JELLYING AND CRYSTALLIZATION OF SIRUPS MADE FROM DIFFERENT PARTS OF THE SORGO STALK AT DIFFERENT STAGES OF MATURITY[1]

By Emil K. Ventre, *chemist*, S. Byall, *assistant chemist*, and C. F. Walton, Jr., *senior chemist, Carbohydrate Research Division, Bureau of Agricultural Chemistry and Engineering, United States Department of Agriculture*

EARLIER WORK

The fact that starch is present in sorgo juices has been known for many years. In 1858 Gilbee (*3*)[2] was granted a British patent on a process for eliminating organic impurities from the juices of saccharriferous plants by the use of alcohol. In 1864 Joulie (*5*), in recommending a method of clarification by the use of alcohol, mentioned that the alcohol precipitate contained starchy materials, and suggested conversion to sugar for production of alcohol. Sylvester (*10*) found the starch granules in sorgo juice to be from one eight-thousandth to one six-thousandth of an inch in diameter, as compared with one five-hundredth to one four-hundredth of an inch for granules of potato starch. Hutchinson (*9*), in discussing the distribution of starch in the sorgo stalk, reported that the iodine test showed no starch in green canes. If any blue color was present it was obscured by an intense brown coloration. With well-matured canes iodine gave an intensely blue color toward the top, which decreased in intensity toward the butt. At the beginning of maturity, as indicated by the presence of sucrose in the lower part of the stalk, more starch was found in the butt than in the top. Janssen, McClelland, and Metzger (*4*) in reporting analyses of sorgo juices from the different internodes of the Honey variety stated that starch occurs throughout the internodes of the stalk, although it is highest in the middle internodes.

Sherwood (*6*, *7*) reported the starch content of the juices of 15 varieties of sorgo and proved that starch is the cause of jellying or clabbering of sorgo sirups. Willaman and Davison (*11*), in studying the jellying of sorgo sirups, found that 0.32 to 1.15 percent of starch is present in normal sirups and 1.17 to 3.36 percent in sirups that jelly. Walton and Ventre[3] found that starch is responsible for slow boiling and scorching in the evaporator, and devised a farm-scale method for preventing this condition.

Many investigators have attempted to develop a practicable process for utilizing sorgo as a commercial source of sugar (sucrose). Among those who also studied the dextrose and levulose content of the stalk were Berthelot and Trannoy (*2*), who reported analyses of sorgo juices at eight stages of plant maturity from August 10 to November 30. At each stage of maturity the juices contained from approximately

[1] Received for publication March 17, 1939.

[2] Italic numbers in parentheses refer to Literature Cited, p. 150.

[3] Walton, C. F., Jr., Ventre, E. K., and Byall, S. How to prevent slow boiling, scorching, clabbering, and sugaring of sorghum sirup. U. S. Bur. Chem. and Soils Cir. 6 pp. 1935. [Mimeographed.]

two to four times as much dextrose as levulose, and at the two earliest stages the dextrose content exceeded the sucrose content. It appears, however, that these earlier investigators did not study the crystallization characteristics of sirups made from the stalk as a whole in comparison with those of sirups made from different parts of the stalk. Moreover, it must have been assumed that the sugar content of the sorgo stalk is similar to that of sugarcane. No report has been found of dextrose crystallization in sorgo sirups.

On the whole the literature indicates that earlier investigators made little attempt to correlate composition of the juice and quality of the sirup, especially the effect of the content of starch, sucrose, and dextrose in the juice on the composition and properties of the resulting sirups.

In the manufacture of sorgo sirups by the customary methods, the effect on the starch and sucrose of continued heating in a slightly acid solution should produce a somewhat greater proportion of dextrose in the sirups than in the juices. Since both the content of starch and sugars in different parts of the stalk and the effect of maturity on the composition of the juice must be considered, it is obvious that farm-made sorgo sirups present a special case of the system sucrose, dextrose, and levulose in a relatively impure solution containing also varying proportions of starch and its degradation products produced by the action of heat and acidity during open evaporation. The purpose of the present investigation was to determine the effect of the sugar and starch content of sirups in relation to crystallization and jellying. Sirups made from different parts of the stalk at different stages of maturity were compared with those made from the whole stalk. Inasmuch as the sirups were made from different portions of the same stalk, an opportunity was afforded to study jellying and crystallization under uniform agronomic conditions.

EXPERIMENTAL PROCEDURE

In continuation of previous work by the authors (*12*) an investigation was conducted in cooperation with the Mississippi Agricultural Experiment Station [4], the four varieties of sorgo (*Holcus sorghum* var. *saccharatus*) most used for sirup production in that section being selected for study. These varieties, designated by the names locally assigned to them, had the following varietal characteristics:

Hodo.—A large-barrel, long-stalk, late-maturing variety.
Iceberg.—A small-barrel, short-stalk, early-maturing variety.
Honey.—A small-barrel, medium-height stalk, intermediate-maturing, sprangle-top variety.
Gooseneck.—A medium-barrel, medium-height stalk, intermediate-maturing variety.

The stages of maturity were determined by the average maturity of the seed heads, described as follows:

Milk stage.—When the seed heads were out of the sheath and well formed, and the contents when pressed out had the consistence of milk.
Dough-to-ripe stage.—When the contents pressed from the seed heads had the consistence of stiff dough, the stage just preceding the hard-dough stage.
Dead-ripe stage.—When the contents of the seed heads were solid, and the glumes were wide open and dry.

[4] The authors wish to express their appreciation for assistance given by the late J. R. Ricks, director, and by W. R. Perkins, formerly assistant director, Mississippi Agricultural Experiment Station, in supplying working facilities and in connection with the agronomic work of this investigation.

When the sorgo had reached the selected stage of maturity, the stalks were cut even with the surface of the ground and stripped of leaves. The seed head and peduncle were removed, and the stalks divided at the nodes. The internodes were numbered consecutively from the peduncle and segregated in batches according to number. Each batch was ground separately in a horse-driven farm mill, being passed through the mill twice in order to extract as much juice as possible with this type of milling equipment.

A whole-stalk sample of each variety at each stage of maturity was used for comparison. An aliquot portion of the stalks was selected and stripped clean, after which the seed heads and peduncle were removed. This sample was subjected to exactly the same treatment as the various batches of internodes.

The juices expressed separately from each of two consecutively numbered batches of internodes were combined, and this juice was made into sirup for comparison with the sirup made from the whole stalk. The experimental method developed by this Division (*8*) for producing farm-made sirups was used in making all the sirups. Samples of the sirups were taken in triplicate and sealed while hot in 4-ounce, screw-top, glass jars. One sample was used for analysis; the other two were stored in a constant-temperature room at 20° C. for about 2 years. The sirups were made and analyzed in the fall of 1935, and the final examination for jellying and crystallization was made in February 1938.

The analytical determinations were made as follows:

Total solids.—Determined directly on the sirups at 20° C. by the use of the Zeiss sugar refractometer.

Sucrose.—Determined by the Association of Official Agricultural Chemists method of double polarization (*1*), invertase being used as the inverting reagent.

Reducing sugars.—Determined by the Munson and Walker method (*1*) and calculated in terms of invert sugar.

Starch was determined by the authors' modification of the official A. O. A. C. method (*1*) of starch determination in the presence of interfering polysaccharides. This method may be described briefly as follows:

For the malt reagent grind well-cleaned, new barley malt of high diastase activity and prepare an infusion of this freshly ground malt, just before it is to be used, by digesting 5 gm. with 100 cc. of water at room temperature for 2 hours, or for 20 minutes if the mixture can be stirred by an electric mixer. Filter to obtain a clear extract, if necessary returning the first portions of the filtrate to the filter. Mix the infusion well.

Wash a 15-gm. portion of the sirup into a beaker with approximately 300 cc. of 70-percent alcohol (by volume) and thoroughly mix by stirring.

Wash the precipitated starch free of sugar with additional 70-percent alcohol by decantation through an alundum crucible (porosity R. A. 360). Return the crucible to the beaker containing the washed starch precipitate and dry in an oven at 105° C. until free of alcohol. Then add 160 cc. of hot water and thoroughly gelatinize the starch at boiling temperature in a water bath. (At this point start a control to determine the quantity of dextrose derived from the malt reagent. This is done by adding 160 cc. of distilled water to a 400-cc. beaker and following the same procedure used for the starch determination.)

Cool to 50° C. or lower. Add 20 cc. of malt extract reagent and place in a temperature-controlled water bath. Keeping the mash thoroughly mixed, gradually raise the temperature to 70° in 20 to 30 minutes. Maintain at 70° for 30 minutes, stirring from time to time; then increase the temperature to 80° and keep it at that temperature for 10 minutes. Finally heat to the boiling point. Keep the mixture well stirred. Cool the contents of the beakers and the water bath to 55°. Add another 20 cc. of the malt-extract reagent, mix well, and hold at 55° for 1 hour, stirring about once every 10 minutes. At the termination of the digestion increase the temperature rapidly to above 80°.

Transfer to a 500-cc. volumetric flask and add 316 cc. of 95-percent alcohol, a little at a time, shaking between additions. Wash the crucible and beaker with hot water and use the washings to make up to the mark. Filter through a dry filter paper and evaporate the filtrate to a volume of 15 to 20 cc. or until free from alcohol.

Transfer the aqueous residue to a 200-cc. volumetric flask. Wash the beaker with hot water, using a rubber-tipped rod to recover any dextrin present, and add the washings to the volumetric flask. Allow it to cool, and then complete the volume to 200 cc. Transfer the contents to a suitable digestion flask, add 20 cc. of hydrochloric acid (specific gravity 1.125), made by diluting 68 cc. of strong acid (sp. gr. 1.19) to 100 cc., and connect the flask with a reflux condenser. Heat in a boiling water bath for 2½ hours Cool and transfer to a 250-cc. volumetric flask. Partly neutralize, while stirring, by adding 10 cc. of strong caustic soda solution (44 gm. NaOH per 100 cc. of H_2O), and complete neutralization with a little anhydrous Na_2CO_3. Cool to room temperature and make up to the 250-cc. mark.

Determine the dextrose in a 50-cc. aliquot. Correct the weight of dextrose obtained by subtracting the weight of dextrose obtained in a 50-cc. aliquot of the malt reagent control. Multiplying this value by 0.9 gives the weight of starch in the 50-cc. aliquot. This value $\times \frac{5}{15} \times 100 =$ the percentage of starch in the sirup.

EXPERIMENTAL RESULTS

Tables 1, 2, 3, and 4 give the sucrose, reducing sugars, and starch content of the sirups, and the calculated amounts of each in grams per 100 gm. of water as determined by the refractometer. The tables also show the ratio of sucrose to reducing sugars, the ratio of reducing sugars to sucrose, and the type of crystallization and jellying, if any.

TABLE 1.—*Solids, sucrose, reducing sugars, and starch content of sirups made from various internodes of Gooseneck variety of sorgo at different stages of maturity*

Sirup No.	Internodes	Total solids [1]	Water [1]	True sucrose (Clerget)			Reducing sugars as invert sugar			Sugar ratios		Crystallization	Starch			Jellying
				On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	Sucrose to reducing	Reducing to sucrose		On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	
	Nos.	*Percent*	*Percent*	*Percent*	*Grams*	*Grams*	*Percent*	*Grams*	*Grams*				*Percent*	*Grams*	*Grams*	
MILK STAGE																
1	1-2	79.30	20.70	16.20	20.43	78.26	52.45	66.13	253.38	0.30	3.23	Dextrose	0.39	0.50	1.91	Negative.
2	3-4	75.10	24.90	19.38	25.80	77.83	50.57	67.33	203.09	.38	2.61	do	.27	.36	1.08	Do.
3	5-6	78.60	21.40	21.95	27.92	102.57	51.90	66.02	242.52	.42	2.36	do	.23	.30	1.10	Do.
4	7-8	77.90	22.10	19.38	24.87	87.69	54.05	69.37	244.57	.35	2.78	do	.19	.25	.88	Do.
5	9-10	76.50	23.50	5.75	7.52	24.46	65.95	86.20	280.63	.08	11.46	do	.16	.21	.70	Do.
6	11-12	74.10	25.90	4.24	5.72	16.37	64.50	87.03	249.03	.06	15.21	do	.07	.09	.27	Do
DOUGH-TO-RIPE STAGE																
7	1-2	73.70	26.30	34.37	46.63	130.68	29.80	40.43	113.30	1.15	0.86	Negative	0.44	0.60	1.68	Negative.
8	3-4	79.30	20.70	39.97	50.40	193.09	32.65	41.17	157.72	1.22	.81	Sucrose	.39	.50	1.91	Do.
9	5-6	74.70	25.30	29.67	39.71	117.27	39.90	53.41	157.70	.74	1.34	Dextrose	.28	.38	1.12	Do.
10	7-8	72.10	27.90	20.14	27.93	72.18	48.24	66.90	172.90	.41	2.39	do	.16	.22	.56	Do.
11	9-10	76.70	23.30	16.65	21.71	71.45	56.11	72.12	240.81	.29	3.37	do	.09	.12	.39	Do.
12	11-12	78.30	21.70	16.50	21.07	76.03	56.63	72.32	260.96	.29	3.43	do	.08	.11	.39	Do.
13	(2)	75.50	24.50	33.46	44.31	136.57	36.80	48.74	150.20	.91	1.10	do	.20	.27	.83	Do.
DEAD-RIPE STAGE																
14	1-2	75.80	24.20	41.33	54.52	170.78	23.78	31.37	98.26	1.74	0.57	Negative	0.86	1.14	3.57	Jellied.
15	3-4	76.90	23.10	44.21	57.48	191.38	24.98	32.48	108.13	1.77	.56	do	.83	1.08	3.59	Do.
16	5-6	77.00	23.00	38.30	49.74	166.52	31.80	41.29	138.26	1.20	.83	Sucrose	.77	1.00	3.34	Do.
17	7-8	75.70	24.30	33.15	43.75	136.41	35.89	47.36	147.69	.92	1.08	Negative	.57	.76	2.36	Do.
18	9-10	74.50	25.50	29.67	39.82	116.35	43.49	58.53	170.54	.68	1.46	Dextrose	.43	.58	1.69	Negative
19	11-12	72.70	7.30	24.83	34.15	90.95	40.81	56.13	149.49	.60	1.64	do	.42	.58	1.54	Do.
20	(2)	74.10	25.90	37.40	50.46	144.40	30.95	41.76	119.49	1.21	.83	Negative	.67	.91	2.60	Jellied.

[1] By refractometer.
[2] Whole stalk.

TABLE 2.—*Solids, sucrose, reducing sugars, and starch content of sirups made from various internodes of Honey variety of sorgo at different stages of maturity*

Sirup No.	Internodes	Total solids [1]	Water [1]	True sucrose (Clerget)			Reducing sugars as invert sugar			Sugar ratios		Crystallization	Starch			Jellying
				On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	Sucrose to reducing	Reducing to sucrose		On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	
	Nos.	*Percent*	*Percent*	*Percent*	*Grams*	*Grams*	*Percent*	*Grams*	*Grams*				*Percent*	*Grams*	*Grams*	
MILK STAGE																
21	1–2	77.60	22.40	24.07	31.03	107.45	38.80	50.01	173.21	0.62	1.61	Dextrose	0.82	1.06	3.67	Jellied.
22	3–4	80.20	19.80	27.40	34.18	138.38	45.55	56.82	230.05	.60	1.66	do	.65	.81	3.28	Do.
23	5–6	81.40	18.60	20.89	25.67	112.31	56.20	69.07	302.15	.37	2.69	do	.49	.61	2.67	Do.
24	7–8	73.50	26.50	13.02	17.72	49.13	57.55	78.33	217.17	.22	4.42	do	.47	.64	1.77	Negative.
25	9–10	71.60	28.40	2.88	4.02	10.14	65.75	91.82	231.51	.04	22.83	do	.39	.54	1.37	Do.
26	11–12	73.40	26.60	1.36	1.85	5.11	65.30	88.96	245.49	.02	48.01	do	.26	.35	.97	Do.
27	[2]	76.40	23.60	20.28	27.85	85.93	50.85	66.58	215.46	.40	2.50	do	.46	.60	1.94	Do.
DOUGH-TO-RIPE STAGE																
28	1–2	79.50	20.50	38.76	48.75	189.07	29.42	37.00	143.51	1.31	0.76	Negative	0.52	0.66	2.56	Jellied.
29	3–4	77.30	22.70	40.58	52.49	178.76	31.77	41.09	139.95	1.27	.78	Sucrose	.51	.66	2.25	Do.
30	5–6	75.90	24.10	34.82	45.87	144.48	38.20	50.32	158.50	.91	1.10	Dextrose	.66	.87	2.74	Do.
31	7–8	73.50	26.50	25.74	35.02	97.13	44.50	60.54	167.92	.57	1.72	do	.55	.75	2.07	Negative.
32	9–10	76.50	23.50	17.11	22.36	72.80	54.95	71.82	233.83	.31	3.21	do	.56	.73	2.37	Do.
33	11–12	72.60	27.40	10.60	14.60	38.68	56.15	77.33	204.93	.19	5.29	do	.40	.56	1.48	Do.
34	[2]	72.80	27.20	24.22	33.26	89.04	45.25	62.15	166.36	.53	1.86	do	.52	.71	1.90	Do.
DEAD-RIPE STAGE																
35	1–2	76.90	23.10	24.22	31.49	104.84	40.75	52.98	176.40	0.59	1.68	Negative	0.55	0.72	2.40	Jellied.
36	3–4	80.00	20.00	31.95	39.93	159.75	40.50	50.62	202.50	.79	1.26	do	.60	.75	3.00	Do.
37	5–6	71.30	28.70	17.87	25.06	62.26	50.02	70.14	174.28	.36	2.78	Dextrose	.48	.67	1.66	Negative.
38	7–8	73.30	26.70	22.41	30.57	83.93	48.10	65.61	180.15	.46	2.14	do	.74	1.01	2.77	Jellied.
39	9–10	71.20	28.80	5.30	7.44	18.40	62.75	88.12	217.88	.08	11.84	do	.66	.92	2.29	Do.
40	11–12	72.30	27.70	5.60	7.74	20.21	63.05	87.19	227.61	.08	11.26	do	.60	.83	2.16	Negative.
41	[2]	78.60	21.40	37.40	47.58	174.76	35.47	45.12	165.75	1.05	.95	Negative	.80	1.02	3.74	Jellied.

[1] By refractometer.
[2] Whole stalk.

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TABLE 3.—*Solids, sucrose, reducing sugars, and starch content of sirups made from various internodes of Iceberg variety of sorgo at different stages of maturity*

Sirup No.	Internodes	Total solids [1]	Water [1]	True sucrose (Clerget)			Reducing sugars as invert sugar			Sugar ratios		Crystallization	Starch			Jellying
				On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	Sucrose to reducing	Reducing to sucrose		On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	
	Nos.	*Percent*	*Percent*	*Percent*	*Grams*	*Grams*	*Percent*	*Grams*	*Grams*				*Percent*	*Grams*	*Grams*	
DOUGH-TO-RIPE STAGE																
42	1–2	81.90	18.10	52.38	63.95	289.39	21.55	26.31	119.06	2.43	0.41	Sucrose	0.84	1.03	4.66	Jellied.
43	3–4	78.30	21.70	51.32	65.53	236.49	22.70	28.99	104.60	2.26	.44	do	.75	.96	3.45	Do.
44	5–6	77.90	22.10	47.24	60.63	213.76	27.17	34.87	122.94	1.74	.57	do	.51	.66	2.32	Do.
45	7–8	77.40	22.60	41.79	53.99	184.91	31.52	40.72	139.47	1.32	.75	do	.49	.64	2.19	Negative.
46	9–10	74.30	25.70	31.79	42.78	123.69	38.37	51.64	149.30	.82	1.21	Dextrose	.44	.59	1.70	Do.
47	[2]	77.50	22.50	46.33	59.77	205.91	26.32	33.96	116.97	1.76	.57	Sucrose	.66	.86	2.96	Jellied.
DEAD-RIPE STAGE																
48	1–2	71.00	29.00	47.99	67.62	165.48	14.92	21.02	51.45	3.21	0.31	Sucrose	1.15	1.63	3.98	Jellied.
49	3–4	80.00	20.00	55.41	69.29	277.05	18.32	22.91	91.60	3.02	.33	do	1.04	1.30	5.20	Do.
50	5–6	76.80	23.20	49.21	64.10	212.11	22.45	29.24	96.77	2.19	.46	do	.96	1.25	4.13	Do.
51	7–8	79.30	20.70	45.12	56.92	217.97	31.60	39.86	152.66	1.42	.70	do	.82	1.04	3.98	Do.
52	9–10	78.30	21.70	35.58	45.46	163.96	38.32	48.96	176.59	.92	1.08	Negative	.69	.88	3.17	Do.
53	[2]	76.70	23.30	52.08	67.93	223.51	17.62	22.98	75.62	2.95	.34	Sucrose	1.06	1.38	4.54	Do.

[1] By refractometer.
[2] Whole stalk.

TABLE 4.—*Solids, sucrose, reducing sugars, and starch content of sirups made from various internodes of Hodo variety of sorgo at different stages of maturity*

Sirup No.	Internodes	Total solids [1]	Water [1]	True sucrose (Clerget)			Reducing sugars as invert sugar			Sugar ratios		Crystallization	Starch			
				On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	Sucrose to reducing	Reducing to sucrose		On sirup basis	Per 100 gm. of solids	Per 100 gm. of water	Jellying
	Nos.	*Percent*	*Percent*	*Percent*	*Grams*	*Grams*	*Percent*	*Grams*	*Grams*				*Percent*	*Grams*	*Grams*	
MILK STAGE																
54	1–2	73.70	26.30	25.59	34.72	97.30	40.40	54.81	153.61	0.63	1.58	Dextrose	0.43	0.59	1.65	Negative.
55	3–4	78.00	22.00	35.58	45.61	161.72	37.17	47.65	168.95	.95	1.04	do	.29	.38	1.34	Do.
56	5–6	73.30	26.70	28.61	39.03	107.15	40.50	55.24	151.68	.70	1.41	do	.17	.24	.66	Do.
57	7–8	72.00	28.00	21.80	30.27	77.85	47.25	65.62	168.75	.46	2.17	do	.16	.23	.59	Do.
58	9–10	71.50	28.50	14.38	20.11	50.45	54.95	76.84	192.80	.26	3.82	do	.15	.22	.55	Do.
59	11–12	71.80	28.20	11.96	16.66	42.41	56.60	78.82	200.70	.21	4.73	do	.07	.10	.25	Do.
60	13–14	72.20	27.80	8.33	11.54	29.96	60.15	83.30	216.36	.14	7.22	do	.04	.06	.14	Do.
61	[2]	72.10	27.90	21.50	29.82	77.06	46.70	64.76	167.38	.46	2.17	do	.13	.19	.49	Do.
DOUGH-TO-RIPE STAGE																
62	1–2	78.20	21.80	46.78	59.83	214.58	25.15	32.17	115.36	1.86	0.54	Sucrose	0.52	0.67	2.40	Negative.
63	3–4	81.40	18.60	48.15	59.16	258.87	29.37	36.09	157.90	1.64	.61	do	.44	.55	2.40	Do.
64	5–6	77.70	22.30	39.36	50.66	176.50	34.20	44.02	153.36	1.15	.87	do	.40	.52	1.81	Do.
65	7–8	77.60	22.40	32.85	42.34	146.65	40.77	52.55	182.00	.80	1.24	Dextrose	.26	.34	1.17	Do.
66	9–10	78.90	21.10	25.44	32.25	120.56	49.85	63.19	236.25	.51	1.96	do	.21	.27	1.01	Do.
67	11–12	72.60	27.40	16.96	23.36	61.89	51.35	70.74	187.40	.33	3.03	do	.13	.19	.50	Do.
68	13–14	73.80	26.20	16.19	21.94	61.79	54.95	74.47	209.73	.29	3.39	do	.11	.16	.45	Do.
69	[2]	75.00	25.00	36.49	48.66	145.96	34.22	45.63	136.88	1.06	.94	Negative	.32	.43	1.29	Do.
DEAD-RIPE STAGE																
70	1–2	80.30	19.70	52.23	65.04	265.12	20.87	25.99	105.93	2.50	0.40	Sucrose	0.76	0.95	3.87	Negative.
71	3–4	81.30	18.70	53.44	65.72	285.77	24.02	29.54	128.44	2.22	.45	do	.54	.67	2.91	Do.
72	5–6	77.30	22.70	47.54	61.49	209.42	26.62	34.43	117.26	1.78	.56	do	.49	.64	2.17	Do.
73	7–8	77.40	22.60	42.69	55.15	188.89	31.92	41.23	141.23	1.33	.74	do	.31	.41	1.40	Do.
74	9–10	75.20	24.80	35.43	47.11	142.86	37.07	49.29	149.47	.95	1.04	Dextrose	.25	.33	1.00	Do.
75	11–12	75.10	24.90	33.16	44.15	133.17	38.97	51.88	156.50	.85	1.17	do	.20	.27	.81	Do.
76	13–14	76.00	24.00	35.28	46.41	147.00	37.65	49.53	156.87	.94	1.07	do	.18	.24	.76	Do.
77	[2]	75.30	24.70	39.36	52.26	159.35	33.05	43.89	133.80	1.19	.84	Sucrose	.42	.56	1.76	Do.

[1] By refractometer.

[2] Whole stalk.

Sucrose crystallized from 21 sirups, dextrose crystallized from 45, and 26 jellied. Only 2 sirups neither crystallized nor jellied. Eleven of the 26 sirups that jellied crystallized sucrose, 6 crystallized dextrose, and 9 jellied without crystallization.

With the exception of sirups from the Honey variety at the two later stages of maturity, when this variety dropped its seed and branched prolifically (*12*), the different sirups showed the following general tendencies.

DEXTROSE CRYSTALLIZATION

Reducing sugar content and its ratio to sucrose content were higher in sirups made from less mature sorgo and, in general, in sirups made from the lower portions of the stalk at each stage of maturity.

Dextrose crystallization was correlated with the ratio of reducing sugars to sucrose, occurring in 45 sirups in which this ratio was greater than 1. It did not occur, however, in 4 sirups in which this ratio was greater than 1.

The number of portions of the stalk giving sirups that crystallized dextrose decreased with maturity.

Dextrose crystallization occurred in sirups from all four varieties, but the extent varied.

SUCROSE CRYSTALLIZATION

Sucrose content and its ratio to reducing sugars were higher in sirups made from more mature sorgo and, in general, from the upper portions of the stalk.

Sucrose crystallization was correlated with the ratio of sucrose to reducing sugars, occurring in 21 sirups in which this ratio was greater than 1. It did not occur, however, in 7 sirups in which the ratio was greater than 1.

The number of portions of the stalk giving sirups that had ratios of sucrose to reducing sugars greater than 1 and which crystallized sucrose increased with maturity.

Sucrose crystallization occurred in sirups from all four varieties, but the extent varied in sirups from the different varieties.

STARCH

In general, the starch content was highest in sirups made from the upper internodes, and decreased progressively in sirups made from internodes toward the bottom of the stalk. As a rule, both the number of sirups that jellied and their starch content increased with maturity of the sorgo.

Jellying was correlated largely with high starch content of the sirups, although it also depended on the content of starch per 100 gm. of water. The minimum amount of starch per 100 gm. of water in sirups in which jellying occurred was 2.25 gm. (in sirup No. 29).

High starch content and the resulting jellying appear to have inhibited sucrose crystallization in five sirups (Nos. 14, 15, 20, 28, and 41) and dextrose crystallization in four (Nos. 17, 35, 36, 52).

In tables 5 and 6 the sirups are regrouped according to their crystallizing and jellying characteristics.

TABLE 5.—*Sucrose and starch content of sirups grouped according to tendency to jelly and to crystallize sucrose*

SIRUPS THAT CRYSTALLIZED SUCROSE BUT DID NOT JELLY

Sirup No.	Sucrose			Rates of sucrose to reducing sugars	Starch per 100 gm. of water	Sirup No.	Sucrose			Ratio of sucrose to reducing sugars	Starch per 100 gm. of water
	On sirup basis	Per 100 gm. of solids	Per 100 gm. of water				On sirup basis	Per 100 gm. of solids	Per 100 gm. of water		
	Percent	*Grams*	*Grams*		*Grams*		*Percent*	*Grams*	*Grams*		*Grams*
8	39.97	50.40	193.09	1.22	1.91	70	52.23	65.04	265.12	2.50	3.87
45	41.79	53.99	184.91	1.32	2.19	71	53.44	65.72	285.77	2.22	2.91
62	46.78	59.83	214.58	1.86	2.40	72	47.54	61.49	209.42	1.78	2.17
63	48.15	59.16	258.87	1.64	2.40	73	42.69	55.15	188.89	1.33	1.40
64	39.36	50.66	176.50	1.15	1.81	77	39.36	52.26	159.35	1.19	1.76
SIRUPS THAT CRYSTALLIZED SUCROSE AND ALSO JELLIED											
16	38.30	49.74	166.52	1.20	3.34	49	55.41	69.29	277.05	3.02	5.20
29	40.58	52.49	178.76	1.27	2.25	50	49.21	64.10	212.11	2.19	4.13
42	52.38	63.95	289.39	2.43	4.66	51	45.12	56.92	217.97	1.42	3.98
43	51.32	65.53	236.49	2.26	3.46	53	52.08	67.93	223.51	2.95	4.54
44	47.24	60.63	213.76	1.74	2.32	47	46.33	59.77	205.91	1.76	2.96
48	47.99	67.62	165.48	3.21	3.98						
SIRUPS THAT HAD RATIOS OF SUCROSE TO REDUCING SUGARS GREATER THAN 1 BUT DID NOT CRYSTALLIZE SUCROSE											
7	34.37	46.63	130.68	1.15	1.68	28	38.76	48.75	189.07	1.31	2.56
14	41.33	54.52	170.78	1.74	3.57	41	37.40	47.58	174.76	1.05	3.74
15	44.21	57.48	191.38	1.77	3.59	69	36.49	48.66	145.96	1.06	1.29
20	37.40	50.46	144.40	1.21	2.60						

TABLE 6.—*Sucrose and starch content of sirups grouped according to tendency to jelly and to crystallize dextrose*

SIRUPS THAT CRYSTALLIZED DEXTROSE BUT DID NOT JELLY

Sirup No.	Reducing sugars as invert sugar			Ratio of reducing sugars to sucrose	Starch per 100 gm of water	Sirup No.	Reducing sugars as invert sugar			Ratio of reducing sugars to sucrose	Starch per 100 gm. of water
	On sirup basis	Per 100 gm. of solids	Per 100 gm. of water				On sirup basis	Per 100 gm. of solids	Per 100 gm. of water		
	Percent	*Grams*	*Grams*		*Grams*		*Percent*	*Grams*	*Grams*		*Grams*
1	52.45	66.13	253.38	3.23	1.91	37	50.02	70.14	174.28	2.78	1.66
2	50.57	67.33	203.09	2.61	1.08	40	63.05	88.12	227.61	11.26	2.16
3	51.90	66.02	242.52	2.36	1.10	46	38.37	51.64	149.30	1.21	1.70
4	54.05	69.37	244.57	2.78	.88	54	40.40	54.81	153.61	1.58	1.65
5	65.95	86.20	280.63	11.46	.70	55	37.17	47.65	168.95	1.04	1.34
6	64.50	87.03	249.03	15.21	.27	56	40.50	55.24	151.68	1.41	.66
9	39.90	53.41	157.70	1.34	1.12	57	47.25	65.62	168.75	2.17	.59
10	48.24	66.90	172.90	2.39	.56	58	54.95	76.84	192.80	3.82	.55
11	56.11	72.12	240.81	3.37	.39	59	56.60	78.82	200.70	4.73	.25
12	56.63	72.32	260.96	3.43	.39	60	60.15	83.30	216.36	7.22	.14
13	36.80	48.74	150.20	1.10	.83	61	46.70	64.76	167.38	2.17	.49
18	43.49	58.53	170.54	1.46	1.69	65	40.77	52.55	182.00	1.24	1.17
19	40.81	56.13	149.49	1.64	1.54	66	49.85	63.19	236.25	1.96	1.01
24	57.55	78.33	217.17	4.42	1.77	67	51.35	70.74	187.40	3.03	.50
25	65.75	91.82	231.51	22.83	1.37	68	54.95	74.47	209.73	3.39	.45
26	65.30	88.96	245.49	48.01	.97	74	37.07	49.29	149.47	1.04	1.00
31	44.50	60.54	167.92	1.72	2.07	75	38.97	51.88	156.50	1.17	.81
32	54.95	71.82	233.83	3.21	2.37	76	37.65	49.53	156.87	1.07	.76
33	56.15	77.33	204.93	5.29	1.48	27	50.85	66.58	215.46	2.50	1.94
34	45.25	62.15	166.36	1.87	1.90						
SIRUPS THAT CRYSTALLIZED DEXTROSE AND ALSO JELLIED											
21	38.80	50.01	173.21	1.61	3.67	30	38.20	50.32	158.50	1.10	2.74
22	45.55	56.82	230.05	1.66	3.28	38	48.10	65.61	180.15	2.14	2.77
23	56.20	69.07	302.15	2.69	2.67	39	62.75	88.12	217.88	11.84	2.29
SIRUPS WITH RATIOS OF REDUCING SUGARS TO SUCROSE GREATER THAN 1 THAT DID NOT CRYSTALLIZE DEXTROSE BUT JELLIED											
17	35.89	47.36	147.69	1.08	2.36	36	40.50	50.62	202.50	1.26	3.00
35	40.75	52.98	176.40	1.68	2.40	52	38.32	48.96	176.59	1.08	3.17

The sirups that crystallized sucrose but did not jelly are grouped in the first part of table 5. Sirup No. 77 had the lowest sucrose content per 100 gm. of water, 159.35, and sirup No. 64 had the lowest ratio of sucrose to reducing sugar, 1.15. In four sirups, Nos. 62, 63, 70, 71, the content of starch per 100 gm. of water was greater than 2.25 gm., which as previously noted was the minimum starch content of the sirups that jellied. High ratios of sucrose to reducing sugar and a relatively high content of sucrose per 100 gm. of water characterized these particular sirups.

The sirups that crystallized sucrose and likewise jellied are grouped in the second part of table 5. The minimum ratio of sucrose to reducing sugars in this group was 1.20, and the minimum content of sucrose per 100 gm. of water was 165.48, both of which are higher than the minimum values in part 1 of this table. The higher values for these sirups were probably due to the influence of jellying on crystallization.

Seven sirups with ratios of sucrose to reducing sugars greater than 1 which did not crystallize sucrose are listed in the last part of table 5. Five of these seven sirups jellied, which probably inhibited crystallization. Thirty-nine sirups that crystallized dextrose, but did not jelly are listed in the first part of table 6. The minimum content of dextrose per 100 gm. of water was 149.30 gm., with a minimum ratio of reducing sugar to sucrose of 1.04.

Data on six sirups that crystallized dextrose and also jellied are given in the second part of table 6. In these the proportion of reducing sugars per 100 gm. of water was greater than the minimum value, 149.30, in part 1. The ratios of reducing sugars to sucrose were also greater than the minimum value, 1.04, in the first part of this table. Probably the increase was due to the influence of jellying on dextrose crystallization.

The last part of table 6 presents data on four sirups with ratios of reducing sugar to sucrose greater than the minimum in part 1. All four of these sirups jellied, as will be noted by referring to tables 1 to 4, and three exhibited a greater content of reducing sugars per 100 gm. of water than the minimum in part 1. Here, again, the effect of jellying on crystallization is apparent.

CONCLUSIONS

From data obtained in a critical examination of 67 samples of sirup made from parts of the sorgo stalk and of 10 samples made from the whole stalk, the following conclusions are drawn.

The starch content and jellying of sorgo sirups are correlated and increase with maturity of the sorgo. The upper portions of the stalk produce sirups higher in starch content. The number of parts of the stalk yielding sirups that jelly increases with maturity.

Sucrose crystallization occurs most frequently in sirups made from the upper part of the sorgo stalk. The number of parts of the sorgo stalk yielding sirups from which sucrose crystallizes increases with maturity.

Dextrose crystallization occurs most frequently in sirups made from the lower portions of the stalk. The number of portions of the stalk yielding sirups from which dextrose crystallizes decreases with maturity.

Within the range of densities applying to farm-made sorgo sirups, either sucrose or dextrose may crystallize from sirups from different parts of the same stalk. In this study it was found that when the ratio of sucrose to reducing sugars in the sirup was 1.15 or greater sucrose crystallized and when the ratio of reducing sugars to sucrose was 1.04 or greater dextrose crystallized.

As a factor in the quality of sorgo sirup, crystallization of dextrose is as important as crystallization of sucrose.

By proper selection of the parts of the stalk for milling, either sucrose or dextrose may be obtained from the sorgo plant.

Jellying and either sucrose or dextrose crystallization may occur in the same sirup.

LITERATURE CITED

(1) ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS.
1935. OFFICIAL AND TENTATIVE METHODS OF ANALYSIS . . . Ed. 4, 710 pp., illus. Washington, D. C.

(2) BERTHELOT, DANIEL, and TRANNOY, RENÉ.
1918. SUR LA TENEUR EN SUCRE DU SORGHO AUX DIVERS STADES DE SA VÉGÉTATION. Compt. Rend. Acad. Sci. [Paris] 166: 824–827.

(3) GILBEE, W. A.
1858. TREATING SACCHARINE FLUIDS. Brit. Patent 655, granted Mar. 27, 1858.

(4) JANSSEN, GEO., MCCLELLAND, C. K., and METZGER, W. H.
1930. SAP EXTRACTION OF SORGHUM AND THE LOCALIZATION OF JUICE AND SUGARS IN INTERNODES OF THE PLANT. Jour. Amer. Soc. Agron. 22: 627–639.

(5) JOULIE, H.
1864. ÉTUDES ET EXPÉRIENCES SUR LE SORGHO À SUCRE CONSIDÉR[*e*] AU POINT DE VUE BOTANIQUE, AGRICOLE, CHIMIQUE, PHYSIOLOGIQUE ET INDUSTRIEL. 200 pp., illus. Paris.

(6) SHERWOOD, SIDNEY F.
1923. STARCH IN SORGHUM JUICE. Indus. and Engin. Chem. 15; 727–728.

(7) ———
1923. JELLYING OF SORGHUM SIRUP. EFFECT OF MALT DIASTASE UPON THE FILTRATION OF SORGHUM JUICE. Indus. and Engin. Chem. 15 : 780–782, illus.

(8) WALTON, C. F., JR., and VENTRE, E. K.
1937. EVAPORATOR FOR RESEARCH ON SIRUP MANUFACTURE. Internatl. Sugar Jour. 39 : 430–31.

(9) WILEY, H. W.
1889. RECORD OF EXPERIMENTS CONDUCTED BY THE COMMISSIONER OF AGRICULTURE IN THE MANUFACTURE OF SUGAR FROM SORGHUM AT RIO GRANDE, NEW JERSEY; KENNER, LOUISIANA, CONWAY SPRINGS, DOUGLASS, AND STERLING, KANSAS, 1888. U. S. Dept. Agr. Div., Chem. Bull. 20, 162 pp.

(10) ———
1892. RECORDS OF EXPERIMENTS WITH SORGHUM IN 1891. U. S. Dept. Agr., Div. Chem. Bull. 34, 132 pp., illus.

(11) WILLAMAN, J. J., and DAVISON, F. R.
1924. STARCH IN SORGHUM SIRUP. Indus. and Engin. Chem. 16 : 609–610.

(12) VENTRE, EMIL K., and BYALL, S.
1937. DISTRIBUTION AND VARIATION WITH MATURITY OF DISSOLVED SOLIDS, SUCROSE, AND TITRATABLE ACIDITY IN THE SORGO STALK. Jour. Agr. Research 55 : 553–562.