NOTES ON THE DETERMINATION OF FAT IN MILK.

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In a previous paper* I described a modified form of Soxhlet's extractor, as used in connection with Babcock's method, for the determination of fat in milk.

In the use of this method a few questions arise with regard to which some experiments are here given.

1st. *How long is it necessary to dry the milk?*

This question is of importance where it is desired to ascertain the amount of total solids in connection with the determination of fat. The following results were obtained by drawing ordinary air over the milk in the tube of asbestos, held upright, and heating at 100° C. After each period of drying, the tube was removed to a dessicator, allowed to cool, and weighed:

	Grammes.	Percentage.	Loss %.
Milk taken	5.1286	100	
After drying 20 minutes	4.0948	79.8	20.2
" 40 "	3.0296	59.1	20.7
" 1 hour	2.0443	39.8	19.3
" 1 hr. 20 min	1.2282	23.9	15.9
" 1 " 40 "	.6738	13.1	10.8
" 2 hours	.6624	12.9	0.2
" 2 hrs. 20 min	.6594	12.857	0.043
" 2 " 40 "	.6591	12.850	0.007
" 3 hours	.6584	12.830	0.02
" 3 hrs. 20 min	.6584	12.830	0
" 3 " 40 "	.6584	12.830	0

In this experiment it will be seen that nothing was lost after drying for three hours, and after two hours the loss amounted to less than 0.1%.

Some variation in the conditions of the process is to be expected, as in the packing of the asbestos and in the rapidity with

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which air is drawn through the tube; a certain latitude should therefore be allowed in taking the results of one or more experiments, but from these results and those obtained by Dr. Babcock,* it may be considered that drying for two hours is sufficient, even where it is desired to determine the total solids.

2d. *How long is it necessary to dry the fat?*

The length of time required for drying the fat may depend, of course, upon the use of ether or petroleum spirit, respectively, in the extraction. With each of these, experiments have been made. The fat was fully extracted by a dozen or more discharges of the extractor, and then dried for periods of 10 minutes each, at 100° C., and weighed, after allowing to cool, in a dessicator. In the process of drying, a circulation of air through the flask was effected by inserting the end of the tube from the air pump into the flask, through the hole in the top of the bath.

First. When extracted with ether.

The ether for extraction was bought for ordinary washed ether, and distilled once before using.

Milk taken						5.0908 gms.
Fat after drying	10	minutes	.....	.2274	gm. or	4.467%
" "	20	"	.....	.2273	"	4.465%
" "	30	"	.....	.2272	"	4.463%

The process of drying and weighing was then continued periodically until it had been dried 2 hours and 15 minutes, but without further change in weight. There was, therefore, no loss after 30 minutes, and after 10 minutes it amounted to less than .01%.

Second. When extracted with petroleum spirit.

The lightest petroleum spirit (rhigolene) was redistilled and the part boiling between 30° and 60° C. taken for extraction. The milk used in this case was the same as that of the preceding.

Milk taken						5.0626 gms.
Fat after drying	15	minutes	.....	.2257	gm. or	4.458%
" "	30	"	.....	.2252	"	4.448%
" "	45	"	.....	.2251	"	4.446%
" "	1	hour	.....	.2251	"	4.446%

*Second Annual Report, N. Y. Agricultural Experiment Station, p. 168.

Fat after	drying	1	hr.	15	min. . .	.2250	gm. or	4.444%
"	"	1	"	30	" ...	.2249	"	4.442%
"	"	2	"	15	" ...	.2248	"	4.440%

In this case more time is necessary to reach a constant weight than before, though, after 30 minutes drying, the total loss is less than .01%.

Another trial was then made with petroleum spirit, using another sample of milk.

Milk taken.....								5.0867 gms.
Fat after	drying	10	minutes.....			.1807	gm. or	3.552%
"	"	20	"	.....		.1805	"	3.548%

Entirely uniform results were obtained by further drying, though continued for 2 hours and 15 minutes, weighing at periods of 15 minutes.

Fifteen to twenty minutes may, therefore, be considered sufficient in ordinary analysis, whether ether or petroleum spirit is used, and no harm is done if dried a much longer time. Some one has said that, by prolonged drying, the fat becomes oxidized and that an increase in weight is obtained. This has not resulted by drying 2¼ hours in these experiments.

In the use of ether it is generally thought preferable to use anhydrous ether, and where a relatively large amount is used, as in the Wanklyn method, this precaution is the more necessary, but by the Soxhlet method of continuous extraction where a small amount is used, and where only the part with least water is brought into contact with the dried milk, it is doubtful if the difference would be of any practical importance. In the above case where washed ether was used, which was afterwards once distilled, it will be noticed that the result is only .02% higher than in the following case where; with the same milk, the extraction was made with petroleum spirit.

3d. *How much extraction is necessary?*

One advantage in the Soxhlet extractor arises from the fact, that by its periodic working a rough measure is afforded as to the amount of extraction performed. In other forms the best that can be done is to note the time and apparent rapidity of the process.

In each discharge of the Soxhlet extractor a part of the extracting fluid remains in contact with the cartridge of asbestos. The amount that remains may vary with the looseness or closeness of the asbestos, the size of the opening in the tube, the form of the extractor, etc., so the operator must use his own judgment and allow some latitude if he would be sure of correct results. With these considerations in view, it was thought, however, to be of some service to make a few experiments which might show the rapidity with which the fat is removed; this could be done by determining the amount of fat withdrawn by each discharge. Accordingly, when the extractor had been once discharged, the extracting fluid was withdrawn through the spoon-shaped exit tube and the flask with the fat thoroughly dried in the manner of the preceding experiment. After weighing the fat the flask was again connected with the extractor and the fat of the second discharge, etc., withdrawn and determined.

FAT REMOVED BY EACH DISCHARGE OF THE EXTRACTOR.

No. of Discharge.	WITH ETHER. (Milk—5.0730 gms.)				WITH PETROLEUM SPIRIT. (Milk—5.0460 gms.)			
	Milligrammes.		Percentage.		Milligrammes.		Percentage.	
	Total.	Separate.	Total.	Separate.	Total.	Separate.	Total.	Separate.
1	199.1	199.1	3.925	3.925	199.1	199.1	3.945	3.945
2	219.0	19.9	4.317	.392	213.9	14.8	4.239	.294
3	224.5	5.5	4.425	.108	222.4	8.5	4.407	.168
4	227.3	2.8	4.481	.056	224.6	2.2	4.451	.044
5	228.6	1.3	4.506	.025	225.7	1.1	4.473	.022
6	229.4	0.8	4.522	.016	226.3	0.6	4.485	.012
7	229.7	0.3	4.528	.006	226.6	0.3	4.491	.006
8	230.0	0.3	4.534	.006	226.9	0.3	4.497	.006
9	230.2	0.2	4.538	.004	227.1	0.2	4.501	.004
10	230.5	0.3	4.544	.006	227.4	0.3	4.507	.006
11	230.8	0.3	4.550	.006	227.7	0.3	4.512	.005
12	231.1	0.3	4.556	.006	227.7	0	4.512	0
13	231.3	0.2	4.559	.003	227.8	0.1	4.514	.002
14	231.4	0.1	4.561	.002	227.9	0.1	4.516	.002
15	231.5	0.1	4.563	.002	227.9	0	4.516	0
16	231.5	0	4.563	0	------	------	------	------
17	231.5	0	4.563	0	------	------	------	------
18	231.6	0.1	4.565	.002	------	------	------	------
19	231.7	0.1	4.567	.002	------	------	------	------
20	231.8	0.1	4.569	.002	------	------	------	------

These experiments would go to show that the amount of fat withdrawn after the fourth or fifth discharge is less than 0.1%, and that what is withdrawn after ten or twelve discharges may be disregarded. The milk in both parts of this experiment was the same, hence, it appears that washed ether, which has been once distilled, again gives a little higher results than petroleum spirit, though only about .05%.

A few reasons occur why petroleum spirit may be preferred to ether for milk analysis. At the present rates petroleum spirit is cheaper than ether, though the small amount used in the Soxhlet extractor makes this of minor importance. Petroleum spirit is known to be an excellent solvent for fat, and there is no doubt that by its use all fat is removed. From these experiments it appears that the extraction is performed a little more rapidly with petroleum spirit than with ether. Milk sugar is entirely insoluble in petroleum spirit. This was shown in an experiment mentioned in the Report of the Brooklyn Department of Health for 1885, which may here be more fully given. Some finely powdered, pure, milk sugar was agitated for a considerable time with petroleum spirit. 25 c.c of the clear liquid were then withdrawn and evaporated in a weighed porcelain dish. On full evaporation the dish was again weighed but no increase was found. 25 c.c. more were again evaporated and still no increase was found.