DETECTION AND ESTIMATION OF COTTON-SEED OIL IN LARD.

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THE method about to be described is a modification of that devised by Milliau, of which an abstract appears in the *ANALYST* (vol. xiii., p. 95). As there described, Milliau's process has failed, in the hands of the present writer, to give satisfactory results; but he does not ascribe the irregularities observed to anything but want of detail; for which, probably, Milliau is in no way to blame, since the article in the *ANALYST* is not an original communication. As worked in the present writer's laboratory the process stands as follows:—

15 grms. of the sample are saponified in a 7-inch porcelain basin with a mixture of 15 c.c. of 30 per cent. NaHO, and 15 c.c. of 92 per cent. alcohol. To commence, the fat is heated to 110°C. The alkaline alcohol must be added in quantities not exceeding 1 c.c. at a time, the temperature not being allowed to fall below 95°C. to 100°C., constant stirring at this part of the operation being most important. If the saponification has been successful, the resultant soap is a smooth thick paste. Boiling distilled water is now added drop by drop, a thin flexible spatula being used to break down the paste. When this has the appearance of smooth starch, water may be run in till a volume of 500 c.c. is reached. Complete solution should follow. 40 c.c. of diluted sulphuric acid (1—10) are now added to the contents of the basin, the liquid is stirred gently and brought to boil for 7 to 12 minutes, then kept just below boiling, until the separated fatty acids fuse to a clear oily layer. The greater bulk of the acid watery liquid is siphoned off, the remainder, with the fatty acids being poured into a clean warm flask with a somewhat long and narrow neck. The fatty acids are freed as nearly as possible by siphonage from the watery under layer, and the flask is filled up with boiling water so as to bring the fatty acids into the neck, by which operation a partial washing is given. 5 c.c. of the fused fatty acids are now transferred by means of a dry, warm, fast-running pipette, into a clean, dry, wide test tube. 20 c.c. of absolute alcohol are added, care being taken to wash the pipette by running the alcohol through it. The contents of the test tube are heated to incipient ebullition in a vessel of boiling water. 2 c.c. of a 30 per cent. solution of silver nitrate are now rapidly poured into the tube, when if even 2 per cent. of cotton-seed oil be present in the sample the characteristic cedar-brown colour is at once developed. Pure lard gives absolutely no colour.

To quantify this reaction, known mixtures of pure lard and refined cotton-seed oil are treated exactly as above, and the colours in the different tubes compared by reflected light against a white background. This must be done simultaneously, for in about seven minutes the colouring matter begins to fall out, and correct comparison is then impossible. In careful hands excellent results are obtainable.

DISCUSSION.

Mr. HEHNER read the following letter from Prof. J. Campbell Brown:—

University College, Liverpool, June 19th, 1888.

DEAR SIR,—As I cannot attend the meeting of Public Analysts' Society tomorrow, I am anxious that if the paper does not deal with the following points, you

should call the attention of the meeting for me to the two errors analysts are liable to make if they trust to the iodine equivalents already published.

1. They are liable to greatly under-estimate the proportion of cotton-seed oil and other foreign fats in adulterated lard. The substance used by about twenty-five of the American firms is a mixture of cotton-seed oil and beef-stearin, the residue from the manufacturer of oleo-margarine. Now, while the iodine equivalent is high, 105 to 110, the iodine equivalent of commercial stearin is low, under 30. And the iodine equivalent of any mixture that can be used for mixing in large proportions with lard is much lower than that of cotton-seed oil—not higher, if so high, as between 80 and 90. Now if any one calculates the quantity of cotton-seed oil in mixed lard giving an iodine equivalent of say 76, using 105 as the iodine equivalent of the foreign fat, instead of 90 or under, it is clear that he will greatly under-estimate the quantity of foreign fat. He will even under-estimate the cotton-seed oil portion of the foreign fat.

2. They are liable, on the other hand, to condemn genuine lard which is more oily than pork fat or lard rendered in this country.

American lard contains as a rule naturally much more olein than our own. If some of the lard oil has not been pressed out, the high iodine equivalent of lard oil—from 75 to 80—so raises the one equivalent of the thin oily lard, that an analyst judging mainly from the iodine equivalent would infer the presence of cotton-seed oil where there was only excess of lard oil. It is necessary, therefore, to be very careful in determining, first the presence of some cotton-seed oil by safe qualitative tests before determining the iodine equivalent; and it is further necessary, in order to avoid both the errors I have mentioned, to take into consideration the consistency of the sample, and attend to tests for beef-stearin, in drawing any conclusion from its iodine equivalent.

Kindly bring these points before the meeting as from me, unless they are fully gone into in the paper.—I am, yours truly,

The Secretary, Society of Public Analysts.

J. CAMPBELL BROWN.

Mr. HARLAND said that the American manufacturers' plan was to express the lard oil from the lard and replace it by cotton-seed oil. Working on these lines in the laboratory, he had found that about ten per cent. of lard oil can be squeezed out of genuine lards, but that adulterated samples of various American manufacturers gave thirty, forty, or fifty per cent. of what appeared to be, by its sp. gr. and solidifying point, cotton-seed oil.

Mr. WATSON GREY gave the following figures for the percentage of iodine absorbed by various kinds of lard when treated by Hübl's process:—

Kind of lard.	Iodine absorption.
From omentum of hog	49·5
Market lard (bought in Liverpool)	49
From omentum of sow	55·5
From back of pig	65

He would fix the average at 57. If this was accepted, it would account for the fact that the President, having taken 62 as the standard number, had found that, whereas the sp. gr. of one adulterated sample corresponded to thirty per cent. of cotton-seed oil, the iodine absorption indicated only fifteen per cent., without having recourse to the supposition that tallow was present as well as cotton-seed oil. His experience went to show that the silver nitrate test—at least, in its unmodified form (without the addition of free nitric acid)—was rather unsatisfactory.

Mr. Fox stated that he had recently found fifty per cent. of earth-nut oil in lard oil, detecting it by the altered sp. gr. and the presence of arachidic acid.

Mr. JONES, in reply to a remark of the President's on his practice of taking the sp. gr. of fats at 100° F., admitted that it was rather an old-fashioned plan, and contemplated changing it. He had not found it necessary to use concentrated aqueous caustic alkali and fresh alcohol when saponifying oils in order to get white fatty acids, as proposed by the President, the alcohol he obtained in Wolverhampton not turning brown from the action of caustic alkali, even when kept for several months.