If to the solution to be tested are added an equal volume of concentrated hydrochloric acid and two drops of a 0.5 per cent. solution of resorcinol and the whole is boiled one or two minutes, characteristic flocks will appear, if sufficient formaldehyde is present.

The advantage of using hydrochloric acid instead of sulphuric acid is that no darkening of color from overheating or charring is possible. The disadvantage is that the delicacy of the test is lessened. It is not possible to get flocks in mixtures of methyl and ethyl alcohols when less than 5 per cent. methyl alcohol is present. The boiling, of course, prevents any formation of a contact ring. But when sulphuric acid is used, the color of the flocks is often destroyed by too rapid shaking, with consequent overheating, especially when only a small amount of formaldehyde is present.

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A METHOD FOR THE DETERMINATION OF LEAD NUMBER IN MAPLE SYRUP AND MAPLE SUGAR.

BY A. L. WINTON AND J. LEHN KREIDER.

Received July 5, 1906.

THE copious precipitate formed in maple products by basic lead acetate furnishes a means not only of clearing the solution for polarization but also of detecting the admixture of refined cane-sugar. Jones[1] and also Hortvet,[2] working independently of each other, have devised methods for measuring the bulk of this precipitate after separation with the aid of a centrifuge. The method devised by Jones was especially designed to test the products delivered by farmers to wholesalers, while Hortvet's method was devised with reference to the official inspection of the commercial products. Both serve well the purpose for which they were intended; they are easily carried out and give comparative results of great value. These results, however, can hardly be accepted as fixed constants, since the precipitate varies not only in composition but its volume is also dependent on the speed of the centrifuge and other details of manipulation.

Although volumetric methods of a similar nature are successfully employed for the determination of the gravimetric per-

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[1] Vt. Agr. Expt. Sta. Rep. 17, 454 (1904).

[2] This Journal, 26, 1532 (1904).

centages of fat (*e. g.*, Babcock method) and essential oil (*e. g.*, Mitchell's method for determining the oil in lemon extract), it is impracticable to convert the volume of a solid precipitate into percentages even when the precipitate is of definite composition and the task is obviously impossible when it is a variable mixture.

It also appears to us impracticable to weigh this precipitate, owing to difficulties in filtering, washing and drying.

The plan which seems to best serve the purpose is not to determine the total percentage of the precipitate but the amount of lead contained in it. This lead could be determined in the precipitate itself but this would involve careful washing and separation from the organic matter, both of which are laborious operations.[1] We have employed an indirect method, using a definite volume of standard lead subacetate and determining the lead remaining in the solution after precipitation. In order to still further reduce the labor of the operation the solution of the material after precipitation is made up to a definite volume, filtered through a dry paper, and the lead determined as sulphate in an aliquot portion of the filtrate.

The method adopted by us is in detail as follows:

Description of the Method.[2]—Weigh 25 grams of the material (or 26.048 grams if it is desired to determine sugars polariscopically in the same portion) into a 100 cc. flask. Add 25 cc. of standard lead subacetate solution, fill to the mark, shake, allow to stand at least one hour, and filter. From the clear filtrate pipette off 10 cc., dilute to 50 cc., add a moderate excess of sulphuric acid and 100 cc. of 95 per cent. alcohol. Let stand over night, filter on a Gooch crucible, wash with 95 per cent. alcohol, dry at a moderate heat, ignite at low redness for three minutes, taking care to avoid the reducing cone of the flame, and weigh. Calculate the amount of lead in the precipitate (factor 0.6829), subtract this from the amount in 2.5 cc. of the standard solution, and divide the remainder by 2.5 thus obtaining the "*Lead Number.*"

The standard lead subacetate is prepared as follows:[3] Boil for half an hour 430 grams of normal lead acetate and 130 grams of

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[1] Since this paper was read at the Ithaca meeting of the Society (June 28, 1906) Prof. Albert P. Sy has published a paper describing a method of determining the lead directly in the precipitate (J. Franklin Inst. July, 1906, p. 71).

[2] Devised by A. L. W.

[3] U. S. Dept. Agr., Div. Chem. Bull. **65**, p. 84.

litharge with 1000 cc. water. Cool the mixture, allow to settle, and dilute the supernatant liquid to 1.25 specific gravity. To a measured amount of this solution add four volumes of water and filter if not perfectly clear.

The standard of the solution is determined in 25 cc. by the lead sulphate method as above described. On standing it deposits a slight precipitate, but our tests show that the strength is not appreciably affected in a month or six weeks.

Given sufficient Gooch crucibles about 24 determinations of the lead number can be made in eight hours distributed through two days.

Test of Different Standard Solutions, Time Required for Precipitation.—As basic lead acetate solution varies somewhat in composition dependent on impurities in the chemicals used and irregularities in the process of preparation, it is important to ascertain whether these variations affect the precipitating power. It is equally important to determine the time required for complete precipitation. To decide these points comparative analyses were made on three samples of pure and three of adulterated maple syrup, using three solutions prepared from different lots of chemicals and allowing to stand after the precipitation one and eighteen hours. Solutions A and B were prepared from different lots of commercial lead acetate and litharge, solution C from the chemically pure materials.

The results are given in Table I.

TABLE I.—COMPARISON OF RESULTS ON LEAD NUMBER IN PURE AND ADULTERATED MAPLE SYRUPS, USING DIFFERENT STANDARD SOLUTIONS OF LEAD SUBACETATE AND STANDING ONE AND EIGHTEEN HOURS AFTER PRECIPITATION.

	Pure.						Adulterated.					
	No. 15988.		No. 15989.		No. 16037.		No. 15967.		No. 15970.		No. 15993.	
	1 hr.	18 hrs.	1 hr.	18 hrs.	1 hr.	18 hrs.	1 hr.	18 hrs.	1 hr.	18 hrs.	1 hr.	18 hrs.
Solution A	1.88	2.04	1.52	1.62	1.63	1.84	0.00	0.00	0.16	0.13	0.46	0.44
" B	2.03	2.12	1.60	1.64	1.71	1.93	0.02	0.02	0.13	0.18	0.31	.....
" C	2.14	2.05	1.70	1.82	1.94	1.93	0.00	0.04	0.23	0.31	0.34	.....

From these results it appears that no fear need be apprehended as to variations in the precipitating power of the standard solution, also that standing one hour gives virtually the same results as standing eighteen hours.

It should here be stated that in the case of all the pure samples, which we have examined, the solution filtered from the lead precipitate after standing one hour remained clear on further standing. The same is also true of all the adulterated samples but one. The latter, No. 16036 Table III, gave a lead number of 0.13 on standing one hour and of 0.42 on standing eighteen hours. Since one hour was found sufficient for all pure samples it is reasonable to assume that the extra amount obtained on long standing from this adulterated sample is due to some foreign constituent and may be justly disregarded.

Results on Authenticated Samples.—The following results, obtained on samples of known purity kindly supplied by Mr. C.

TABLE II.—RESULTS ON LEAD NUMBER AND TOTAL SOLIDS IN AUTHENTICATED SAMPLES OF MAPLE SYRUP AND MAPLE SUGAR.

No.		Total solids.	Lead number.
1	Maple syrup, excellent quality, composite sample	64.16	1.54 1.54
2	" " " " " "	64.07	1.39 1.38
3	" " " " " " (Ohio)	68.49	1.21 1.19
4	" " " " (made in 1893)	64.53	1.49 1.47
5	" " good quality, composite sample	68.28	1.66 1.66
6	" " medium quality, " "	66.21	1.57 1.57
7	" " " " " "	64.86	1.40 1.40
8	" " poor quality	65.59	1.77 1.77
9	Maple sugar (American), good quality	99.24	2.07 2.10
10	" " " " "	99.20	2.37 2.38
11	" " " medium quality	98.99	2.30 2.29
12	" " " " "	98.43	2.47 2.48
13	" " " composite sample	99.28	2.18 2.17
14	" " (Canadian), good quality	94.99	2.35 2.36
15	" " " " "	94.63	1.83 1.84

H. Jones, chemist of the Vermont Station, give a general idea of the variation in the lead number in the normal product although these results should be supplemented later by others on samples from other localities before the range in the lead number can be finally decided.

Results on Samples Found on the Market.—We have recently made analyses of 38 brands of maple syrups found on sale in Connecticut which show that only three were the genuine products, the remainder being mixtures consisting largely of refined cane-sugar. The determinations made include not only lead number but also, for comparison, total solids, cane-sugar (by Clerget's formula), Hortvet number (volume of the lead precipitate), and total ash. The lead number in the samples classed as pure ranged from 1.61 to 2.03, while in the adulterated samples it ranged from 0.02 to 0.92. In Table III are given such of these analyses as serve to show the range in composition of the samples.

It is a remarkable fact that every result in this table, except those for total solids and sucrose, furnishes decided evidence of purity or adulteration. Even the percentages of total solids and sucrose are useful, since the difference between the two is usually greater in the case of the pure syrups.

For a consideration of other methods of value in detecting adulteration the reader is referred to the exhaustive papers of Hortvet[1] and Jones.[2]

It has been suggested that the lead number of adulterated maple products can be so increased by the addition of various substances as to equal that of the genuine products. That this is true can not be denied, but it is also true that the results for ash and other constituents of these products as well as many of the constants of other food products can be rendered valueless as a means of detecting adulteration by skilful manipulation. The task, however, becomes more difficult with the introduction of new constants, especially when these are based on different principles.

The determination of ash and its characters and of lead number will usually suffice for the detection of the adulterants now in common use.

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[1] Loc. cit.

[2] Loc. cit.; Vt. Agr. Expt. Sta. Rep. 18, 315 (1905).

TABLE III.—ANALYSES OF MAPLE SYRUP FOUND ON THE MARKET.

Serial number.		Total solids. Per cent.	Sucrose by Clerget's formula. Per cent.	Hortvet number.[1]	Lead number.	Total ash. Per cent.
15988	Pure	66.98	62.4	1.00	2.03	0.75
15989	Pure	67.29	61.6	0.69	1.61	0.60
16037	Pure	66.40	59.5	1.24	1.89	0.54
15929	Adulterated[2].	66.40	65.4	0.15	0.66	0.14
15932	Adulterated	66.90	65.4	0.15	0.48	0.16
15945	Adulterated	65.81	.....	0.22	0.87	0.23
15948	Adulterated	67.61	67.8	0.07	0.52	0.04
15961	Adulterated	63.18	.....	0.07	0.10	0.02
15967	Adulterated	68.62	67.6	0.07	0.02	0.04
15970	Adulterated	66.58	65.2	0.11	0.19	0.19
15971	Adulterated	69.49	68.4	0.11	0.43	0.07
15982	Adulterated	64.53	61.9	0.15	0.17	0.17
16005	Adulterated	65.57	.....	0.15	0.12	0.04
16006	Adulterated	66.13	65.2	0.11	0.66	0.08
16035	Adulterated	65.33	60.0	0.12	0.92	0.14

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ON THE DETERMINATION OF CARBON DISULPHIDE AND TOTAL SULPHUR IN COMMERCIAL BENZENE.

BY EDWARD S. JOHNSON.

Received July 7, 1906.

CRUDE, unwashed benzene, or the so-called "benzol light oil," contains as certainly identified sulphur compounds, hydrogen sulphide, carbon disulphide, thiophene and its homologues. There are doubtless others among the unsaturated compounds which become eliminated in the first stage of benzene purification, the washing with concentrated sulphuric acid. There is left after this treatment in the lower-boiling fractions derived from the washed benzene, as a main impurity carrying sulphur, carbon disulphide. Thiophene, as well-known, represents the chief of other sulphur bodies present.

I. THE DETERMINATION OF CARBON DISULPHIDE.

The principle underlying the method seemingly most widely practiced is that of the transformation of carbon disulphide by means of alcoholic potash into potassium xanthate, utilizing this

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[1] Cubic centimeters of lead precipitate from 5 grams of the material.

[2] The adulterated samples were mixtures of maple syrup and refined cane-sugar. Some were labeled as compounds and therefore were not legally adulterated.