Determination of Nonvolatile Acids and Sugars from Fruits and Sweet Potato Extracts by Capillary GLC and GLC/MS

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An analytical technique has been developed to determine nonvolatile acids and sugars in fruit and sweet potato extracts based on gas-liquid chromatography using a single capillary column. After simple extraction of fresh apples, pears, peaches, and sweet potato tissue with 75% ethanol, the sugars were converted to their oxime-TMS derivatives and the nonvolatile acids to their TMS derivatives. Identifications of sugars and acids were obtained by comparison of retention times and mass spectra with authentic compounds. The quantitative results obtained with the procedure agreed well with literature values of nonvolatile acids and sugars from similar cultivars of fruits and sweet potatoes. The mass spectra of the major sugar oxime-TMS sugars found in fruits and sweet potatoes are given, which previously have not been reported.

Many GLC techniques have been described in the literature for the determination of sugars from a variety of plant material. In most of these procedures the sugars are converted to volatile derivatives as alditol acetates, TMS, and oxime-TMS derivatives (Sweeley et al., 1963; Mason and Slover, 1971; Tanowitz and Smith, 1984; Traitler et al., 1984; Robards and Whitelaw, 1986; Van Den et al., 1986). Numerous columns, both packed and capillary, and GC conditions have been adequately described in an excellent review on the subject by Robards and Whitelaw (1986).

The determination of nonvolatile acids from various sources have been conducted by HPLC of the free acids (Marsili et al., 1981; Guerrant et al., 1982) and GLC, either as methyl esters or TMS derivatives (Li and Woodroof, 1968; Horrii et al., 1965; Fernandez-Flores et al., 1970).

All of the GLC analytical methods so far described by the determination of sugars or nonvolatile acids utilize GC columns and conditions designed for either one or the other class of compounds. Currently, there are no analytical methods described that would allow the determination of both nonvolatile acids and sugars on a single GC column.

This paper presents a method for the identification and quantification of the major sugars and nonvolatile acids found in apple, pear, peach, and sweet potato extracts with use of a single GC capillary column.

We also report the major mass ions and their abundance for oxime-TMS sugars, data previously unavailable due to limitations of published methods.

MATERIALS AND METHODS

Plant Material. Apples (var. Red Delicious), peaches (var. Loring), and pears (var. Danjou) used in this study were obtained from a local supermarket. Sweet potatoes (var. Jewel and Tai 57) were obtained through cooperation of Dr. Stanley Kays, University of Georgia Horticultural Department, Athens, GA.

Extraction of Sugars and Acids. About 5 g of mesocarp tissue from each fruit and the parenchyma tissue of sweet potatoes were diced with a sharp knife and glass rod in a small mortar. Approximately 5 mL of 75% ethanol was added and the tissue finely ground with a pestle. Additional 75% ethanol was added to the ground mesocarp to give a final volume of 25 mL. The tissue was extracted for 10 min (room temperature) and filtered through Whatman No. 4 filter paper into a small flask. Total yield of sugars and acids with this extraction procedure was approximately 100%. One-half-milliliter aliquots of these extracts were used for sugar and acid analysis.

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TMS Derivatization and Capillary GC Analysis. A $12 \times 100 \text{ mm}$ test tube containing 0.5 mL of extract was placed on a TEMP-Blok heater at 75 °C and the solvent evaporated to dryness with a stream of dry nitrogen. Sugars were initially converted to their oximes by the addition of 0.5 mL of hydroxyle mine (25 mg/mL of pyridine) containing phenyl β -D-glucoside as internal standard (6 mg/mL of pyridine) and heated to 75 °C for 30 min. Sugars and nonvolatile acids were then converted to their TMS derivatives by addition of 0.5 mL of BSTFA + 1% TMCS (Pierce Chemical Co.), and the resultant mixture was heated for an additional 20 min. After reaction, a small amount of anhydrous sodium sulfate was added to ensure dryness. After cooling, 0.5-1.0- μ L injections were made.

Capillary GC analysis of oxime-TMS sugars and TMS acids were conducted on a Hewlett-Packard 5890A gas chromatograph with a 3392A reporting integrator (Avondale, PA). The capillary column (J&W, Alltech, Inc., Deerfield, IL) was a 15×0.25 mm (i.d.) fused silica, DB-1, 0.25- μ m film thickness. Injector and detector temperatures were 225 and 280 °C, respectively. Oven temperature was held at 150 °C for 4 min, then programmed to 192 °C at 4 °C/min and held for 0.5 min, and then programmed to 240 °C at 10 °C/min and held for 7 min. Hydrogen was used as carrier gas with a linear flow rate of 42 cm/s. The split injection mode was used with a split flow rate of 80 mL/min. Septum purge flow rate was 5–6 mL/min. Nitrogen was used as makeup gas at a flow rate of 43 mL/min. Air and hydrogen flow rates to the detector were 250 and 24 mL/min, respectively.

All authentic compounds used in this study were obtained from reputable chemical companies. Individual sugars and acids were identified by comparison of their retention times and mass spectra with those of TMS derivatives of authentic compounds prepared in the same manner. Sugars and acids were quantitated with use of phenyl β -D-glucoside (Sigma Chemical Co., St. Louis, MO) as internal standard. Sugar and acid composition were expressed at weight percent of fresh and dry matter.

Mass Spectral Analysis. An Extrel Model C50/400 (Pittsburg, PA) quadrapole mass spectrometer interfaced with a Perkin-Elmer Sigma 300 GC (Norwalk, CT) equipped with a cold on-column injector was used. Chromatographic separations were made on a 20 m \times 0.32 mm (i.d.) fused silica column coated with SE-54. Helium was the carrier gas with a head pressure of 0.5 psi. Initial column temperature was 130 °C, which was then programmed to 190 °C at 4 °C/min and held for 0.5 min and then programmed to 250 °C at 10 °C/min and held for 38.5 min, for a total run time of 60 min. Mass spectrometer conditions: ion source temperature, 150 °C; scan rate, 200 amu/s; ionization energy, 70 eV. The interface temperature between the GC and mass spectrometer was 200 °C. Data were acquired with Technivent software (St. Louis, MO) on an IBM PC (640K, 40MB disk, Boca Raton, FL).

RESULTS AND DISCUSSION

A typical GLC chromatogram of a synthetic mixture of derivatized sugars and nonvolatile acids is shown in Figure

 Table I. Retention Times of Oxime-TMS Sugars and TMS

 Acids on DB-1 Capillary Column

component ^a	time, min	component ^a	time, min
succinic acid	1.29	mannitol	12.33
glyceraldehyde	1.67	sorbitol	12.51
dihydroxyacetone	1.69	fructose	12.71
malic acid	2.63	galactose	13.67
erythrose	3.78	mannose	13.78
pyruvic acid	4.39	glucose	13.95
tartaric acid	5.10	galacturonic acid	14.71
desoxyribose	6.18	caffeic acid	15.62
xylitol	7.02	inositol	15.76
xylose	8.04	G-1-P	18.92
arabinose	8.14	phenylglucose	18.93
shikimic acid	8.73	F-6-P	19.82
ribose	8.79	G-6-P	20.25
citric acid	8.89	sucrose	24.24
isocitric acid	8.92	sorb-6-P	25.96
rhamnose	9.59	cellobiose	28.38
fucose	9.78	maltose	29.92
quinic acid	10.34	isomaltose	34.81
ascorbic acid	11.87		

^aAbbreviations: G-1-P, glucose 1-phosphate; F-6-P, fructose 6phosphate; G-6-P, glucose 6-phosphate; sorb-6-P, sorbitol 6-phosphate.

1. A total GC run time of 26.5 min was sufficient to elute all components, with sucrose (peak 11) being last. If detection and quantitation of maltose, isomaltose, and larger oligosaccharides are required, then run times can simply be extended. Table I shows the retention times of standard acids, sugars, and sugar phosphates, which can be derivatized and separated with the procedure and capillary GC conditions described in Materials and Methods. Base-line separations were achieved when peak heights were only 0.1 min apart under these conditions. Only four compounds could not be completely resolved by the column: glyceraldehyde, dihydroxyacetone, phenylglucose, glucose 1-phosphate (G-1-P) (Table I). However, this is not a problem for quantitation, since glyceraldehyde, dihydroxyacetone, and G-1-P were not detected in fruit and sweet potato extracts of this study.

All the nonvolatile acids, sugar alcohols, phenylglucose, and sucrose were resolved as single peaks (1, 2, 4-6, 9-11;Figure 1). Xylose, galactose, mannose, glucose, maltose, and isomaltose yielded two isomeric peaks in a ratio of approximately 4:1, with the larger peak of each sugar used for quantitation. We have observed that this ratio remained consistent regardless of sugar and acid composition and total sample amounts from 0.5 to 15 mg under the reaction conditions described. Galactose, mannose, and glucose yielded an identical isomeric peak under these

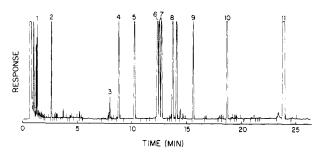


Figure 1. Capillary GLC separation of oxime-TMS derivatives of sugars and TMS derivatives of acids from Loring peach on a $15 \text{ m} \times 0.25 \text{ mm}$ (i.d.) DB-1 column. Peak identification: 1, succinic acid; 2, malic acid; 3, xylose; 4, citric acid; 5, quinic acid; 6, sorbitol; 7, fructose; 8, glucose; 9, inositol; 10, phenylglucose; 11, sucrose.

reaction conditions, which eluted immediately after peak 8. The isomeric peak did not interfere with other sugars and was not integrated; consequently it had no effect on quantitation. Fructose (peak 7) yielded two peaks, possibly α and β isomers, in a 1:1 ratio and could easily be summed for quantitation with the 3392A integrator (Figure 1).

Sugar and nonvolatile acid compositions from fruits and sweet potatoes used in this study are shown in Tables II and III. The results for apples, peaches, and pears were based on dry weight and computed with moistures determined by Lee et al. (1984). Similar values for Jewel and Tai-57 sweet potatoes were also computed on the basis of moistures determined by Kays (personal communication, 1988) (Table III).

The major nonvolatile acids common in all fruit and sweet potato samples as identified by GC retention times and GC/MS were succinic, malic, citric, and quinic. Major sugars identified were xylose, fructose, glucose, and sucrose. Low levels of maltose were detected in raw sweet potato, and traces of galactose were also detected in all samples tested. In addition, fruit samples contained two sugar alcohols, sorbitol and inositol, while the two sweet potato samples contained only the latter (Table II). The quantitative results using the techniques described showed excellent reproducibility, with standard deviations for sets of four samples given in Table II. Replicate injections of a sample yielded essentially identical values. The oxime-TMS derivatives for the sugars and the TMS derivatives of nonvolatile acids were found to be stable for 20 h.

Weight percent values for sugars on dry- and freshweight basis, in this study (Tables II and III), are in good agreement with values found in the literature for similar

Table II. S	ugar and Nonvo	latile Acid Composi	ition of Fruit	s and Sweet Potatoes ^a
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		fruits		sweet potatoes		
component	apple	pear	peach	Jewel	Tai-57	
		A	cids			
succinic	0.01 ± 0	0.01 ± 0.006	0.02 ± 0.005	0.05 ± 0.0	0.06 ± 0.02	
malic	0.43 ± 0.02	0.29 ± 0.03	0.23 ± 0.03	0.16 ± 0.02	0.26 ± 0.02	
citric	0.01 ± 0		0.36 ± 0.17	0.05 ± 0	0.02 ± 0.03	
quinic	0.03 ± 0.005	0.01 ± 0.005	0.19 ± 0.02	0.06 ± 0.01	0.06 ± 0.03	
		S	ugars			
xylose	0.02 ± 0	0.12 ± 0.02	0.02 ± 0			
sorbitol	0.35 ± 0.005	3.01 ± 0.32	0.42 ± 14			
fructose	4.98 ± 0.12	5.22 ± 0.40	1.64 ± 0.10	0.66 ± 0.20	0.17 ± 0.01	
galactose	0.05 ± 0	0.01 ± 0	0.02 ± 0	0.02 ± 0	0.02 ± 0	
glucose	2.51 ± 0.07	1.29 ± 0.19	1.47 ± 0.05	0.84 ± 0.20	0.22 ± 0.02	
inositol	0.01 ± 0	0.02 ± 0	0.02 ± 0	0.06 ± 0.005	0.06 ± 0.008	
sucrose	2.29 ± 0.07	0.27 ± 0.05	5.89 ± 0.41	4.43 ± 0.24	2.69 ± 0.46	
maltose				0.04 ± 0.005	0.04 ± 0.005	

^a Based on fresh weight of mesocarp and parenchyma tissue (g/100 g); average of four extracts; \pm SD.

Table III. Sugar and Nonvolatile Acid Composition Based on Dry Matter $(g/100 g DM)^{\alpha}$

fruits sweet potatoe					
component	apple	pear	peach	Jewel	Tai-57
		Acid	s		
succinic	0.08	0.09	0.18	0.20	0.24
malic	3.31	2.63	2.10	0.64	1.04
citric	0.08		3.31	0.20	0.08
quinic	0.23	0.09	1.69	0.24	0.24
		Suga	rs		
xylose	0.15	1.09	0.23		
sorbitol	2.69	27.39	3.78		
fructose	38.34	47.50	14.92	2.64	0.68
galactose	0.38	0.09	0.18	0.08	0.08
glucose	19.32	11.74	13.38	3.36	0.44
inositol	0.08	0.18	0.18	0.24	0.24
sucrose	17.63	2.45	53.60	17.72	10.76
maltose				0.16	0.16
total S + A	82.3	93.2	93.5	25.4	13.9
S/A^b	21.2	32.2	12.0	19.5	7.7

^a The moistures used were from Lee et al. (1984) for apples, pears, and peaches and from Kays (personal communication, 1988) for sweet potatoes. ^bSugar to acid ratio, includes sorbitol and inositol as sugars.

Table IV. Comparison of Milligrams/100 g of Nonvolatile Acids from the Present Study with Fernandez-Flores et al. (1970)

	apple		pear		peach	
acid	this study	ref	this study	ref	this study	ref
succinic	10	5	10	35	20	10
malic	430	203	290	291	230	486
citric	10	98		100	360	320
quinic	30		10	42	190	178

fruit and sweet potato varieties (Wrolstad and Shallenberger, 1981; McDonald and Newson, 1970; Van Den et al., 1986). The ranges of values of Lee et al. (1984) for glucose, fructose, and sucrose on a dry-weight basis for apples were 7.65-18.71%, 30.2-43.7%, and 13.6-23.0%, respectively. Our values for Red Delicious were 19.3%, 38.3%, and 17.6%, respectively. The dry weight percent in this study for glucose and sucrose in Danjou pears was slightly lower than the range of Lee et al. (1984); however, fructose values were within their range: viz. 11.7% (glucose) and 2.5% (sucrose) vs 23-32.9% (glucose) and 5.4-13.7% (sucrose) and 47.5% vs 33-61.5% for fructose

(Table III). Wrolstad et al. (1981) reported 23%, 65%, 18%, and 16.8% of total sugars for glucose, fructose, sorbitol, and sucrose, respectively, in pears. This compares favorably with values of 19%, 77%, 31%, and 3.9%, respectively, obtained when our data (Table II) are converted to percent total sugars.

The composition ranges (dry basis) found in four varieties of peaches for glucose, fructose, and sucrose by Lee et al. (1984) were 5.3-10.2%, 6.2-11.8%, and 36.6-59.4%, respectively, compared to values found in this study for Loring, which were 13.4%, 14.9%, and 53.6% (Table III). The percent total sugars for peaches found by Wrolstad et al. (1981) were 12.4\% glucose, 11.9% fructose, 7.2% sorbitol, and 76% sucrose. Loring peach values in this study were 16.3\% glucose, 18.2% fructose, 4.4% sorbitol, and 65% sucrose.

The compositional values based on fresh weight for glucose, fructose, sucrose, maltose, and inositol (McDonald and Newson, 1970) in sweet potato were 0.33%, 0.15%, 3.47%, 0.0%, and .01%, respectively. Values found for Jewel were 0.84% glucose, 0.66% fructose, 4.43% sucrose, 0.04% maltose, and 0.06% inositol (Table II). In one of several varieties of sweet potatoes analyzed by Van Den et al. (1986), the amounts of sugars on a dry basis were 1.69% glucose, 1.42% fructose, 16.25% sucrose, and 0.22% maltose. Our values for Jewel were 3.36% glucose, 2.64% fructose, 17.72% sucrose, and 0.16% maltose (Table III).

The major nonvolatile acids found in this study are in agreement qualitatively with Worlstad et al. (1981); however, the levels observed were somewhat different from those reported by Fernandez-Flores et al. (1970). The acids were found in Red Delicious apples based on percent total acids were 89.6% malic, 2.1% citric, and 6.2% quinic, which were similar to those reported for apple juice: 91.3% malic, 2.0% citric, and 4.0% quinic (Wrolstad et al., 1981). To better compare our nonvolatile acid results from apple, pear, and peach samples with that of Fernandez-Flores et al. (1970), data from Table II were converted to milligrams/100 g (fresh basis). The most apparent difference was the absence of citric acid in Danjou pears; however, significant levels of quinic in Red Delicious apples were found in our study (Table IV). The nonvolatile acid data obtained with our procedure (Table II) can also be summed and readily converted to milliequivalents/100 g (fresh basis). Results we found for Loring peaches were within the range of titrated values of Li and Woodroof (1981): 11.9 (as malic) vs 9.3-16.0.

Table V. N	Major Mass I	Ion and Relative	Abundance of Suga	r Oxime-TMS Derivatives
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xylose	73 (100)	103 (77)	217 (43)	72 (42)	102 (36)	147 (29)
	307 (21)	218 (13)	75 (10)	525 (0) M ⁺		
arabinose	73 (100)	103 (75)	217 (60)	147 (48)	307 (27)	75 (26)
	104 (25)	218 (18)	189 (15)	525 (0) M ⁺		. ,
sorbitolª	73 (100)	103 (59)	147 (30)	217 (29)	205 (26)	
	319 (18)	307 (17)	75 (11)	117 (9)	614 (0) M ⁺	
fructose	73 (100)	103 (65)	217 (51)	147 (30)	307 (20)	
	75 (13)	218 (10)	205 (7)	117 (7)	627 (0) M ⁺	
galactose	73 (100)	205 (45)	319 (37)	147 (31)	103 (33)	
0	217 (16)	75 (13)	320 (13)	218 (13)	627 (0) M ⁺	
glucose	73 (100)	205 (69)	319 (57)	147 (51)	103 (34)	
0	218 (25)	217(21)	320 (20)	75 (19)	627 (0) M ⁺	
inositolª	73 (100)	305 (97)	217 (97)	318 (60)	191 (46)	147 (44)
	306 (44)	319 (35)	218 (28)	204 (26)	612 (0) M ⁺	(/
phenylglucose ^a	73 (100)	361 (36)	217 (20)	147 (19)	103 (15)	
	129 (13)	362 (12)	169 (9)	544 (0) M+		
sucrose ^a	73 (100)	361 (96)	217 (37)	362 (32)	103 (20)	147 (18)
	271 (16)	363 (15)	169 (14)	129 (13)	918 (0) M+	(==)
maltose	73 (100)	361 (55)	204 (37)	147(29)	217 (24)	
	362 (19)	360 (18)	103 (14)	1005 (0) M ⁺		

^aCompounds included in table since these spectra can be useful to an investigation using our technique. These compounds do not form oximes.

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It has been observed that the levels of nonvolatile acids differ widely within the same fruit variety, most likely due to variation in fruit maturity and analytical techniques (Wrolstad et al., 1981). We found that citric was the major acid in the Loring peaches analyzed (Tables II and III), whereas other studies (Souty and André, 1975) report malic as being the major acid in most peaches. Citric acid, however, has been reported as the major acid in shipping ripe peaches, while malic acid is predominant in the ripe maturity stage (Li and Woodroof, 1968).

Summation of the sugars, sugar alcohols, and acids for each fruit and sweet potato allows determination of the percentage of these groups of components within total dry matter and calculation of sugar/acid ratios (Table III). Dry weight percentage values ranged from about 14% for Tai-57 sweet potato to 93% for pears and peaches. It is interesting to note that although pears and peaches had the same total dry matter, the sugar/acid ratio of pears was almost 3-fold higher. Such values are considered a measurement of fruit quality (Green, 1971); thus, the procedure allows another important parameter to be readily calculated. The procedure thereby eliminates the necessity for separate analytical methods.

The mass spectra of TMS derivatives of acids, polyhydroxy compounds, and sugars as their oximes from fruit and sweet potato extracts were used for identification by comparison of their spectra with authentic compounds. The mass spectra of the sugar oxime-TMS derivatives did not show molecular ions; the base peak was m/e 73. The more prominent ions for the sugars were at m/e 75, 103, 147, 217, 307, and 319. The oxime-TMS derivative of maltose and the TMS derivative of sucrose yielded additional ions at m/e 204, 360, 361, and 362 (Table V). Polysilyl derivatives of polyols and sugars are reported to possess ions at m/e 73, 75, 103, 147, 204, 217, and 320 (Pierce, 1968). It would appear that the oxime-TMS group is similar to the TMS group and is not a consistent director of characteristic fragmentation, since the same series of ions were found to be common to both TMS and oxime-TMS sugars in this study.

The advantages of the procedures herein reported for the analysis of sugars and nonvolatile acids from fruits and sweet potatoes include simplicity, reproducibility of reactions, stability of the derivatives, and applicability for both qualitative and quantitative analysis.

Registry No. xylose, 58-86-6; sorbitol, 50-70-4; maltose, 69-79-4; succinic acid, 110-15-6; malic acid, 6915-15-7; citric acid, 77-92-9; quinic acid, 36413-60-2; inositol, 87-89-8; sucrose, 57-50-1; fructose, 57-48-7; galactose, 59-23-4; glucose, 50-99-7; glyceraldehyde, 367-47-5; dihydroxyacetone, 96-26-4; erythrose, 583-50-6; deoxyribose, 533-67-5; cellobiose, 528-50-7; arabinose, 147-81-9; ribose, 50-69-1; rhamnose, 3615-41-6; fucose, 2438-80-4; isomaltose, 499-40-1; mannose, 3458-28-4; glucose 1-phosphate, 59-56-3; fructose 6-phosphate, 643-13-0; glucose 6-phosphate, 56-73-5; sorbitol 6-phosphate, 20479-58-7; pyruvic acid, 127-17-3; tartaric acid, 87-69-4; xylitol, 87-99-0; shikimic acid, 138-59-0; isocitric acid, 320-77-4; ascorbic acid, 50-81-7; mannitol, 69-65-8; galacturonic acid, 685-73-4; caffeic acid, 331-39-5; xylose oxime-TMS derivative, 103516-44-5; arabinose oxime-TMS derivative, 103516-45-6; sorbitol-TMS derivative, 14199-80-5; fructose oxime-TMS derivative, 120788-24-1; galactose oxime-TMS derivative, 120850-88-6; glucose oxime-TMS derivative, 120850-89-7; inositol-TMS derivative, 2582-79-8; phenylglucose-TMS derivative, 120788-25-2;

sucrose-TMS derivative, 19159-25-2; maltose oxime-TMS derivative, 120788-26-3.

LITERATURE CITED

- Fernandez-Flores, E.; Kline, D. A.; Johnson, A. R. GLC determination of organic acids in fruits as their trimethylsilyl derivatives. J. Assoc. Off. Anal. Chem. 1970, 53, 17-20.
- Green, A. Soft Fruits. In The Biochemistry of Fruits and Their Products; Hulme, A. C., Ed.; Academic Press: London, New York, 1971; Vol. 2.
- Guerrant, G. C.; Lambert, M. A.; Moss, C. W. Analysis of short chain acids from anaerobic bacteria by high-performance liquid chromatography. J. Clin. Microb. 1982, 16, 355–360.
- Hellener, S. R., Milne, G. W. A., Eds. EPA/NIH Mass Spectral data base; U.S. Department of Commerce, National Bureau of Standards: Washington, DC, 1978; Vol. 1-4.
- Horrii, Z.; Makita, M.; Tamura, Y. Gas-liquid chromatographic separation of acids of krebs cycle as trimethylsilyl derivatives. *Chem. Ind.* **1965**, *34*, 1494.
- Kays, S., personal communication, 1988.
- Lee, E.; Koo, J.; Lee, J.; Ha, J. Determination of free sugars in some fruits by liquid chromatography. J. Korean Agric. Chem. Soc. 1984, 27, 158-162.
- Li, K. C.; Woodroof, J. G. Gas chromatographic resolution of nonvolatile organic acids in peaches. J. Agric. Food Chem. 1968, 16, 534-535.
- Marsili, R. T.; Ostapenko, H.; Simmons, R. E.; Green, D. E. High performance liquid chromatographic determination of organic acids in dairy products. J. Food Sci. 1981, 46, 52-57.
- Mason, B. S.; Slover, H. T. A gas chromatographic method for the determination of sugars in foods. J. Agric. Food Chem. 1971, 19, 551-554.
- McDonald, R. E.; Newson, D. W. Extraction and gas-liquid chromatography of sweet potato sugars and inositol. J. Am. Soc. Hortic. Sci. 1970, 95, 299-301.
- Pierce, A. E. Silylation of Organic Compounds; Pierce Chemical: Rockford, IL, 1968.
- Robards, K.; Whitelaw, M. Chromatography of monosaccharides and disaccharides. J. Chromatogr. 1986, 373, 81-110.
- Souty, M.; André, P. Composition biochimique et qualite' des peaches. Ann. Technol. Agric. 1975, 24, 217-236.
- Sweeley, C. C.; Bentley, R.; Makita, M.; Wells, W. W. Gas-liquid chromatography of trimethylsilyl derivatives of sugars and related substances. J. Am. Chem. Soc. 1963, 85, 2497-2507.
- Tanowitz, B. D.; Smith, D. M. A rapid method for qualitative and quantitative analysis of simple carbohydrates in nectars. Ann. Bot. 1984, 53, 453-456.
- Traitler, H.; Del Vedovo, S.; Schweizer, T. F. Gas Chromatographic separation of sugars by on-column injection on glass capillary columns. HRC CC, J. High Resolut. Chromatogr. Chromatogr. Commun. 1984, 7, 558-562.
- Van Den, T.; Beirmann, C. J.; Marlett, J. A. Simple sugars, oligosaccharides, and starch concentrations in raw and cooked sweet potato. J. Agric. Good Chem. 1986, 34, 421-425.
- Wrolstad, R. E.; Shallenberger, R. S. Free sugars and sorbitol in fruits—A compilation from the literature. J. Assoc. Off. Anal. Chem. 1981, 64, 91–103.
- Wrolstad, R. E.; Cornwell, C. J.; Culbertson, J. D.; Reyes, G. R. Establishing criteria for determining the authenticity of fruit juice concentrates. In Quality of selected Fruits and Vegetables of North America; Teranishi, R., Barrera-Benitez, H., Eds.; American Chemical Society: Washington, DC, 1981.

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