

treated vegetables in the raw or canned forms did not produce any changes in flavor. If a product had exhibited a significant change in flavor due to insecticide treatment when evaluated immediately, it usually had the same rating when evaluated after storage. Possible exceptions to this generalization were insecticide-treated canned onions which developed a flat flavor after 9 months' storage.

Some changes in color were noted to occur along with the changes in flavor. Canned beets were markedly affected by applications of endrin, the color turning from bright red to deep red-black. Some slight increases in the redness of yellow squash, pumpkin, carrots, and rutabagas as determined by the Hunter color difference meter were also noted.

The rates of application of insecticides used in these tests were approximately four times normal. In most cases when a flavor change occurred at four times the normal rate of application, a similar change occurred at normal rates of application (76). Lesser rates of application of the insecticides should, therefore, be made with caution. The selection of insecticides is greatly narrowed by consideration of flavor effects as well as tolerance specifications but this enforced decrease in utility will

ultimately result in higher quality and purity of vegetable crops.

#### Acknowledgment

The authors wish to acknowledge the project support of this study by Allied Chemical and Dye Corp., California Spray Chemical Corp., Chemagro Corp., Diamond Alkali Co., Ethyl Corp., Hercules Powder Co., Shell Chemical Corp., and Velsicol Corp.

#### Literature Cited

- (1) American Can Co., Research Division, "Manual for Institutional Canning," Barrington, Ill., 1945.
- (2) Boswell, V. R., Clore, W. J., Pepper, B. B., Taylor, C. B., Gilmer, P. M., Carter, R. L., U. S. Dept. Agr. Tech. Bull. **1121** (1955).
- (3) Gilpin, G. L., Dawson, E. H., Geissenhainer, E. L., Reynolds, E. H., *Food Technol.* **7**, 132-5 (1953).
- (4) Gould, W. A., Slesman, J. P., Rings, R. W., Lynn, M., Krantz, F., Brown, H. D., *Ibid.*, **5**, 129-33 (1951).
- (5) Greenwood, M. L., Tice, J. M., *J. Agr. Research* **78**, 477-82 (1949).
- (6) Hening, J. C., Davis, A. C., Robinson, W. B., *Food Technol.* **8**, 227-9 (1954).

- (7) Hinreiner, E., Simone, M., *Hilgardia* **26**, 76-85 (1956).
- (8) Kirkpatrick, M. E., Linton, G. S., Mountjoy, B. M., Albright, L. C., *J. Agr. Food Chem.* **3**, 409 (1955).
- (9) Lindgren, D., Anderson, L., Frost, M. H., Jr., *Calif. Agr.* **8**, 9 (1954).
- (10) Merrill, L. G., Michigan Agr. Expt. Sta. Quar. Bull. **36**, 169-72 (1953).
- (11) Ristich, S. S., Schwardt, H. H., *J. Econ. Entomol.* **42**, 77-80 (1949).
- (12) Roessler, E. B., Warren, J., Guymon, J. F., *Food Research* **13**, 503 (1948).
- (13) Shell Chemical Corp., "Agricultural Handbook for Aldrin and Dieldrin," Denver, Colo., 1954.
- (14) Stitt, L. L., Evanson, J., *J. Econ. Entomol.* **42**, 614-17 (1949).
- (15) Stone, M. W., Foley, J. B., Bixby, D. H., U. S. Dept. Agr. Dept. Circ. **926** (1953).
- (16) Weckel, K. G., Birdsall, J., Chapman, R. K., "Organoleptic Studies of Vegetables Treated with Chlorinated Hydrocarbon Insecticides," interdepartmental report, unpublished. University of Wisconsin, Madison, Wis., June 6, 1956.
- (17) Weckel, K. G., Chapman, R. K., *Food Packer* **34**, 32-4 (1953).

Received for review August 4, 1956. Accepted November 23, 1956.

## SORGO ANALYSES

### Methods for Extraction of Sucrose from Sorgo

CHARLES PRICE and J. M. FIFE  
Field Crops Research Branch,  
Agricultural Research Service,  
U. S. Department of Agriculture,  
Salinas, Calif.

Accurate selection of commercial varieties of sorgo for sugar production required a dependable method for determining sucrose content. A study was made of three methods, using the chips, shreds, and extracted juice of the Rex variety, for sucrose and marc determinations. Either the hot- or the cold-water digestion method is suitable for the determination of sucrose in shredded sorgo. The sucrose yield from sorgo chips by the hot-water digestion method was 86% and by the cold-water method was only 40% of the amount extracted from the shreds. Purity and nitrogen determinations are also presented.

SORGO RESEARCH was started in the Imperial Valley of California during 1942 to evaluate available commercial varieties for sugar production. None of the commercial varieties was satisfactory; therefore a breeding program was started to develop high-yielding varieties possessing high sucrose content and adapted to growing conditions in the Imperial Valley. Since then a suitable variety has been developed which is promising for commercial cul-

ture and for sugar processing in sugar beet factories at a time when these factories otherwise are idle.

The accurate evaluation of commercial varieties and promising selections for sugar production required a dependable laboratory method for determining the sucrose content. A comparison of three laboratory methods for sucrose and marc determinations, as well as reducing sugars, purity, and nitrogen determinations are presented.

#### Materials Used and Preparation of Samples

Although better varieties are now available, Rex was used for this work because of its relatively high sucrose content.

The sorgo material used in these tests was selected and prepared in the following manner: Twenty-one sorgo stalks were taken at random, when the seed was in the hard-dough stage, from a sorgo experimental plot in the Imperial

Valley. These were cut off between the first and second nodes above the soil level. The leaves were stripped from the stalks, but the leaf sheath was not removed. The seed head was removed immediately below the top node and each of the 21 stalks was cut into three equal lengths and marked A, B, and C, respectively, beginning at the base of the stalk. The stalks were then divided so that a section of each stalk was placed in each of three samples. The stalks in one of three samples were sectioned with a band saw into relatively thin cross sections, 2 to 5 mm. thick. The stalks in the second sample were shredded with a small laboratory shredder commonly used for preparing sugar cane samples. The stalks in the third sample were run through a laboratory mill and the juice was extracted from them by means of a series of rollers. These three lots of samples were removed to the quick-freeze locker and stored at  $-5^{\circ}$  F. for approximately 2 weeks, until the analyses were begun. For convenience, the sectioned stalks are referred to as "chips" and the shredded stalks as "shreds."

### Methods and Results

**Determination of Marc.** The marc was determined on both shreds and chips of the stalk by three standard methods used for sugar beet roots and by one method used for sugar cane with only slight modifications, which were found necessary owing to the texture of the material used.

**Method I** Fifty to 60 grams of shreds or chips were transferred to a beaker and tap water was added. The ratio of water to sample in all extractions was approximately 8 to 1. The sample was placed in a partial vacuum for 5 minutes to remove the air. The entire sample then remained completely submerged in the extracting solutions. After 30 minutes of extraction, the solution was removed by suction through a sheet of felt stretched over a 6-cm. Büchner funnel. The samples were extracted three times (each for 30 minutes) with cold tap water followed by two extractions with boiling water (each for 5 minutes). Following the last extraction, the residue was collected on a weighed, dried filter paper. The filter paper and the marc were dried to a constant weight at  $100^{\circ}$  to  $105^{\circ}$  C. No sand or other extraneous matter was found in the samples, consequently, no correction was made for such material.

**Method II** The samples were extracted in the same manner, except that boiling water was poured over the samples and allowed to digest for 10 minutes for each extraction.

**Method III** The samples were extracted once with 70% alcohol for 20 minutes, followed by three

**Table I. Sorgo Marc as Determined from Uniform Samples**

Method	Marc	
	Chips, %	Shreds, %
I <sup>a</sup>	20.89	12.88
	18.56	12.85
	18.49	12.34
	16.00	12.67
	18.48 <sup>b</sup>	12.68 <sup>b</sup>
II	17.44	14.29
III	20.12	13.24
IV	17.99	12.33

<sup>a</sup> Four replicate determinations of a uniform sample of sorgo show the variability of marc in chips while in shreds this variation is not shown.

<sup>b</sup> Mean.

cold-water extractions each for 20 minutes and, finally, one extraction with boiling water for 10 minutes.

**Method IV** The samples were extracted for 24 hours in running tap water in a calico bag of double thickness. The excess water was squeezed from the bag by hand and the samples were dried to a constant weight. (This method is used for sugar cane.)

Table I shows the per cent of marc obtained by the various methods of extraction. It was much higher from chips than from shreds, because the chips were so coarse that the extraction did not completely remove all of the soluble substances. This was substantiated by sucrose determinations made on each successive extract in certain tests and on the last extraction made in most of the tests. Even with seven extractions with hot water, the last extract, when polarized, showed sucrose to be present, while with shreds the extract, in most cases, was free of sucrose after the third extraction. The variation in the per cent of marc of the shredded samples does not appear to be due to incomplete extraction or to leaching out of hemicelluloses or pectins, but probably to lack of uniformity among the different samples. For example, the sample which was extracted seven times with hot water gave the highest marc and yet, by test, was free of sucrose after the fourth extraction. Further extraction in this test did not appear to dissolve significant amounts of the hemicelluloses or pectins as indicated by the high value for the marc.

**Determination of Sucrose.** A mean marc content of 12.95% was found for the shreds, indicating that the samples contained 87.05% of juice. The juice was found to have a specific gravity of 1.1010 (at  $25^{\circ}$  C.), or a volume of 20.55 ml. of juice in 26 grams of pulp. With this sample of shreds of this weight, the proper amount of water to add to 26 grams of pulp for a sucrose determination is 179.45 ml.

A number of sucrose determinations were made on the shreds and also on the chips cut from the same lot of sorgo stalks to determine the completeness of extraction of sucrose from the two samples. The one-solution method devised by Bachler (2) was used. The sucrose determinations were carried out by hot-water digestion and also by cold-water digestion for the same length of time, 45 minutes. Samples of 26 grams each were digested in 200 ml. of water containing sufficient dry lead to clarify the solution. The correction was made to each of the sucrose percentages after the true marc had been established. The air was removed from the samples by placing them under a partial vacuum for 5 minutes to obtain complete submersion of the sample during the digestion.

The per cent of sucrose found in the shreds and chips by both hot- and cold-water digestion and also the sucrose in the extracted juice is shown in Table II.

The slight difference in the percentage of sucrose in favor of the hot-water digestion over that of the cold-water digestion in the shredded samples is not significant. The coefficient of variation for sucrose by the hot-water digestion method was slightly higher than 1% for both shreds and chips. This indicates that the sample of shreds and chips were uniform, and that the significantly greater amount of sucrose from the shreds by hot water was due to a more complete extraction.

Although the chip samples appear to be uniform, the percentage of sucrose obtained by the cold-water digestion was highly variable, showing that extraction was incomplete. The coefficient of variation in sucrose for replicate samples of chips extracted with cold water was 16% as compared with 1% by the hot-water digestion method. The hot-water digestion of the chips yielded approximately 86% of the sucrose in the

**Table II. Sugars and Purities of Shredded and Chipped Sorgo Stalk Samples and on Extracted Juice**

Test	Replicates	Mean Sucrose, %				Juice Sucrose, %
		Hot Water Digestion		Cold Water Digestion		
		Shreds	Chips	Shreds	Chips	
1	10	14.31 <sup>a</sup>	12.25			
2	4	14.50 <sup>a</sup>	12.47	14.15 <sup>a</sup>	5.80	17.8

<sup>a</sup> Significantly greater than chips at 1% level.

**Table III. Nitrogen Fractions and Dry Matter in Shredded Sorgo Samples**

Fraction	G. of N/1000 G. of Dry Tissue
Total nitrogen	4.41
Soluble nitrogen	3.19
Nitrogen soluble in copper hydroxide	1.96
Ammonia plus amide nitrogen	0.417
Harmful nitrogen (1.960 - 0.417)	1.543
Dry matter	32.87%

shredded samples, while with cold-water digestion the sucrose in the chips was only 40% of that in shredded samples.

The extracts from the chips with hot water filtered in approximately 5 minutes, whereas the extracts from the shredded samples required approximately 30 minutes. The filtrates obtained by hot-water digestion gave a very light, straw-colored solution, whereas the clear extract from the shredded samples was a light amber color. This slight coloration of the clarified filtrates did not interfere with the saccharimeter readings.

**Determination of Harmful Nitrogen.** The methods used for the determina-

tion of harmful nitrogen in sorgo are essentially those developed by the European investigators for sugar beets. The term "harmful nitrogen" is here applied to the nitrogen not precipitated by copper hydroxide minus the total ammonia nitrogen (ammonia plus amides). Briefly, the methods used for the nitrogen studies are as follows:

Approximately 350 ml. of water was added to 100 grams of shredded sample in a calibrated 500-ml., wide-mouthed Erlenmeyer flask and placed under a vacuum to remove the air from the tissue, then transferred to a water bath which was held at 85° C. After the sample had reached the temperature of the bath, 50 ml. of copper sulfate solution (60 grams of copper sulfate made up to 1000 ml. with water) and 50 ml. of sodium hydroxide (0.312*N*) were added and thoroughly mixed. The samples were allowed to digest for 15 minutes with frequent shaking, after which they were cooled to room temperature and made up to 500 ml. and filtered, clear, through dry paper pulp. Aliquots were removed from the clear extract to determine the nitrogen not precipitated by copper hydroxide. The ammonia-plus-amide nitrogen was determined in aliquots, made 1*N* with sulfuric acid, after hydrolysis for 2 hours. The samples were neutralized with sodium hydroxide and then made alkaline with an alkaline borate mixture (0.5*N* sodium hydroxide

in 5% borax) and the ammonia distilled off at atmospheric pressure. Total nitrogen was determined on a 5-gram sample of the shreds. The total soluble nitrogen content of the samples was determined by placing 20 grams of sample in a 200-ml. Kohlrausch flask with 180 ml. of water, removing the air by vacuum, and then placing the flask in a water bath at 90° C. After the sample had attained the temperature of the bath, sufficient 1*N* acetic acid was added to bring the pH to 4.7. The extract was cooled to room temperature, made up to volume, and filtered through dry paper pulp. The nitrogen determinations were carried out using official methods which did not include the nitrates (7). The nitrogen fractions are shown in Table III.

The harmful nitrogen content of the sorgo canes used in these tests is only approximately one half that found in sugar beets grown in the same general locality in 1944 and 1945.

#### Literature Cited

- (1) Assoc. Offic. Agr. Chemists, Washington, D. C., "Official and Tentative Methods of Analysis," 6th ed., pp. 572-74, 1945.
- (2) Bachler, F. R., *Facts About Sugar* 28, 420-3 (1933).

Received for review August 25, 1956. Accepted November 24, 1956.

## XANTHOPHYLL OIL STABILIZATION

### Protective Starch Matrix for Xanthophyll Oil and Vitamin A

Oxygen-sensitive materials such as corn xanthophyll oil, vitamin A, or white phosphorus can be protected against air oxidation by emulsifying the materials in a cooked paste of a suitable modified starch, and drying the emulsion as a thin film or flake. The starchy base must be a good protective colloid and must give molecularly dispersed solutions, free from swollen granules or retrograded starch. Suitable starch types include hydrolyzed corn-starch of 5 to 20 dextrose equivalent, high-soluble canary dextrans, thin-boiling waxy starches, and thin-boiling oxidized or etherified starches.

THE FOOD AND AGRICULTURAL INDUSTRIES need means of protecting water-insoluble substances from deterioration by air oxidation or by evaporation. The stabilization of vitamin preparations for human use or for stock feed supplements, the protection of essential oils and flavoring agents, and the production of dry insecticidal dusting powder are examples. There are methods for protecting such materials as vitamin A and flavoring oils, many of which merely involve soaking these substances into a suitable carrier—e.g., flour or a roll-

flaked cereal base. However, the added substance is still accessible to the air and the degree of protection is low. Olsen and Seltzer (2) described the emulsification of citrus oils in a warm gelatin solution, cooling to produce a rigid gel, then mechanically subdividing the latter and drying the gel particles to give a stable dispersion of the oil in a solid gelatin matrix. Taylor (1) protected oxygen-sensitive vitamin A and D concentrates in a somewhat similar fashion. More recently, Schlenk, Sand, and Tillotson (3) reported the protection

of vitamin A by the formation of a molecular complex with the cyclic Schar-dinger  $\alpha$ - and  $\beta$ -dextrans.

Corn xanthophyll oil, a by-product isolated from corn gluten during the manufacture of zein, contains approximately 1 gram of xanthophyll and 0.5 gram of carotene per pound. This study was undertaken to find a cheap and simple method for protecting xanthophyll oil in a starchy matrix, primarily for use as a poultry feed supplement. Protection is provided only by certain specific types of starch products

EILEEN C. MAYWALD and  
THOMAS J. SCHOCH  
George M. Moffatt Research  
Laboratories, Corn Products  
Refining Co., Argo, Ill.