

The Manufacture of Condensed Milk, Casein, Etc.—I*

Discussion of Methods of Analyses

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AS EARLY as 1810 an English Patent was granted to De Heine covering the evaporation of part of the water from milk, and its preservation with cane sugar. Other early English Patents on condensed milk were granted to Newton in 1835, Grimway in 1847, De Lignac in 1847, and in France to Appert in 1827. The first patent for a vacuum pan for evaporating milk was granted, I believe, to Green in England in 1813 (English Patent 3,754).

The object of these early inventors is clearly shown in Newton's English patent No. 6,787, 1835, which reads: "For preparing animal milk so that it may be preserved for any length of time with its nutritive properties, and capable of being transported into any climate for domestic and medicinal purposes, this being effected by adding to the milk a certain amount of sugar and evaporating it by any suitable means, using only a gentle heat to quicken the operation. It may be brought to the consistency of cream, honey, or soft paste, or even into dry cakes. Cocoa, coffee, or tea may be evaporated with it."

The first instance of the successful manufacture of condensed milk on a commercial scale in America was in 1856 by Gail Borden, founder of the present firm of Borden's (U. S. Patent 15,553 in 1856; English Patent 509, 1856). Up to the year 1861 there was little demand for condensed milk, but during the civil war a great demand was created, and from that time on it has steadily increased.

The Anglo-Swiss Condensed Milk Company was started by U. S. Consul Page, of Zürich, Switzerland, who first made the product in 1865, and in 1866 incorporated the Anglo-Swiss Company, which now produces over 80 per cent of all the condensed milk manufactured in Australia and Europe.

Milk was first preserved by sterilization in 1856, and unsweetened concentrated milk, or what is now known as evaporated milk, was first successfully manufactured in Highland, Ill., in 1885.

The U. S. Census Report of 1909 shows a production of 214,518,310 pounds of sweetened condensed milk and 280,278,234 pounds of unsweetened evaporated milk, increases of 8 per cent and 154 per cent, respectively, over 1905. The 1913 output in the United States amounted, I am told, to about 250,000,000 cans of sweetened and about 390,000,000 pounds evaporated.

The milk supply for the condensing must be ample for the factory's capacity, and must be of the highest quality. It is under the rigid inspection of the factory, and constant inspection is also conducted of the herds, pastures, barns, and dairies. Rigid instructions are enforced in regard to cleanliness of the animals and dairies. No feeding or other operation which would raise dust is allowed for one hour previous to milking. Strict instructions are issued against the use of certain foods, such as turnips, garlic, etc., and only limited amounts of ensilage are allowed. Immediately after milking the milk is cooled and aerated by running it over block tin coils through which cold brine or water circulates, and then stored in a cool place till brought to the factory. Each can as delivered at the factory is opened by the receiver, who is able to detect any undesirable odor in the milk. In addition to the flavor, the temperature is noted. Tests are also made for acidity, any milk above 0.2 per cent being usually refused. As the farmer is usually paid on the basis of percentage of fat, a small sample is taken each day and tested for fat, or sometimes the test is made on a composite sample of a week's delivery, the sample being preserved with mercuric chloride.

It is customary to make frequent bacteriological tests on the supply from each farmer. A good practical test for the number of organisms present in the milk is the reductase test, which is based on the fact that normal cow's milk has the power of decolorizing certain dye-stuffs, the time of decolorization (reduction) depending on the number of micro-organisms it contains. The test is conducted as follows: 1 cubic centimeter of methylene blue solution is mixed in a glass tube with 10 cubic centimeters of the milk to be examined, 2 cubic centimeters of paraffin oil is floated over it to exclude air, and the tube is kept at about 115 deg. Fahr. (46 deg. Cent.). If the dye is decolorized within one hour, the milk is regarded as very bad from a hygienic standpoint; if decolorized within three hours it is of second quality; if the color persists for more than three hours the milk is good.

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Another test depends on the decomposition of hydrogen peroxide by an enzyme present in milk, known as catalase, the catalytic power of which is increased by the bacteria present. This method has not come into general use in Canada.

To determine the nature of the bacteria, so-called "fermentation tests" are applied. The best known of these is the "Wisconsin curd test," in which a pint of milk is heated in a sterilized jar to about 98 deg. Fahr., 10 drops of standard rennet extract is added, and the jar is closed and incubated for about eight hours at 98 deg. to 102 deg. Fahr. The jar is then opened and the odor observed, the curds are cut with a knife, and the appearance is noted. Good clean milk contains practically no organisms but the lactic acid bacteria, which produce no gas and no bad odors, and the curd formed, therefore, should have only an occasional irregular hole. Milk produced under insanitary conditions will produce more or less gas, and the curds will be full of large irregular holes.

Another "fermentation test" is conducted by incubating samples of the milk in sterilized tubes at about 100 deg. Fahr. After twelve hours the samples are examined, and the bacterial content of the milk is judged by the character of the curd, etc.

The milk after weighing is poured through a large strainer and run into large enameled vats, fitted with stirrers, on the floor below. The room in which these vats are placed is kept as cool as possible. The treatment of the milk from now on depends on the product to be made.

Condensed Milk.—The Canadian standard is not less than 28 per cent of milk solids and not less than 7.7 per cent of milk fat. The last inspection of milk made by the authorities and recorded in Bulletin 257, Inland Revenue Department, shows that the product on the market complies exceptionally well with this standard.

The fresh milk is drawn from the receiving vats into large copper vats known as fore-warmers, where it is heated after addition of the requisite amount of cane sugar. Some of the fore-warmers are heated by copper jackets, while others depend on live steam from an open pipe; the latter method introduces some condensed water from the steam, but the amount is very small. The amount of sugar added varies from 16 to 19 pounds to 100 pounds of raw milk. In some cases the sugar is added to the whole quantity of milk before it goes into the pan, while in others one half or three quarters of the milk is run in, and the sugar added to the remainder. The temperature to which the milk is heated before it goes to the pan also varies in different factories. These variations by different operators are mainly directed toward overcoming crystallization in the finished product.

The milk is now sucked into the vacuum pan and the "condensing" operation begins. The concentration is carried out at a vacuum of about 28 inches and a temperature about 140 deg. Fahr. The vacuum pans used are single type evaporators made of copper, round at each end, with straight sides. They are heated by a steam jacket at the bottom and also internally by large coils, and are connected to a water-jacketed condenser.

The "condensing" takes about two hours. Samples are drawn off at frequent intervals through a double sealed test cup. The consistency is tested by means of hydrometers, the end point being 35 deg. to 36 deg. Baumé. More often, however, the sample is cooled to 70 deg. Fahr., and the appearance and the way the milk "strings" are noted. The operators get quite expert in judging the concentration of condensed milk in this way. As soon as the right consistency is attained, the valves are opened, and the finished product is run into ordinary milk cans (about 14 gallons), which are then placed in a shallow iron tank through which cold water circulates; here they are stirred and cooled, to make the product smooth and free from lumps. The tanks are provided with a series of can wheels, driven from a central shaft, and so constructed that the milk cans fit rigidly on them. A stationary wooden paddle is placed in each can, and as the cans revolve the milk is stirred and cooled. After cooling, the syrupy product is filled into cans and sealed. This product is not sterilized, preservation being dependent on the low percentage of moisture present and the absence of air in the cans. Table 1 shows the average composition of the product compared with that of other evaporated milks and milk powders. If properly made and kept air-tight, it keeps for several years. It contains too much sugar for use as an infant food.

TABLE 1.
Average composition of condensed milks.

	Solids. %	Ash. %	Fat. %	Protein. %	Lactose. %	Cane sugar. %
Condensed whole milk	72.6	1.6	10.0	8.0	12.0	41.0
Condensed skim milk	70.0	2.0	1.0	10.5	14.5	42.0
Evaporated milk	26.3	1.6	7.9	7.7	9.1	—
Whole milk powder	98.3	5.6	26.8	32.0	31.9	—
Skim milk powder	91.7	6.9	1.7	33.8	49.3	—

The product obtained as outlined is made from whole milk and complies with the standard in every way. A skim milk product is also made, but this is always sold as "skim condensed milk." It is manufactured in the same way. (See Table 1 for composition.)

The great difficulty in the manufacture of condensed milk is to prevent "sandiness," due to crystallization of the lactose or cane sugar. A decrease in concentration to prevent this is impossible, as the successful keeping of the product depends on its having less than 30 per cent of moisture, and to effect this the sugar must be added in a proportion to give about 41 per cent cane sugar. Over-concentration and the use of too much sugar increases this sandy condition, but even with proper conditions of amount of sugar and percentage of solids, the lactose generally crystallizes. Each manufacturer seems to have his own method of overcoming this, and for this reason practice varies in regard to the method of and time of adding sugar, temperature, etc., in the pan, and the method of cooling.

On opening a can of condensed milk a layer of sugar is often found on the bottom. This may either be lactose which has settled, or cane sugar separated out owing to an excess being used. It is not detrimental to the product, except that the layer of milk above may not have enough solids to preserve it as long as it should be kept.

Another defect in condensed milk is so-called "buttons." On opening a can and pouring out the contents, one or more small lumps, varying in size from a pin-head to a bean, may be found, attached to the side of the can. These are called "buttons," and are not easy to explain. Some state that they are insoluble casein compounds formed by the action of the metals of the tin plate. The peculiar feature, however, is that they appear to grow. A satisfactory explanation for this trouble is yet to be given, I believe.

Occasionally a can of milk is found to be in a thick, gelatinous condition. This denotes too much milk solids and not enough cane sugar to make a syrup.

Other defects are rancidity, brown color, blown tins, or putrid odor. These conditions are due to too low percentage of solids to prevent bacterial changes, but at most they are rarely very marked until the milk is from two to four years old.

Thus, it is seen that the manufacturer is faced with a difficulty as regards this product. Too little sugar may result in a gelatinous product or one that will not keep, while too much may cause excessive "sandiness" and separation.

Evaporated milk in Canada is defined as milk from which a considerable portion of water has been evaporated, and contains not less than 26 per cent of milk solids and not less than 7.2 per cent of milk fat. This product, therefore, does not contain any added sugar, and is preserved by sterilization.

The milk is drawn from the receiving tanks into the fore-warmers as before. Here it is brought to a boil, and then drawn into the pan and condensed as before, the operator drawing off an occasional sample and testing the solids by hydrometer; when the proper density is reached the valves are opened, and the milk is run over a series of cooling coils to prevent any rapid multiplication of organisms. From the coolers the milk runs into large copper vats, and a sample is tested for fat and solids. If these tests are satisfactory, the milk is next homogenized by breaking up the fat globules into very minute particles and mixing them so intimately with the caseous matter of the milk that the cream does not rise to the top. It is claimed that homogenizing makes the milk more digestible. This process is accomplished by heating the milk to about 185 deg. Fahr. and forcing it through fine metal capillary tubes under a pressure of about 250 atmospheres against a conical surface of agate or metal. Homogenized milk cannot be separated by means of a separator. From the homogenizer the milk goes to the filling machines, where it is run into cans, which are

sealed and transferred to the sterilizer. The sterilizers are large circular iron drums, with a revolving framework inside. The cans, in crates, are placed in compartments of the revolving framework. When filled, the door is closed, the retort nearly filled with water, and the temperature raised to the desired point and maintained for the necessary time, the can revolving all the time. The steam is then turned off and cold water run in till the cans are cold. The time and temperature are very important and are very variable, so that this part of the work is in charge of experts. The factors governing the sterilization are acidity, solids, fat content, the season of the year, and size of cans. Milk of high acidity (over 0.4 per cent) will easily curdle with high temperature, hence the reason for keeping the acidity as low as possible on the fresh supply. The effect of acidity on the milk is shown by Table 2, made from

actual experiment. The same milk was used in each instance and the same temperature and time.

TABLE 2.
Effect of increased acidity on milk. Fresh milk
0.17% lactic acid.

Concentration.	Acidity.	Condition of milk.
1:58:1	0.30	Not precipitated
1:74:1	0.34	" "
1:90:1	0.40	" "
1:99:1	0.43	" "
2:11:1	0.48	Small lumps of curd.
2:25:1	0.54	Large " "

NOTE.—Chemical changes during concentration and sterilization apparently affect the acidity.

Milk of different seasons will stand different temperatures, fortunately the highest in the summer. The fresh

milk varies in composition with the seasons, and hence the concentration also has to be varied to bring the product up to standard. Table 3 illustrates this:

TABLE 3.
Variation in concentration with seasons.

	Solids in fresh milk.	Concentration.	Solids in condensed milk.	Condition of condensed milk.
June	12.68	2:00:1	25.88	Smooth, no separation or curdiness.
August	11.75	2:21:1	26.01	" "
November	13.40	1:99:1	26.62	" "

NOTE.—Lower concentration causes separation of fat, and higher causes curdy milk.

(To be concluded.)

Principles of Coal Sampling*

WITHOUT question the principle point in the entire range of coal inspection and analysis is in the sampling. If the sample taken is truly representative of the entire lot, the results, if accurate in themselves, furnish correct information as to the larger mass of which the sample is a part. If, on the other hand, the sample is in error, the results of the analysis though correct in themselves will be in error so far as they relate to the mass under consideration. Throughout the process of sampling two points must be observed with scrupulous care:

First—The sample taken must be representative of the whole, that is, the distribution of the various substances which go to make up the original mass must be maintained without any change in the relative amount of the various constituents.

Second—The moisture content, which changes readily, must be under exact control so that at any stage the ratio of moisture present to the original moisture of the mass may be definitely known.

As stated above, the first essential in a sample is that it shall truly represent the mass of which it is a part. To secure this result a few fundamental conditions must be observed, as follows:

The gross sample must be representative of the various kinds of material present. That is to say, a mass of coal consists of fine stuff, lump, bone, slate, pyrites, and other constituents. As a rule the "fines" differ in composition from the lump, hence the sample must have these two sorts of material in their proper proportion. The same is even more true of slate or pyrites, of which the composition differs so widely from that of the major part of the mass. An undue amount of such material would cause a serious disturbance in the accuracy of the sample.

In procuring a representative sample a large element of safety resides in the quantity taken. In general, the larger the amount, the more representative it will be. However, conditions differ. It is easier, for example, to procure an even sample from the face of a working vein or from a carload of screenings than from a carload or other mass or lump of run-of-mine coal. In the latter cases larger amounts should be taken than in the former.

The limits of practicability for the proper handling of the sample must, however, be considered. In general, the gross sample should weigh approximately from 200 to 600 pounds. Doubtless 200 pounds of screenings, taken with fairly good distribution throughout the unloading of a 40- or 50-ton car, will yield a very true sample. The difficulties increase greatly with the increase of the size of the particles, as in the case of lump or mine-run coal. If mechanical appliances for grinding are available, the larger amount should be taken, but a smaller sample well crushed down before quartering is better than a greater mass quartered down while the particles are still in larger pieces.

Assuming that the sample as taken is made up of the various kinds of material in proper proportion, the next important item is to maintain these variables in their ratios throughout the process of reducing the gross amount to a small working or laboratory sample. To insure this result, there must be maintained a certain ratio of size of the particles to size or weight of the mass. This, as a rule, is based on a formula which provides that the weight of the largest piece of impurity shall have a ratio to the weight of the mass of about 2: 10,000. For example, a mass weighing 10,000 grammes, or about 22 pounds, should contain no particles weighing more than 2 grammes. This would mean that the largest particle, as for example, a piece of iron pyrites, must not be over one quarter inch in its greatest diameter.

The final ratios of sizes, however, should be determined by the methods available for grinding. With mechanical appliances for obtaining the smaller sizes, a table of ratios with greater safety limits can be adopted than is perhaps practicable where the crushing is done by hand. If a power crusher is available, the entire sample should be passed through the mill and reduced to a size which will pass a 1/4-inch screen. If the crushing must be done

by hand, the first reduction in size of the particles should be such that the entire mass will pass through a 1-inch screen. When by quartering, the sample is reduced to 100 pounds, the size of the particles should be further reduced to a size that will pass a 1/2-inch screen, and with a 50-pound sample in hand the crushing should be carried to 1/4-inch mesh. The subdivisions with their respective sizes are shown in tabular form as follows:

TABLE 1—Size of Mesh for different subdivisions of sample.

Weight of subdivision of sample (pounds).	Size of mesh to which each subdivision should be broken (inches).
500	1
250	3/4
125	1/2
60	3/8
30	1/4

Illinois coals are easily crushed in mills which are available at little expense. Hence it is entirely reasonable to require that gross samples when reduced in mass to 50 or 75 pounds, shall be passed through a mill set for grinding to approximately one eighth inch. For this work, a mill which is not of the jaw-crusher or roller type is preferred, since these types produce too large a percentage of fine material, and the harder pieces of slate, especially those of flaky or plate-like structure, are liable to pass in pieces having inadmissably large dimensions in two directions, even though the adjustment used would seem to be fine enough to prevent the passage of such material. A grinder of the coffee-mill type or one with projecting teeth on the grinding surfaces will be found to produce a more uniform size and the minimum amount of dust.

As a further precaution in maintaining a correct distribution of the various constituents, emphasis is placed upon the necessity of thorough mixing, followed by even selection of the remaining subdivisions. It is true that fine grinding contributes materially to this end but further care is necessary. It is entirely practicable to mix a 50-pound sample, ground as above described, by rolling in an oilcloth about five feet square. This is accomplished by taking one corner of the cloth and carrying it over the pile towards the diagonally opposite corner so as to cause the mass to roll over upon itself, then reversing the motion and repeating the process with the other two corners. Fifteen or twenty such alternations, depending somewhat upon the size of the sample, should be sufficient to effect an even mixture. Where available, however, especially in commercial sampling, a mixer is to be preferred. Such a device is most conveniently made in the form of a drum having cone-shaped ends capable of being closed air-tight, and mounted so as to revolve endwise.

The subdividing of the larger sample, to reduce it to a convenient size for transmission to the laboratory, requires special consideration as having an important bearing on the maintenance of the correct ratio of constituents. This may be best shown by the data given in Table 2.

Note in this table that series 1 and 2 are 3-pound samples taken by subdividing in the same manner the same gross sample of about 30 pounds. Each sample was ground to 8-inch mesh and sized. It will be seen that in series 1, duplicates a and b had 16.6 and 13.7 per cent of the 60-mesh size, whereas in series 2 the duplicates a and b had 22.5 and 23.1 per cent respectively. Note in Table 2 the great increase in ash in the fine size as compared with ash in the coarse material. For example, series 1 having an average of 14 per cent of ash in the coarse size has an average of 23.75 per cent in the fine portion. A similar increase in ash is seen in the corresponding sizes in series 2. The ultimate ash average for series 1 is 16.09 per cent and for series 2 it is 17.85 per cent. These values vary consistently with the variation in the percentages of fine material in the respective series. On the other hand, the duplicate halves a and b throughout, because of their uniformity resulting from the sizing process, show results in the several pairs which check very closely.

The values as presented in the table, therefore, show

TABLE 2—Ash variations in different sizes obtained from duplicate 3-pound samples.

Series	Mesh	Duplicate halves	Percent of each size	CO ₂ in "dry" coal	Ash corrected for CO ₂ in "dry" coal	Ash corrected for a and b composed by calculation
1	On 20	a b	41.7 48.4	.40 .37	14.11 14.00	
1a	Through 20 On 60	a b	41.7 37.9	.85 1.00	15.55 15.42	a...16.32 b...15.86
1s	Through 60	a b	16.6 13.7	1.31 1.38	23.89 23.65	Average...16.09
2	On 20	a b	29.1 25.0	.53 .46	15.91 15.68	
2a	Through 20 On 60	a b	48.4 51.9	.94 .98	16.23 16.06	a...17.90 b...17.80
2s	Through 60	a b	22.5 23.1	1.32 1.28	24.09 23.98	Average...17.85

clearly that in the process of subdividing the gross sample and in the further reduction of the sample as received at the laboratory, great care must be exercised to see that no part of the manipulation is of such a nature as will promote segregation of the constituents.

A riffle may be used to advantage after the sample has been reduced by quartering to about 30 pounds. At this stage the sample is ground to 1/4-inch size, hence the riffle openings may be 1/2-inch in width.

The second essential in taking and preparing a sample relates to the free moisture present, and requires that the changes in moisture content "must be under exact control so that at any stage the ratio of the moisture present to the original moisture of the mass may be definitely known."

In coals of this region especially, where the moisture in the coal as it comes from the mine averages from 10 to 15 per cent, the tendency toward moisture changes is very marked. For example, the process of crushing down the larger sizes affords an opportunity for the escape of moisture. Again, if the coal is spread out on the floor of a hot boiler room or left exposed to currents of air for any length of time there will be a serious change in the moisture factor. Another practice sometimes followed is that of assembling the various increments of the gross sample in a sack or other receptacle permitting a relatively free transmission of air. Samplers kept in this manner for any length of time or shipped in such containers will have a moisture content quite different from the original.

The methods employed, therefore, in collecting and reducing a gross sample must have special reference to this tendency on the part of the free moisture to escape. The work should be done rapidly in a room at or below the normal temperature and, so far as possible, with the use of closed apparatus which admits of the least possible exchange of the contained air. Precautionary measures of this sort should be made at the very outset. The gross sample, which is made up of small increments collected usually over a considerable length of time, should be inclosed in a tight box or clean garbage can having a tightly fitting cover which can be closed and locked against the possibility of change until the time for grinding and reducing.

FACE SAMPLING.

Since the procedure for obtaining the face samples described in this report serves as a good illustration of methods adapted to meet the principles above enumerated concerning the uniformity of composition and control of moisture, the details of the process for collecting, and subsequent preparation for shipment in the small container are here given in full. The methods thus described are applicable in principle to the collection of any and all samples from whatever source.

DIRECTIONS FOR COLLECTING FACE SAMPLES IN THE MINE.¹

Selection of the face.—Choose three faces in the mine as widely separated as possible in order to give a good average of the coal for that mine. An attempt should always be made to get faces which have not been exposed more than 48 hours.

¹Substantially as given in Bureau of Mines Technical Paper No. 1 with the exception of the preparation, grinding, etc., of the sample.

*Bulletin 29 of the Illinois State Geological Survey, by S. W. Parr.