

THE " NEUSAL " MILK TEST.

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THE difficulties which the " Neusal " milk test (invented by O. Wendler) is designed to overcome do not exist for the analyst working in his own laboratory. Certain points of chemical interest are, however, brought out by an investigation of the method, and also the greatly-extended use to which the mechanical testing of milk is now put, frequently involves the calling in of expert advice on the methods chosen and the results obtained.

It is only necessary to mention, as an example, the valuable work done by the Milk Record Societies, where men are employed to travel round a circuit of farms testing and weighing the milk from each cow in the herd both night and morning, to give an instance of a case where great advantage would accrue were it possible to replace the large quantities of sulphuric acid required for Gerber's acid test by some more portable reagent. To meet this difficulty various methods have been tried, such as the " Sin-acid " method, and Gerber's " Sal " method. In these, solutions of alkaline mixtures of salts were used with amyl or butyl alcohol (added separately), to dissolve the milk.

In 1905 I investigated these methods, and came to the conclusion that both methods were unreliable, because, when the milk is dissolved with an alkaline solution, the fat which separates consists not only of butter-fat, as in Gerber's acid method, but that the volume is swollen by the alcohol which the fat dissolves from these alkaline solutions of the milk. For this reason slight inaccuracies which could hardly be avoided in measuring the alcohol resulted in considerable variations in the volume of fat to be read off; also, there was nothing to show that this had taken place. Allowance is made in Gerber's " Sal " method by taking 10 c.c. of the milk in the same bottles in which 11 c.c. were required for the acid test. Variations of 0.5 to 1.5 c.c. of the amyl alcohol made no difference in the acid test, but in the " Sal " test such variations caused the fat layer to be more than doubled. It is, therefore, not surprising that these methods have not replaced the older acid methods.

The patent of Dr. O. Wendler for the " Neusal " method states that the milk is treated with a mixture consisting of one or more salts of a hydroxybenzoic acid (*e.g.*, sodium salicylate), a sucrate, and a fat-clarifying agent (butyl alcohol); the quantity of fat separating from the milk is then measured. The salt is sent out in a bag containing 260 grms., which is directed to be dissolved in 600 c.c. of water. The salt gives a blue coloured solution; with this is also a bottle containing 255 c.c. of isobutyl alcohol of a certain purity. This has to be added to the blue solution, and the bottle rinsed out with the liquid. The alcohol, though fairly insoluble in water, is very soluble in the solution of the salt. I have also found that a solution of sodium salicylate readily dissolves the alcohol with frothing, making a clear solu-

tion. The total volume of the solution obtained by dissolving the alcohol in the salt solution is about 1,000 c.c.

Two sizes of butyrometer tubes are supplied; to the smaller of these 2 c.c. of this "Neusal" solution are added, and 4.85 c.c. of milk. To the larger 4 c.c. of "Neusal" solution, and 9.7 c.c. of milk. The bottles are then corked, well shaken, and placed in water at 50° C. for four minutes, shaken again, and spun in the centrifugal machine for three minutes. It is then directed to place them in water at 45° C. for three minutes, after which the fat may be read off. If the acid butyrometers are to be used, the "Neusal" solution is diluted with an equal volume of water, and 12 c.c. of the mixture used with 9.7 c.c. of milk.

The sodium salicylate and butyl alcohol completely digest the milk at this temperature, and a very clear reading of the fat is obtained. Examples of the results obtained are given in the following table :

TABLE I.

Sulphuric Acid.	—	—	—	10 c.c.	Rose Gottlieb (a).
Milk.	4.85 c.c.	9.7 c.c.	9.7 c.c.	11 c.c.	Extraction (b).
"Neusal."	2 c.c.	4 c.c.	12 c.c. diluted.		
Milk, No. 1 ...	Small Tubes. { 3.75 3.85 3.45 3.40	Medium. 3.75 3.80 3.45 3.45	Large. 3.70 3.70 3.45 3.45	Acid. 3.75 3.75 3.40 3.45	3.82 (a) 3.82 (b) 3.56 (a) 3.54 (b)
„ No. 2 ...	4.55	4.45	—	4.50	4.57 (b)
„ No. 3 ...	1.45	—	—	—	1.53 (b)
„ No. 4 ...	3.00	3.00	3.15	3.00	3.00 (b)
„ No. 5 ...	—	—	6.00	6.00	6.09 (b)

I find that it is necessary to read the "Neusal" tubes to the bottom of the meniscus, while with the acid tubes I get results agreeing most nearly with the extraction method by reading to the middle of the meniscus. The "Neusal" tubes seem to require slightly longer spinning than the acid tubes; but if they are not spun sufficiently, a pearly appearance is seen at the bottom of the fatty layer, which serves as a valuable warning. Similar results are reported by M. Siegfeld in the *Molkerei-Zeitung* for May, 1910.

It is obvious from the fact that 9.7 c.c. of milk are taken in the same tubes in which 11 c.c. are taken for the acid method, and in both the same volume of fat is read off that in this method as with the "Sal" method we are reading off a volume of fat plus dissolved alcohol; and, as would be expected, experiments in which varying quantities of "Neusal" were taken with the same quantity of milk showed this to be the case.

TABLE II.—ACID BUTYROMETER TUBES TAKEN WITH MILK CONTAINING 3·8 PER CENT. OF FAT.

" Neusal. "	Water.	Milk.	Fat.
c.c.	c.c.	c.c.	Per Cent.
5·5	6·5	9·7	3·40
6·0	6·0	9·7	3·75
6·5	5·5	9·7	3·80
7·0	5·0	9·7	3·90
7·5	4·5	9·7	3·90

The experiments were repeated in tubes to which 0·5 c.c. of melted butter-fat had been added (see Table III.).

TABLE III.—0·5 C.C. BUTTER-FAT AT 50° C., TAKEN IN GERBER TUBES WITH—

Water.	Dilute " Neusal. "	" Neusal " Alcohol.	50 per Cent. H ₂ SO ₄ .	1 per Cent. H ₂ SO ₄ .	1 per Cent. NaOH.	Fat.
21·5	—	—	—	—	—	3·60
9·5	12·0	—	—	—	—	4·50
15·5	6·0	—	—	—	—	3·70
—	21·5	—	—	—	—	5·35
19·5	—	2	—	—	—	4·50
—	—	2	19·5	—	—	3·60
—	—	2	—	19·5	—	4·50
—	—	2	—	—	19·5	4·50

TABLE IV.—EFFECT OF VARYING THE QUANTITY OF ALCOHOL : MILK OF 3 PER CENT. ; FAT, 9·7 C.C. ; 88 GRMS. OF SODIUM SALICYLATE MIXTURE DISSOLVED IN 200 C.C. OF WATER.

Solution.	Water.	Alcohol.	Result.
c.c.	c.c.	c.c.	
5	about 6	less than 1·4	cloudy fat
5	" 6	1·4	3 per cent. fat
5	" 6	1·5	3 per cent. fat, clear
5	" 6	1·6	3 per cent. fat
5	" 6	1·8	3·5 per cent. slightly milky
5	" 6	2·0	3·8 " " "
5	" 6	2·5	5 per cent. cloudy "
5	" 5	3·0	about 6 per cent. cloudy

The variations are not so great as when more alkaline solutions are used (over 7 per cent. obtained). The table shows that the fat dissolves the alcohol from the solution. Varying the quantity of "Neusal" salt produced no alteration in the percentage of fat even if the quantity recommended was doubled; with less salt the readings of fat could not be obtained. In these experiments the alcohol was added separately (1.5 c.c.). The effect of varying the quantity of alcohol is shown in Table IV.

In conclusion, while the method gives satisfactory results when the exact quantity of alcohol is used, the fact that it is not pure fat which is being measured, but a solution, the volume of which will vary with the quantity of alcohol used, makes the method inferior in accuracy to the acid Gerber method, a fault which is likely to be increased if it is put into the hands of inexperienced persons. On the other hand, it is cheaper, safer, and more portable, and might be safely recommended to dairy farmers and others, to replace the acid method where this offers serious disadvantages; but the introduction of another possible error will prevent it from replacing the original Gerber method for more accurate work, though the method is a great advance on the previous alkaline methods.

DISCUSSION.

Mr. RICHMOND remarked that the fact that the method depended upon the differential solubility of iso-butyl alcohol in the fat and in the aqueous portion seemed to be a disadvantage. According to Mr. Golding's figures, the butyl alcohol appeared to be twice as soluble in the fat as in the aqueous portion, and it was obvious that such approximation to correctness as was obtained was due simply and solely to strict adherence to the conditions specified. This meant that one was entirely in the hands of the manufacturers, and, since the composition of the reagents in such cases was generally more or less secret, there was no means of ascertaining for oneself whether the correct conditions were really being fulfilled. The acid method was much more reliable, but here also, even in experienced hands, the results were liable to vary, as was instanced by the fact that, whereas Mr. Golding obtained correct results by reading half-way down the meniscus, he (Mr. Richmond) found it necessary to read, if anything, a little below the meniscus. Both Mr. Golding and himself knew what were the causes of these differences and could allow for them, but the unskilled person could not. As a matter of fact, all these methods—even the acid method—were merely approximate ones, and although in trained hands they might be satisfactory, the results were necessarily less trustworthy than those of actual analysis.

Mr. GOLDING said that the difference in the reading point was probably due to the fact that, while he used a hand-worked apparatus, Mr. Richmond had the advantage of mechanical power, and thus probably was able to extract the fat more completely.

