity of solids.

In 100 grams of milk

$$\frac{100}{s} = \text{c c of milk in 100 grams.}$$

$$\frac{100}{s} - \frac{t}{S} = \text{c c of water in 100 grams}$$

$$100 - \frac{100}{s} - \frac{t}{s} = t$$

$$S = \frac{t}{\frac{100}{s} \quad t - 100}$$

Applying this formula to the case of skim milk powder which was used in the analysis following, the per cent of solids and specific gravity of skim milk are fairly constant being 9 and 1.036 respectively. The specific gravity of the solids (S) is therefore 1.63. From this it is seen that the volume of the solids is 5.52 cc, and hence in 100 c c of milk there would be 94.48 grams of water, and the 100 c c would weigh 103.48 grams.[1]

Milk Powder.

The analysis for total constituents in milk powder was made as outlined. The results are given in Table VIII as grams per 100 of the original substance.

After this was completed the solution for serum analysis was made up by weighing out enough of the powder to make a solution with 9 per cent solids. The powder contained 6.79 per cent moisture, therefore 9.65 grams were put in for every 100 c c made up. The weight per 100 c c of this mixture was 103.48 gms.

The solution stood four hours at room temperature and then the serum was drawn off and centrifuged for 15 minutes. After carefully drawing off a portion of the serum from the centrifugal tubes, a small but quite definite sediment was thrown down with further centrifuging, showing that some of the finest particles of sediment remained in suspension for a very long time. Hence the serum was allowed to stand 24 hours and was re-centrifuged to extract the last traces of sediment. 100 c c of this clari-

[1] Due to volume change with partial solution, this is not strictly correct with milk powder.

fied serum was taken for analysis.

The results for milk powder, both original substance and serum obtained therefrom, are shown in Table VIII. It will be noted that the per cent of sediment is very high, being 14.55 of the total solids in the original substance, or 13.57 per cent of the original substance. Most noteworthy, however, are the results obtained for ash and ash constituents. The per cent of ash in the sediment was double that in the original substance; the per cent of calcium oxid almost three times as much; that of magnesium oxid seven times, and the phosphorus three times. On the other hand the potash decreased to about one half, and the soda remained about the same.

It is evident that the results show a rearrangement in ash constituents. The fact that the ash content of the sediment is high points to a change in its constituents, and the per cent of change in each ash constituent verifies the total change.

The calcium, magnesium and phosphorus seem to be chiefly concerned in the change from soluble to insoluble condition. Just what the nature of this change is, can only be conjectured, but it would seem that it is a combination of calcium, magnesium, phosphates and carbonates, and that the small amount of original magnesium was reduced fifty per cent to produce these insoluble compounds.

As would naturally be expected the soluble potash was reduced in the sediment. Further, in Table IX the effect of making up the solution to 140° Fahr. reduces the alkali content of the ash to almost nothing.

The protein and ash together make up practically all the sediment. It would seem that this protein consists of all the albumin and a small

1. Previous paper "Detection of Condensed Milk in Ice Cream."

amount of casein. The precipitation of this small amount of casein may be caused by the lactic acid concentration which occurs in condensing.

Tables 8 to 17 show the results of analyses of powdered milk at 60° and 140° Fahr.; of five samples of evaporated milk and three samples of sweetened condensed milk. A summary of these tables is given in tables 18, 19, and 20.

Table 18 deals with the amounts of each constituent that were found to be soluble, and the figures given are the grams of each constituent that were soluble for every 100 grams of the constituent put in the original mixture.

Table 19 gives the number of parts insoluble for every 100 parts of the constituent put in the original solution. This represents the sediment.

Solids. The milk powder sample had the greatest amount of insoluble total solids or sediment. The sample of sweetened condensed milk that gave the high per cent of sediment had some decomposed lumps in it. Disregarding this analysis, the amount of sediment in all the evaporated and condensed milk samples was practically the same.

Table VIII.

Milk Powder at 60° Fahr.

	Water	Solids	Protein	Ash.	Ash Constituents.				
					CaO	MgO	P_2O_5	K_2O	Na_2O
Per cent of Constituents on original Substance	6.79	91.21	32.20	8.04	1.7929	0.2460	2.0992	2.2737	0.8241
Grams of Constituents in 100 c c Mixture		9.00	3.02	0.7758	1.1730	0.0237	0.2026	0.2194	0.0795
Grams of Constituents soluble in 100 c c Serum		7.69	1.89	0 5639	0.1105	0.0119	0.1242	0.1990	0.0645
Per cent of Constituents soluble in 100 c c serum		85.45	62.22	73 21	63.9	50.21	61.30	90.70	81.1
Grams of Constituents insoluble in 100 c c Serum.		1.31	1.13	0.2119	0.0625	0.0118	0.0784	0.0204	0.0150
Per cent of Constituents insoluble in 100 c c Serum		14.55	37.78	27.31	36.1	49.79	38.70	9.30	18.9
					Per cent of Ash.				
Per cent of Constituents in Insoluble substances.		100.00	86.2	16.2	29.5	5.6	37.0	9.6	7.1

Table X

Evaporated Milk.

	Solids not fat.	Protein	Fat.	Ash	Ash Constituents.				
					CaO	MgO	P_2O_5	K_2O	Na_2O
					Per cent of ash				
Per cent Constituents in original substance.	23.68	6.56	4.77	1.73	23.02	2.82	26.64	27.41	7.98
Grams of Constituents in 100 c c of mixture.	10.72	2.9375		0.7842	0.1800	0.0221	0.2088	0.2149	0.0628
Grams of Constituents soluble in 100 c c serum.	10.5440	2.8705		0.7622	0.172	0.0216	0.2053	0.2090	0.0600
Per cent of the Constituents soluble in 100 c c.	98.4	97.7		97.2	95.5	97.7	98.3	97.2	95.8
Grams insoluble in 100 c c	0.1760	0.0670		0.022	0.008	0.0005	0.0035	0.0039	0.0026
Per cent insoluble in 100 c c.	1.6	2.3		2.8	4.5	2.3	1.7	2.8	4.2
					Per cent of ash				
Per cent of Constituents in sediment.	100.00	38.1		12.5	36.36	2.27	15.9	17.7	11.8

Table XI.

Evaporated Milk.

	Solids not fat	Protein	Fat	Ash	Ash Constituents				
					CaO	MgO	P_2O_5	K_2O	Na_2O
					Per cent of Ash.				
Per cent of Constituents in original substance	20.86	7.14	6.45	1.702	21.09	2.90	28.08	26.6	8.62
Grams of Constituents in 100 c c of misture	9.7554	3.3400	1.86	0.767	0.1678	0.0231	0.2234	0.2117	0.0686
Grams of Constituents soluble in 100 c c of serum	9.6195	3.2429		0.717	0.1470	0.0187	0.2158	0.2007	0.0636
Per cent of Constituents soluble in 100 c c serum	98.6	97.1		93.4	86.6	80.9	96.6	94.8	92.7
Grams of Constituents insoluble in 100 c c of serum	0.1359	0.0971		0.0500	0.0208	0.0044	0.0076	0.011	0.005
Per cent of Constituents insoluble in 100 c c of serum	1.4	2.9		6.6	12.4	19.1	3.4	5.2	7.3
					Per cent of Ash.				
Per cent of Constitutent in sediment	100.00	71.4		36.7	41.6	8.8	15.2	22.0	10.0

Table XII

Condensed Milk Sweetened

	Solids not fat.	Protein	Fat	Ash	Ash Constituents				
					CaO	MgO	P_2O_5	K_2O	Na_2O
					Per Cent of Ash				
Per cent of Constituents in original Substance	71.66	7.51	8.35	2.18	20.82	2.91	25.90	26.52	9.05
Grams of Constitutents in 100 c c of mixture	9.91	1.9502		.5638	0.1130	0.0164	0.1465	0.1495	0.0510
Grams of Constituents Soluble in 100 c c of Serum	8.5389	1.7859		.4593	0.0860	0.0108	0.1198	0.1204	0.0401
Per cent of Constituents soluble in 100 c c of serum	85.1	86.4		81.5	76.1	65.8	81.8	80.5	80.0
Grams of Constituents insoluble in 100 c c of serum	0.3711	0.1643		.1045	.0270	0.0056	0.0267	0.0291	0.0109
Per cent of Constituents insoluble in 100 c c of serum	13.9	13.6		18.5	23.9	34.2	18.2	19.5	20.00
					Per Cent of Ash.				
Per cent of Constituents in sediment	100.0	44.3		28.2	26.8	5.36	25.4	27.8	10.4

Table XIII.

Evaporat ed Milk.

	Solids not fat	Protein	Fat	Ash.	Ash Constituents.				
					CaO	MgO	P_2O_5	K_2O	Na_2O
					Per cent of Ash				
Per cent of Constituents in original substance	21.60	6.99	7.28	1.68	27.2		27.7	28.49	10.7
Grams of Constituents in 100 c c of Mixture	10.92	3.54		0.8495	0.3192		0.2353	0.2420	0.0909
Grams of Constituents soluble in 100 c c serum	10.4634	3.294		0.8243	0.3087		0.2249	0.2365	
Per cent of Constituents soluble in 100 c c of serum	9.58	93.0		97.	.97		95.5	97.6	
Grams of Constituents insoluble in 100 c c serum	0.4566	0.246		0.0252	0.0105		0.0104	0.0055	
Per cent of Constituents insoluble in 100 c c serum	4.2	7.0		3.0	3.0		4.5	2.4	
					Per cent of ash				
Per cent of Constituents in sediment	100.0	53.9		5.5	41.6		41.5	21.8	

Table XIV.

Sweetened Condensed Milk

	Solids not fat	Protein	Fat	ash	Ash Constituents.				
					C_aO	M_gO	P_2O_5	K_2O	Na_2O
Per cent of Constituents in original substance.	66.34	7.95	8.14	8.86	24.54	1.55	27.75	24.56	16.42
Grams of Constituents in 100 c c of mixture	17.9959	2.1582		0.5047	0.1238	0.0083	0.1400	0.1239	0.0421
Grams of Constituents soluble in 100 c c of serum	17.3222	2.1054		0.4239	0.0990	0.0078	0.1220	#	0.0413
Per cent of Constituents soluble in 100 c c of serum	96.2	97.5		84.0	79.9	94.0	87.1		98.1
Grams of Constituents insoluble in 100 c c of serum	0.6239 *	0.0528		0.0808	0.0248	0.0005	0.0180		0.0008
Per cent of Constituents insoluble in 100 c c of serum	3.8	2.5		16.0	20.1	6.0	12.9		1.9
					Per Cent of Ash.				
Per cent of Constituents in sediment	100.0	8.5		12.0	30.7	0.6	22.3		1.0

* Owing to high sucrose, the solids determination

Results negative.

Table XV.

Evaporated Milk

	Solids not fat	Protein	Fat	Ash	CaO	MgO	P_2O_5	K_2O	Na_2O
					Per Cent of Ash.				
Per cent of Constituents in original substance	18.45	6.74	7.26	1.57	14.6		28.5	26.5	9.72
Grams of Constituents in 100 c c of mixture	8.89	3.2477		0.8100	0.1276		0.2142	0.1992	
Grams of Constituents soluble in 100 c c of serum	8.7475	3.1500		0.7754	0.1150		0.1991	0.1960	*
Per cent of Constituents soluble in 100 c c of serum	98.4	96.7		95.7	90.1		93.0	98,4	
Grams of Constituents insoluble in 100 c c of serum	0.1425	0.0977		0.0346	0.0126		0.0151	0.0032	*
Per cent of Constituents insoluble in 100 c c of serum	1.6	3.3		4.3	9.9		7.0	1.6	
					Per cent of Ash.				
Per cent of Constituents in sediment	100.0	68.6		24.3	36.4		43.6	9.2	

* No results.
Note-Per cent CaO is low in original as well as sed.

Table XVI.

Evaporated Milk.

	Solids not fat.	Protein	Fat.	Ash.	CaO	MgO	P_2O_5	K_2O	Na_2O
					Per Cent of Ash.				
Per cent of Constituents In original sample	18.17	6.26	6.75	1.585	21.79	.	28.32	33.48*	*5.38
Grams of Constituents in 100 c c of mixture	10.152	3.4172	.	0.8661	0.1887	.	0.2453	0.2899	
Grams of Constituents soluble in 100 c c of serum	9.9266	3.3227	.	0.7748	0.1506	.	0.2011	0.2560	
Per Cent of the Constituents soluble in 100 cc serum	97.7	97.3	.	87.4	80.0	.	82.0	88.4	
Grams of the Constituents insoluble in 100 c c of serum	0.2254	0.0945	.	0.0913	0.0381		0.0442	0.0339	
Per Cent of the Constituents insoluble in 100 c c serum	2.3	2.7	.	12.6	20.0	.	18.0	11.6	
					Per cent of Ash				
Per Cent of Constituents in Sediment	100.0	41.9	.	40.5	41.7	.	48.4	37.5	

Table XVII.

Sweetened Condensed Milk

	Solids not fat.	Protein	Fat	Ash	Ash Constituents				
					CaO	MgO	P_2O_5	K_2O	Na_2O
					Per cent of ash				
Per cent of Constituents in original sample	67.84 **	8.1	9.76	1.986	20.52	.	28.27	24.89	14.3
Grams of Constituents in 100 c c of mixture	16.4908	1.9333		0.4821	0.0991	.	0.1365	o.1202	0.0690
Grams of Constituents soluble in 100 c c of serum	16.0982	1.7800		0.4340	0.0837	.	0.1261	0.1169	0.0546
Per cent of the Constituents in serum.	97.6	79.3		90.0	84.4	.	92.4	97.2	79.1
Grams of Constituents insoluble in 100 c c of serum	0.3926	0.1533		0.o481	.0154	.	o.0104	0.0033	0.0144
Per cent of the Constituents insoluble in 100 cc serum.	2.4	20.7		10.0	15.6	.	7.6	2.8	20.9
					Per cent of ash.				
Per cent of Constituents in sediment	100.0	39.0		12.3	32.0	.	21.6	6.8	29.9

* sucrose included

Table XVIII

Summary of Soluble Constituents.

Parts per 100 soluble in 100 c c.

	Solids	Protein	Ash	CaO	MgO	P_2O_5	K_2O	Na_2O
Milk Powder.	85.45	62.22	73.21	63.9	50.21	61.30	90.70	81.1
Evaporated Milk	98.4	97.7	97.2	95.5	97/7	98.2	97.2	95.8
"	95.8	93.0	97.0	97.0		95.5	97.6	
Evaporated Milk	98.6	97.1	93.4	86.6	80.9	96.6	94.8	92.7
"	98.4	96.7	95.7	90.1		93.0	98.4	
	97.7	97.3	87.4	80.0		82.0	88.4	
Condensed Milk	86.1	86.4	81.5	76.1	65.8	81.8	80.5	80.0
"	96.2	97.5	84.0	79.9	94.0	87.1		98.1
"	97.6	79.3	90.0	84.4		92.4	97.2	79.1

Table XIX.

Summary of Insoluble Constituents

Parts per 100 insoluble in 100 c c.

	Solids	Protein	Ash	CaO	MgO	P_2O_5	K_2O	Na_2O
Milk Powder	14.55	37.78	27.51	36.1	49 79	38.70	9.30	18.9
Evaporated Milk	1.6	2.3	2.8	4.5	2.3	1.8	2.8	4.2
" "	4.2	7.0	3.0	3.0		4.5	2.6	
Evaporated Milk	1.4	2.9	6.6	12.4	19.1	3.4	5.2	7.3
" "	1.6	3.3	4.3	9.9		7.0	1.6	
" "	2.3	2.7	12.6	20.0		18.0	11.6	
Sweetened Condensed	13.9	13.6	18.3	23.9	34.2	18.2	19.5	20.0
	3.8	2.5	16.0	20.1	6.0	12.9		1.9
	2.4	20.7	10.0	15.6		21.6	6.8	29.9

Table XX

Summary of Per Cent Constituents in Insoluble Material

	No.	Protein	Ash	CaO	MgO	P_2O_5	K_2O	Na_2O
Milk Powder	VIII	86.2	16.	29.0	5.6	37.0	9.6	7.1
		(75.3)	(24.7)					
Evaporated Milk	X	38.1	12.5	36.36	2.27	16.4	17.7	11.8
		(90.7)	(9.3)					
	XIII	53.9	5.5	41.6		41.5	21.8	
		(73.8)	(26.2)					
Evaporated Milk	XI	71.4	36.7	41.6	8.8	15.2	22.0	10.0
		(73.8)	(26.2)					
	XV	68.6	24.3	36.4		43.6	9.2	
		(51.4)	(48.6)					
	XVI	41.9	40.5	41.7		48.4	37.1	
		(61.1)	(38.9)					
Sweetened Condensed Milk	XII	44.3	28.2	26.8	5.36	25.5	27.8	10.4
	XIV	8.5	12.0	30.7		22.3		1.0
		(78.0)	(22.0)					
	XVII	39.0	12.3	32.0		21.6	6.8	29.9

Figures in parenthesis represent per cent, when protein plus ash equals total insoluble material.

Conclusions.

Insoluble sediments are contained in all the samples of evaporated milk, sweetened condensed milk, and milk made from powdered milk solids that were examined.

The amount of sediment was greates in the milk powder milk, being over 13 per cent of the original substance. With the sweetened condensed milk it averaged 6.6 per cent, and 2.2 per cent with evaporated milk.

The sediments from evaporated milk were coarse and granular. The coarse material had a high per cent of calcium.

Granulation occurred in the milks of the least density. The granular particles are the result of recrystallization, and this takes place more quickly in fluids of low density.

The sediments are characteristic of highly heated and condensed milk products, and their presence in milk would show that condensed milk or powdered milk had been used.

The protein probably forms from one-half to two-thirds of this sediment.

The results of the calcium, phosphorus and magnesium determinations indicate that insoluble compounds are formed with these constituents.

Some of the calcium is evidently in the form of carbonates and phosphates, since effervescence took place on addition of an acid to the granular sediment, and phosphorus was shown to be in the sediment in greater proportion than normal.