THE ANALYST.

OCTOBER, 1899.

THE ANALYSIS OF GOLDEN SYRUP.

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As the analysis of "golden syrup" has recently called for considerable attention on the part of public analysts, we venture to think that an account of the methods employed and the results obtained in the laboratory of Dr. Thomas Stevenson and in that of one of us (R. B.) may be of service to other members of the Society.

It would seem from the convictions which have been obtained under the Sale of Food and Drugs Act that "golden syrup" is required to be the product of the partial hydrolysis of cane-sugar (sucrose), and should therefore consist only of cane- and invert-sugar, besides water, mineral matter, and small quantities of organic impurities. A large proportion of the syrups now sold are, however, either mixtures of cane-sugar syrup with "glucose syrup" or else consist merely of "glucose syrup" sweetened by the addition of cane-sugar. These products are characterised by the presence of dextrin, maltose, and excess of dextrose. Since these last-named compounds are dextro-rotatory, dextrin being so to a very high degree, a determination of the rotatory power of the sample will at once afford a clue to its nature. Assuming genuine golden syrup to have a specific rotation $[\alpha]_D = +16^\circ$ and glucose syrup $[\alpha]_D = +110^\circ$, the percentage of glucose syrup present will be :

$$\frac{100([\alpha]_D - 16)}{110 - 16}.$$

A more extended examination is, however, usually desirable, and the following method of analysis has been found to be convenient.

Water.—A 10 per cent. solution of the syrup is made, and from the specific gravity (water=1000) of this solution the solid matter, and hence the water, in the original syrup is calculated by using the well-known factor 3.86.

Rotatory Power.—The 10 per cent. solution is clarified, if necessary, by means of a little animal charcoal, placed in the tube of the polarimeter, and the angle of

rotation (α) observed. The specific rotation of the syrup is then calculated by the well-known formula,

$$[\alpha]_D = \frac{100\alpha}{lc},$$

where l is the length of the observation tube in decimetres and c is the concentration (grammes per 100 c.c.). Using a 2 decimetre tube and a 10 per cent. solution, the observed angle has simply to be multiplied by 5.

Reducing Power.—This is determined (a) in the original solution after suitable dilution, (b) after inversion of the cane-sugar, (c) after inversion of the cane-sugar, dextrin, and maltose.

(a) Ten c.c. of the 10 per cent. solution are diluted to 100 c.c., and the reducing power determined by means of Fehling's solution. We prefer the gravimetric process, using 20 c.c. of Fehling's solution for 10 c.c. of the diluted sugar solution.

(b) Ten c.c. of the 10 per cent. solution are diluted to 50 c.c. with water containing 5 c.c. of concentrated hydrochloric acid, heated for ten minutes at a temperature not exceeding 68° , cooled, rendered alkaline, and made up to 100 c.c. Ten c.c. of this solution may be treated with 25 c.c. of Fehling's solution.

(c) Ten c.c. of the 10 per cent. solution are diluted to 50 c.c. with water containing $1\frac{1}{2}$ c.c. of concentrated sulphuric acid, heated on the water-bath for two to three hours, cooled, rendered alkaline, and diluted to 100 c.c. Thirty c.c. of Fehling's solution should be used for 10 c.c. of this liquid.

The reducing powers thus determined are expressed as percentages of glucose in the original syrup, and denoted by the symbols K_1 , K_2 , and K_3 respectively.

Ash.—Ten c.c. or 20 c.c. of the solution of the syrup are evaporated on the water-bath with a drop or two of concentrated sulphuric acid, and the residue ignited and weighed. The usual deduction of one-tenth is to be made from this "sulphated" ash.

Dextrin.—In the presence of dextrin, a turbidity is produced on adding alcohol to the solution of the syrup, but no great importance is to be attached to this test, since a similar turbidity has been noticed in the case of syrups made from coarse sugar, where there was no other evidence of the presence of dextrin. The quantitative estimation of dextrin by precipitation with alcohol was, as might be expected, found unreliable. The fermentation method is probably more accurate, but we have had no personal experience of this.

In the following table are given the results obtained by the application of the methods described to a number of samples received for analysis under the Sale of Food and Drugs Act. All were sold as "golden syrup" with the exception of No. 2, which was labelled as "amber syrup."

No.	Water.	Ash.	Specific Rotation. [α] _D .	Reducing Power.		
				Original. K ₁ .	After Inversion of Cane-sugar. K ₂ .	After Prolonged Hydrolysis. K ₃ .
1 ...	16.0	2.0	+15°	45.7	80.8	
2 ...	20.5	1.2	+86°	39.5	49.2	79.9
3 ...	16.9	1.4	+68°	39.4	52.9	73.0
4 ...	17.9	0.5	+105°	33.2	38.5	80.3
5 ...	20.6	0.5	+62.5°	38.2	60.7	81.5
6 ...	19.4	1.0	+100°	36.8	42.0	78.1
7 ...	20.4	0.5	+67.5°	34.4	64.0	76.5
8 ...	17.6	1.8	+82.5°	37.7	50.7	74.7
9 ...	18.5	2.4	+92.5°	30.2	44.9	79.7
10 ...	21.9	1.1	+100°	35.5	38.8	78.7
11 ...	19.6	1.5	+99°	34.2	41.8	78.1
12 ...	13.8	8.2	+17.5°	32.5	66.6	72.8
13 ...	16.2	4.1	+11.5°	45.6	72.8	70.8
14 ...	16.3	2.1	+13°	45.3	81.2	77.4
15 ...	19.7	1.8	+95.5°	35.7	43.1	82.4
16 ...	20.1	7.9	+25°	22.0	64.9	57.2
17 ...	23.7	0.6	+90°	39.5	42.2	74.4
18 ...	19.8	1.5	+94.5°	32.2	40.6	74.4
19 ...	19.8	1.6	+82°	29.1	46.8	75.6
20 ...	20.5	1.8	+91°	31.7	36.7	71.2
21 ...	16.5	2.0	+12.5°	44.0	73.9	
22 ...	16.9	1.8	+16°	40.8	77.5	78.0
23 ...	19.8	1.5	+90°	29.0	41.7	65.3
24 ...	19.6	1.4	+100°	34.0	39.0	70.3
25 ...	15.4	3.4	+57°	35.8	56.2	78.9
26 ...	16.0	3.5	+75°	32.9	48.8	75.1

Of these samples, Nos. 1, 12, 13, 14, 16, 21 and 22 are, in all probability, unadulterated syrups prepared from more or less refined sucrose. Assuming the original reducing power to be due to invert-sugar and the increase in the reducing power on inversion to be due to cane-sugar, the composition of the genuine syrups may be expressed as in the following table :

	1.	12.	13.	14.	16.	21.	22.
Water	16.0	13.8	16.2	16.3	20.1	16.5	16.9
Ash	2.0	8.2	4.1	2.1	7.9	2.0	1.8
Cane-sugar	33.3	32.4	25.8	34.1	40.8	28.4	34.9
Invert-sugar	45.7	32.5	45.6	45.3	22.0	44.0	40.8
Organic matter other than sugar (by difference)	3.0	3.1	8.3	2.2	9.2	9.1	5.6
	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Specific rotation, calculated ...	11.6°	14°	6.6	12.2°	22.1°	8.7°	13.8°
„ „ observed ...	15°	17.5°	11.5	13°	25°	12.5°	16°

It will be noted that the specific rotation calculated on the above assumption is always lower than that actually observed, the difference being probably caused by substances included under the head of "organic matter." No. 13 was shown to be a beet-sugar product by the development of a slight fishy odour in the solution on keeping, by the high ash, and by the very small amount of phosphoric acid present (0.009 per cent. P_2O_5).

All the remaining samples are, to a greater or less extent, adulterated with glucose syrup, as is shown by the high rotatory power and the increase in reducing power after prolonged hydrolysis. The original reducing power (K_1) is here due, not only to invert-sugar, but also to dextrose (from starch) and maltose, whilst the increase in reducing power on prolonged heating with acid is caused by the inversion of dextrin and maltose. It should be theoretically possible to calculate the amounts of all these substances from the values obtained for the reducing power and the specific rotation of the mixture before and after the two inversions described above, but up to the present we have found that the errors of experiment and the disturbing influence of impurities are too great to allow of the necessary number of equations being obtained with sufficient accuracy for the desired purpose. Failing this, however, a convenient expression of the composition of the sample may be arrived at by treating anhydrous commercial "glucose" as an individual substance having a cupric reducing power of 53 and a specific rotation $+134^\circ$ for sodium light. (See L. K. Boseley, ANALYST, 1898, p. 125.)

If s is the percentage of cane-sugar ($K=O$, $[\alpha]_D = +66.5^\circ$), i that of invert-sugar ($K=100$, $[\alpha]_D = -23.1^\circ$), x the commercial glucose, then the following relations hold:

$$K_1 = i + 0.53x.$$

$$K_2 = i + 0.53x + \frac{100s}{95}.$$

$$[\alpha]_D = \frac{66.5s + 134x - 23.1i}{100}.$$

Whence:

$$s = \frac{95(K_2 - K_1)}{100}.$$

$$x = 0.685[\alpha]_D + 0.16K_1 - 0.455s.$$

$$i = K_1 - 0.53x.$$

The following table shows the results obtained by the application of these formulæ to a few typical samples:

	5.	3.	2.	15.	24.	4.
Water	20.6	16.9	20.5	19.7	19.6	17.9
Ash	0.5	1.4	1.2	1.8	1.4	0.5
Cane-sugar	21.4	12.8	9.2	7.0	4.8	5.0
Invert-sugar	17.3	14.4	7.2			
"Glucose"	39.4	47.1	61.0	67.9	71.8	75.0
Organic matter (by difference)	0.8	7.4	0.9	3.6	2.4	1.6
	100.0	100.0	100.0	100.0	100.0	100.0

Since cane-sugar syrup contains from one-third to one-half its weight of cane-sugar, and glucose syrup contains an average of 82 per cent. of solid matter, these samples may be represented as containing approximately :

			5.	3.	2.	15.	24.	4.
Cane-sugar syrup	...		55	40	25	20	15	10
Glucose syrup	...	...	45	60	75	80	85	90