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A PRACTICAL METHOD FOR THE DETECTION OF BEEF FAT IN LARD.

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The regulations of the Secretary of Agriculture governing meat inspection require that all trade labels for meat food products shall show the true names of the products to which they are affixed. All products labeled "lard" must consist only of the rendered fat from edible portions of the carcasses of hogs. Products which contain lard mixed with other edible animal or vegetable fats are known as "lard compounds," or "compounds," depending upon the proportion of lard contained in them. The regulations referred to have necessitated the examination of a very large number of samples of prepared fats, and in the course of such examinations one of the most difficult problems presented is the detection of small quantities of beef fat. This may be added in the form of beef tallow or oleostearin, usually the latter, and is employed to secure a greater degree of firmness in a lard of poor grade, particularly for use in warm climates. Beef fat is also added to give the consistency of lard to products to which cotton-seed oil or some other vegetable oil has been added.[a]

EXISTING METHODS OF TESTING LARD.

THE BELFIELD TEST.

Aside from the determination of the chemical and physical constants, the test which until quite recently has been almost universally employed depends chiefly upon a microscopic examination of the solid glycerids crystallizing from solutions of the fat in ether under certain conditions of temperature and concentration. This method, known as the Belfield test, has been subjected to many modifications at the hands of various investigators, chief among the number being Pattinson,[1b] Goske,[2] Stock,[3] Cochran,[4] and Solstein.[5]

[a] The possibility of animal fats other than beef tallow and oleostearin being used as adulterants in lard in this country is so extremely slight that a discussion of methods for their detection will not be undertaken in this paper.

[b] The figures refer to list of literature at end of circular.

In case the percentage of beef fat in lard is comparatively high, this method and its modifications are applicable with a reasonable degree of accuracy, but in samples of lard where the amount of added beef fat is small the test is not always so satisfactory. Extensive trials of the Belfield test by the writer and others in the Biochemic Division of the Bureau of Animal Industry upon lard containing small quantities of added beef fat have led us to doubt its entire reliability, as samples of known pure lard have deposited crystals, which, when subjected to the usual treatment and examination under the microscope, have shown forms in some fields so closely resembling those obtained in a like manner from mixtures of small amounts of beef with lard as to render their differentiation extremely difficult. Goske states that German home-rendered lard does not yield the crystalline plates, but crystallizes instead in needles, which are not readily distinguishable from those yielded by beef stearin under the same conditions.

RECENT METHODS.

In an exhaustive article by Polenske[6] the difference in degrees between the melting point and the solidifying point of the fat under examination is utilized as a means for the detection of foreign admixture. The author does not, however, claim to detect additions of beef tallow to lard in less amount than 10 per cent, or of oleostearin to lard in less amount than 5 per cent.

In a recent contribution to this subject by Alexander Leys[7] the melting points of the solid glycerids is taken as a basis for the detection of beef suet in lard. In this method the elimination of olein is accomplished by means of its oxidation or modification by the action of mercuric acetate in glacial acetic-acid solution. The saturated solid glycerids are unaffected, and are rendered free from the modified olein by washing with absolute alcohol, in which menstruum the latter is readily soluble. The low iodin value of these solid glycerids is an indication of their purity. A number of samples have been examined by this method and good comparative results have been obtained.

It has been the present writer's custom since the appearance of this method to employ it as a means of confirming the method described later in this paper when the melting point of the glycerids obtained in the crystallization from ether has so closely approximated the limit prescribed for purity as to carry a feeling of slight uncertainty regarding the actual presence of a small amount of adulterant. Such cases have been comparatively few, but when they occurred the indicated presence of the foreign fat has been confirmed.

The writer was unable to determine the melting point when using the method employed by Leys, and after numerous attempts this was

abandoned and the method described in this paper (page 4) was substituted. The standard temperature for lard as given by Leys, 60.4° to 61° C., was found by this method to be from 62° to 62.2° C., and the presence of beef fat was positively indicated when the glycerids melted below 61.5° C. In the determination of the melting point with the use of a tube the phenomenon of the so-called double melting point is observed, the body melting clear while the particles remain separate, then later become opaque, and finally perfectly transparent again and then unite readily.

To this feature was probably due the difficulty experienced in using the mercury bath advocated by Leys for determining the melting point, as even with the use of a hand lens and careful observation it was impossible to observe these changes in the particles on the surface of the mercury with sufficient accuracy to prove satisfactory.

EXPERIMENTAL WORK.

In consideration of the uncertainty in the results obtained by some of the foregoing tests and the time required for the completion of others, it became necessary to devise some new method or to adopt some modification of an already existing one, which would permit the more rapid detection of an adulterant of this nature beyond reasonable doubt. Considerable experimental work was carried on with this end in view, and crystallizations from a large number of solvents and mixtures were tried at various degrees of temperature and with various dilutions, in the hope that the crystalline structure of the glycerids deposited would be more clearly defined and that more marked differences would be noticeable in the crystals obtained when a small percentage of beef fat was known to be in the mixture. The work met with but little success, however, and it was finally decided that a microscopic examination alone would fail to yield the desired results when only small amounts of this adulterant were present.

Attention was then directed to the melting points of the solid glycerids crystallized in the manner indicated from rendered beef fat and from lard, and it was observed that under certain conditions of crystallization a difference of several degrees existed in their points of fusion. With this in mind, efforts were made to ascertain whether or not this fact could be utilized in the determination of the presence in lard of an adulterant of the character named.

By determining the melting point of the solid glycerids crystallized from a large number of different lards it was established that under like conditions of crystallization the melting point of such glycerids was practically the same. It was further established that under the same conditions of crystallization the melting point of the solid

glycerids crystallizing from beef tallow or oleostearin was also uniform.

It was noted, however, that the melting point of the glycerids obtained from beef fat was several degrees lower than the melting point of the glycerids obtained from lard, while various mixtures of the two fats yielded crystalline deposits with melting points registering between these two constants. The point of fusion of the crystals from pure lard was lowered in a degree proportional to the amount of beef fat present, but this ratio was maintained only up to a certain percentage. (See Table 1.)

With these facts established it remained to determine the most favorable conditions under which the crystallizations should take place and the most efficient method for subsequent treatment of the crystals in order that the greatest difference in melting point between the glycerids obtained from the two fats could be secured. After a large number of experiments the following method was decided upon:

TECHNIQUE OF METHOD.

Five grams of the warm filtered fat is weighed (on a balance sensitive to 0.1 gram) in a glass-stoppered graduated cylinder of 25 c. c. capacity, 150 to 175 mm. in height, with an internal diameter of about 18 mm., and warm ether is added until the 25 c. c. graduation is reached. The glass stopper is securely replaced and the cylinder is shaken vigorously until complete solution of the fat takes place. The cylinder with its contents is then allowed to stand in a suitable place where a constant temperature, at which it is desired to have the crystallization proceed, may be maintained. (An apparatus described by Rogers proved efficient for the maintenance of this constant temperature.)[a] After eighteen hours the cylinder is removed and the supernatant ether solution carefully decanted from the crystallized glycerids, which are usually found in a firm mass at the bottom of the vessel. Cold ether is then added in three portions of 5 c. c. each from a small wash bottle, care being taken not to break up the deposit while washing and decanting the first two portions. The third portion is, however, actively agitated in the cylinder with a sharp rotary motion and by a quick movement transferred, with the crystals, to a small filter paper. The crystals are then washed with successive small portions of the cold ether, with the use of the wash bottle, until 10 to

[a] It is necessary to observe great caution in the use of this form of apparatus, as the sparking of the thermo-regulator is a source of danger if the solutions are carelessly handled. A better form for this work would be one in which the temperature is controlled by a circulating hot-water system heated by a small lamp outside of the box, the regulation of which could be adjusted by using one of the many forms of gas regulators on the market.

15 c. c. has been used, dependent on the amount of crystals. Then by means of a slight exhaust the small amount of remaining ether is rapidly removed. The paper with its contents is then transferred to a suitable place, where it should be spread out and any large lumps of the glycerids broken up by gentle pressure. When dry the mass is thoroughly comminuted and the melting point of the crystals determined.

As the difference between the melting points of the glycerids obtained in this manner from beef fat and lard is not very great, being only about 3.5 degrees, and as the writer has mentioned a standard melting-point temperature for the glycerids of pure lard obtained under certain conditions, a description of the apparatus used in determining the melting points, together with its manipulation, is essential and may be of some assistance.

DETERMINATION OF THE MELTING POINT.

A large test tube approximately 150 by 25 mm., containing water (free from air) into which the bulb of a thermometer [a] with the melting-point tube attached is immersed, is placed in a beaker of water and so adjusted that the surface of the liquid contained in the two vessels is at the same level. The water in the beaker should be heated rapidly to about 55° C. and that temperature maintained until the thermometer carrying the melting-point tube registers between 50° and 55° C., then heat is again applied and the temperature of the outer bath carried somewhat rapidly to 67° C., when the lamp is removed. The melting point of the crystals is regarded as that point when the fused substance becomes perfectly clear and transparent. The use of a dark background placed about 4 inches from the apparatus will prove of advantage.

The melting-point tube should be of about 1 mm. internal diameter, sealed at one end and with a slight flare at the other extremity, in order that the loading may be expedited. The amount of the substance taken for each determination should be approximately the same and should occupy a space about 9 mm. in length, being somewhat firmly packed in the lower end of the tube by tapping it sharply on a hard surface. The water in the outer bath should be agitated frequently during the determination.

POSSIBLE SOURCES OF ERROR.

In applying the foregoing method too great care can not be exercised with the preparation of the sample. The presence of water, the incomplete solution of the fat in the ether, or the presence of small

[a] The thermometer used was one graduated in one-fifth degrees and extending from 0° to 100° C.

particles of extraneous matter may interfere with the process of crystallization, frequently causing it to proceed too rapidly and resulting in the formation of a large mass of small fluffy crystals instead of the compact mass of larger crystals desired. These fine crystals render the preliminary washing by decantation with ether difficult, and they also persistently hold the unsaturated glycerids in larger amount than is desirable. The temperature at which the crystallization should be allowed to proceed should not be less than 15° C. nor more than 20° C., with the best results obtainable in the neighborhood of an average between the two. Although larger crystals are formed at the higher temperature (20° C.), only lards of high grade afford crystalline deposits in working quantity, and in many cases where lards of inferior grades are tested the amount of solid glycerids entering into their composition is so reduced as not to yield any deposit at all.

SOME RESULTS OF PRACTICAL APPLICATION OF THE METHOD.

A large number of samples of lard of known purity as well as samples rendered in the laboratory from selected pork fat were used in this work, while beef tallow and oleostearin known to be pure were used as the adulterant. Below are given the records of a few results with varying percentages of beef fat and lard:

TABLE 1.—*Melting points of glycerids obtained from lard and beef fat, alone and in mixtures.*

Fat.	Melting point.	Fat.	Melting point.
	°C.		°C.
Pure lard	63.8	Lard with 6 per cent beef fat added	62.2
Lard with 1 per cent beef fat added	63.4	Lard with 7 per cent beef fat added	61.6
Lard with 2 per cent beef fat added	63.2	Lard with 8 per cent beef fat added	61.6
Lard with 3 per cent beef fat added	63.0	Lard with 9 per cent beef fat added	61.6
Lard with 4 per cent beef fat added	62.8	Lard with 10 per cent beef fat added	61.5
Lard with 5 per cent beef fat added	62.5	Beef fat	60.6

The lard used in this case was a pure leaf lard with melting point of 42.4° C., and iodin number 59.73. The beef fat was a sample of beef tallow with iodin number 40.13 and melting point 47° C.

Thirty samples of absolutely pure leaf lard rendered under the most careful supervision in twenty different packinghouses throughout the United States when examined by this method gave a maximum range in temperature of 0.5° C. between the highest (64.1° C.) and the lowest (63.6° C.).

Among the large number of samples of all varieties of lards examined only one failed to yield crystals of glycerids when the ether solution was allowed to stand in the cold box for the required num-

ber of hours. This lard was obtained from pigs' feet rendered at 283°F. in a closed tank. The amount of solid glycerids entering into its composition was evidently unusually low, as the iodin value (72.31) closely approximated that of samples of lard oil.

That the presence of added lard stearin does not materially interfere with the temperature reading for pure lard is shown in Table 2.

Table 2.—Melting points of the solid glycerids of lard and mixtures of lard and lard stearin.

Fat.	Melting point.
	°C.
Lard	63.6
Lard and 10 per cent lard stearin	63.8
Lard and 15 per cent lard stearin	63.6

This same lard, with 5 per cent oleostearin added, gave a melting point for its solid glycerids of 61.5° C.

The lard used in this instance was a head lard, rendered at 283° F. in a closed tank for 5 hours, with an iodin value of 67.8 and melting point of 35° C. The lard stearin used had an iodin value of 47.3 and melted at 45° C.

Cotton-seed oil does not interfere when present in reasonable amount, but when in considerable quantity the crystallized glycerids are not deposited at the prescribed temperature in the ice box.

A sample containing both lard and oleostearin (about 15 per cent of the latter) in mixture with cotton-seed oil (about 30 per cent) gave deposits in which beef fat predominated to such an extent as to cause a reduction of the melting point to 60.6° C. These crystals were rewashed in order to eliminate any possibility of retained cotton-seed oil, but when dried in the usual way and their melting point determined it was found to be unchanged.

Samples of lard and mixtures of the same lard with beef fat (oleostearin) were submitted to two assistants unacquainted with the percentages of beef fat contained in each sample, and the following results were obtained:

Fat.	Melting point of crystallized glycerids.	
	A.	B.
	°C.	°C.
Lard	63.6	63.6
Lard and 1 per cent beef fat	63.0	63.0
Lard and 2 per cent beef fat	62.6	62.4
Lard and 3 per cent beef fat	62.4	62.6
Lard and 5 per cent beef fat	61.9	62.2

It is essential that great care be exercised in washing the crystals, as otherwise the unsaturated glycerids would be retained in the deposit in varying amounts in the hands of different experimenters and a standard temperature could not be established.

The method should be applied to samples of the animal fats mentioned, and to mixtures of these fats, and standard temperatures obtained for use as an index of comparison with the melting point of the samples under investigation. If possible this should be done in all cases.

The presence in the crystals of a small proportion of glycerids containing olein is readily shown by the percentage of iodin absorbed by the deposit. This percentage has been found to be practically constant both in high and in low grade lards: it is quite low, and the small amount of these glycerids present does not materially interfere with the interpretation of results, as they are of a comparative nature.

COMPARISON WITH THE LEYS METHOD.

The following results were obtained for a sample of pure lard and for the same with 1 per cent and 5 per cent of added beef fat (oleostearin) when examined for comparative purposes by the Leys method (using a tube in determining the melting point) and by the method described in this paper:

Fat.	Leys's method.	Writer's method.
	°C.	°C.
Pure lard	62 to 62.2	63.6
Pure lard and 1 per cent beef fat	61.6 to 61.8	63 to 63.2
Pure lard and 5 per cent beef fat	59.2	60.8

CONCLUSION.

Where in the daily routine of work a large number of lard samples are to be investigated for suspected adulteration with beef fat the time consumed in the process of examination, as well as the expense involved, must be taken into consideration in conjunction with its efficiency. The method as described by the writer has afforded very good results both in his hands and in those of a number of his coworkers and is considerably shorter than the method proposed by Leys.

The application of both of these methods has proven efficient in detecting added beef in samples of suspected "pork sausage." The fat was obtained by subjecting the meat to a temperature of 125° C., and then pouring off and filtering the separated melted fat.

In samples in which the amount of fat as obtained in this manner was not sufficient, extraction of the fat from the water-free finely minced product by means of ether and evaporation of the solvent was necessary. The subsequent procedure was then carried out as described in the method given.

Finally it may be said that when the crystallized glycerids of any questionable sample of lard examined by the method described in this paper show a melting point below 63.4° C., the presence of beef fat should be suspected, while a melting point of 63° C., or below, can be regarded as positive evidence that the sample under examination contains beef fat as an adulterant.

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