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ESTIMATION OF FAT IN CONDENSED MILK.

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THE use of canned, sugar-preserved, condensed milk has increased considerably during the last few years, no less than forty brands of the article being on sale in Massachusetts at the present time. In view of the fact that of all its ingredients the fat is the only one that can be conveniently tampered with to produce an inferior or cheaper product, it becomes highly important to have at hand a ready and accurate means of fat determination, especially where samples have to be examined systematically and in large numbers for adulteration.

The Babcock centrifuge method, so convenient for estimating fat in ordinary milk, was long thought to be out of the question for use with sugar-preserved milk, by reason of the fact that the cane-sugar, which is present to the extent of some 40 per cent. in the average condensed milk, becomes so charred by the action of the sulphuric acid employed in the test as to produce nothing but a black mass in place of the desired column of clear fat. This has been the common experience of all who tried it.

The Adams-Soxhlet method, which is the recognized standard for fat determination in ordinary milk, has been largely used in the case of condensed milk, but, in the writer's experience, can not be depended on for the latter purpose, on account of the fact that the large amount of cane-sugar present, even when the sample is diluted for analysis, encloses the fat particles so firmly. when dried on the extraction coil, as to render its removal by the solvent ether a very difficult matter.

In 1895 the writer devised a scheme whereby the Babcock machine could be satisfactorily used for condensed milk fat, and an outline of the process as then carried out was published in the "Annual Report of the Massachusetts State Board of Health for 1896." This process, with certain minor modifications which experience has from time to time suggested, has been in successful use in the Food and Drug Department of the Board for over five years, and has proved itself to be not only much quicker

than the Adams-Soxhlet extraction method and easier of manipulation, but, indeed, more accurate, by reason of the fact that the cane-sugar with all its attendant troubles is first eliminated.

If, in the case of condensed milk, ordinary ether is used as a reagent for the Soxhlet extraction, the amount of extract weighed as fat may not appear too low, because the alcohol and water present in the ether dissolve not only fat, but also sugar, which goes in with, and is weighed as fat. On the contrary, if the ether be dehydrated with calcium chloride and distilled over sodium, to free it completely from alcohol, the extracted fat will be found to be far too small. The same incomplete extraction results from the use of benzine or petroleum ether as a solvent.

Parallel determinations of fat in sugar-preserved milk by the Adams-Soxhlet process, using ether carefully dehydrated and freed from alcohol, and by the writer's method involving the use of the Babcock machine, show in all cases a larger fat content by the latter or modified Babcock process. Indeed, in one instance an extraction of sixty hours was required in the case of the Soxhlet process to equal the percentage of fat found by the modified Babcock process, so firmly were the fat particles enclosed by the cane-sugar on the extraction coil, thus resisting the action of the ether. It is obvious that, in the case of the modified Babcock process, no more fat can be shown by the final result than actually exists in the milk; indeed, if anything, one would expect a slight loss, so that, when compared with the Soxhlet method, if the latter shows lower figures, it can safely be presumed that the process is unreliable. This, of course, applies only to the sugar-preserved variety of condensed milk.

The improved method of fat estimation with the Babcock centrifuge is as follows: Having first insured a homogeneous sample of the contents of the can by stirring, 40 grams are weighed out preferably in a weighing tray for sugar, transferred by washing to a graduated 100 cc. sugar flask, and made up to the mark with water. Twenty-five cc. of the thoroughly mixed diluted sample, corresponding to 10 grams of the original condensed milk, are measured by a pipette into an ordinary test-bottle of the Babcock centrifuge. This is filled nearly to the neck with water, and 4 cc. of a solution of copper sulphate of the strength of Fehling's copper solution are added. The contents are thor-

oughly shaken, and the precipitated proteids, carrying with them the fat, are rapidly separated out by whirling the fat bottle in the centrifuge, preferably (though not necessarily) without heating. The writer prefers an electric centrifuge of the Robinson type for this purpose, as the heat of the steam-driven machine cakes the precipitate down, so that it is harder to wash. If desired, the precipitate may be allowed to settle out of itself, which it does more quickly in the cold.

The supernatant liquid containing the sugar is drawn off by means of a pipette of large capacity, having a stem sufficiently small to pass easily into the neck of the milk bottle, a small wisp of absorbent cotton being first twisted over the bottom of the pipette to serve as a filter. If many samples are to be treated, a suction-pump, connected by rubber tubing with the pipette, is a great convenience. On withdrawing the pipette with the sugar solution, the cotton is wiped off into the bottle by rubbing against the inner side.

The precipitated proteids and fat are given two additional washings, as above, by shaking thoroughly with water introduced nearly to the neck of the bottle, separating out in each case by centrifuge or by settling, and finally removing the washings with the pipette, two of such extra washings being found nearly always sufficient to remove all the sugar. If the precipitate is caked down hard after treatment with the centrifuge, it may be necessary to employ a stiff platinum wire as a stirrer to aid in mixing with the wash-water.

Finally, enough water is added to amount approximately to the normal volume of 17.6 cc. usually employed for the Babcock test, 17.5 cc. of sulphuric acid are added, and the test continued from this point on as in the ordinary Babcock process of milk testing, multiplying the reading obtained by 1.8 to give the correct percentage of fat in the sample.

For condensed milk containing no added cane-sugar, these precautions are, of course, unnecessary, the ordinary Babcock method being directly employed with a weighed portion of the milk.