out changing the quantities of the other ingredients. If this be done, the precipitate obtained is not pure white. Moreover, the end-point is not sharp enough when highly colored products are analyzed.

DETERMINATION OF SUGAR IN MEATS.[1]

By A. Lowenstein and W. P. Dunne.

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The determination of "Reducing Sugar" in meats as published in several bulletins of the United States Department of Agriculture and the Association of Official Agricultural Chemists is substantially as follows:

"Boil 100 grams of the finely divided meat for 15 or 20 minutes in a 500 cc. graduated flask, with a convenient volume of water. Add a few cubic centimeters of normal lead acetate, cool to room temperature, make up to the mark with water, and filter through a folded filter. Remove the lead, and determine reducing sugar as *d*-glucose, etc." (*Bulletin* **107**, *Bureau of Chemistry*, page 111).

Upon considering this method one will note that there are 100 grams of meat present in a 500 cc. flask and in addition a voluminous precipitate which results upon the addition of lead acetate. It is obvious at a glance that to use a portion of the filtrate from this mixture and regard it as an aliquot part involves an error, which in the present method is of considerable magnitude, inasmuch as the percentage of reducing sugar present in meats is usually small, so that the error involved becomes of considerable importance. As this is the only method on record known to the writers for the determination of sugar in meats, its use is naturally also extended to the determination of sucrose in meats as well, and here the same error is involved, the magnitude of which is shown in Tables I, II and III. We find the method as outlined above to be rather difficult to manipulate, particularly in the case of meats of high fat content, such as bacon. The introduction of 100 grams of meat into a 500 cc. flask is tedious, when a large number of determinations are to be made; filtration after clarifying with lead acetate is difficult and the use of the lead salt causes a precipitate error, and another precipitate error is involved on removing the excess of lead.

In view of the above we desire to offer a method which has been used satisfactorily in this laboratory, the principal advantages of which are as follows: That it eliminates the error indicated above; that one can conveniently and accurately determine the percentage of nitrates (saltpeter) in the same portion; that it is not necessary to use clarifying agents,

[1] Read before the Agriculture and Food Sections of the American Chemical Society at the New Haven Meeting.

and consequently eliminates the precipitate errors attending their use; and in the case of fat meats the fat is easily removed, so that the determination of sugar in this kind of meat is less difficult than in the case of lean meats. For the final determination of the reducing sugar or inverted sucrose we employ the Pavy method, which we find rapid and accurate, and well adapted to this kind of work.

But very little has ever been published concerning the presence of sugar in meats. In the cured meat products of commerce one finds both sucrose and reducing sugar present, although not always at the same time. Some fresh meat products, notably livers, show considerable reducing sugar when determined by the present method. It is the intention of the authors at a later date to publish a paper, showing the quantities and form in which sugar exists in the commercial cuts of fresh and cured meats in the uncooked, smoked and cooked states. In the present paper all results have been calculated to sucrose ($C_{12}H_{22}O_{11}$), principally because this is the form in which sugar was originally added to the meat.

It is found convenient to employ 300 grams of meat for the determinations and the extract from this quantity, when brought to a volume of 200 cc., as described below, yields a solution of a satisfactory concentration for the determination of reducing sugar, sucrose, and nitrates in aliquots thereof. Considerably smaller quantities can be successfully employed, but when the percentages of reducing sugars and nitrates are very low, as is frequently the case, the above quantity is found to be very satisfactory.

Table I shows results obtained by adding known amounts of pure sucrose to *fresh* meat, and determining the percentage of sugar, by the A. O. A. C. method given above, and also by the method herein described. The A. O. A. C. method was followed in detail, employing the largest aliquot portion of the filtrate practicable, as described in *Bulletin No.* **13**, *Bureau of Chemistry*, p. 1403. The solutions were inverted by the official method of the A. O. A. C. For the determination of nitrates the Schloesing-Wagner method was employed. The saltpeter and sugar were added to the meat at the same time.

TABLE I.

Lab. No.	Wt. of meat. Gms.	$C_{12}H_{22}O_{11}$ added. Wt.	Per cent.	Vol. of solution.	cc. Pavy sol.	Titration. cc.	Per cent. $C_{12}H_{22}O_{11}$ found.	KNO_3 added. Wt.	Per cent.	cc. NO gas.	cc. orig. sol. used.	Per cent. KNO_3 found.
33647	300	1.0	0.33	200	50	4.7	0.34	0.3	0.10	34.4	100	0.101
33648	300	1.0	0.33	200	50	5.4	0.30	0.9	0.30	26.1	25	0.306
33649	200	1.5	0.75	200	100	6.45	0.73	...	....	....	...	.....
33650	150	1.5	1.00	200	100	6.1	1.03	...	....	....	...	.....

A. O. A. C. METHOD.

Lab. No.	Wt. of meat. Gms.	$C_{12}H_{22}O_{11}$ added. Wt.	$C_{12}H_{22}O_{11}$ added. Per cent.	Vol. of solution.	cc. of orig. sol. evaporated, inverted and diluted to 100 cc.	cc. Pavy sol.	Titration, cc.	Per cent. $C_{12}H_{22}O_{11}$ found.	$C_{12}H_{22}O_{11}$ increase over amt. added. Per cent.
33652	100	0.00	0.00	500	300	50	No reduction.	0.00	0.00
33653	100	0.50	0.50	500	390	100	8.5	0.72	0.22
33654	100	0.50	0.50	500	250	50	5.95	0.79	0.29
33655	100	1.00	1.00	500	145	50	6.4	1.29	0.29
33656	100	1.00	1.00	500	250	50	3.65	1.30	0.30

100 cc. Pavy solution will reduce 0.050 g. glucose, equivalent to 0.0475 g. sucrose.

The fresh meats employed in the above experiments contained practically no reducing sugar, a phenomenon which is observed particularly after meat has been kept for some time, and which will be discussed in a future paper. The following table shows results of adding known amounts of sucrose to *fresh* meat which contains an appreciable amount of reducing sugar, but no sucrose.

TABLE II.

A. O. A. C. METHOD.

Lab. No.	Wt. of meat. Gms.	$C_{12}H_{22}O_{11}$ added. Wt.	$C_{12}H_{22}O_{11}$ added. Per cent.	vol. of solution.	cc. of orig. sol. evaporated, inverted and diluted to 100 cc.	cc. Pavy sol.	Titration. cc.	Per cent. $C_{12}H_{22}O_{11}$ found.	$C_{12}H_{22}O_{11}$ increase over amt. added. Per cent.
33663	100	0.00	0.00	500	300	50	14.7	0.22	0.00
33664	100	0.25	0.25	500	250	50	7.3	0.65	0.18
33665	100	0.50	0.50	500	250	50	5.0	0.95	0.23
				METHOD HEREIN DESCRIBED.					
33666	300	0.00	0.00	200	...	50	8.0	0.20	0.00
33667	300	0.75	0.25	250	...	50	4.3	0.46	+0.01
33668	300	1.50	0.50	250	...	50	2.9	0.68	—0.02

Table III shows results of sugar determinations on several samples of *cured* meat, and the increase in sugar found after a known percentage of pure sucrose has been added to the cured meat.

TABLE III.

Lab. No.	Kind of meat.	Wt. of meat. Gms.	Vol. of solution.	Aliquot used.	cc. Pavy sol.	Titration. cc.	$C_{12}H_{22}O_{11}$ added. Wt.	$C_{12}H_{22}O_{11}$ added. Per cent.	Per cent. $C_{12}H_{22}O_{11}$ found.	$C_{12}H_{22}O_{11}$ increase over amt. added. Per cent.
33657	Pork ham	300	200	75 diluted to 100 cc.	100	9.6	0.00	0.00	0.44	0.00
33658	" "	300	200	75 " " 100 cc.	100	5.75	0.75	0.25	0.72	0.03
33659	" "	300	200	75 " " 100 cc.	100	8.65	0.00	0.00	0.48	0.00
33660	" "	300	200	75 " " 100 cc.	100	4.25	0.75	0.25	0.76	0.03
33661	Beef ham	300	200	.....	100	5.4	0.00	0.00	0.56	0.00
33662	" "	300	200	.....	100	3.8	0.75	0.25	0.83	0.02
				A. O. A. C. METHOD.						
33661	Beef ham	100	500	300 cc. evap. in 100 cc.	50	4.5	0.00	0.00	0.81	0.00
33662	" "	100	500	300 cc. " " 100 cc.	50	3.0	0.25	0.25	1.31	0.25

The method in detail is as follows: Place 300 grams of the finely divided meat in a white enameled iron casserole of about a liter capacity, add 200–300 cc. of water, and boil for 30 minutes over a free flame. Allow to cool, then strain liquid through muslin, thoroughly express the liquid from the meat, evaporate in another casserole, similar to the one described above, over a free flame; return the partially extracted meat to the original casserole, add 200 cc. more water and boil for 15 minutes, cool, and strain as above, and add the expressed liquid to the first portion. When saltpeter is determined, this operation is continued until the extract ceases to respond to the diphenylamine test for nitrates. Two extractions are usually sufficient to remove all sugar and saltpeter, unless the latter is present in abnormal quantity. Thoroughly wash the meat and cloth with hot water, and evaporate the united extract to about 300–400 cc. Then transfer to a 500 cc. separatory funnel, separate the fat, returning the aqueous solution to the casserole in which the evaporation was conducted—wash the fat several times with portions of 50 cc. each of hot water, until the washings cease to respond to the diphenylamine test. Add the washings to the main portion, continue to evaporate over free flame to about 150 cc. There is no danger of scorching if ordinary precautions are observed and no bumping when the enameled metal casseroles are used. Transfer to a 200 cc. graduated flask. If it is desired to determine reducing sugar, the solution can at once be made up to the mark, filtered and titrated. If total invert sugar is desired, add 5 cc. of concentrated hydrochloric acid and invert by the official method of the A. O. A. C. Then make up to the mark, filter and titrate. An aliquot can be used for the saltpeter determination. In case reducing sugar has been determined and the solution made up to the mark prior to inversion, an aliquot can be inverted and total invert sugar determined. By deducting the percentage of reducing sugar, the percentage of sucrose can be readily calculated.

The evaporation in the metal casseroles is very rapid; several samples can be started in the morning and the sugar and saltpeter determinations completed the same day. Inversion with hydrochloric acid sufficiently coagulates the proteins and satisfactorily clarifies the solution for this determination. If the solution is made up to the mark without filtering, a slight precipitate error results, but this is negligible.

The Pavy solution is made up as follows:

Fehling solution 1 { 69.28 grams $CuSO_4.5H_2O$ / 1 cc. pure H_2SO_4 } dilute to 1 liter.

Fehling solution 2 { 300 grams potassium sodium tartrate in 700 cc. water; filter if necessary. 100 Grams pure sodium hydroxide in about 200 cc. water; dilute to 1 liter.

60 cc. solution No. 1
60 " " No. 2.
100 " 10 per cent. NaOH solution.
362 " aqua ammonia sp. gr. 0.90.
Dilute to 1 liter.

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METHYLSALICYLATE. THE ANALYTICAL SEPARATION AND DETERMINATION OF SALICYLIC ACID AND METHYLSALICYLATE, AND THE HYDROLYSIS OF THE ESTER.

BY H. D. GIBBS.

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Since salicylic acid and salicylates have been prohibited in foods,[1] it becomes necessary in many cases to separate salicylic acid and its metal salts from its esters.

The methyl ester, either the synthetic preparation, or oil of gaultheria, or oil of betula, is often found to be a constituent of many non-alcoholic beverages, such as the so-called root beers and sarsaparillas, and soda water flavors. The United States Pharmacopœia and the National Formulary[2] authorize its use as a flavoring agent, and it is therefore often found in emulsions, the most common of which is cod-liver oil, and other pharmacopœial preparations.

Salicylic acid or its salts and its methyl ester may be, and often are, found together in the above preparations: *first*, through the incorporation of both in the original mixture; *second*, when methylsalicylate, either natural or synthetic, alone is used, the ester may contain varying amounts of free salicylic acid as an impurity; *third*, when a comparatively pure ester is employed, free salicylic acid may subsequently become a constituent of the compound through the hydrolysis of the ester.

An examination of all of the different samples of methylsalicylate available in this laboratory and in the city of Manila, eight in all, has revealed the presence of the free acid in every case. Two of these samples were represented to be genuine oil of gaultheria, and six were synthetic preparations. All were of European exportation and had been in stock in this city from a few days to over a year. The amounts of free salicylic acid varied from a trace in one laboratory sample to 0.025 per cent. by weight in a genuine oil of wintergreen. These small amounts do not wholly account for the larger quantities of salicylic acid or its salts which have been found in a number of different preparations upon the local markets and entering the Port of Manila.

[1] *U. S. Dept. Agr., Food Inspection Decision* 76 (1907).
[2] *3rd Ed.* (1906), 46.