the polarizations were made within 5 or 6 minutes after adding the reagent.

(6) Acid mercuric nitrate as employed for the inversion of sucrose has no influence on the polarization of lactose. This reagent can therefore be employed with safety in the analysis of sweetened condensed milk or in other solutions containing sucrose and lactose, provided the temperature of the solution is kept at or below 15° and the polarization is made as soon as possible after the addition of the reagent.

To three samples of milk in which the percentage of lactose had previously been determined by polarization known quantities of sucrose were added and the percentage of sucrose in each sample then determined by the use of acid mercuric nitrate as a clarifying and inverting agent.

Sample 1, contained 12.28%, found 12.14%.

Sample 2, contained 12.80%, found 12.80%.

Sample 3, 6.40%, found 6.33%.

These figures show that the method is capable of giving reliable results. Its simplicity and ease of execution commend it as superior to the more tedious and complicated gravimetric processes.

[CONTRIBUTION FROM THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE.]

ESTIMATION OF LACTOSE AND BUTTER FAT IN MILK CHOCOLATE.

BY W. L. DUBOIS.

Received January 18, 1907.

Genuine milk chocolate consists of an intimate mixture of cacao mass or ground cacao nibs, sucrose, milk powder and cacao butter. In order to determine whether a so-called milk chocolate be true to name it is necessary to establish the presence of desiccated milk. This is accomplished by the estimation of lactose and butter fat. Reliance upon the lactose determination alone would lead to error in case glucose or other reducing sugar beside lactose were used in preparing the chocolate under consideration. Hence the estimation of butter fat becomes a valuable confirmation of conclusions drawn from the lactose figures.

In examining milk chocolate with the above purpose in view a rapid and accurate method for the determination of sugars is important. It is also desirable to determine lactose and sucrose in the same solution. Polarizing at 86° after inverting the sucrose was suggested and a method worked out on that basis.

It was first necessary to determine the effect of heating to 86° on the optical rotation of lactose. A number of solutions of lactose were polarized at 20° and at 86° and the necessary correction determined. The results appear in Table I.

TABLE I

Specific rotatory power of sucrose ..... = 66.44[1]
" " " " lactose ..... = 52.53[1]
Normal weight of sucrose ..... = 26.00 grms.
∴ " " lactose ..... = 32.884 "

Lactose in 100 Grams	Per cent. of normal wt.	Polarization at 20°	Polarization at 86°	Factor to reduce readings at 86° to 20°	Average factor	Readings at 86° x 1.11
0.8221	2.5	2.55	2.13	1.19	1.11	2.36
1.6441	5.0	5.07	4.50	1.12	...	5.00
2.4663	7.5	7.45	6.75	1.10	...	7.49
3.2884	10.0	10.00	9.0	1.11	...	9.99
4.1105	12.5	12.35	11.3	1.09	...	12.54
4.9326	15.0	15.10	13.7	1.10	...	15.20
6.5768	20.0	20.00	18.13	1.10	...	20.12

Each of the above polarimetric readings is an average of five observations. These figures show that heating to 86° causes a decrease in the right-handed reading of lactose which may be corrected by multiplying the reading at 86° by 1.11.

Solutions containing lactose and sucrose were next prepared by dissolving certain percentages of the normal weights of these sugars in water and diluting to 100 cc. As is well known the normal weight is that amount of pure sugar contained in 100 cc. of solution which polarizes at 100° or 100% on the Ventzke scale. Accordingly the amounts of sugars used in preparing the solutions represent actual percentages.

Polarizations were made before and after inversion and at 86°. Inversions were made by the German official method except that the acid was neutralized before diluting to volume. The procedure was as follows:

To 50 cc. of the solution contained in a 100 cc. flask were added 25 cc. of water and 5 cc. hydrochloric acid (sp. gr. 1.2). The flask was heated in a water-bath at such a rate that the contents attained a temperature of 68° in three minutes. This temperature was maintained for seven minutes longer. The flask was then quickly cooled, the acid nearly neutralized with sodium hydroxide and the flask filled to the mark. Invert readings were accordingly doubled to correct for dilution. Sucrose was calculated by the following formula:

$$\frac{(a-b)\ 100}{142.66-t/2} = \%\ \text{sucrose}$$

in which a is the direct reading and b the corrected invert reading.

Experiment showed that the above method of inverting did not destroy enough of the lactose to justify the introduction of a correction. This sugar was accordingly calculated by doubling the readings made on the invert solution at 86° and multiplying this figure by the factor 1.11 derived in Table I.

[1] Principles and Practice of Agricultural Analysis—Wiley.

In Table II are given the composition of the sugar solutions used and polarimetric results on the same:

TABLE II

Lactose grams in 100 cc.	Lactose Per cent. of normal wt.	Sucrose Grams in 100 cc.	Sucrose Per cent. of normal wt.	Polarization Direct	Invert	Temp.	at 86°	Sugar found Per cent. sucrose	Per cent. lactose	Lactose x 1.11
3.2884	10	6.5	25	34.64	— .7	20	+4.5	25.05	9.0	9.99
2.6307	8	7.8	30	37.74	— .8	24	+3.52	29.96	7.04	7.84
1.9730	6	9.1	35	40.74	—2.7	24	+2.62	35.18	5.24	5.82
1.3154	4	10.4	40	43.64	—4.94	20	+1.89	40.20	3.78	4.19

The figures in this table show that sucrose and lactose may be accurately estimated in the presence of each other by the procedure outlined above.

PREPARATION OF CHOCOLATE SAMPLES.

Two samples of chocolate containing known quantities of sucrose and lactose were prepared by rubbing the melted chocolate with the desired amounts of the sugars until a homogeneous mass was obtained. The composition was as follows:

TABLE III

No.	Chocolate grams	Sucrose grams	Lactose grams	Per cent. sucrose	Per cent. lactose
1	118.45	95.0	24.0	40.0	10.10
2	107.50	82.0	26.0	38.05	12.06

POLARIZATION OF CHOCOLATE SAMPLES.

Thirteen grams of the sample were shaken and centrifuged twice with 100 cc. of gasoline to extract fat and the gasoline poured off. One hundred cubic centimeters of water were then added and the sample shaken for 10 minutes. Five cubic centimeters of a basic lead acetate solution were added to clarify the solution and the excess of lead removed from the filtrate by potassium sulphate. For direct readings 25 cc. of the filtrate were boiled to destroy birotation, cooled, diluted to 50 cc. and polarized. Inversion was carried out by allowing 25 cc. of the above filtrate to stand over night with 2.5 cc. of hydrochloric acid. The acid was then neutralized and the solution diluted to 50 cc. All readings were accordingly multiplied by four.

Several other corrections must be introduced into the calculation. When sugar dissolves in water an expansion in volume takes place. The amount of this expansion for various amounts of sucrose dissolved in 100 cc. of water at 20° is shown in Table IV.

TABLE IV

Sucrose grams	Water cc.	Total volume after solution	Temperature
2	100	101.2	20°
4	"	102.5	"
6	"	103.6	"
8	"	104.8	"
10	"	106.05	"
15	"	109.40	"
20	"	112.40	"

Chocolate samples Nos. 1 and 2 contained approximately 50 per cent. of sugars which in this connection may be regarded as all sucrose. The 13 grams taken for analysis accordingly contained 6.5 grams of sugar. Interpolating between 6 and 8 in Table III 103.9 cc. is found as the volume after dissolving 6.5 grams of sugar in 100 cc. of water. All polarimetric results on the above chocolates were accordingly multiplied by 1.039 to correct for this dilution. The results were also multiplied by 1.05 to correct for dilution due to the addition of 5 cc. of lead acetate solution. Sucrose was calculated by the following formula:

$\frac{(a-b)\ 1.05\,x}{144-t/2}$ = per cent. sucrose, in which x is the volume obtained by dissolving the sugar present in 100 cc. of water. In the above case $x = 103.9$.

Beside being corrected as indicated above the lactose results were multiplied by $\frac{32.884}{26.00}$ or 1.264 to correct for the difference between the normal weight of lactose and that of sucrose, and also by 1.11 to reduce readings at 86° to the corresponding figures at 20°. Lactose was accordingly calculated by the following formula:

$C \times 4 \times 1.11 \times 1.05 \times x = (4.662c)\ x =$ per cent. lactose, in which x has the same value as in the sucrose calculation and c is the reading at 86°.

The results are set forth in Table V.

TABLE V

Number	Polarizations				Sugars Found		
	Direct	Invert	Temp.	at 86°	Lactose per cent.	Sucrose per cent.	Sucrose average
1	+ 10.6	— 1.7	23°	+ 1.5	9.17	40.42	
1	+ 10.7	— 1.6	"	+ 1.5	9.17	40.42	40.42
2	+ 10.8	— 0.9	"	+ 2.0	12.22	38.45	
2	+ 10.7	— 0.9	"	+ 2.0	12.22	38.14	38.30

These figures show that lactose and sucrose in such materials as the chocolate samples described may be accurately estimated by the method outlined.

Preparation of Milk Chocolate.—Two samples of milk chocolate were prepared by thoroughly incorporating Baker's plain chocolate, pulverized sugar, whole milk powder and cacao butter, the mixing being done in a porcelain dish on the steam-bath. The sugar used was 99.5 per cent. pure. The milk powder contained 36.73 per cent. lactose determined by Soxhlet's method, using alkaline copper solution, and 25.56 per cent. fat. The milk chocolate prepared had the following composition:

TABLE VI

Number	Sugar grams	Chocolate grams	Milk powder grams	Cacao butter grams	Sucrose per cent.	Lactose per cent.
3	36	28	24	12	35.82	8.82
4	40	30	16	14	39.80	5.88

Polarization of Milk Chocolate.—Thirteen grams were taken for analysis and treated as described for samples Nos. 1 and 2, except that direct readings were made on the filtrate without further dilution and inversions were carried out by the modified German official method. Direct readings were accordingly doubled and invert readings and those at 86° multiplied by four. Other corrections were made as noted above.

The results shown in Table VII prove the method to be accurate for milk chocolate.

TABLE VII

Number	Polarizations				Sugars found	
	Direct	Invert	Temp.	at 86°	Sucrose per cent.	Lactose per cent.
3	+ 19.0	- 1.5	20	+ 1.40	35.99	8.52
3	+ 19.0	— 1.4	20	+ 1.47	35.67	8.95
4	+ 19.7	— 2.2	21	+ 0.99	39.84	6.03
4	....	....	..	+ 0.98	....	5.97

Sugars in Commercial Milk Chocolate.—Five samples of well known brands of milk chocolates were analyzed for sucrose and lactose by the procedure just described. Results are given in Table VIII.

TABLE VIII

Number	Polarizations				Sugars found		Remarks
	Direct	Invert	Temp.	at 86°	Sucrose per cent.	Lactose per cent.	
15591	+ 21.0	— 2.0	24	+ 1.36	40.9	8.24	
15592	+ 20.88	- 1.8	24	+ 1.24	40.06	7.51	Same brand
15593	+ 23.44	— 2.14	23	+ 1.46	45.83	8.88	as No. 15591
15594	+ 23.22	— 2.22	23	+ 1.50	45.73	9.12	Same brand
15595	+ 23.88	— 2.20	23	+ 1.36	46.78	8.24	as No. 15593
15595	+ 23.84	— 2.18	23	+ 1.36	46.62	8.24	

In the opinion of the writer this method for determining sugars in chocolates is satisfactory both as regards speed and accuracy. The second shaking with gasoline is only necessary with samples which are not thoroughly disintegrated by the first treatment.

Butter Fat in Milk Chocolate.—An approximate estimation of butter fat in milk chocolate may be made by determining the Reichert-Meissl number of the fat extracted from the chocolate with gasoline or a similar solvent. Owing to the very low Reichert-Meissl number of cacao butter (0.45) that fat does not materially interfere. The Reichert-Meissl determination on the fats extracted from the milk chocolates described above were made by Prof. G. E. Patrick and appear in Table IX. In the calculations 24 is taken as the Reichert-Meissl number of butter fat.

TABLE XI

Number	Reichert-Meissl number	Approx. per cent. butter fat
15591	5.3	22.1
15592	5.2	21.7
15593	6.0	25.0
15594	5.5	22.9
15595	5.8	24.2
3	4.85	20.1
4	3.48	14.5

In these figures the approximate amount of butter fat present may be calculated. Of course it is not possible to tell how much milk powder has been used in the manufacture of the chocolate, but this is not essential. It is sufficient to establish the presence of butter fat in approximately the amount which would be expected from the quantities of milk powder usually employed in these products.

SUMMARY.

1. In order to prove a milk chocolate true to name it is necessary to establish the presence of desiccated milk. This may be done by estimation of lactose and butter fat.

2. Heating a solution of lactose to 86° decreases the polarimetric reading by an amount which may be corrected by multiplying by 1.11.

3. Sucrose and lactose may be determined in the same solution by polarizing the sucrose in the usual way and lactose at 86°, after the inversion of the sucrose.

4. These sugars may be rapidly and accurately determined in sweet and milk chocolates by the optical method described.

5. Butter fat in milk chocolate may be approximately estimated from the Reichert-Meissl number of the fat extracted therefrom.

THE ESTIMATION OF HIGHER ALCOHOLS ("FUSEL OIL") IN DISTILLED LIQUORS.

By Philip Schidrowitz.

Received Jan. 9, 1907.

In a paper entitled "Results of Fusel Oil Determination According to the Allen-Marquardt Method as Modified by Schidrowitz" A. Lasche[1] comes to the conclusion that "* * * the results obtained by the Allen-Marquardt Method do not represent the quantities of higher alcohols or Fusel Oil contained in distilled liquors, whereas the Roese Method results are very reliable and practically correct." This conclusion is somewhat remarkable, for Lasche finds by the Roese Method in two series of experiments the following percentages of the Higher Alcohols added to the spirit:

Propyl Alcohol	49%	and	70%
Iso-Butyl Alcohol	66%	"	80%
Amyl Alcohol	88%	"	112%

These results Lasche calls "practically correct." These six experiments together with six similar experiments with the Allen-Marquardt Method practically constitute the whole of the work with which Lasche attempts to disprove the value of the Allen-Marquardt process.

If Lasche's experiments had been conducted on what I consider to be sound scientific principles, the results obtained by him would certainly not

[1] Lasche's Magazine, September, 1906.