THE ANALYST.

JULY, 1889.

FAT EXTRACTION FROM MILK SOLIDS.

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(*Read at Meeting, May, 1889.*)

THE estimation of fat in milk, simple though it seems at first sight, has perhaps given more trouble to analysts than anything else, and on it, consequently, a very large amount of original work has been done. Happily, now the subject is much better understood than it was, and by the improved methods now in use something like concordance between separate analysts is to be reasonably expected. But there is still a want to be supplied, which is well expressed by Vieth (*ANALYST*, xii., 63; xiii., 49), where he says, "The difference between the percentages of fat extracted if the coil process is used . . . and the fat extracted if the plaster process is used . . . are smallest with skim milks, and increase with the increasing percentage of fat; or, in other words, the differences are small where great difficulties for the complete extraction of fat exist, and comparatively large where no such difficulties exist." Vieth gives two tables illustrating this: one in *ANALYST*, xii., 63, and the other comparing the results obtained by Hehner and myself in working out our formula (*ANALYST*, xiii., 49). A third table showing the same thing is found in *ANALYST*, xi., 62; and a glance at the diagram annexed to our paper (*ANALYST*, xiii., 24) shows it in a graphic form.

To explain this fact I undertook this investigation. I only propose to consider here the methods of extraction of fat from milk solids, and shall not, therefore, refer to such methods as Soxhlet's areometric process, etc.

The methods of fat extraction from milk solids are many, among them being:—

1. Wanklyn's direct extraction, and modifications by Carter Bell, Waller, etc.
2. Evaporation on Sea Sand.
3. „ „ Asbestos. Babcock (*Dept. Agric. Rept.*, 13, pt. I., 81).
4. „ „ Asbestos Cloth or Paper. Wiley (*Foods and Food Adulterations*, pt. I., p. 81), also used by Johnstone (*ANALYST*, xii., 234).
5. Evaporation on Plaster of Paris. Soxhlet.
6. „ „ Pumice, with grinding to powder. Storch (*ANALYST*, xii., 6).
7. „ „ Copper Sulphate. Morse, Piggott, and Burton (*ANALYST*, xiii., 18).
8. Evaporation on Sponge. Duclaux (*Le Lait*, p. 176).
9. „ „ Lint. Abrahams (*ANALYST*, ix., 20).
10. „ „ Paper. Adams (*ANALYST*, x., 46).
11. „ „ Powdered Glass. Baynes.
12. „ „ Kieselguhr.
13. „ „ Wood Fibre. Gantter (*Z. f. Anal. Chem.*)
14. Extraction from Milk Solids in pasty condition. J. Bell (*Foods*, vol. ii., and *ANALYST*, viii., 141).
15. Extraction from precipitated Casein.

It was at once seen that the investigation of all these methods would serve no useful purpose. It may be laid down as an axiom of milk analysis, that only methods which extract the whole of the fat are applicable, and no other should be tolerated as an exact estimation. The following published statements were found to assist in choosing suitable methods for investigation:—

1. Wanklyn's method and modifications are now universally admitted to be faulty.
2. By the use of sea-sand, Schrott and Hengold find results very nearly the same as Soxhlet's plaster and areometric methods; other experimenters find rather less.
3. Kent and Grothe (*ANALYST*, x., 106) find that Babcock's method gives only slightly more than Wanklyn's, and $\cdot 2 - \cdot 3$ less than Soxhlet's.
4. Wiley has discarded his asbestos paper method on account of it not giving results as high as Soxhlet's areometric method; Johnstone has published figures (*ANALYST*, xii., 234) showing that results obtained by asbestos cloth fall appreciably below those given by Fleischmann's formula.
5. Soxhlet's method, which is the usual method of fat estimation on the Continent, gives very concordant results, which are higher than any of the preceding.
6. Storch's method gives on an average $\cdot 27$ higher than Soxhlet's (*ANALYST*, xiii., 6; *Versuch. Stat.*, 1888, p. 316).
- 7, 8, 9, 11, 13, and 15. No comparative figures published for any of these methods.
10. Adams' paper methods gives higher results than any other, except Storch's (*ANALYST*, x., 46; Baertling, *Rep. f. Anal. Chem.*, 41 (1886), No. 31, 411, etc.).
12. Neither method nor figures yet published.
14. Bell's method has been examined by Wigner (*ANALYST*, viii., 211), and gives $\cdot 2 - \cdot 3$ more than Wanklyn's.

From these data I decided to submit to investigation the following methods:—

Soxhlet's plaster method.

Adam's paper „

and either Storch's pumice method or the Kieselguhr method, which are practically identical.

An objection has been urged against Adams' method on the ground that paper contains a substance which is soluble in ether, but difficult to extract; thus Vieth (ANALYST, x., 86) found that one coil gave, after soaking over night in ether, .0157 grs., and on a second extraction .0072 grs. more; and Faber (ANALYST, xii., 8); shows that in one case a first extraction gave .023 grs., and a second .010 grs. The instructions of the Milk Committee of the Society of Public Analysts (ANALYST, x., 217) are to previously extract the coils with ether, but nothing is said about there being any difficulty in extracting them completely, it being implied that twelve siphonings are sufficient. In a former paper, in which Hehner and I used this method for working out a formula, we stated that we did not previously extract our coils, but made a subtraction of .012 grs., that being the average amount extracted by blank experiments. Having now another batch of paper, the usual blank determinations were made, twenty-eight coils giving on an average .0102 grs. each, giving

a probable error for one coil of .0006 grs.

and a maximum error „ „ of .0025 grs.

Two coils were submitted to further extraction, with the following results, each being extracted for $1\frac{1}{2}$ hours at a time:—

1st Ext.	2nd Ext.	3rd Ext.	4th Ext.	5th Ext.	6th Ext.	7th Ext.
grs.	grs.	grs.	grs.	grs.	grs.	grs.
.0102	.0086	.0076	.0074	.0012	—	—
.0127	.0025	.0041	.0032	.0030	.0020	.0020

giving a total of .0350 grs. and .0295 grs. respectively. These results showed that although the subtraction of certain quantity found by blank experiment might do for every-day work, yet for a scientific investigation it is absolutely inadmissible. I tried the use of various other solvents; neither benzene, petroleum ether, or carbon disulphide showed any advantage over ether, but alcohol completely extracted the papers. As it has been stated that soap is used in the manufacture of the paper, I also tried the effect of acid, either by soaking the coils in dilute acetic acid, allowing to dry and extracting with ether, or by mixing a small quantity of glacial acetic acid with the ether or alcohol used for extraction, and found that in every case the paper was completely extracted. I found that the most convenient and expeditious method of extracting papers was to use acid alcohol, *i.e.*, alcohol containing 10 per cent of acetic acid, and extract in a large Soxhlet apparatus for three or four hours. Paper extracted in this way yielded to ether after about six hours' extraction a very small quantity of extract, consisting chiefly of fibres, amounting to perhaps one milligram., which if necessary may be estimated and subtracted, but which is practically negligible. After the completion of this stage of the research, my attention was drawn to a paper by Professor Soxhlet (*Molkerei Zeitung*, 43, 439), who had come to the same conclusions, and as the outcome of his work, Messrs. Schleicher and Schüll are placing on the market a fat-free paper, of which their agents in England, Messrs. Townson and Mercer, 89, Bishopsgate Street, E.C., hold a stock.

To prevent an objection similar to that urged against the paper process being brought forward against the plaster or Kieselguhr processes, I discarded the use of paper to wrap the cartridges in, and substituted wide test-tubes, having a hole at the bottom, and containing a wad of cotton wool which had been previously extracted with ether, until it gave no further extract. The Kieselguhr was ignited before use. The plaster or Kieselguhr, after being evaporated, was ground in the basin in which it had been dried, and *not, as is usually done, simply crushed to a coarse powder*; in many cases it was, after being extracted, ground a second time. The extractions in all cases lasted *at least three hours*, and in most cases more, as it was found that less time did not suffice, especially in the plaster extractions. For the sake of comparison, a series of results with unextracted coils were made, a correction of .010 grs. being used, and the extraction being prolonged for only $1\frac{1}{2}$ hours (our usual custom hitherto).

The fine and repeated grinding and long extraction are essential points of Storch's method, and should always be adopted if very exact results are wanted.

The following are the results obtained:—

No.	G.	$\frac{G}{D}$	T.	Ad. Ext.	Ad. Un.	P.	K.	Calc.	
								New F.	Old F.
1	32.3	31.3	11.62	2.89	2.95	2.85	*2.94	2.90	2.93
2	33.1	32.0	12.41	3.30	3.40	3.25	*3.31	3.41	3.43
3	29.6	28.7	11.05	2.81	2.91	2.76	*2.86	3.00	3.03
4	32.1	31.1	12.99	4.00	4.10	3.85	—	4.11	4.14
5	32.0	31.0	12.90	4.02	4.15	3.96	—	4.06	4.09
6	31.5	30.5	12.57	3.78	3.89	3.74	*3.85	3.90	3.92
7	28.2	27.4	15.08	6.77	6.91	6.73	—	6.73	6.78
8	31.2	30.3	11.07	2.66	2.81	2.66	—	2.65	2.69
9	27.1	26.4	16.80	8.37	8.47	8.35	—	8.43	8.49
10	30.2	29.3	11.82	3.45	3.49	3.41	—	3.51	3.56
11	29.7	28.8	13.92	5.45	5.54	5.41	—	5.43	5.47
12	31.1	30.2	11.10	2.75	2.88	2.70	—	2.70	2.74
13	31.2	30.3	11.29	2.77	2.89	2.77	—	2.84	2.89
14	30.8	29.9	12.57	3.94	4.49	—	—	4.03	4.07
15	31.8	30.8	12.06	3.35	3.54	—	—	3.38	3.42
16	30.3	29.4	10.52	2.41	2.62	2.40	*2.49	2.38	2.42
17	30.7	29.8	12.49	4.03	4.22	—	—	3.98	4.03
18	31.6	30.6	12.24	3.61	3.78	—	—	3.58	3.62
19	29.8	28.9	11.98	3.80	4.00	—	—	3.74	3.78
20	31.5	30.5	10.35	1.99	2.10	1.96	*2.02	2.00	2.02
21	32.8	31.8	12.69	3.86	—	3.80	*3.86	3.70	3.73
22	32.0	31.0	13.61	4.75	—	*4.75	*4.79	4.67	4.69
23	33.8	32.7	9.21	.54	—	*.54	*.60	.52	.53
24	35.4	34.2	9.41	—	—	*.46	*.44	.36	.37
25	35.6	34.4	9.29	—	—	*.26	*.27	.21	.22
26	35.3	34.1	9.37	.26	—	—	*.26	.34	.35
27	32.5	31.5	11.47	2.68	—	—	*2.71	2.71	2.76
28	35.6	34.4	9.71	.42	—	—	*.45	.56	.58
29	33.9	32.8	10.08	1.13	—	—	*1.13	1.24	1.26
30	—	—	—	.35	—	—	*.35	—	—
31	—	—	—	—	—	*.40	*.44	—	—
32	—	—	—	—	—	*.20	*.21	—	—
33	—	—	13.87	5.00	5.07	4.96	†4.95	—	—
34	35.4	34.2	9.28	.18	$\left\{ \begin{array}{l} .15 \\ .28 \end{array} \right.$	†.10	†.12	.25	.25

* Re-ground and extracted a second time.

† Extracted $1\frac{1}{2}$ hrs. only.

1-20 were extracted for about three hours, the others 5—6 hours.

These results lead to the following conclusions:—

1. If a sufficiently long time is given for the extraction, and if the plaster and Kieselguhr are ground to a fine enough powder, there is, practically speaking, no difference between the paper, plaster, or Kieselguhr methods.

2. The use of unextracted coils induces an error which is by no means constant varying from +.06 to +.55. The observation that acids rendered a larger portion of the extractive matter of the coils soluble than would otherwise have dissolved led me to the conclusion that the free acid in milk (always present) increased the amount extracted from the coil itself, and this was further shown by the following experiment:—A skim milk, when fresh, gave as a mean of three results .15 per cent. fat. A day or two afterwards, when beginning to turn sour, the fat had increased to .28 per cent., the correction of .010 grs. being made for each coil.

I certainly recommend that the instructions of the Milk Committee should be modified, and should state that the coils should be extracted with acid alcohol, and that at least three hours or more extraction should be allowed.

As I have shown that the paper method, as Hehner and I worked it (and, we believe, as it was worked by many others), was open to objection, I deemed it advisable to see how my results agreed with the formula we had previously worked out, and which was based upon that method.

A discussion of the various formulæ which have been proposed will not be out of place here.

Behrend and Morgen (*J. f. Landw.*, xxvii., 250) were the first to point out the exact relation between sp. gr. fat and total solids, and published a table. In the same year, 1879, Clausnitzer and Mayer (*Forchngen auf dem Gebeite*, ii., 265) gave a formula, constructed from one sample of milk, which was analysed, allowed to stand during the night, and the skim milk again analysed. From this they found that 1 per cent. fat lowered the gravity .001, while 1 per cent of solids not fat raised it .00375. Hehner (*ANALYST*, viii., p. 129), from the result of 22 analyses of milk, compiled a formula to represent Wanklyn's method; he took the gravity of fat to be .9275, and assumed that 1 per cent decreased the gravity (water = 1,000) by .725, while each per cent. of solids not fat raised the gravity by 3.6. This assumption is not absolutely correct, though the error introduced is well within the limits of experimental error. Fleischmann and Morgen (*J. f. Landw.*, xxx., 293) constructed a formula to represent the plaster process. They assumed that each per cent. by volume (*i.e.*, gram. per 100 c.c.) of fat lowered the gravity by a constant factor; while each per cent. by volume of solids not fat raised it by a constant factor. They took the gravity of fat at 15.5 to be .94. From a series of experiments, Fleischmann found the gravity of butter fat to average .93, and consequently altered his formula in accordance with this fact. Hehner and myself last year published a formula for the Adams' process, in which we made the same assumption as in Hehner's original formula, but obtained our constants entirely from our observations. None of these formulæ are absolutely free from objection, as they either contain an assumption that is not strictly correct, or the gravity of the fat is assumed to be the same in milk as it is in the solid state.

On account of this, and the fact that the method employed by Hehner and myself is now shown to be faulty, I have considered it desirable to obtain a new formula.

If we assume that $1+x$ is the gravity of a substance in solution, and that we have a solution containing y grs. in 100 c.c., the y grs. will occupy $\frac{y}{1+x}$ c.c., and as the total bulk is 100 c.c., the water occupies $100 - \frac{y}{1+x}$ c.c., and weighs $100 - \frac{y}{1+x}$ grs.

$$\begin{aligned} \text{** The specific gravity of solution is } & \frac{100 - \frac{y}{1+x} + x}{100} \\ & = \frac{100(1+x) - y + y + xy}{100(1+x)} \\ & = 1 + \frac{xy}{100(1+x)} \end{aligned}$$

** 1 gram. in 100 c.c. raises the gravity by $\frac{x}{100(1+x)}$ which is a constant for each substance.

In a milk each gram. of fat in 100 c.c. lowers the gravity by a certain amount, (ϕ), while the solids not fat raise it by a certain amount (σ); and if F =per cent. of fat, $F \times D$ (density)=per cent. of fat by volume, and $S \times D$ =per cent. of SNF by volume, then $G = S \times D \times \sigma - F \times D \times \phi$, or $\frac{G}{D} = S\sigma - F\phi$; or as $S = T - F$: $\frac{G}{D} = (T - F)\sigma - F\phi$;

$$\text{or } \frac{G}{D} = T\sigma - F(\sigma + \phi); \text{ or } \left(\frac{\sigma + \phi}{\sigma}\right) - \frac{G}{D} \frac{1}{\sigma}; \text{ or } 1 = \frac{F}{T} \left(\frac{\sigma + \phi}{\sigma}\right) - \frac{G}{D} \frac{1}{T\sigma},$$

and, by the same process as used in the paper by Hehner and myself, I calculate from my results the following formula: $T = 1.17 F + .263 \frac{G}{D}$, which gives the following values:—

$$\phi = .65, \text{ sp. gr. of fat } .939$$

$$\sigma = 3.8, \text{ sp. gr. of SNF } 1.613.$$

In calculating this formula only the Kieselguhr results and the paper and plaster results of Nos. 22—29 have been used; the formula when applied to the paper results of Nos. 1—21 averages about .02—.03 per cent. higher.

It is a curious fact that the sp. gr. of fat in milk should be .939, which is distinctly higher than the observations of Blyth, Fleischmann, and others. This number is practically identical with the number calculated from the results obtained by Hehner and myself, and with that used by Fleischmann and Morgen. With regard to this it is worthy of note that Besana, from an extended series of analyses by Soxhlet's method, found that Fleischmann and Morgen's old formula expressed the results better than Fleischmann's new one; an examination of the results published in Fleischmann's paper (*J. f. Landw.*, xxxiii., 251) shows the same thing; and Vieth practically confirms this in a paper where he states that with skim milks from 141 analyses he finds there is an average difference of + .04, which is about the difference between the two formulæ. It is, therefore, a remarkable fact that butter-fat has a higher gravity in milk (when presumably liquid) than it has when solid. Taking the gravity of milk-sugar to be 1.65—a number

obtained by many observers—of casein, etc. at 1·31 (Hegner), and ash at 3·0 (Dupré), and the proportions of these constituents in milk at 6 : 5 : 1 (Vieth), the gravity of SNF should be

$$\begin{array}{rcl}
 1\cdot65 \times 6 & 9\cdot90 \\
 1\cdot31 \times 5 & 6\cdot55 \\
 3\cdot0 \times 1 & 3\cdot0 \\
 \hline
 19\cdot45 \div 12 = 1\cdot62
 \end{array}$$

which is nearly identical with that given above.

When working out our formula, Hegner and myself attributed to lower results obtained with skim milk to the fact that a change in composition (due to formation of hydrates) took place as less and less fat was present, or as the amount of water increased. To ascertain if this was so, a sample of skim milk was taken and diluted with weighed quantities of water, the mixtures allowed to stand several hours, and the density taken. The density of the mixtures was calculated from the density of the original milk, and the amount of water added, with the result that the two were identical within experimental error.

Percentage, by Milk.	Volume of Water.	Observed Sp. Gr.	Calculated Sp. Gr.
100	—	1·03544	—
94·4	5·6	1·03343	1·03345
89·3	10·7	1·03170	1·03165
83·3	16·7	1·02950	1·02952
79·8	20·2	1·02829	1·02828
68·8	31·2	1·02439	1·02438

In the table I have given in the two last columns, the numbers calculated by the new formula, and Hegner's and my old formula respectively, it will be seen that the difference between them is very small, and that for all practical purposes they are identical. By permission of the directors of the Dairy Supply Co., Mr. Faber has applied the new formula to over 200 analyses made by him in 1885 in the laboratory of that company by Storch's method, with the result of almost absolute agreement, there being an average difference of ·02 per cent., with maximum differences of + ·34 and — ·26 respectively, showing that this method, which has been in use by Storch and other Scandinavian chemists for several years, gives complete extraction.

As the total solids are used for the calculation of the fat, it is, of course, important that they should be accurately estimated. The usual mode of doing this is to evaporate 5 grs. of milk to dryness in a small platinum, glass, or porcelain basin, these being used indiscriminately. With a view of shortening the process Gerber and Radenhausen in 1876 proposed the use of acetic acid (*Vorschläge zur einer einheitliche Untersuchungsmethod der Milch* 7), but rejected it ultimately on account of the approximate character of the results, it being impossible to remove all the acetic acid, and at the same time to prevent change in the milk residue. Sea-sand and other powders have been used, Duclaux (*Le Lait*, 176) uses sponge, Babcock asbestos, and Adams paper, all with the idea of spreading the residue out in a fine condition, so that perfect desiccation may take place. When performing duplicate experiments, I was struck by the fact that when the milk was in a small basin the results were lower, and the residues darkened more than when a larger basin was used, and this was especially the case when the small basin was of

porcelain. A series of experiments was made to ascertain the influence of mass on the estimation, by comparing the results obtained with from 1–1·5 grs. of milk in a large basin and 5 grs. in a small basin. The results are as follows:—

T. S. 5 grs.	T. S. 1 – 1·5 grs.	Diff.
10·57	10·62	+ ·05
12·33	12·41	+ ·08
10·89	11·05	+ ·16
12·79	12·99	+ ·20
12·79	12·90	+ ·11
12·53	12·57	+ ·04
14·97	15·08	+ ·11
11·06	11·07	+ ·01
16·69	16·80	+ ·11
11·70	11·82	+ ·12
13·77	13·92	+ ·15
11·05	11·10	+ ·05
11·22	11·29	+ ·07
10·25	10·35	+ ·10
12·60	12·69	+ ·09
13·42	13·61	+ ·19

In every case the results obtained with small quantities in large basins were higher than the large quantities in small basins, and the residues in the former case were quite white, while the latter were more or less brown. Using small quantities only, from one to one and a-half hour's drying is requisite, although longer drying produces no change in weight, one, No. 2 in the list, having been allowed to remain in for six hours, showing not the slightest change; using the larger quantity constancy of weight is not obtainable. I am unable to account satisfactorily for this, but it seems to be connected with the rate of drying, the bulk of the water being evaporated in two or three minutes in the case of the small quantities, while it takes sometimes half an hour with the larger quantities. There is not the slightest difficulty in weighing out a small quantity of milk on a quick weighing balance, the loss by evaporation rarely being sufficient to influence the second place of decimals, and agreement between duplicates being extremely small (not exceeding ·05 per cent.). Wiley has made the same observation, and recommends in his book (*Foods and Food Adulteration, part i.*) either to dry about 2 grs. in a wide platinum basin or to dry 5 grs. on asbestos (*Babcock's method*). He finds that these two methods give identical results, which are higher than the usual method. Mr. Faber (private communication) informs me that he has dried 10 grs. of milk on 10 grs. of pumice, and finds that there is no loss of weight on leaving this overnight in the water oven. It is seen that there is a tendency to under-estimate the total solids, and, therefore, to over-estimate the water in a milk; this I mention because it has been used as an argument in favour of Soxhlet's method of fat extraction, that by its use complete analyses added up to about 100 per cent., Vieth (*Anal. xii.* 59) stating that his analyses add up to 100·15 on the average. I do not, however, propose to discuss this question here, beyond mentioning that Wiley (*Foods, etc., pt. i.*, 115) gives 121 complete analyses of milk, of which the average is

Water.	Fat.	Proteids.	Sugar.	Ash.	Total.
87·22	4·07	2·73	4·90	·67	99·59

which is ·41 below 100 per cent. His estimations were made as follows:

Water: by drying 2 grs. in wide basin, or 5 grs. on asbestos.

Fat: Soxhlet's areometric process.

Proteids: nitrogen by either soda-lime or Kjeldahl, $\times 6.38$. (N.B.—Wiley uses 6.25, but I have recalculated his figures).

Sugar: polarised (Wiley's figures corrected for volume of fat).

Ash: as usual.

THEORY OF FAT EXTRACTION.

The difference between Adams' method and Soxhlet's method having now been shown, to be the result of, on the one hand, extraction of something, not milk-fat, and on the other, incomplete extraction, it only remains to explain, if possible, the theory of the subject. If an ordinary milk is filtered through filter or blotting paper, we find the portion that passes through is almost entirely free from fat, it usually containing .1-.2 per cent.; showing that by capillary attraction between the fibres of the paper the milk is almost entirely separated into two parts, one rich in fat, which remains on the surface of the paper, and the other poor in fat, which passes into the paper itself. Here are the conditions favourable for the complete extraction of the fat. Then again, the milk is spread out over an enormous surface, so that a very thin film is presented for extraction, but I consider that this condition is far less effective in promoting complete extraction than the first separation. In Soxhlet's process the milk is absorbed almost as a whole by the plaster of Paris, and the mass is dried together, so that there is by no means the separation that occurs in the Adams process. This mass is roughly broken up only, and consequently it is not surprising that complete extraction does not take place. With regard to the increasing difference with the increase of fat, the difference is only apparently an increasing one; for instance, say with a milk containing .5 per cent. of fat, there is a difference of .2 per cent., and with a milk containing 5 per cent. there is a difference of .4 per cent. At first sight there appears to be an increasing difference, while in reality, in the first case 40 per cent. of the fat is left behind, while in the second, only 8 per cent. And this is by no means surprising when we consider that almost the whole of the fat is distributed equally through the plaster, and that there is a great tendency for the plaster, etc., to enclose some of the fat, and the enclosing action would be greater the less the quantity of fat, and consequently there would be a tendency for a larger proportion of the fat to be left behind, although on superficial observation the amount of fat left in appears smaller. It is but natural to expect that by fine grinding and regrinding, and long extraction the enclosed portions of the fat should be brought into contact with the ether and become dissolved. It might be brought forward as an argument that the fat by long extraction becomes oxidised and therefore increases in weight; but this is excessively unlikely, and seems to be contradicted by the results of Johnstone (*Anal.* xi. 22), and the figures obtained by the use of the Abbé refractometer (for the loan of which I am indebted to Mr. Faber). Fats extracted by—

Adams' method.	Soxhlet's (modified).	Kieselguhr process.
1.464	1.464	1.463
1.4655	1.462	1.465
1.463	1.463	1.462

These figures are all normal for butter-fat. When, however, fats are heated in

contact with the air, the refractive index rises very perceptibly, thus a sample of butter-fat gave at first 1.463, after six hours in water-bath at 98°-99° C although the weight had not perceptibly changed, the refractive index was 1.467. I do not therefore think that that objection can be maintained; there was nothing extracted by ether from any of the materials used, the paper being extracted beforehand, the plaster giving nothing, and the Kieselguhr, having been ignited before use, also giving nothing.

In conclusion, I would recommend that extracted papers be always used for the Adams process, and that the extraction should be continued for 3-4 hours as a minimum; or that the Kieselguhr process should be used as an alternative; Kieselguhr presenting many advantages over plaster, it being of a porous nature, and easy to reduce to a fine powder, through which ether percolates with comparative ease; and that total solids be taken by drying 1-2 grs. of milk in a wide platinum basin on the water-bath for about an hour and a half, cooling and weighing; this on returning to the bath for another half hour, rarely shows more than one or two tenths of a milligram. difference.

On account of the practical identity of the new formula and the old one worked out by Hehner and myself, I do not give a table for facilitating calculation, the one published in our paper (*Anal. xiii.* 32), and also as a separate sheet, and the "milk scale" (*Anal. xiii.* 65) adequately serving that purpose.

I wish to express my thanks to Mr. Hehner, in whose laboratory this research was carried out, for the kind way in which he has advised and superintended the work, and to Dr. Vieth and Mr. Faber for the loan of instruments, books, etc., and for the way in which they have placed at my disposal a large amount of valuable information.

DISCUSSION.

MR. ALLEN said that Mr. Richmond had brought before them a large number of valuable results, which they would study at their leisure. It was a matter for great satisfaction that they had a young chemist amongst them who was not content simply with accumulating figures, but who also strove to ascertain how far they were due to avoidable experimental error, and how far to some error inherent in the process. Mr. Richmond's suggestion as to the presence of soapy matter in the paper was very plausible, but at the same time he (Mr. Allen) would like to know whether Mr. Richmond had succeeded in extracting any resinous or fatty matter from the paper. As to No. 14 experiment he should think that either the paper was full of soap or else the milk was.

With regard to the notes on the different factors for calculating fat, he thought that was a refinement which they scarcely required. He always used Hehner and Richmond's slide-rule, which usually gave satisfactory results, but after all the method was only one of a preliminary kind.

The questions of the size of the basins and so on were all eminently practical, and they were all indebted to Mr. Richmond for bringing the matter before them.

DR. VIETH said that as Mr. Richmond had alluded to him as being partially responsible that the paper was brought before the Society, he wished to say that he took with pleasure his full share of responsibility upon himself. He thought they had heard an extremely useful and important paper. The figures given bore out a fact which had never been shown before. They all knew that something could be extracted from paper, but here, for the first time, they had a large number of comparative experiments showing that a larger quantity of ether-extract was obtained when milk was dried up on paper extracted in the usual way, than in case the paper was extracted under certain precautions. The experiments answered a number of other questions, for instance, they proved that

plaster of Paris was after all a very good medium for drying milk previous to extraction. He thought the results with regard to the determination of the total solids were of almost more importance than the fat results. He was very much struck to hear that when a very small quantity of milk was dried in a large basin the results should come out higher than when a larger quantity was dried in a small dish. The experiments were most interesting and important, and opened up a new field for inquiries which were really wanted in that direction. With regard to the figures quoted from Dr. Wiley's report to the Commissioner of Agriculture, U.S., he must say that he considered the percentage of proteids so extremely low that he had some doubt whether the results could be correct. Personally he wished to thank Mr. Richmond for his valuable paper, which cleared up some hitherto rather obscure points.

MR. STOKES said that in the figures given of the three processes, if the fat was below 4 per cent. or thereabouts, those methods always gave a lower result than the calculation, whether by the new or the old table, whereas when you come to fats higher than 4 per cent., you always extract more than the calculation shows. With regard to dishes, Mr. Stokes thought some people made a mistake when they said "large"; and he illustrated this on the blackboard, showing that so-called "large" dishes had only higher rims but exposed no larger surface of the fluid. He advocated the use of flat-bottomed dishes.

MR. RICHMOND in reply said that he had not yet collected sufficient of the extract from the coils, but hoped to be able shortly to give some information respecting its composition; with regard to the formulæ he had brought forward a new one, more from the scientific point of view than from its having any practical advantage over the old one. Dr. Vieth had pointed out that the proteids in Wiley's complete analyses were very low, and he agreed with him there, but did not consider that the factor for converting nitrogen into proteids (6.38) was one on which any doubt could be shown, it having been established by Hammarsten and others for both casein and albumen in milk; he considered rather that, if there were a deficiency, it was more to be explained by Atwater's recent work on the soda-lime method, where he found that even experienced observers are often led into grave under-estimations. He did not consider that in plaster extraction a rich milk after one extraction was under the same conditions as a poor milk, for it must be remembered that in a rich milk the proportion of molecules of fat to molecules of diluent (in this case plaster and other milk solids) was much greater than in a poor milk, and consequently in the poor milk each molecule of fat was confined by many more molecules of diluent than in the rich milk, and so would be less easy to extract, and the extraction of some of the fat molecules in the rich milk could not make the others less easy to any marked degree. He had observed that with plaster the fat was extracted less readily than with either paper or Kieselguhr.

With regard to Mr. Stoke's objections to the formula, he thought that they arose from insufficient perusal of the results, as the formula had been calculated from the the results themselves by a method which could not give other than agreement all along the curve, and was convinced that Mr. Stokes would see it in the same light when he had the paper before him and could study it at his leisure; he need hardly say, however, that he must not expect formulæ to agree to .012. As to size of basins, he called a large basin one which exposed a large surface of milk to be evaporated, and a small one in which the opposite was the case. In reply to Dr. Voelcker he admitted that when milk drawn from the cow was not acid, he believed that it had the property of seeming both acid and alkaline at the same time, but by the time that it usually reaches the hands of the analyst it has become slightly but distinctly acid, due, no doubt, to the commencement of lactic fermentation.

[NOTE.—Since reading the paper I have examined the extract from the coils, and find it to consist chiefly of the calcium salt of a resinous acid, containing also sodium and small quantities of aluminium. —H. D. R.]