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ON THE DETECTION OF COTTON-SEED OIL IN LARD.

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I HAVE recently had occasion to look into the question of the adulteration of lard with cotton-seed oil, and for the purpose of comparison have analysed certain samples, known

and suspected to be adulterated, together with certain specimens of which the genuine nature was beyond suspicion.

The following table shows some of the results obtained. The figures given by the lard marked "Omentum" were yielded by a specimen rendered in my laboratory :—

	Omen- tum Lard.	Leaf Lard.	English Lard.	American Lard containing Cotton-seed Oil.	Mixture of unknown nature.	Suspected sample.
ORIGINAL FAT.	Melting point; °C	39.0	40.0	39.0	37.5	40.0
	Solidifying point; °C	26.5	32.0	27.0	27.5	30.5
		rising to 27.5				
	Plummet gravity at 99°C	.8602	.8620	.8608	.8648	.8628
FATTY ACIDS.	Iodine absorption, per cent.	55.4	60.5	62.0	82.5	68.8
	Melting point; °C	39.0	39.5		39.5	
	Solidifying point; °C	38.7	38.5		37.5	
		rising to 39.0	rising to 38.8		rising to 38.5	
	Plummet gravity at 99°C	.8372	.8385		.8450	.8385
	Mean combining weight	274.5			276.8	
	Iodine absorption, per cent.	58.3	65.3		70.4	64.8
	Oleic acid, etc., per cent.	58.4			57.8	
	" " Iodine absorption	87.4			(94.6)	
	Milliau's nitrate of silver test	white	white	grey	Marked black- ening.	Marked black- ening Sensible dark- ening.

The following figures are the recorded results of experience with tallow, lard, and cotton-seed oil :—

	Tallow.	Lard.	Cotton-seed Oil.
ORIGINAL FAT.	Melting point; °C	28—45	fluid
	Solidifying point; °C	33—48	
	Specific gravity at 99°C	.862	.872
	Iodine absorption, per cent	40	59—62
FATTY ACIDS.	Melting point; °C	45	35—36
	Solidifying point; °C	43	30
	Specific gravity at 99°C	—	846.7
	Iodine absorption, per cent.	41.3	64.2

In addition to these Müller has recorded the following iodine-absorptions :—Beef-suet, 38.4 ; lard, 55.0 ; cotton-seed oil, 107.9.

Mr. J. Carter Bell informs me that he recently found 51·5 as the iodine-absorption of a sample of leaf-lard which he prepared himself. The same sample yielded 51·2 in the hands of Mr. R. Williams, while Mr. E. W. T. Jones found 46·57.

I have this day received from Mr. Chas. M. Blades a sample of cotton-seed stearin, which is now a commercial product. I have attempted in vain to obtain this article for years past, in consequence of the result recorded by Dr. Muter (*ANALYST*, vii., 93), who described the adulteration of lard with this product. The *ordinary* cotton-seed stearin of commerce is really the stearic acid from cotton-seed oil, and quite a different article from the sample of neutral fat now before you. The figures given in the following table, in the column headed "Cotton-seed Fat," are those yielded by the sample in question :—

	A. Cotton-seed Fat.	B. Cotton-seed Oil.	C. Cotton-oil Acids from B.
Plummet gravity at 99°C.....	·8684	·8725	·8476
Melting point.....	40°C	—	35°C
Solidifying point	31 rising to 32·5°	—	32°
Iodine absorption	89·8	108—110	115·8
Saponification equivalent	—	285—294	289*
Acidity (= Oleic Acid).....	·34	trace	97·6

The results I have obtained substantially confirm those of Mr. Jones, Mr. Rowland Williams, and other previous observers, and appear to show that lard differs materially in its iodine-absorption from beef-stearin on the one side and cotton-seed oil on the other. There is also a marked difference in the specific gravity of lard and cotton-seed oil, and this difference is also noticeable in the fatty acids. On the other hand, lard and beef-fat are substantially of the same density. This difference is very important, as it would enable one to distinguish a mixture of beef-stearin and cotton-seed oil, having an iodine-absorption of about 60, from genuine lard. Thus, while the proportion of the adulterant in a mixture composed of lard and cotton-seed oil *only* can be ascertained with considerable accuracy by determining the iodine-absorption, the estimation will be below the truth if beef-stearin be present. On the other hand, the presence of beef-stearin does not interfere with the deduction to be drawn from the increased specific gravity of the melted sample. Hence this method, though not affording more than approxi-

* Mr. Rowland Williams, in his recent paper on the characters of certain fatty acids (*ANALYST*, xiii., 89), quotes "other observers as recording a range of 275 to 337 for the mean combining weight of the fatty acids from cotton-seed oil." On reference, I found, to my surprise, that the anomalous figure 337·2 was one recorded by myself. I feel sure this figure is erroneous, though I am unable, at this distance of time, to ascertain the cause with certainty; but the specimen said to have that combining weight was of considerable age when analysed.

mate results, is calculated to do very good service in conjunction with the iodine-absorption.

As a qualitative test, I have found Milliau's modification of Becchi's nitrate of silver reaction to be very valuable. It is quite possible that it may be used for obtaining rough quantitative results, but I have not had sufficient experience of it in this direction to express a positive opinion. Milliau's test certainly seems to be a great improvement on that originally proposed by Becchi, in which rape-oil and amylic alcohol were used with no obvious purpose.

My experiments did not indicate that either the melting point or solidifying point of the original sample, or of the fatty acids therefrom, is capable of affording any useful information.

The iodine-absorption of a sample of lard-oil was found by Mr. Rowland Williams to be 73 (private communication). A small quantity of lard-oil which I obtained by pressure from lard of known purity showed an iodine-absorption of 74. On the other hand, a sample of lard-oil several years old, believed to be pure, gave only 41, and Müller has recorded an iodine figure of 47.2.

It will be observed that I have determined the iodine-absorptions of the fatty acids of some of the samples, in addition to making the determinations on the original fats. This plan has been recommended by Rose, and Mr. R. Williams has recently recorded some experiments made in the same way. There is one important practical advantage in operating on the fatty acids, and that is that in consequence of their solubility in alcohol, the use of chloroform can be entirely dispensed with. Chloroform is liable to have more or less reducing action on Hübl's iodine solution, and hence a quantity equal to that used in the test must be employed in the blank experiment for ascertaining the strength of the iodine solution. If, as sometimes happens, more chloroform has to be added during the analysis to maintain proper solution, an uncertainty is introduced, unless another blank experiment be made with a larger quantity of chloroform. All this is avoided if the fatty acids be employed instead of the original fat, and the chloroform wholly omitted. The fatty acids are best prepared by saponifying the fat with rectified spirit (not methylated), and the saturated aqueous solution of caustic soda, recommended by Wollny for use in Reichert's butter process.* A perfectly colourless product is thus obtained.

I may take this opportunity of cautioning employers of Hübl's iodine process, of the importance of adhering strictly to the prescribed process, unless the modification adopted be fully described and its effects allowed for. Already there are indications of deviation in detail in manipulation which are liable to cause much trouble and confusion at a moment when analysts are trying to ascertain within what range the absorptions of lard, cotton-seed oil, and other fats are liable to vary.

I have made some experiments with a view of isolating from adulterated lard the fatty acids forming lead salts soluble in ether. The high iodine-absorption of cotton-seed oil being presumably due to the presence of the glyceride of linoleic or similar acid, it would seem to follow that the iodine figure should be higher after the separation of

* I have been using Wollny's modified butter method extensively, and with the most satisfactory results. The improvement in sharpness and certainty amply compensates for the little extra time and trouble involved.

the acids of the stearic series. My experiments in this direction have not been very satisfactory, and the tedious nature of the process is such as to discourage one from following up the matter, unless absolutely necessary. I think that the experience we now have shows that cotton-seed oil can be detected with certainty in lard, even when present in very moderate quantity, if it be the sole adulterant. The determination can, at present, only be affected with tolerable accuracy in the absence of beef-stearin. The detection of cotton-seed oil is quite easy, in any case, but the determination becomes more uncertain if beef fat be present. From the combined results of the determinations of iodine-absorption and specific gravity it would be possible to form an idea of the proportion of beef-stearin present in a mixture of that substance with lard and cotton-seed oil.

Of course it is necessary to multiply results and to avoid the error of laying down, at this stage, too rigid limits of variation; but the tools we now possess, if used skilfully, appear to be amply sufficient to solve the problem presented to us.

I have to thank Mr. W. Chattaway and Mr. W. H. Barraclough for their zealous co-operation and assistance.