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ANALYSIS OF SUGAR, MOLASSES, AND SYRUP.

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Having a number of sugars and syrups to test, and rapidity of work being desirable, I have modified some existing processes to suit my case. As my principal object was the detection of starch syrup, the analysis of the whole of the samples had to be completed as soon as possible, so as to sort them out. The process I used is as follows :—

The reagents, etc., employed, were the following :—1. Animal charcoal in fine powder after twelve hours digestion in dilute hydrochloric acid, washing and re-ignition. 2. Alumina cream, prepared by precipitating a solution of alum with ammonia, washing thoroughly, and allowing the precipitate to consolidate to a cream. 3. Fehling solution which consisted of a mixture of solutions A and B in equal volumes; A contained 34·639 grammes of pure copper sulphate dissolved in water, filtered and made up to 500 cubic centimetres; B contained 173 grammes Rochelle salt dissolved in 300 c c water, with 40 grammes sodium hydrate added, dissolved and made up to 500 c.c., and then filtered through glass wool or asbestos fibre. 4. A nearly saturated solution of carbonate of soda. 5. The asbestos filters used for obtaining and weighing the cuprous oxide were constructed in the following manner: A "Macfarlane milk tube" is used for containing the filtering materials. Such tubes are specially made for this laboratory, but may be made from a 50 c.c. pipette with long bulb, by cutting the latter across in the middle, and leaving enough of the small tubes at the other ends to pass through a perforated cork. A piece of platinum gauze or perforated platinum foil is placed at the bottom of the wide part of the tube, sufficiently large to prevent the asbestos from being drawn through, and the tube is inserted in the perforation of a rubber stopper, closing a Bunsen filtering flask, which is connected with an exhausting apparatus. The latter is put in operation, and a loose layer of common asbestos fibre (crysotile), about an inch deep, placed upon the platinum gauze. A thin pulp made of Italian or hornblendic asbestos is then poured into the tube in sufficient quantity to form a filtering bed. This pulp is made by scraping down a piece of hornblendic asbestos in the direction of the fibre, digesting it for one hour with 4 per cent. soda solution, and washing thoroughly. Sufficient pulp must be poured into the tube to form a layer one-eighth to one-fourth of an inch in thickness. The filter is then washed once with alcohol and once with ether, dried thoroughly in the water-bath, cooled in the dessicator, and weighed. With regard to the details of the processes used, the following may be mentioned :—

Saccharimeter Readings.—The saccharimeter employed is manufactured by Schmidt and Haensch, Berlin, and is known as the Soleil-Ventzke-Scheibler apparatus, the reading giving the percentage of cane sugar direct. 13·024 grammes of the sugar are taken and dissolved and washed, by means of 50 or 60 c.c. warm water, into a flask of 100 c.c. capacity; 1 gramme animal charcoal is added, and agitated occasionally for half an hour; then 20 to 30 c.c. alumina cream is introduced into the flask, mixed with its contents, cooled and made up to the 100 c.c. mark. The solution is then filtered through a dry ribbed filter into a dry flask, introduced into the 200 millimetre tube, and read in the saccharimeter. The reading × 2 gives the percentage. Syrups and molasses are treated in the same way as the sugars, but sometimes require more animal charcoal or have to be read in the 100 m.m. tube.

Clerget Inversion.—50 c.c. of the same solution prepared for the saccharimeter reading are placed in a 50 c.c. flask provided with a mark at 55 c.c.; 2·5 c.c. of strong hydrochloric acid are added and mixed with the solution. The flask is then immersed in water, the temperature of the latter raised to 70° C. and kept between 60° and 70° for twenty minutes. The flask is next cooled to room temperature, made up to 55 c.c., and the solution placed in a 200 m.m. tube fitted with a thermometer and observed in the saccharimeter. The temperature of the solution can also be determined in the ordinary tube immediately after its removal from the saccharimeter. The reading should be (unless in the case of samples containing dextrose of starch syrup) to the left (or—), and its amount, which in the case of sugars varies from—13 to—14 at 20° C., should be increased one-tenth, on account of the dilution. The Clerget formula is then applied, viz.:—

$$\frac{(\text{Direct reading} + \text{invert reading}) \times 200}{142{\cdot}7 - \frac{1}{2}\text{ temp. of invert.}} = \text{percentage of cane sugar.}$$

Reducing Sugars.—In the case of samples of white or yellow sugar, $2\frac{1}{2}$ grammes are taken, dissolved and washed in a 50 c.c. flask by the use of about 25 c.c. warm water. 50 c.c. of undiluted Fehling solution are placed in a small beaker, and the latter, as well as the 50 c.c. flask, are immersed in boiling water, and allowed to remain until their contents have about the same temperature as the surrounding water. The sugar solution is then poured into the beaker, and the flask washed out with hot water, not more than about 25 c.c. being used, so that the resulting mixture may not exceed 100 c.c. in bulk. The beaker is allowed to remain in the boiling water for exactly ten minutes longer, and then the solution containing the precipitated cuprous oxide is poured into the weighed asbestos filter, standing in connection with the exhausting apparatus. If the filter has been properly made the filtration proceeds as rapidly as the liquid can be poured in, and the precipitate can be very rapidly washed into it with hot water. The filter is then filled up three or four times with hot water, and, when the filtrate gives no alkaline reaction with litmus paper, the tube is filled up once with alcohol and a second time with ether. While this is going on, it is convenient to proceed with the precipitation of another assay, which has been previously heated up in the boiling water. After the washing with ether, the

tube is heated in a steam bath, to complete dryness (about two hours is usually sufficient), cooled in the desiccator, and weighed. The increase in weight of the tube multiplied by 0·4861 and 40 gives the percentage of reducing sugars.

Cane Sugar by Inversion and Fehling's Solution.— In testing white and yellow sugars, 0·25 gramme is washed into a 50 c.c. flask by using 25 to 30 c.c. of warm water; 0·5 c.c. of strong hydrochloric acid is added and the mixture inverted by heating, as in the Clerget inversion process. It is then neutralised by solution of soda carbonate, the the quantity necessary having been previously ascertained in a blank experiment. Excess of the soda carbonate must be avoided, as it interferes with the correctness of the result. The precipitation, filtration and washing are conducted as described above, under reducing sugars, but 60 c.c. of Fehling solution are used instead of 50, and the bulk of the mixture made up to 110 instead of 100 c.c.m. The increase of weight which the tube experiences, × 0·4861, × 400 gives the percentage of invert sugar resulting from the cane sugar. This, minus the percentage of reducing sugar × 0·95, gives the percentage of cane sugar.

With syrups and molasses 5 grammes of the sample are washed into a 250 c.c. flask and made up to the mark; 25 c.c. are used for the reducing sugars, and the same quantity is inverted for the cane sugar. Precipitation, filtration and washing are conducted in the same way as in the case of the yellow and white sugars, excepting that 60 c.c. Fehling's solution are used in determining the reducing sugars and 70 to 80 c.c. of the Fehling when the cane sugar is to be estimated, and that, further, 2 c.c. of the strong hydrochloric acid are used for the inversion, and a corresponding quantity of soda carbonate for neutralising it.

Moisture and Water.—The moisture in the white and yellow sugars was determined by drying 2 grammes to constant weight. In the case of the syrups and molasses, the quantity of water was ascertained by dissolving 20 grammes in warm water, and diluting to 100 c.c. and introducing 20 c.c. of this solution (=4 grammes of the sample) into ordinary asbestos fibre (crysotile) contained in a milk tube, the tube and contents having been previously dried and weighed. The milk tubes used in this case had an inside diameter of 40 m.m., a depth of about 35 m.m., with an opening at bottom 5 m.m. in diameter, into a small tube 8 m.m. in length. The tube, with contents, is placed in a water-bath and dried at from 90° to 100° C. The drying is complete in 48 hours, and the increased weight of the tube and contents gives the amount of dry substance contained in 4 grammes of the sample, water found by difference.

As regards the rapidity of this process, six samples can be completely finished in duplicate, with the exception of the moisture, in eight hours, and by taking aliquot parts of the solution prepared for the polariscope reading for the precipitation, the weighing would be avoided and the work expedited; but I have preferred to make a separate weighing, as a more complete check.

As regards the accuracy, that depends, of course, on the manipulation, and probably the factor obtained may vary for the same reason, but if the same method is adhered to, the results obtained will be at all events comparable. The following table gives the data of some work done for that purpose.

	Number of determinations.	Direct polariscope in per cent.	Invert polariscope in per cent.	By Clerget.	Direct.				Inverted.				Cane sugar by difference of Cu_2O.	Other sugars by difference of Cu_2O.	Water.
					Amount taken.	Cu_2O obtained.	Difference, max. and min.	Factor.	Amount taken.	Cu_2O obtained.	Difference, max. and min.	Factor.			
Cane sugar	10	+100	..	..	2·5	·001	·0005	..	·25	541	·002	·4861	99·92	..	0.
Grape sugar (corn) ..	4	+79·6	+79·7	..	·25	·530	·002	·4717	25	·5385	·001	..	..	100·00	0.
Equal weights grape and cane sugars	4	+88·92	+25·84	49·40	·50	·532	·003	·4717	·25	·547	·004	..	50·35	50·18	0.
Milk sugar	4	+76·8	+78·6	..	·25	·361	·001	·6944	·25	·363	·004	..	..	100·26	0.
Starch syrup.. ..	4	+153·8	+151·0	..	·5	·463	·004	1·08	·5	·477	·044	..	..	..	..
50 pts. starch syrup 50 „ cane syrup 50 „ water 33·3 per cent. of each.	4	+43·1	+21·0	34·3	·5	·166	·002	1·08	·5	·536	·002	..	34·6	35·86	..

In addition to the foregoing particulars regarding methods of examination and manipulation, I may state that the constant for the Clerget inversion, 142·7, is taken from the redeterminations by Wohl, given in "Spencer's Handbook for Sugar Manufacturers" (p. 116). The half normal weight for the quantity of sugar was taken because, with the saccharimeter used, accurate readings to the left were not obtainable with the normal weight. It must also be mentioned that the factor, 0·4861, used for calculating the amount of reducing sugars from the cuprous oxide, was ascertained from a number of determinations of the quantity of the oxide yielded by pure cane sugar after inversion. These determinations agreed closely, but it is quite possible that changes in the manipulations by other operators would alter the factor. Basic lead acetate was not used for clarifying in any of the above analysis, in order to avoid the lead error, or the trouble of removing the lead previous to inversion.

Some of the syrup and molasses samples show greater differences than usual between the results obtained by direct and inverted saccharimeter reading. The differences reappeared even when basic lead acetate was employed to clarify, and were probably due to the presence of unusually large amounts of syrup-forming substances different from sugar. All these analyses were done in duplicate, but I do not consider this to be necessary, as the optical and chemical results check each other and are equivalent to a duplicate analysis.

In giving these details regarding the processes used, it is not intended to suggest that these should be substituted for others now in use. But since, in the examination of saccharine substances, many different methods of analysis are in use, the results of which may vary slightly according to the nature of the process and the mode of manipulation, I have thought it necessary to give these particulars, in order that they may be taken into consideration when comparisons are being made with the results above stated and the work of other analysts.