

uble. You will note that the ether extract could not be dried down to a dry solid on account of its oily nature. Further experiments along this line have been planned, but have as yet not been completed.

EXPERIMENT WITH THE EXTRACTION OF CASCARA BARK WITH A SERIES OF MENSTRUUMS.
Experiment No. 1.

Menstruum and order of extraction.	Yield of dry extract, %.	Remarks.
Ether	4.0	Bitter, tarry, insoluble in water, melts on heating.
Acetone	10.0	Bitter, 60.7% water-soluble.
Alcohol	11.0	Bitter-sweet, then acrid bitter, 82.5% water-soluble.
Dilute alcohol	9.0	Flat, faintly bitter, 83% water-soluble.
Hot water	1.7	Flat, faintly bitter, 82.2% water-soluble.
Total extractive all solvents	35.7	

Experiment No. 2.

Menstruum and order of extraction.	Yield of dry extract, %.	Remarks.
Hot water	27.0	90.8% water-soluble.
Dilute alcohol	4.5	Bitter oily taste, 63.2% water-soluble.
Alcohol	3.0	Tarry, oily taste.
Acetone	.45	Waxy, taste flat.
Ether	(.30)	Amount estimated at 0.3% extract was lost through accident in drying.
Total extractive all solvents	35.25	

One of the objects of making these experiments was to determine if some other method of removing the bitter principle from cascara would be more satisfactory than the one now employed as by the present method a large amount of the activity of the drug is lost. I have heard that one may exhaust cascara with certain organic solvents, thereby obtaining a bitter cathartic principle and rendering the bark bitterless. This bitter cathartic principle I am told is made use of under the name of Cascarin and the bitter free bark is then available for the preparation of an aromatic fluidextract. However, I have no definite information on this subject and do not know whether this practice is really followed.

Further work on this subject is under way and contemplated and I hope to make an additional report at next year's meeting.

LABORATORIES OF E. R. SQUIBB & SONS.

A COMPARISON OF CANE AND BEET SUGAR FOR PHARMACEUTICAL PURPOSES.*

BY ADRIAN THOMAS.

For a long time there has been a question, especially among manufacturers of pharmaceutical preparations, whether there is a difference between products made with a cane or beet sugar. The experiments recorded here were conducted in order to ascertain if any marked difference existed between sugars from the two sources or between solutions of them. Four samples of cane sugar and four samples of beet sugar were examined, each sample being representative of a car lot.

* Scientific Section, A. Ph. A., Cleveland meeting, 1922.

CHEMICAL EXAMINATION.

Polariscopic examinations were made but revealed no difference between the two kinds of sugar. Direct readings, and also readings after inversion were taken, the sucrose being then calculated by the formula of Clerget. The sucrose content of all samples was found to be above 99% and no differences were observed which might not be considered within the experimental error and within the limits of accuracy of the polariscope used. Therefore these results were not considered to be of any great value by themselves.

Determinations were made of the moisture, ash, nitrogen and reducing sugar, calculated as *invert* sugar, in the eight samples. The results are given in Table I.

TABLE I.

Sample.	Moisture, %.	Ash, %.	Nitrogen \times 6.25, %.	Invert sugar, %.
1-Cane	0.030	0.020	0.075	0.013
4-Cane	0.035	0.005	0.026	0.027
5-Cane	0.030	0.010	0.052	0.030
6-Cane	0.020	0.010	0.018	0.021
2-Beet	0.030	0.010	0.022	0.004
3-Beet	0.050	0.005	0.043	0.003
7-Beet	0.044	0.005	0.000	0.020
8-Beet	0.050	0.005	0.020	0.008
Average for cane	0.029	0.011	0.043	0.023
Average for beet	0.044	0.006	0.021	0.009

The averages would make it appear that the beet sugars contained less of these impurities than the cane sugars, but if the analyses of the individual samples are examined, it will be seen this does not entirely hold true; however, any differences in the sugars could not be attributed to these small amounts of impurities.

The reducing sugars were in such small quantities that the ordinary methods of determination were not applicable. The method used was that given by Bates and Jackson¹ and employed a modified Soldaine reagent containing 300 Gm. KHCO_3 and 1 Gm. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ per liter. Bates² found that most samples of both beet and cane sugar examined were of remarkable purity; he even found some beet sugar to polarize above 100° owing to the presence of raffinose. This high degree of purity is in accordance with the present findings.

FERMENTATION WITH YEAST.

A series of solutions of different concentrations was made from each of the eight sugars. These were put in fermentation tubes and inoculated with 0.1 cc of a yeast suspension containing 10 Gm. of compressed yeast per 100 cc. The rate of growth of the organisms was recorded after 48 hours, the amount of gas formed, rather than a count of yeast cells, being taken as an index of growth. The gas was then released and the solutions inoculated with an additional 1.0 cc of a yeast suspension of the same concentration and the growth as indicated by the amount of gas formed, was noted after 24 hours. The tubes were kept at room temperature (20°C. to 25°C.). The results of these fermentation tests are given in Table II.

¹ U. S. Bur. of Standards, *Sci. Paper* No. 268.

² F. J. Bates, *Abs. in Int. Sugar J.*, 22, 654, 1920.

In this table the customary bacteriological notation is used. Minus (-) indicates the absence of gas, plus (+) the presence of gas, the relative amount being expressed by the numerical coefficient 6+ indicating that the arm of the fermentation tube was filled with gas.

TABLE II. FERMENTATION OF SUGAR SOLUTIONS WITH YEAST.

Concentration, per cent.	Sample.								
	No. 1. Cane.	No. 4. Cane.	No. 5. Cane.	No. 6. Cane.	No. 2. Beet.	No. 3. Beet.	No. 7. Beet.	No. 8. Beet.	
10	4+	3+	3+	+	3+	5+	3+	5+	} Gas produced in 48 hrs. after inoculation with 0.1 cc yeast suspension.
20	4+	2+	4+	2+	5+	3+	2+	3+	
30	+	+	4+	2+	5+	+	3+	6+	
40	4+	a	+	+	3+	+	2+	4+	
50	a	4+	3+	a	—	—	+	5+	
60	b	2+	2+	—	—	—	4+	3+	
70	—	a	—	—	b	—	a	a	
80	—	b	—	—	—	—	—	—	
*85	—	—	—	a	—	—	—	—	
10	6+	6+	6+	6+	6+	6+	6+	6+	
20	6+	6+	6+	6+	6+	6+	6+	6+	
30	6+	6+	6+	6+	6+	6+	6+	6+	
40	6+	6+	6+	6+	6+	6+	6+	6+	
50	6+	6+	6+	5+	a	+	6+	6+	
60	5+	6+	5+	2+	b	b	6+	6+	
70	3+	3+	+	+	6+	2+	2+	2+	
80	—	b	—	—	b	+	b	b	
85	+	—	—	b	+	—	—	—	

a indicates a small bubble.

b indicates a minute bubble.

* 85% sugar solution corresponds to the U. S. P. simple syrup.

Upon examination of Table II it will be seen that as the concentration of the sugar solution approaches 85%, the fermentation decreases. Only three samples showed any fermentation at all at this concentration, one being a beet sugar and two, cane; one of the latter, however, exhibited only a small bubble of gas.

MOULDS AND BACTERIA.

It was desired to know to what extent organisms were present in the sugar as received. For this purpose fifteen grams of sugar were dissolved in sterile distilled water and made to 100 cc in sterile flasks. One cubic centimeter of the resulting solution was plated out in sterile Petri dishes with nutrient agar. Duplicate solutions and plates were made of each sugar, one set being cultured in an incubator at 30° C., the other being allowed to stand at room temperature (20° C. to 25° C.). After three days the plates and solutions were examined for growth and then allowed to incubate for twenty-four hours longer when counts of the plates were made. The results of this set of experiments is recorded in Table III.

From the bacteria and mould counts which were made the average number of bacteria and mould spores per gram of sugar may be calculated. These calculated numbers are given in Table IV. While only approximate, they may serve for comparison.

TABLE III.—BACTERIA AND MOULDS IN SUGAR.

Sample.	Growth in solutions.						Growth on plates.			
	After 3* days.		After 4 days.		After 3* days.		Mould.		Colonies after 4 days.	
	Room temp.	30° C.	Room temp.	30° C.	Room temp.	30° C.	Room temp.	30° C.	Room temp.	30° C.
1-Cane	±	—	+	+	—	+	0	4	1	1
4-Cane	—	—	+	+	±	+	1	2	2	0
5-Cane	+	+	+	+	+	±	1	0	0	1
6-Cane	+	—	+	+	—	±	0	X	1	X
2-Beet	+	+	+	+	+	±	4	X	1	1
3-Beet	±	±	+	+	+	+	1	2	4	2
7-Beet	±	+	+	+	—	+	2	3	6	11
8-Beet	+	+	+	+	+	+	1	1	8	7

* For samples No. 7 and No. 8 data are recorded after two days instead of three.
X indicates colonies having spread to such an extent as to make count impossible.

TABLE IV.—BACTERIA AND MOULD SPORES PER GRAM OF SUGAR.

Sample.	Mould spores incubating at		Bacteria incubating at	
	Room temp.	30° C.	Room temp.	30° C.
1-Cane	0	26	6	6
4-Cane	6	13	13	0
5-Cane	6	0	0	6
6-Cane	0	X	6	X
2-Beet	26	X	6	6
3-Beet	13	6	16	13
7-Beet	13	20	73	40
8-Beet	6	6	53	46
Average for cane sugar	3	13	6	4
Average for beet sugar	11	14	26	39

X indicates colonies having spread to such an extent as to make count impossible.

It would appear from these figures that the samples of beet sugar were on the average contaminated with a greater number of organisms than were the samples of cane sugar. The bacteria were not identified but the moulds belonged to the *mucor*, *aspergillus* and *penicillium* groups.

KEEPING QUALITIES OF SYRUPS.

In order to observe any difference in the keeping qualities of syrups made from the two sugars under ordinary conditions, a series of syrups ranging in concentration from 10% to 85% were made from each sample of sugar, ordinary distilled water being used as a solvent. Each syrup was then divided into three portions. One portion was put in Erlenmeyer flasks, plugged with cotton and sterilized 20 minutes under 15 lbs. steam pressure. A second portion was put into bottles, which had been thoroughly washed, and tightly corked. The third portion was put into bottles the same as the second portion, but was afterwards inoculated with a mixed culture of moulds and bacteria taken from the plates upon which the counts were made. It is needless to say that in no case was any growth observed in the series which had been sterilized.

In Table V are shown the relative growths obtained in the inoculated syrups and in syrups where growth was allowed to develop spontaneously. All these syrups were held at room temperature. No active fermentation was noted and no pressure was developed in the tightly corked bottles so it may be concluded that gas-producing organisms were absent. The growths appeared to be chiefly due to moulds. These were largely confined to the surface.

A close examination of Table V discloses that in the case of the syrups in which the organisms were allowed to develop spontaneously, there is but little difference in the rate of growth in syrups made from beet or cane sugar, but in the case of the inoculated syrups organisms developed at a slightly more rapid rate in beet sugar syrups.

TABLE V.—RATE OF GROWTH OF ORGANISMS IN SYRUPS.

Sample.	Period of growth, days.	Concentration.									
		10%.	20%.	30%.	40%.	50%.	60%.	70%.	80%.	85%.	
Spontaneous growths	1-Cane	7	+	+	+	+	+	+	+	+	—
	14	+	+	2+	2+	2+	2+	2+	2+	±	
	42	+	+	2+	2+	2+	2+	2+	3+	+	
	4-Cane	7	—	±	+	+	2+	—	—	—	
	14	±	±	+	+	2+	±	—	—	—	
	42	+	+	2+	2+	2+	2+	2+	±	+	
	5-Cane	7	+	+	2+	2+	2+	2+	±	—	
	14	+	+	2+	2+	2+	+	+	+	±	
	42	+	+	2+	3+	3+	2+	2+	+	+	
	6-Cane	7	+	2+	2+	2+	2+	2+	±	—	
	14	2+	2+	2+	2+	2+	2+	+	±	+	
	42	2+	3+	3+	3+	2+	2+	2+	2+	2+	
	2-Beet	7	±	±	±	±	+	+	+	—	
	14	+	+	+	2+	2+	2+	2+	—	—	
	42	2+	2+	2+	3+	4+	4+	3+	2+	2+	
	3-Beet	7	+	+	2+	2+	2+	+	±	—	
	14	+	+	2+	2+	2+	+	+	+	—	
	42	+	+	3+	4+	4+	4+	4+	2+	2+	
	7-Beet	7	2+	—	+	2+	2+	+	±	—	
	14	2+	—	+	2+	3+	2+	+	—	—	
	42	2+	+	+	2+	3+	2+	+	±	±	
	8-Beet	7	2+	+	+	±	+	±	—	—	
	14	2+	+	+	+	+	±	—	—	—	
	42	2+	+	+	+	+	+	±	±	±	
Growth after inoculation	1-Cane	2	2+	2+	2+	2+	2+	+	+	±	
	10	2+	2+	2+	2+	2+	+	+	2+	+	
	42	2+	3+	2+	2+	2+	+	+	3+	+	
	4-Cane	2	+	+	+	2+	+	+	+	±	
	10	+	+	+	2+	+	2+	+	+	+	
	42	2+	+	2+	2+	2+	2+	2+	+	+	
	5-Cane	2	+	+	+	+	±	±	±	±	
	10	2+	2+	2+	+	+	+	+	+	+	
	42	2+	2+	2+	2+	3+	2+	2+	2+	+	
	6-Cane	2	+	2+	+	+	+	+	+	±	
	10	2+	2+	2+	2+	+	+	+	+	+	
	42	3+	3+	3+	3+	2+	2+	2+	2+	2+	
	2-Beet	2	+	+	+	2+	2+	2+	2+	±	
	10	+	+	2+	3+	3+	2+	2+	+	+	
	42	2+	2+	4+	4+	4+	4+	3+	2+	2+	
	3-Beet	2	+	+	2+	2+	2+	2+	+	±	
	10	+	+	3+	2+	2+	2+	2+	+	2+	
	42	2+	2+	2+	4+	4+	4+	4+	2+	2+	
	7-Beet	2	2+	+	2+	2+	2+	2+	+	±	
	10	2+	+	2+	2+	3+	2+	2+	+	±	
	42	3+	2+	2+	3+	3+	3+	2+	2+	+	
	8-Beet	2	2+	2+	2+	2+	2+	±	±	—	
	10	2+	2+	2+	2+	2+	+	+	±	±	
	42	3+	3+	3+	3+	3+	2+	+	+	+	

When first made, no difference in color could be detected between beet or cane sugar syrups of the same concentrations. Upon the development of moulds, some of the syrups became discolored; this, however, was noted in both cane and beet sugar syrups.

Shaw³ has compared the merits of beet and cane sugar for preserving fruits and making jellies and has found the two sugars to have almost the identical sucrose content, which would be expected. He also found no difference between jellies made with beet and cane sugar, and of 2000 cans of preserved fruit he found but 6 from the beet sugar lot and 7 from the cane sugar lot to spoil during two years of storage. This loss, it is stated, was due to imperfect sealing.

From the evidence at hand it appears that beet and cane sugar may be used with equally good results for all purposes. The process of refining sugar has been so perfected that a uniform product can be supplied. The greatest source of danger, it seems, is contamination by moulds and bacteria, but with modern processes of refining both beet and cane sugar may be produced equally free from contamination by organisms.

SUMMARY.

Chemical and polariscopic examination failed to establish any difference between the beet and cane sugars examined, though of the almost negligible amounts of ash, nitrogen and invert sugar found in both the beet and cane sugar samples, the cane sugar, on the average, contained the most.

Fermentation with yeast showed the process to proceed the same for both sugars.

Of the eight samples examined, more organisms were found in beet than in cane sugar. Any difference between given samples of the two sugars must be concluded to be due to the degree of refinement or to contamination by moulds and bacteria, the latter being the more probable.

PAPER NO. 19.
CHEMICAL RESEARCH DEPT.,
PARKE, DAVIS & COMPANY.

A METHOD FOR MANUFACTURING ETHYL OENANTHYLATE.*

BY CHARLES H. ROGERS.

Nearly all fruits possess very distinctive flavors and these flavors may be imparted to such substances as confections, jellies, ice cream, etc., by simply mixing in sufficient quantities the fruit, either in preserved or fresh form or the juice therefrom, with the material. In many instances, however, it is not practical to prepare an extract from the respective fruits which is sufficiently concentrated to give the desired fruit flavor when used in moderate quantities. These conditions have led to the use of artificial fruit essences which are made up of esters, alcohols, aldehydes, lactones, etc., mixed in various combinations and proportions to imitate more or less the various fruit flavors. In concentrated form these artificial flavors are usually more pungent and crude smelling than the sapid and odorous principles

³ G. W. Shaw, *Univ. of Cal. College of Agr., Cir. No. 33, 1907.*

* Northwestern Branch A. Ph. A., February meeting, 1923.