Commercial Casein
Michigan State University Agricultural Experiment Station
Technical Bulletin
A.C. Weimar, John Taylor, Dairy
Issued June 1927
15 pages

Oakley Lardie

Technical Bulletin No. 82 June, 1927

COMMERCIAL CASEIN

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FOREWORD

The problem of utilization of skim milk during certain seasons of the year when dairy plants have a surplus of this product has absorbed the attention of practically every operator of a dairy plant. The reason for this study of methods of manufacture of commercial casein was augmented by the recent increase in the tariff on this product. According to the trials reported here, as well as the results obtained in commercial practices followed in the College Creamery, casein manufacture does not offer an especially profitable means of skim milk disposal, but, as between allowing the skim milk to be wasted and manufacturing it into casein, the latter is to be highly recommended. Casein manufacture can be especially recommended to Michigan dairy plants because of the available market for it in the state in the manufacture of paper and furniture.

O. E. Reed.

COMMERCIAL CASEIN

A. C. WEIMAR AND JOHN TAYLOR

The utilization of skim milk is fast becoming a serious problem for the average dairy products manufacturer, and its economic importance cannot very well be overlooked. The market for skim milk products has not been developed to the point of complete utilization of surplus, and, though these products are being absorbed by the market in increasing amounts, the total wasted each year is very high.

The small and medium sized plants in possession of a surplus of skim milk at certain times of the year are usually equipped to manufacture one or more of the following products: skim milk cheese; powdered skim milk; sweetened, plain, or superheated condensed skim milk; commercial buttermilk; or casein with perhaps the further by-products of milk sugar and albumen. Unless the surplus is consistently large, the equipment will be limited to the manufacture of one or two of the simplest above named products. As market conditions fluctuate, the smaller plants are not equipped to turn their surplus into more profitable channels, with the result that skim milk is wasted. The larger plants, being well equipped, can divert their surplus into any of the above channels and, by careful analysis of market conditions, are prepared to turn to the manufacture of the product that at the time, is the more profitable.

When a suitable market can be found for skim milk products, there is no cause for waste, but when the market is not satisfactory there is one by-product that can be made to relieve the situation. This by-product is casein. There are several very large plants which prefer the manufacture of casein, not alone for the profit in it, but for the further by-products of lactose and albumen. The average size plant does not have the volume of skim milk, however, to warrant venturing into the making of the milk sugar and albumen.

The cost of equipment for casein manufacture is within the means of the average size plant and it can remain idle at less expense than the equipment for any other product. This does not mean that casein is the last product to resort to in the utilization of skim milk, nor does it mean that in all cases where a surplus of skim milk exists, casein should be made. If no other means of utilizing the skim milk is available, the profit realized from it when made into casein should be reckoned as that amount remaining after deducting from the manufacturing costs the selling price of the casein. However, the increasing demand for casein and the unsteady supply has often raised the price to a point where skim milk is worth more than thirty cents per hundred after deducting manufacturing costs. Spasmodic attempts to make this product have been harmful to the interests of domestic casein. The great variation in methods of making by inexperienced men has resulted in a lack of uniformity and quality.

Unfortunately, the casein manufacturers of the United States have been unable to agree upon a uniform method of manufacture, with the result that there is a wide range of quality. Casein buyers found their efforts to purchase American casein of uniform quality rather discouraging, and have, therefore, sought elsewhere for their supply. Argentine casein has met their demand for quality as well as quantity and uniformity. A protective tariff has not affected imports. The records of the U. S. Department of Commerce show that the United States took 66 per cent of their 1921 supply, 88 per cent of that of 1922, and during the first six months of 1923, 81 per cent of their supply.

The practical value of casein depends upon its solubility, adhesiveness, and general working qualities. Casein of desired quality should dissolve with certain limits of alkalinity, giving a clear, uniform solution free from any form of sediment, have a fairly heavy body, and possess good adhesive qualities. The general working properties of the dissolved casein are determined by its viscosity and spreading qualities. Any factor that has an influence upon one of the requirements for making a good grade of casein will necessarily affect the quality of the final product.

Some of the factors controlling the quality, usefulness, and uniformity of casein are:

1. Coagulating reagent—There are three acids used in coagulating casein, namely: hydrochloric acid; sulfuric acid; and lactic acid, or natural souring. Rennet is also used in coagulating commercial casein but its use is not general.

2. Irregular grinding—Dried casein should be ground to pass through a twenty mesh sieve.

3. Presence of foreign particles which prohibit the complete solubility of casein.

4. Presence of burned casein particles—usually caused by excessive heat in the drying tunnel.

5. Irregular ash content—This defect is more or less important depending upon the purpose for which the casein is used. Manufacturers of glue or adhesives can not use casein of high ash content.

6. Irregular acid content—caused by an excess or an insufficient amount of acid or by the extent of washing.

7. Life of the casein—The length of time casein remains in solution before solidifying.

8. Marketing of green curd.
   (a) Distance to drying plant.
   (b) Temperature of wet curd during shipping.
   (c) Acidity of wet curd.
   (d) Condition of curd.

Experimental Work

In view of the fact that casein is manufactured by various processes, which produce a wide variation in quality and uniformity, this experimental work was planned to determine which of the commercial methods of manufacture produced the best quality and greatest uniformity under ordinary factory conditions. A secondary object was the study of the operation and results with a view to selecting arguments for and against each method of manufacture. During this work a few additional points of interest to the manufacturer were observed and the results have been added as supplements.

Review of Literature of American Casein

In 1919, there were six commercial methods of manufacturing casein, namely: Natural Sour, Hydrochloric, Sulphuric, Cooked Curd, Grain Curd, and Rennet. In reality four are represented since the cooked curd method is a modification of the Sulphuric, while the grain curd is a modification of the Hydrochloric. Rennet curd has its use in the making of plastic casein products and is not marketed as generally as the acid caseins because of its extremely high ash content and its inactivity.

During the World War the grain curd method was developed through the necessity for a low ash content in casein for glue purposes. Dr. Clark (1) found that by coagulating casein with hydrochloric acid at a pH of 4.6 and washing it well with water at pH 4.8, the curd produced was very porous and allowed free removal of the salts. His method was adopted by several large plants so that enough low ash casein could be made to furnish the demands created by the aviation board for aeroplane production. Following the war this method was found to be too technical for ordinary usages, and, though the old methods were again used, some of the steps in the making of grain curd were retained. The necessity of washing the curd two or more times is perhaps the outstanding improvement over the original methods. Another improvement adopted from the grain curd method is the conditioning of the curd during and immediately after coagulation. More attention is now paid to the size of the curd particles after coagulation, conditions being regulated so as to produce smaller lumps which in turn allows the wash water to come in contact with more surface. Washing the curd not only removes much ash but also gives the casein a lighter color when dried. One of the largest manufacturers of casein in this country stated that the thorough washing of casein increased its value from 1.5 to 3.0 cents per pound.

Cooked curd, which is a modification of the sulphuric acid method, is produced by heating the curd in water until it gathers into a semisolid plastic mass, impermeable to air and water. In this form it can easily be molded into barrels and shipped in the wet or green condition to some central drying plant. This method eliminates expenditure for drying equipment.

The Sulphuric Acid Method

A wooden vat of suitable size is usually used. The skim milk is run in and heated to 115 to 125 degrees F. Commercial sulphuric acid with a specific gravity of 1.83 is added at the rate of one pint to 1,000 lbs. of milk. Before being added to the milk the acid must be diluted with ten parts of water. The usual precautions must be taken in diluting the acid if accident is to be avoided. The diluted acid is added slowly while the milk is constantly stirred until a clear separation occurs. This requires but a few minutes. If the curd does not separate clearly or produce a clear whey, more acid is added until the desired results are obtained.

The whey is then drained through the gate while the curd remains to be washed with cold water to remove the free acid and excess whey. After draining the wash water, the curd is placed in press cloths and put to press. These cloths are usually of heavy duck and the pressing period usually continues over night or until the curd is thoroughly free from excessive moisture and is dry and mealy in body.

The pressed curd is then run through the curd grinder and is spread evenly on to the drying trays. The thickness of the curd upon the tray, to insure rapid drying, should not be over 3/16 of an inch. The time required for proper drying depends upon three factors which are the condition of the curd, the uniformity of spreading on the trays, and the velocity and temperature of the air passing through the tunnel. A temperature of 125° to 130° F. has been found to give the best results. When properly dried the casein should not adhere to the trays and should crumble easily when removed. Exessively wet casein on the trays will form a hard brittle mass when dried. High drying temperatures will cause the color of the casein to darken. This is true, especially, when the casein contains fat or impurities that are the results of either poor raw materials or faulty manufacture. Grinding the dried curd should be done carefully. A uniform size of particles passing through a 20 mesh sieve is desired by buyers but the finer the casein is ground the lighter and more attractive the color becomes.

The finished casein is shipped to the market in large burlap sacks and in this form is sometimes stored for many months. Insects and rodents must be kept from it while in storage.

The average creamery may be fitted easily for casein manufacture by including the following equipment:

1. Press cloths (heavy duck or burlap),
2. Curd press with dividing boards,
3. Curd mill,
4. Drying trays with a few tray trucks,
5. Drying tunnel with blower and heater.

Hydrochloric Acid Method

The original hydrochloric acid method differs from the sulphuric only in the acid used for coagulation. Hydrochloric acid is more expensive and slightly more is needed for complete separation. Its use is limited to those manufacturers who utilize the whey for making lactose. The cheaper sulphuric acid forms a precipitate of calcium sulphate, which interferes with the filtering and purifying of milk sugar. Hydrochloric acid forms a soluble calcium salt which is removed during washing.

Natural Sour Method

Skim milk is run into a vat and usually allowed to set over night to curdle. Usually no starter is added. However, if the milk has been pasteurized a starter must be added. After firm coagulation the curd is cut with cheese knives, and is then heated to about 125 degrees F., or until it becomes very firm and brittle. The whey is drained off and from this step the pressing and drying are the same as that used in the sulphuric acid method.

Rennet Method

There are two distinct methods of manufacturing rennet casein. Much depends upon its use in the commerial form. As previously mentioned, rennet casein is used in the manufacture of plastic materials such as imitation ivory products. It is known as an inactive casein because it does not react as quickly with dilute alkalies or acids and requires more concentrated solutions to dissolve it. Its composition differs from that of the acid casein. Rennet casein exists as para casein, while acid caseins are in the form of calcium caseinates.

The cold method of manufacture is that of setting the milk at 90 degrees F. with enough rennet to coagulate it in 30 minutes. The curd is cut into half inch cubes and the whole heated quickly to 115 degrees or 120 degrees F. while the curd is stirred continuously to avoid matting. The whey will be expelled from the casein in a comparatively short time, and, as the curd becomes firm, the vat gate is opened to drain it, and the casein allowed to settled at the bottom. Cold water is then poured over the matted curd to retard the rennet action. The casein is then run through the curd mill and immediately put back into a vat of cold water. From this step the pressing and drying are identical to that of the sulphuric acid method.

The hot rennet method consists of setting the skim milk as in the cold method, cutting the curd very fine, and at the same time running the temperature up to 135 degrees. After holding at this temperature for a short period, the action of the rennet is completely stopped and at the same time the curd has become thoroughly dried. The draining, washing, pressing, and drying can be carried on as in the sulphuric acid method.

There are other methods of manufacturing casein but none of them are used on a large commercial scale. It is true that some casein is first manufactured in a crude form and then altered to meet requirements.

The four commercial methods of manufacture studied in the experimental work were the Hydrochloric Acid, Sulphuric Acid, Natural Sour, and Rennet.

Procedure

No definite amount of skim milk was available and therefore batches varied from 50 to 250 pounds. Each batch of milk was analyzed for total solids, specific gravity, and fat, using the Mojonnier machine and methods.

Eight batches were run in the first group using each of the four methods of making casein, namely: Hydrochloric Acid, Sulphuric Acid, Natural Sour, and Rennet, as before described. However, in order to obtain more accurate results in the making of acid caseins, the coagulation was controlled by using pH 4.6 as the stopping point in the adding or developing of acid. It was found that when the curd in the natural sour method was firm for cutting, its whey was very close to pH 4.6. The washing was accomplished by replacing the amount of whey drained off by an equal amount of water. The acid caseins were washed with water acidified to pH 4.8. This is necessary where well water is distinctly alkaline in order to keep the casein in a better condition for washing.

After washing and draining, the curd was put to press. Square pieces of heavy duck were used as press cloths. From the press the curd was broken up finely by hand and spread on drying trays. A cloth was placed under the last tray of each batch to catch any loose particles of curd. These trays were then placed in the drying tunnel and the curd allowed to dry over night by the blowing of air over it at a temperature of from 115 to 120 degrees F.

Each batch was then taken out, weighed and ground. A sample was taken to be analyzed for moisture, fat, and ash. To determine the moisture content, a two gram sample was heated to 100 degrees C. in a vacuum oven for thirty minutes. The loss in weight was recorded on a percentage basis as loss in moisture. To determine the fat content, a two gram sample was used and tested by the Mojonnier modification of the Roese-Gottlieb method. For ash, a one gram sample was slowly heated in a porcelain crucible. Later more heat was applied until all char disappeared. This required from 20 to 24 hours. The residue was calculated as ash.

In the second group, the batches of casein were made up from skim milk by each of the four methods and at the same time many batches were run on the same day. This was done to avoid errors and variations in the skim milk supply. Batches 9, 10, 11, 12, and 13 of the four methods were all from the same milk. Batches 14 to 22 inclusive of the Hydrochloric Acid and Rennet methods were from the same milk while the same numbered batches of the Sulphuric Acid and Natural Sour were from another milk. The data shows that all the skim milk used in the second group was practically identical in total solids and specific gravity and varied only .02 per cent in fat.

SECOND GROUP—RESULTS

The manufacturer is primarily interested in the yield he obtains, while the user of casein must pay particular attention to the fat, moisture, and ash it contains, and at the same time he must observe its purity and later its general working qualities. If the casein has a good appearance and the fat, moisture, and ash content are within reasonable limits, it can safely be bought for general commercial use. In drawing conclusions from the data, the yield, fat, moisture, ash, and general appearance were the points taken into consideration.

Table I.*—Showing yields and analyses of caseins by varying methods of manufacture.

BATCH	HCL									H_2SO_4								
	1	2	3	4	5	6	7	8	Ave	1	2	3	4	5	6	7	8	Ave
Amount of skimmilk	216.75	223	224	233	228	217	225	209	222.3	203.5	212.	208	182	72.	211.5	168.	166.5	177.7
Lactometer reading	34	35	34.5	34.5	35	33	33.5	35	34.5	33	33	34	34	34	35	33	34	33.7
Fat in skim	.06	.02	.10	.08	.05	.04	.05	.03	.066	.03	.03	.03	.03	.03	.06	.02	.03	.0325
% Total solids in skim	9.03	8.24	9.72	8.48	8.15	7.73	8.81	8.67	8.60	8.98	9.12	7.72	7.36	7.55	6.80	7.32	7.29	7.77
% Yield of casein	2.76	2.24	2.90	2.30	2.79	2.88	2.77	2.93	2.63	2.76	2.82	2.82	2.95	2.24	2.36	2.41	2.40	2.59
% Moisture in casein	7.53	4.66	4.41	4.75	4.06	4.39	5.37	5.16	5.04	7.01	6.07	8.11	8.07	7.70	4.55	4.83	4.67	6.37
% Ash in casein	4.95	2.54	2.23	2.04	2.86	2.92	3.26	3.75	3.07	1.97	3.46	1.23	2.0	2.53	1.42	1.34	1.94	1.98
% Fat in casein	3.47	2.25	2.33	2.88	1.76	1.01	.51	1.09	1.93	2.25	2.27	1.34	1.68	1.83	2.82	1.85	1.63	1.96
General Quality	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

BATCH	NATURAL SOUR									RENNET								
	1	2	3	4	5	6	7	8	Ave.	1	2	3	4	5	6	7	8	Ave.
Amount of skimmilk	167	210	226	193	210	224	220	209	207.3	212	210	221	219	231	220	197	238	218.5
Lactometer reading	35	33	33	32	31	34	33	33	32.7	32	34	33	33	32	33	33	33	33
Fat in skim	.04	.07	.05	.05	.07	.09	.07	.06	.118	.04	.05	.05	.05	.07	.06	.05	.07	.0556
% Total solids in skim	8.65	8.73	8.57	8.17	8.48	8.31	9.03	8.41	8.54	9.28	8.72	8.89	8.95	8.49	8.76	8.71	8.60	8.8
% Yield of casein	2.47	2.82	2.70	2.76	2.68	2.34	2.5	2.71	2.62	2.71	2.67	2.83	2.88	2.81	3.07	2.66	2.86	2.80
% Moisture in casein	4.37	4.76	4.36	5.05	5.00	5.38	5.89	5.92	5.99	6.41	7.0	6.75	6.70	6.39	6.85	6.50	7.59	6.77
% Ash in casein	2.30	1.28	1.55	1.35	—	1.63	1.69	1.43	1.60	8.31	8.54	8.37	8.57	8.53	8.46	8.52	8.16	8.34
% Fat in casein	1.54	1.66	5.32	2.34	2.10	2.19	2.11	2.28	2.02	.62	.32	.57	.92	.77	1.07	.21	.46	.61
General Quality	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

*The writers wish to acknowledge the valuable assistance and advice of Dr. E. J. Miller and Mr O. B. Winter of the Chemistry Section of the Experiment Station in making the analyses recorded in these tables.

Table II.—Showing yields and analyses of caseins by varying methods of manufacture.

METHOD	HCl.														
BATCH NO.	9	10	11	12	13	14	15	16	17	18	19	20	21	22	Ave.
Amount of skim	76	71	78	77	68	50	50	50	50	50	50	50	50	50	....
Lactometer Reading	33.4	—	—	—	—	33.4	—	—	—	—	—	—	—	—	....
Fat in skim	.04	—	—	—	—	.02	—	—	—	—	—	—	—	—	....
% Total solids in skim	8.79	—	—	—	—	8.92	—	—	—	—	—	—	—	—	....
% Casein yield	2.87	2.90	2.56	3.08	2.71	2.97	3.13	2.48	2.99	3.13	3.02	2.68	2.94	3.01	2.88
% Moisture in casein	7.19	7.18	7.06	8.95	6.39	6.38	5.91	6.02	6.34	6.18	5.81	6.14	6.42	6.11	6.57
% Ash in casein	2.67	2.19	2.37	2.80	2.03	2.18	2.38	2.49	1.84	2.23	2.42	2.21	2.09	2.17	2.29
% Fat in casein	1.05	1.44	1.40	.99	1.44	1.21	1.01	.97	.84	1.11	.89	.83	.94	.97	1.07
General quality	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....

METHOD	NATURAL SOUR														
BATCH NO.	9	10	11	12	13	14	15	16	17	18	19	20	21	22	Ave.
Amount of skim	67	69	75	61	70	60	60	60	60	60	60	60	60	60	....
Lactometer Reading	33.4	—	—	—	—	33.3	—	—	—	—	—	—	—	—	....
Fat in skim	.04	—	—	—	—	.03	—	—	—	—	—	—	—	—	—
% Total solids in skim	8.79	—	—	—	—	8.9	—	—	—	—	—	—	—	—	—
% Casein yield	2.89	2.99	2.75	3.28	3.21	2.99	3.02	3.1	2.99	3.01	3.03	3.12	3.28	3.21	3.06
% Moisture in casein	6.38	5.77	6.43	5.62	6.30	5.91	5.77	5.43	5.91	6.02	5.71	5.84	5.69	5.97	6.90
% Ash in casein	2.35	2.37	2.46	2.45	2.21	1.98	2.14	2.17	2.15	2.21	2,05	1.94	2.11	2.17	2.19
% Fat in casein	1.53	2.71	1.88	2.40	2.53	1.35	1.88	1.22	1.38	1.41	1.28	1.53	1.71	1.80	1.75
General quality	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

Table II.—Continued

METHOD	H_2SO_4														
BATCH NO.	9	10	11	12	13	14	15	16	17	18	19	20	21	22	Ave.
Amount of skim	71	74	77	76	56	60	60	60	60	60	60	60	60	60	....
Lactometer Reading	33.4	—	—	—	—	33.3	—	—	—	—	—	—	—	—	....
Fat in skim	.04	—	—	—	—	.03	—	—	—	—	—	—	—	—	....
% Total solids in skim	8.79	—	—	—	—	8.9	—	—	—	—	—	—	—	—	—
% Casein yield	2.73	2.70	2.68	2.63	3.23	3.03	2.79	2.84	2.91	2.68	2.71	3.01	3.0	2.84	2.84
% Moisture in casein	6.41	5.43	5.98	5.46	5.51	5.50	5.43	5.21	5.09	5.41	5.58	5.37	5.60	5.48	5.53
% Ash in casein	2.59	1.71	2.16	2.55	1.97	2.55	2.16	2.59	1.98	2.53	2.45	2.91	2.19	2.38	2.34
% Fat in casein	2.03	2.04	2.11	1.35	1.22	1.23	1.31	1.11	1.17	1.31	1.28	1.16	1.24	1.28	1.42
General quality	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....

METHOD	RENNET														
BATCH NO.	9	10	11	12	13	14	15	16	17	18	19	10	21	22	Ave.
Amount of skim	76	69	74	74	77	50	50	50	50	50	50	50	50	50	....
Lactometer Reading	33.4	—	—	—	—	33.4	—	—	—	—	—	—	—	—	....
Fat in skim	.04	—	—	—	—	.02	—	—	—	—	—	—	—	—	....
% Total solids in skim	8.79	—	—	—	—	8.92	—	—	—	—	—	—	—	—	....
% Casein yield	2.82	3.31	2.87	2.78	3.00	3.23	3.12	3.19	3.01	3.14	3.18	3.14	3.01	3.19	3.07
% Moisture in casein	7.19	7.88	7.90	7.30	6.90	7.30	6.91	6.94	7.43	7.31	7.88	7.19	7.14	7.09	7.31
% Ash in casein	8.43	8.33	8.27	8.56	9.60	6.91	7.43	8.01	6.39	7.27	7.74	7.19	7.15	7.48	7.97
% Fat in casein	.61	.60	.54	.34	.40	.71	.64	.78	.48	.53	.74	.61	.67	.66	.59
General quality	....	....	....	....	....	....	....	....	....	....	....	....	....	....	....

The Yield of Casein in Table I

It will be noticed that the yields by all the methods used are below normal. This is due to the fact that the skim milk used was below normal in solids. The difference or variation in the yield of the four methods was found to be very small. The highest yield was obtained by the rennet method. Hydrochloric acid was second, natural sour third, and sulphuric lowest.

The Yield of Casein in Table II

When using milk normal in solids and fat somewhat different results were obtained. Again the averages are not especially far apart. Rennet yield is the highest, natural sour second, hydrochloric acid third, and sulphuric acid fourth.

Fat in Casein

In both table I and II the fat content follows in definite order. This is interesting from the users' standpoint because high fat content in casein is very undesirable in the manufacture of glues, adhesives, and paints. Rennet casein contained the lowest amount of fat, hydrochloric acid second, sulphuric acid third, and natural sour the highest per cent.

During analysis it was discovered that the analytical method adopted during the World War for determining the fat in casein was far from accurate. It was found that the smaller the particles of casein the more fat could be extracted. The Air Craft Board method, adopted in 1918, is as follows: A five gram sample is extracted with ether for sixteen hours in either a Caldwell or Soxhlet extractor. The receiver is dried for thirty minutes at 89 degrees C. before weighing.

This method was discarded for the Roese-Gottlieb which Shaw suggested would be more accurate. The analyzing of five samples of casein taken at random from different lots yielded some positive results. The casein was shaken through a set of sieves of 60, 80, 100, 150, and 200 mesh and analyzed for fat using the ether extraction method. The original samples were analyzed by the Roese-Gottlieb method. Table III shows the results.

Table III.—Showing fat content of caseins by two methods of determination.

Method of Analysis	Kind	Ether Extractiom					Roese Gottlieb
Mesh		60	80	100	150	200	20
Sample I	HCL	.68	.71	.94	1.25	1.98	2.14
Sample II	NS	1.01	1.21	1.64	1.71	2.10	2.09
Sample III	H_2SO_4	.91	1.02	1.34	1.66	1.87	1.94
Sample IV	Rennet	.64	.71	.78	.94	1.49	1.61
Sample V	NS	.89	1.01	1.34	1.65	1.93	2.07
Average		.826	.932	1.208	1.442	1.874	1.97

Moisture in Casein

In Table I the hydrochloric acid method produced casein with the lowest moisture while rennet casein was highest.

In Table II, however, sulphuric acid casein was lowest in moisture while rennet casein was again highest. But little significance can be attached to moisture in casein unless it is excessively high or low.

Ash in Casein

The per cent of ash in casein proved to be in favor of the natural sour method. It was consistently low in Table I and II while the ash in rennet casein naturally was the highest. Hydrochloric and sulphuric acid caseins contained no great variation in ash although the results are slightly in favor of the sulphuric acid.

A variation too great for experimental work was found in the skim-milk used in obtaining the results for Table I. This was due perhaps to the fat separation in the milk and the amount of water used in flushing the separator. Some variation may have been due to changes in the milk. It is for this reason that the work, recorded in Table II was planned.

Method of Stirring At Time of Coagulation

It was found necessary to check up on the method of agitation during the coagulation of acid casein due to the fact that the yield seemed to fluctuate too greatly. An experiment was planned to determine if possible the effect of varying degrees of stirring on the yield as well as ash and moisture of the casein. Five lots of skim milk were divided into six portions each. Sulphuric and Hydrochloric acids were used as coagulating reagents. A special arrangement was made to assure uniform addition of acid to the milk during each coagulation.

Table IV.—Showing the effect of stirring on yield, ash, and moisture content.

BATCH NUMBER	YIELD					(ave.)	ASH					(ave.)	MOISTURE					(ave.)
One complete stir in 10 seconds	3.18	3.14	3.07	3.10	3.16	3.13	3.94	3.25	3.64	3.88	3.71	3.68	6.18	7.41	6.90	5.10	7.64	6.64
One complete stir in 4 seconds	3.11	3.15	3.06	3.12	3.12	3.11	3.89	3.95	3.51	3.28	3.75	3.67	5.78	6.40	7.90	5.64	7.01	6.72
One complete stir in 2 seconds	3.13	3.15	3.17	3.03	3.15	3.12	3.14	3.66	3.50	3.19	3.48	3.39	6.52	6.47	6.75	7.09	6.13	6.59
One complete stir in 1 second	3.01	2.97	3.12	3.04	3.09	3.04	3.01	3.28	3.21	2.97	2.77	3.04	6.80	7.64	5.51	6.42	7.42	6.73
One complete stir in ¾ second	2.59	2.84	2.67	2.88	2.74	2.74	2.78	2.69	2.74	2.67	2.46	2.67	5.49	5.94	7.10	6.91	4.85	6.03
One complete stir very fast	1.91	2.45	2.37	2.49	2.41	2.32	2.22	2.19	2.44	2.10	2.00	2.19	6.48	7.61	5.60	7.70	5.87	6.65
Kind of acid used	H_2SO_4	HCL	H_2SO_4	HCL	H_2SO_4	......	......	......	......	......	......	......	......	......	......	......	......	......

The stirring was continued until complete separation took place. The whey was then tested and if the acidity was not high enough, more acid was added. The curd was then drained upon a cheesecloth in the usual way. The pressing and drying were carefully conducted so that there was no loss of curd. The following table contains the results obtained.

It will be observed from the table that the yield and ash content are greatly decreased by excessive agitation during coagulation. This is due to the amount of curd "dust" formed, which is fine enough to pass through the cheese cloth and become lost in the whey. The ash content is reduced more rapidly with increased agitation than is the yield.

Method and rate of Adding Acid

Care must be exercised in adding the acid if a proper curd condition is to result. If the acid is added too rapidly local coagulation takes place and lumpiness cannot be avoided. On the other hand if it is added too slowly there is apt to be an excessive amount of curd "dust" formed. The proper way is to add the acid while stirring briskly at the place where the acid enters the milk while the remainder of the milk is moderately stirred.

Discussion of Four Methods

From the data it can safely be said that the natural sour method produces the most uniformly low ash casein, and, under normal commercial conditions, the yield is satisfactory. It cannot be made in plants where whey is used for recovery of its milk sugar and albumen content, since some of the lactose has already been reduced to lactic acid. As a result the amount of milk sugar obtained would be low. Time and vat space also do not allow this method to become of economic importance. Modern casein manufacturers require rapid coagulation so as to make room for another batch and thereby conserve both space and time.

Another factor to be observed in the making of natural sour casein is the source of skim milk supply. If the skim milk is not pasteurized, harmful bacteria may greatly lower its quality. If protein digesting organisms are in preponderance, yield of casein would be greatly lowered. The making of natural sour casein on a commercial scale, therefore, is not recommended.

Rennet casein has some advantages over all the others, but the fact that it is extremely high in ash content is sufficient in itself to cause it to be discarded as a commercial proposition. A limited quantity only is needed for the manufacture of those articles for which it is adapted.

In sulphuric and hydrochloric acid casein is found the greatest uniformity in methods of manufacture as well as quality of the product. With proper manufacturing methods, reasonable control of the whole process, and with proper washing, this casein should be uniform in quality.

Assuming that both of these caseins can be made with equal degree of uniformity in quality, the fact that the sulphuric method produces whey which cannot be used for sugar manufacture immediately dis-