significantly differ from values for hardwood chips. Data in Table I show the efficiency of the extraction procedure to be concentration independent and to have excellent precision. Quantitation of PCP in unknown samples was conducted by comparison to a standard that was near the sample value and the recovery factor applied to obtain sample concentration. This analytical procedure has been successfully used to analyze 86 samples of hardwood chips between 1977 and 1981. Observed concentrations of PCP residues in the hardwood chips ranged from less than MDL to 240 ppb with an average level of 44 ppb. All of the hardwood chip samples analyzed met the 2-ppm experimental specifications for PCP.

The procedure was also used to analyze for PCP in cardboard, a gray-mist chipboard, used to make animal feeder boxes for rodent feeders (Fullerton et al., 1981). Levels in 11 cardboard samples ranged from 0.13 to 4.4 ppm with an average level of 1.8 ppm. Three of the cardboard samples analyzed were rejected because they exceeded the 2-ppm limitation established at our laboratory.

The method presented provides sensitivity in the analysis of wood products not previously available. In addition, the method has good precision as evidence by the reproducibility of the results reported in Table I, which were obtained from replicate analyses.

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Analysis of Neutral Sugar Hydrolysates of Forage Cell Walls by High-Pressure Liquid Chromatography

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The separation and analysis of monosaccharides were accomplished by high-pressure liquid chromatography using an amine bonded phase column and acetonitrile–water solvent system. Excellent separation was achieved for rhamnose, xylose, arabinose, and glucose, while adequate separation of glucose and galactose was obtained. Monosaccharide samples were obtained by hydrolyzing the cell walls of forage grasses with 2 N trifluoroacetic acid. The hydrolysates were prepared for HPLC analysis with a Waters Associates C_{18} Sep-PAK. The precision of the analysis (10 injections of the same sample preparation) as measured by standard deviation was ±0.38, ±0.24, and ±0.35% for xylose, arabinose, and glucose, respectively. Analysis of variance indicated no difference in recovery of sugars from triplicate preparations of the same sample. Analysis of variance did show significant differences in the composition of three different samples of the same species. Thus, it is possible to accurately analyze for differences in the composition of the component sugars hydrolyzed from forage cell walls and know that the method is sensitive enough to reflect differences in composition that could be related to nutritional qualities of the feed.

One of the differences between temperate and tropical grasses is the amount of readily hydrolyzable polysaccharides in the plant cell wall. Readily hydrolyzable polysaccharides are defined as that portion of the forage cell wall hydrolyzed by dilute acid. Barton et al. (1976) showed that tropical grasses contain 30–35% more "hemicellulose" than temperate grasses (i.e., tropical 28–35%, temperate 22–26%). Further, they showed that this difference in the amount of hemicellulose could account for some of the difference in digestibility of temperate and tropical grasses.

Albersheim et al. (1967) used a gas–liquid chromatographic method to analyze the cell walls of "pinto bean" hypocotyls. This method is an improvement over earlier methods in that 2.0 N trifluoroacetic acid (TFA), which is easily removed by evaporation, replaced 1.0 N sulfuric acid as the hydrolytic agent; however, several steps are still required. In addition, Collings and Yokoyama (1979) used a modification of the alditol acetate technique of Albersheim et al. (1967) to determine cellulose and hemicellulosic sugars of delignified forages and feeds by 1-h hy-

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drolysis with 1 N TFA. The results presented by these authors were extensive, but the complicated presentation of their calculations was extremely difficult to follow.

Linden and Lawhead (1975) described a liquid chromatography method that does not require derivatization of the neutral sugar. Palmer and Brandes (1974) and Palmer (1975a,b) used high-pressure liquid chromatography (HPLC) to analyze simple sugar preparations and enzymatically hydrolyzed cellulose. Gum and Brown (1977) also developed HPLC methods for the analysis of reduced celloligosaccharides from monomer through hexamer. These HPLC procedures, which were faster and required fewer steps than GLC procedures, efficiently separated monomers from oligosaccharides or the monomers and dimers normally found in beverages of high-sugar foods (fructose, glucose, and sucrose). The objective of this study was to develop an HPLC method for the analysis of neutral sugars from cell wall (i.e., Coastal Bermuda grass) polysaccharide hydrolysates.

MATERIALS AND METHODS

Liquid Chromatography: Equipment and Procedures. The liquid chromatograph used for this study was a Waters Associates ALC 100, which was equipped with a U6K injector, a Model 600 solvent delivery system, and a differential refractometer detector, having a sensitivity of 1×10^{-7} refractive index units and an attenuation of 64 $\times$ $^1/_4$. The liquid chromatograph was connected to an Infotronics CRS-24 automatic digital integrator and an Omniscribe 10-mV full-scale, variable chart speed (0.2 in./min) recorder. Sugars were separated by using a 30 cm $\times$ 5 mm i.d. stainless steel column packed with Micromeritics Microsil NH_2 and a Waters Associates guard column packed with LiChrosorb NH_2. Operating conditions were as follows: column temperature, ambient; refractometer detector temperature, 4 °C below ambient; eluent, water–acetonitrile (25:75 v/v) with a flow rate of 2.0 mL/min at pressures from 400 to 1000 psi. The size of the usual sample injected was 10 μL.

Samples: Sugar Standards. Sugar standards were of high purity from Aldrich Chemical Co. Working standards in the range of 10–20 mg/mL (1–2%) were prepared by dilution in a methanol–water (60:40 v/v) solution. The concentration, 10 mg/mL (1%), yielded the optimum resolution for the sugar standards and was used to determine the concentration of the sugars in the hydrolysates.

Forage Samples. Three samples of Coastal Bermuda grass (CBG) [*Cynodon dactylon* (L.) Pers.] were used in this study. Two 4-week regrowth and a spring growth were collected from Tifton, GA, on Sept 23, 1974, Aug 24, 1976, and May 24, 1978, respectively, fertilized at the rate of 56.25 kg of N, 18.75 kg of P_2O_5, and 37.5 kg of K_2O per ha. Samples were handled and prepared as described by Barton et al. (1976).

Chemical Analysis. Neutral detergent fiber (NDF) was isolated from each of the forage samples as described by Goering and Van Soest (1970) with modifications by Barton et al. (1976). Crude protein was determined as 6.25 times Kjeldahl nitrogen.

Sugar analysis was conducted by weighing 200 mg of the isolated NDF into reaction flasks and hydrolyzed in 20 mL of 2 N trifluoroacetic acid (TFA) at 121 °C for 15, 30, 45, 60, 120, 180, and 240 min. On the basis of time studies, a 1-h (60-min) hydrolysis time was chosen for all analytical samples. After hydrolysis, the hydrolysate was filtered through a 15-mL coarse Büchner funnel and washed with 100 mL of boiling distilled water. The soluble portion of the hydrolysate was evaporated to dryness at 40 °C under reduced pressure (30 mmHg). The hydrolysate residue was dried at 100 °C for 12 h to determine the amount of cell wall material hydrolyzed.

To preclude damage to the liquid chromatograph column from residual fluoronated hydrocarbons, we made three additions of distilled water (150 mL) to each sample and evaporated them to dryness. Samples were then diluted in 30 mL of distilled water which resulted in a soluble water fraction and a light brown precipitate. The total hydrolysate was eluted through a 0.45-μm Millipore filter. The water-soluble fraction was evaporated to approximately 4 mL for rapid sample cleanup with a Waters C_{18} Sep-PAK cartiridge to ensure that all noncarbohydrate material was separated from the sample. This step greatly increases the life of the HPLC column. The cartridge was prepared by charging with 2 mL of acetonitrile and a 4-mL water wash. The water-soluble fraction (4 mL) was eluted through the Sep-PAK, followed by a 2-mL water wash of the cartridge and evaporated to dryness. The residue was dissolved in 1 mL of methanol–water (60:40 v/v) for component sugar analysis. Nuclear magnetic resonance spectra were taken as described by Himmelsbach and Barton (1980).

Statistical Analysis. Data were analyzed by analysis of variance for primary effect and orthogonal polynomials for unequally spaced treatments (i.e., time of hydrolysis) according to Steel and Torrie (1960). Differences between means were determined by using Scheffe's multiple comparison procedure as described by Kleinbaum and Kupper (1978).

RESULTS AND DISCUSSION

Sugar analysis are among the larger applications for the HPLC techniques (Palmer, 1975a). However, the separation of monosaccharides that are commonly found in plant material or in cell wall polysaccharides is quite difficult. Palmer's (1975b) work with a Waters μBondapak column shows the difficulty in separating the C-6 monosaccharides. The resolution of glucose from mannose and galactose was about 0.75, marginally adequate for quantitative purposes (Palmer, 1975b). Palmer (1975b) reported that the resolution can be increased to an adequate 0.87 by coupling two μBondapak columns in series. The Micromeretics Microsil amine columns, while only resolving galactose from glucose, have proven to be the most reliable and reproducible with excellent column life. The separation of the 1% standard on the Micromeretics column is shown in Figure 1. The analysis shows excellent separation of the C-5 sugars and good separation of glucose from galactose, all of which are adequate for quantitative purposes. The separation of Figure 1 is comparable to that obtained by Palmer (1975b) using HPLC and Albersheim et al. (1967) via gas chromatography of these sugars as alditol acetate derivatives. The analysis of the 1-h hydrolysis of NDF from Coastal Bermuda grass is shown in Figure 2. The chromatogram indicates the presence of xylose, arabinose, glucose, and trace amounts of galactose that appear as a shoulder on the trailing edge of glucose.

The precision of this method of analysis was determined from repeated injections (10) into the HPLC of the same sample preparation as shown in Table I. The instrument is capable of precisely reproducing sample data as shown by the standard deviation and coefficient of variation. The resolution of galactose in the amounts present is not adequate for quantitative purposes and as such is reported as trace amounts. The data from three different preparations of CBG NDF to determine the overall precision of the method are shown in Table II. These samples are individually run from the forage and entail three different

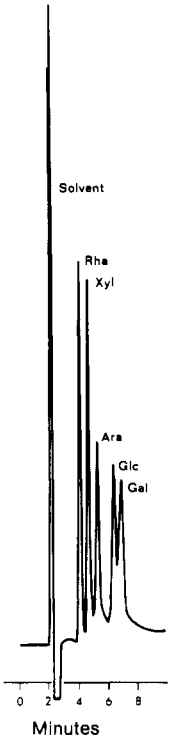

Figure 1. A high-pressure liquid chromatogram of the 1% sugar standard. Rha = rhamnose, Xyl = xylose, Ara = arabinose, Glc = glucose, and Gal = galactose.

Table I. Precision of HPLC Method

	mg of sugar components after repeated injections[a]			
injection no.	xylose	arabinose	glucose	galactose
1	37.13	11.57	13.88	tr[b]
2	37.28	11.74	13.73	tr
3	37.82	11.53	14.45	tr
4	38.14	11.76	14.16	tr
5	37.98	11.46	14.09	tr
6	37.19	11.08	13.91	tr
7	37.35	11.76	13.75	tr
8	37.89	11.55	14.47	tr
9	37.13	11.32	13.51	tr
10	37.64	11.17	13.43	tr
mean	37.55	11.49	13.94	
SD	0.38	0.24	0.35	
coeff of variation	1.02	2.09	2.55	

[a] Milligrams recovered per 100 mg of CBG NDF hydrolyzed. [b] tr = trace; the amount present was inadequate for quantitative purposes.

Table II. Accuracy of Sample Preparation for Coastal Bermuda Grass Neutral Detergent Fiber

	mg[a]			
sample	xylose	arabinose	glucose	galactose
CBGa[d]	37.67 ± 0.44[b]	11.58 ± 0.37	14.06 ± 0.26	tr[c]
CBGb	37.47 ± 0.36	11.46 ± 0.34	14.04 ± 0.36	tr
CBGc	37.35 ± 0.26	11.64 ± 0.39	13.92 ± 0.34	tr

[a] Milligrams recovered per 100 mg of NDF hydrolyzed. [b] Each value is the mean of five observations (i.e., five injections of three sample preparations). [c] tr = trace; the amount present was inadequate for quantitative purposes. [d] CBG = Coastal Bermuda grass.

NDF preparations with separate hydrolysis, sample workup, and HPLC analysis. Analysis of variance showed no difference ($P < 0.05$) in the milligrams recovered of xylose, arabinose, and glucose due to sample preparation. On the basis of results in Tables I and II, the method is suitable for quantitative analysis of component sugars from forage cell wall hydrolysates. The recovery of component sugars in Table III is for three different CBG samples whose compositional analysis data are given in Table IV. Analysis of variance indicated a year of harvest ($P < 0.05$)

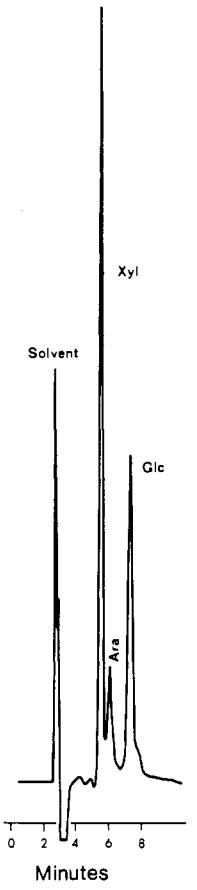

Figure 2. A high-pressure liquid chromatogram of the hydrolysate from NDF of Coastal Bermuda grass. Xyl = xylose, Ara = arabinose, and Glc = glucose.

Table III. Monosaccharide Constituents of Neutral Detergent Fiber for Three Different Coastal Bermuda Grasses

	mg[a]		
sugar	CBGa[g]	CBGb	CBGc
xylose	38.13[b,c] ± 0.45	33.84[d] ± 0.56	36.78[e] ± 0.44
arabinose	11.72[c] ± 0.38	10.86[d] ± 0.40	12.63[e] ± 0.14
glucose	14.24[c] ± 0.27	10.11[d] ± 0.23	13.02[e] ± 0.20
galactose	tr[f]	tr	tr

[a] Milligrams recovered per 100 mg of NDF hydrolyzed. [b] Each value is the mean of six observations (i.e., three injections of duplicate sample workup). [c-e] Dissimilar characters within rows indicate differences between means ($P < 0.05$). [f] tr = trace. [g] CBG = Coastal Bermuda grass.

Table IV. Compositional Analysis of Three Coastal Bermuda Grass Samples

	%				
sample	NDF[a]	ADF[b]	hemicellulose[c]	PML[d]	CP[e]
CBGa[f]	72.66	34.90	37.76	4.34	8.54
CBGb	59.22	28.60	30.62	4.47	10.84
CBGc	63.25	34.59	28.66	3.90	10.34

[a] Neutral detergent fiber. [b] Acid detergent fiber. [c] Hemicellulose = NDF − ADF. [d] Permanganate lignin. [e] Crude protein. [f] CBG = Coastal Bermuda grass.

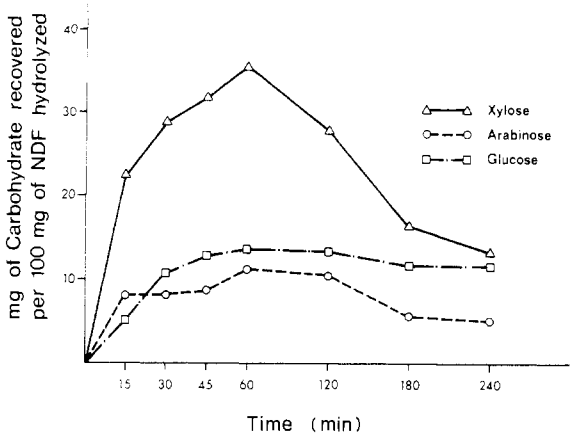

Figure 3. A plot of milligrams of carbohydrate recovered by HPLC per 100 mg of hydrolyzable cell wall with increasing time of hydrolysis.

effect for the component sugars detected. This indicates that the sugar content of a particular grass (CBG) differs due to the year it was harvested, which is possibly due to the environmental conditions during that growing season. On the basis of these results, the method appears to be accurate and sufficiently sensitive to distinguish subtle differences in the component sugars of forage cell walls.

Figure 3 shows the amount of sugars obtained from NDF of CBG on hydrolysis with 2 N trifluoroacetic acid in a sealed tube at 121 °C for various periods of time. There was a difference ($P < 0.05$) in the milligrams of sugars recovered due to the time of hydrolysis. With xylose and arabinose, there was a curvilinear effect ($P < 0.001$) with time of hydrolysis. Linearity appears to be associated with the 15–60-min hydrolysis; after 1 hour (optimal for most of the sugars) the recovery decreases, probably due to destruction of the sugar monomers. With glucose, there was a linear increase ($P < 0.05$) in recovery with increasing time of hydrolysis. These data are in agreement with Albersheim et al. (1967), who found that 1 h was optimal for cell walls of pinto bean hypocotyls and after 1 h the recovery of each sugar began to decrease.

Control experiments were run in which the five monomer sugars were taken completely through the 1-h hydrolysis procedure, the sample workup with a Sep-PAK, and analysis by HPLC. The percentage of recovery of sugars was 97.5. The actual amount recovered in this study from the hydrolysis of CBG cell walls (i.e., milligrams recovered by HPLC per 100 mg hydrolyzed from the cell wall) ranged from 56 to 60%. When the polysaccharides xylan, araban, and galactan were hydrolyzed and analyzed, the percent recovery through the HPLC was 96.8. This means that in NDF there is a fraction that is hydrolyzable or soluble in 2 N trifluoroacetic acid but is not a polysaccharide. This fraction precipitates on the wall of the flask during evaporation. This fraction is yellow to light brown and is soluble in acetone. The amount of this fraction was 40–48% of the hydrolysate and accounts for all the remaining dry matter that was hydrolyzed but not recovered through the HPLC. Preliminary results of the analysis of the precipitated water fraction remaining after removal of trifluoroacetic acid by evaporation by ^{13}C nuclear magnetic resonance (NMR) spectroscopy indicates the likely presence of a lignin–carbohydrate complex (LCC). Based on this observation, it is possible that all of the material that is solubilized or hydrolyzed is not "hemicellulose". Some of the "hydrolyzed or solubilized" material may actually contain an unhydrolyzed but soluble lignin–carbohydrate complex. Figure 4 shows the ^{13}C NMR spectrum of the precipitate from the hydrolysis of a CBG sample. The samples contain *p*-coumaric- and ferulic-type lignin groups and xylose and arabinose in both the α and β forms. The resonances for xylose are the same as the first five carbons for glucose; glucose is present as shown by the resonances at 63 ppm for the C-6 carbon of a glucopyranose. No interpretation as to how the lignins and carbohydrates are bound has been made at this time. These carbohydrates are bound to the "lignin" moieties and, while solubilized by 2 N TFA, are not hydrolyzed. Of the crude protein (CP) that is prsent in NDF, most is solubilized by the TFA. The spectrum in Figure 4 contains carbonyls at 170 ppm and aliphatics at 29–33 ppm These peaks could be partially due to protein. They are, however, a very minor portion of the sample. Further studies are under way to determine the fractions containing the nitrogenous protein of NDF. The data on the sugar content reported in this study are based on the portion hydrolyzed and recovered from the HPLC being the hemicellulose since no finitive data as to its utilization by ruminants are currently available for the LCC.

The HPLC method reported in this study is well suited to the study of carbohydrates comprising the plant cell wall of forages. The speed of analysis is due in part to the resolving power of the Micromeritics Microsil amine columns at high flow rates. These high flow rates in turn allow analysis to be completed in less than 8 min. This method should enable us to readily relate the composition

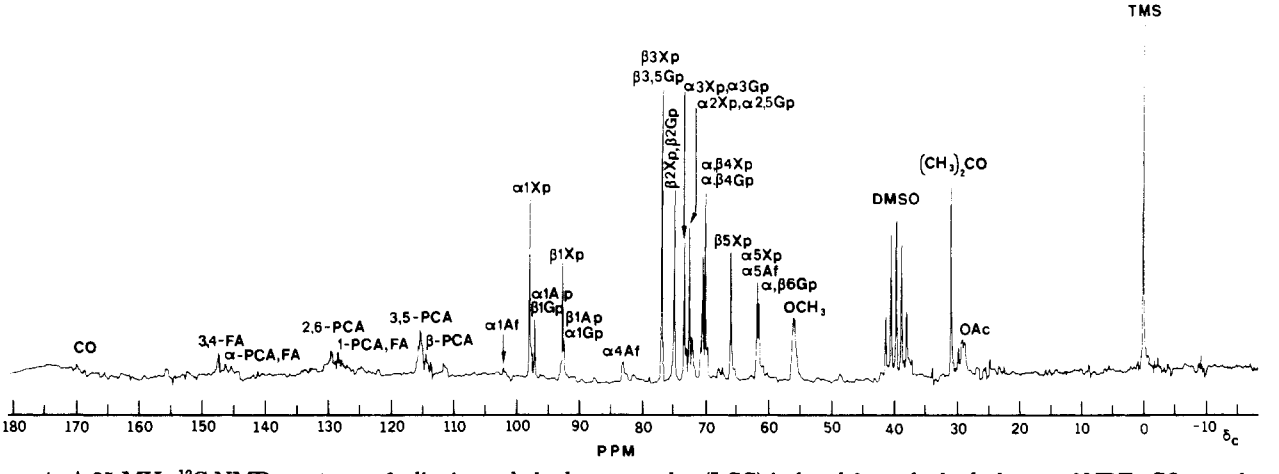

Figure 4. A 25-MHz ^{13}C NMR spectrum of a lignin–carbohydrate complex (LCC) isolated from the hydrolysate of NDF. CO = carbonyl, FA = ferrulic acid, PCA = *p*-courmaric acid, X = xylose, A = arbinose, G = glucose, f = furanoside, p = pyranoside, DMSO = dimethyl sulfoxide, OAc = acetate, TMS = tetramethylsilane, and $(CH_3)_2CO$ = acetone.

of the easily hydrolyzed portion of the cell wall fiber to its rate and percentage digestibility in ruminant animals.

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Dietary Fiber Content of Some Tropical Fruits and Vegetables

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The dietary fiber composition of some common tropical fruits and vegetables was studied. Samples analyzed were pineapple, carambola, sapodilla, papaya, mango, grapefruit, sweet potato, and yam. Values for cellulose, hemicellulose, lignin, cutin, ash, neutral detergent residue (NDR), and enzymatic (insoluble and soluble) fractions were obtained (percent of fresh weight). NDR for most samples was 0.9–1.2%. Cellulose was 0.1–0.9%, except sapodilla (2.4%) and sweet potato peel (1.4 and 1.9%). Lignin was around 0.025–0.17%; carambola (0.3%), sapodilla (2.3%), and sweet potato peel (0.4%) were exceptions. Hemicellulose was about 0.04–0.4%; exceptions were pineapple (0.5%), sapodilla (0.6%), and tropical (Puerto Rican) sweet potato peel (0.9%). In some samples nutritionally valuable fiber components were relatively concentrated, and they may be potential sources of concentrated fiber fractions with useful physiological properties. Sapodilla, in particular, seems to be an abundant source of lignin and cellulose.

The composition of fiber fractions in forages and cereals has been thoroughly investigated, but fiber in fruits and vegetables has not been as well studied. Previous investigations (Kamath and Belvady, 1980; Shipley, 1978) showed some differences between cereal products, fruits, and vegetables. Grain products have relatively high hemicellulose concentrations, vegetables have been found somewhat low in lignin, and the composition of fruits is usually somewhere between. Although some tropical fruits and vegetables have been analyzed, fiber in most of the common examples has not been reported. Tropical fruits and vegetables may contain unusual fiber components, some of which may have unique physiological properties, such as specific lipid binding capacities.

Previous studies have shown that in a few tropical fruits and vegetables fiber components, in general, did not differ significantly from those of other fruits and vegetables. Among the more common tropical species, these authors (Kamath and Belvady, 1980; Shipley, 1978) reported values for sweet potato, yellow pumpkin (*Curcurbita maxima*), plantain, banana, mango, guava, grapefruit, and mandarin oranges. A report on olive fiber has also been published (Moreno and Diez, 1979). The Van Soest detergent procedure or the Southgate procedure was used in the studies cited above. The Van Soest procedure yields values for cellulose, lignin, hemicellulose, and neutral detergent residue (NDR). The Southgate method gives cellulose and lignin values, but the hemicellulose and pectin are usually reported together as "noncellulosic polysaccharides". Most samples had 0.2–6% cellulose, 0.04–2.4% hemicellulose (or noncellulosic polysaccharides), and 0–0.8% lignin. A few samples deviated significantly from the usual composition range. Thus, plantain and guava contained relatively large lignin fractions (1.23 and 0.80%, respectively), and sweet potato had a large amount of noncellulosic polysaccharides (5.24%).

In this report, the dietary fiber content of 15 samples from 8 different fruits and vegetables was determined. Modified forms of the Van Soest detergent and Hellendoorn enzymatic methods were used (see Experimental Section). Differences between varieties, the effects of processing, and variations between different parts of the plant were studied for some samples.

EXPERIMENTAL SECTION

Materials. Pineapple and papaya samples and one sample of sweet potato were purchased at local markets. The yams (five types, listed in Table I) and the tropical sweet potato were obtained from the Mayaguez Institute of Tropical Agriculture, Mayaguez, PR. Carambolas, sapodillas, and mangos were obtained from the USDA Subtropical Horticultural Research Station at Miami, FL. The grapefruit were obtained from USDA Horticultural Field Station, Orlando, FL.

General Procedure. Unless otherwise noted, the edible, fleshy portion of the fruit or vegetable was analyzed. Since the peels of sweet potato are also edible, they were analyzed separately from the flesh. The peel of carambola is normally consumed with the fruit flesh, thus the ana-

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