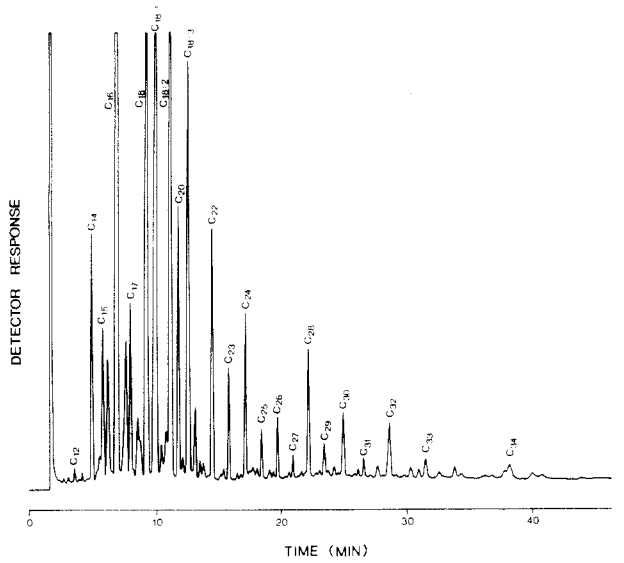

Figure 8. Separation of FAME from cigarette smoke on a FS Silar 10C WCOT column. Conditions: 150–240 °C at 4 °C/min; 30 cm/s H_2 flow; split injection mode; FID; 25 m × 0.25 mm i.d.

oratory.

Registry No. C_{14}, 544-63-8; C_{16}, 57-10-3; C_{18}, 57-11-4; $C_{18:1}$, 27104-13-8; $C_{18:2}$, 60-33-3; $C_{18:3}$, 463-40-1; C_{20}, 506-30-9; C_{22}, 112-85-6; C_{12}, 143-07-7; C_{17}, 506-12-7; C_{23}, 2433-96-7; C_{24}, 557-59-5; C_{15}, 1002-84-2; C_{25}, 506-38-7; C_{26}, 506-46-7; C_{27}, 7138-40-1; C_{28}, 506-48-9; C_{29}, 4250-38-8; C_{30}, 506-50-3; C_{31}, 38232-01-8; C_{32}, 3625-52-3; C_{33}, 38232-03-0; C_{34}, 38232-04-1; Superox-4, 25322-68-3; vitreous silica, 60676-86-0.

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Elimination of Sample Autoxidation by Butylated Hydroxytoluene Additions before Thiobarbituric Acid Assay for Malonaldehyde in Fat from Chicken Meat

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This study tested the influence of the antioxidant butylated hydroxytoluene (BHT) on the thiobarbituric acid (TBA) assay for malonaldehyde in fat extracted from chicken breast and leg meat. It was found that 125 μg of BHT/mg of fat was required to prevent autoxidation during the heating step of the TBA assay. In addition, autoxidation of sample lipid during extraction can be prevented by the addition of 0.01% BHT to chloroform–methanol (1:2) reagent. For samples extracted with BHT, the addition of 75 μg of BHT/mg of fat before heating was sufficient to prevent sample autoxidation. Meat samples analyzed without any BHT additions yielded 6 times higher malonaldehyde concentrations compared to samples that received BHT during extraction and 75 μg of BHT/mg of fat during the TBA assay. It is concluded that antioxidant protection is necessary during TBA assays to prevent sample autoxidation and consequent artifactually high analytical results.

There are many variations of the thiobarbituric acid (TBA) assay for malonaldehyde (MA). However, all variations are similar in that they each have a critical heating step (usually 100 °C for 30–60 min) that is necessary for releasing MA from lipid hydroperoxides. This heating in the presence of air may promote sample autoxidation and lead to artifactually high assay values.

For the analysis of MA in chicken and other meats, the tissue distillation–TBA assay method of Tarladgis et al. (1960) is most commonly used. This original method did not include antioxidants during the distillation step, and neither do many meat distillation assays in current use. However, some investigators have used antioxidants such as propyl gallate, EDTA, and BHA during the distillation step of the TBA assay (Rhee, 1978; Rhee and Ziprin, 1981; Moerck and Ball, 1974). Yamauchi et al. (1982) used 0.3% addition of BHT to meat samples before homogenization and distillation for TBA assays. This recent use of antioxidants in tissue distillation indicates that sample aut-

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oxidation is a serious concern.

Several variations of tissue extraction–TBA assays for meat have been reported. These variations are similar in that the sample extract must yield a clear liquid for spectrophotometric analysis of the TBA assay, in addition to containing quantitative amounts of the MA–TBA chromophore. Most of these assays involve aqueous acid extraction of meat homogenate (Sinnhuber and Yu, 1977; Shamberger et al., 1977; Newburg and Concon, 1980; Webb et al., 1974; Ohkawa et al., 1979; Witte et al., 1970). One major benefit of the extraction–TBA assays is the elimination of the cumbersome and difficult tissue distillation step. It has been suggested that TBA numbers were lower in extraction–TBA assays compared to those in a standard distillation–TBA assay because of reduced sample autoxidation (Witte et al., 1970; Vyneke, 1975; Siu and Draper, 1978).

Outside the meat industry, the TBA assay is used in clinical laboratories for testing blood (Gutteridge and Tickner, 1978; Lee, 1980; Shimizu et al., 1981) and blood platelet (Takayama et al., 1980; Satoh et al., 1981) lipid hydroperoxide activity; but none of these assays include antioxidants before the critical heating step. In basic research involving pure fatty acids, antioxidants such as BHT are sometimes used in conjunction with TBA assays of lipid hydroperoxides (Asakawa and Matsushita, 1980), but more often they are not. Although the specific topic of our report is TBA analysis of chicken meat, the results of our experiments concerning BHT protection of sample autoxidation are relevant and important to all other variations of the TBA assay.

MATERIALS AND METHODS

Reagents. 1,1,3,3-Tetramethoxypropane (TMP) was obtained from the Eastman Kodak Co., Rochester, NY, 2-thiobarbituric acid (TBA) and butylated hydroxytoluene (BHT) were from Sigma Chemical Co., St. Louis, MO, and electrophoresis-grade sodium dodecyl sulfate (SDS) was from Bio-Rad Laboratories, Richmond, CA.

Materials. Whole leg and breast muscles were collected from 4-month-old New Hampshire × Columbian (NH × C) pullets immediately after slaughter. Surface adipose tissue, but not intramuscular fat, was first removed, and then muscle tissue was deboned manually, wrapped in aluminum foil, and kept frozen at −18 °C for 3 months. While still frozen, meat samples were finely minced and weighed, transferred to mortar and pestel, and extracted immediately.

Procedures. The basic procedure of Kates (1972) for tissue lipid extraction was used with several modifications. Typically 5-g samples of meat plus 3 mL of distilled water plus 30 mL of chloroform–methanol (1:2) were ground and blended with a mortar and pestle and then homogenized for 30 s with a Polytron PCU-2 (Brinkman Instruments, Westbury, NY) homogenizer; the homogenate was centrifuged at 1000*g* for 5 min at 10 °C. The supernatant was decanted and saved, and the residue was resuspended in 35 mL of methanol–chloroform–water (2:1:0.8), reextracted with mixing and 30-s homogenization and recentrifuged, with combined supernatants added to separatory funnels together with 20 mL of chloroform and 20 mL of distilled water, mixed thoroughly, and allowed to stand overnight at 4 °C. After standing, the organic phase was collected in volumetric cylinders, then approximately 0.3 g of anhydrous sodium sulfate was added, and the solution was mixed by inversion, filtered, dried by nitrogen evaporation at temperatures not higher than 35 °C, weighed, dissolved by chloroform to give a fat concentration of 10 mg/mL, and then assayed for TBA reactivity.

Table I. Absorbance Values at 532 nm for TBA Assay Reaction with TMP in the Presence of BHT

TMP concn, μg/mL	BHT in TBA reaction mixture, μg/mL				
	0	2.5	25	125	250
0.0275	0.058[a]	0.061	0.059	0.061	0.058
0.055	0.124	0.120	0.126	0.122	0.117
0.110	0.230	0.231	0.237	0.221	0.208
0.220	0.470	0.465	0.473	0.467	0.463
0.330	0.680	0.680	0.679	0.676	0.662
0.440	0.925	0.930	0.927	0.931	0.915
0.550	1.150	1.148	1.163	1.156	1.128

[a] Absorbance data are means of triplicate determinations.

Malonaldehyde (MA) concentration of lipid extracts was determined by TBA assay according to conditions described by Ohkawa et al. (1978, 1979). Their assay did not include BHT before the boiling step and was therefore modified for assays that included BHT. For the TBA assay, 0.8 mL (without BHT method) or 0.7 mL (with BHT method) of distilled water was added to tubes containing 1–8 mg of tissue fat, followed by addition of 0.2 mL of 8.1% SDS, thorough vortexing, and addition of 1.5 mL of 20% acetic acid adjusted to pH 4.0 with sodium hydroxide. Next, all tubes received 1.5 mL of 0.8% aqueous TBA solution, and an additional 0.1 mL of ethyl alcohol, containing 0.001–1.0% BHT, was added for TBA assays with BHT, where 0.1 mL of 0.5% ethanolic BHT solution resulted in 125 μg/mg of fat in tubes containing 4 mg of dried fat. Sample tubes were heated in boiling water for 60 min, cooled in tapwater, and centrifuged at 4000*g* for 15 min at 10 °C. Afterward, the absorbance of the clear supernatant was measured at 532 nm in a Beckman Model 25 spectrophotometer with glass cuvettes of 1-cm light path. TMP was used as the external standard and sample lipid oxidation was expressed as micrograms of malonaldehyde per milliliter of reaction mixture. For some experiments, a sample of dried fat was allowed to autoxidize for 1 week at room temperature and 24 additional h at 56 °C while exposed to air. Peroxide values were determined by the official AOCS method (Link, 1973) and expressed as nanomoles of peroxide per milligram of fat.

RESULTS AND DISCUSSION

BHT Effects on MA Release and MA–TBA Binding. There are many variations of the TBA assay for malonaldehyde in biological samples, but all variations contain a critical heating step that is necessary for releasing MA from fatty acid hydroperoxides so that it can bind to TBA. Since it is likely that high-temperature heating in air can promote sample autoxidation and consequent artifactually high TBA results, the addition of an antioxidant before heating seems desirable. In our study, BHT was selected as a test antioxidant because it is relatively more effective compared to other commonly used antioxidants (Markuze, 1971). Ideally, an acceptable antioxidant should not only prevent autoxidation of samples during an assay but should also not interfere with either the release of MA from preformed fatty acid hydroperoxides or the binding of MA with TBA to produce the measured chromophore. Table I demonstrates that BHT addition to reactions containing the standard TMP has no effect on either the release of MA from TMP or the binding of MA to TBA when BHT concentrations are 125 μg/mL or less; and at 250 μg/mL, in the absence of fat, only a slight amount of interference is apparent.

BHT Addition before the Boiling Step of TBA Assay. In order to determine the optimal concentration of

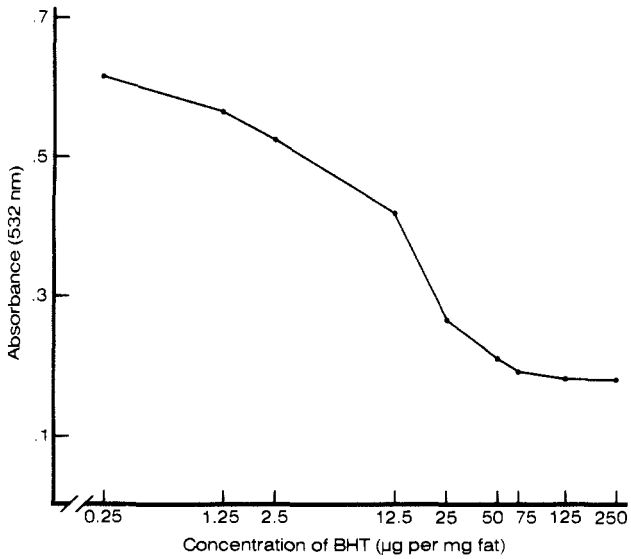

Figure 1. Absorbance values at 532 nm obtained from TBA reactions that contained various concentrations of BHT addition before the assay boiling step. Each tube contained 4 mg of fat from chicken leg muscle.

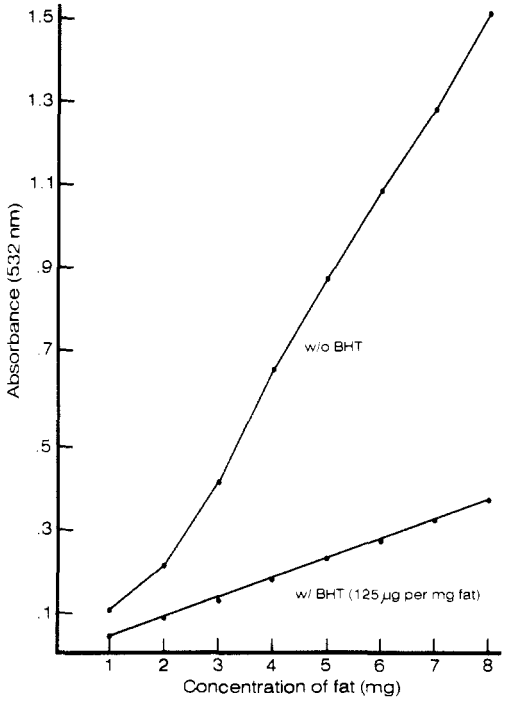

Figure 2. Absorbance values at 532 nm obtained from TBA reactions either with (w) or without (w/o) 125 μg of BHT/mg of fat addition before the assay boiling step. Sample range was 1–8 mg of fat from chicken leg muscle.

BHT necessary to prevent autoxidation during the boiling step of the TBA assay, several concentrations of BHT were tested in assays containing 4 mg of fat as shown in Figure 1. The figure shows that TBA reaction absorbance is stable at 125 and 250 μg of BHT/mg of fat and that at lower concentrations of BHT, the absorbance progressively increases, which indicates incomplete protection against autoxidation. When the TBA assay was performed within a range of 1–8 mg of sample fat, the addition of 125 μg of BHT/mg of fat was sufficient to quench autoxidation at all fat levels tested as shown in Figure 2 where absorbance was not only linear but was also proportional to the amount of sample tested. The same experiment performed simultaneously but without BHT addition gave much higher absorbance that was nonlinear with increasing sample size

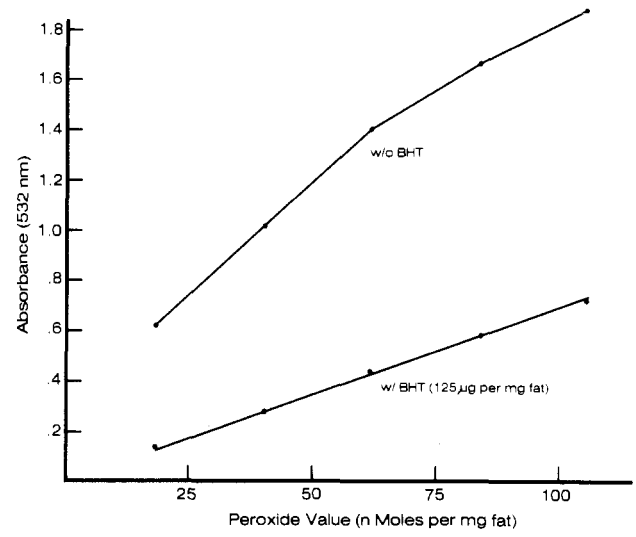

Figure 3. Absorbance values at 532 nm obtained from TBA reactions either with (w) or without (w/o) 125 μg of BHT/mg of fat addition before the assay boiling step by using autoxidized fat samples with different measured peroxide values. Each tube contained 4 mg of fat.

(Figure 2). A final experiment for determining a suitable BHT concentration was performed with mixtures of original and rancid fat mixed in various proportions. When 125 μg of BHT/mg of fat was added to tubes containing 4 mg of sample fat, absorbance was linear and proportional relative to the measured peroxide values of fat samples indicated in Figure 3, while samples without BHT addition gave relatively aberrant readings. The combined results from experiments presented in Figures 1–3 demonstrate that the addition of BHT at a level of 125 μg/mg of fat from chicken skeletal muscle is sufficient to totally control sample autoxidation, even in rancid samples, if sample size is kept between 1 and 8 mg.

BHT Addition before Tissue Extraction Step. Since such large and artifactually high absorbance values due to sample autoxidation were found to occur in TBA assays without antioxidant protection at the critical boiling step, it was interesting to investigate whether significant sample autoxidation occurred during the multiple lipid extraction steps. To test this possiblity, 0.01% BHT was added to the chloroform–methanol (1:2) reagent used at the initial blending step of the extraction. This concentration of BHT is recommended for antioxidant protection of lipids during tissue extractions (Nelson, 1972, 1975). This amount of BHT addition probably represents an upper limit. At this concentration of BHT, dried lipid extracts from leg and breast muscle often contained 1–2% BHT by weight.

For tissue extracted with 0.01% BHT in chloroform–methanol (1:2) reagent, the addition of 75 μg of BHT/mg of fat was enough to protect against sample autoxidation during the TBA boiling step as shown in Figure 4. It is also apparent from Figure 4 that the BHT added during the extraction was far less than required to prevent autoxidation during the TBA assay. Figure 5 shows that when 75 μg/mg of fat is added to the TBA assay, sample autoxidation is effectively prevented within a sample size range of 1–8 mg.

TBA Numbers of Meat Samples. For determining the concentration of MA in fat from various meat samples, TMP was used as the standard. Under the conditions of the assay, 1 mol of MA is released from each mol of TMP (Sinnhuber and Yu, 1977; Gutteridge, 1975). On the basis of 100% equimolar conversion of TMP to TBA–MA, the

Table II. Malonaldehyde Concentrations Measured in Chicken Breast and Leg Muscle with Four Different Procedures[a]

assay conditions	breast muscle[b]		leg muscle[b]	
	μg of MA/g of fat	TBA no.[c]	μg of MA/g of fat	TBA no.[c]
no BHT additions	322.5 ± 9.81	3.29 ± 0.12	367.7 ± 12.51	7.79 ± 0.18
BHT added before boiling step (125 μg/mg of fat)	77.9 ± 3.82	0.79 ± 0.05	90.3 ± 5.29	1.91 ± 0.09
BHT during extraction (0.01% in chloroform-methanol reagent)	91.4 ± 3.27	0.93 ± 0.04	103.1 ± 4.47	2.18 ± 0.07
BHT during extraction and before boiling step (75 μg/mg of fat)	49.8 ± 2.91	0.51 ± 0.03	59.8 ± 3.29	1.27 ± 0.04

[a] Data are presented as mean ± standard deviation; each group had a sample size of six. [b] Breast muscle contained 1.02 ± 0.06% fat and leg muscle contained 2.12 ± 0.13% fat by weight. [c] TBA number units are mg of MA/kg of tissue.

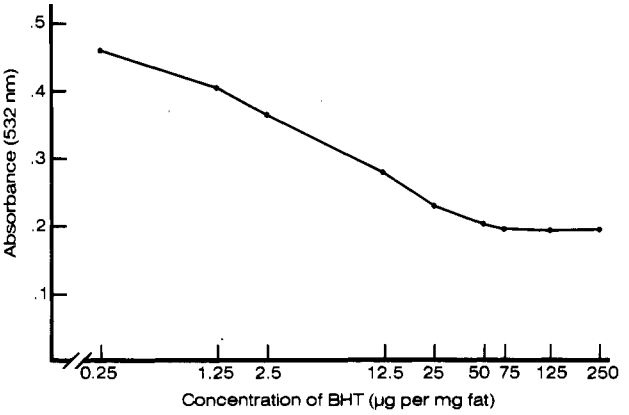

Figure 4. Absorbance values at 532 nm obtained from TBA reactions that included BHT addition (0.01% BHT in chloroform–methanol reagent) during the tissue extraction step as well as various levels of BHT addition before the assay boiling step. Each tube contained 8 mg of fat.

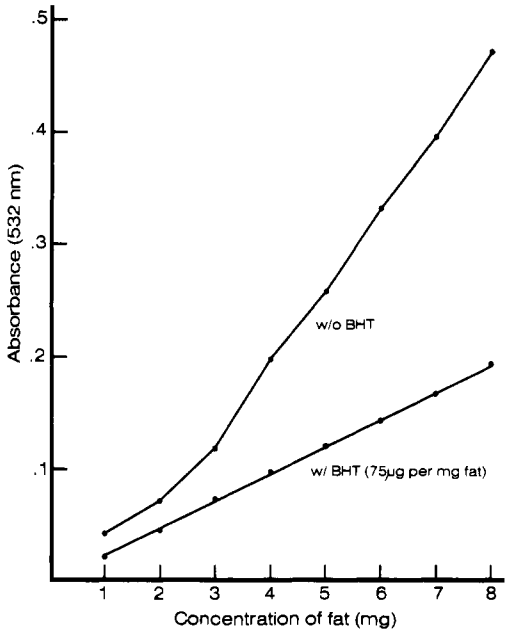

Figure 5. Absorbance values at 532 nm obtained from TBA reactions by using samples extracted with 0.01% BHT in chloroform–methanol reagent but either with (w) or without (w/o) 75 μg of BHT/mg of fat added before the assay boiling step. Sample range was 1–8 mg of fat from chicken leg muscle.

absorbance values at 532 nm can be converted to micrograms of MA as shown in Figure 6.

In order to evaluate the practical use of BHT in the determination of TBA numbers of chicken meat, samples of leg and breast muscles from six healthy 4-month-old pullets were analyzed as shown in Table II. When MA determinations were made per gram of fat, leg muscle fat had slightly more MA than breast. However, on the basis of TBA number, leg muscle was more than twice as high in all four procedures, mainly because leg muscle contained more than twice as much fat as breast muscle (Table II). The lowest values for leg and breast muscle MA were obtained when BHT was added both in the TBA assay and in the initial sample extraction step. Many of the extraction steps, including tissue grinding and homogenization, were done at room temperature; however, even if all extraction steps are performed in a cold room, it is still likely that BHT addition to the extraction is advisable to assure full protection against autoxidation.

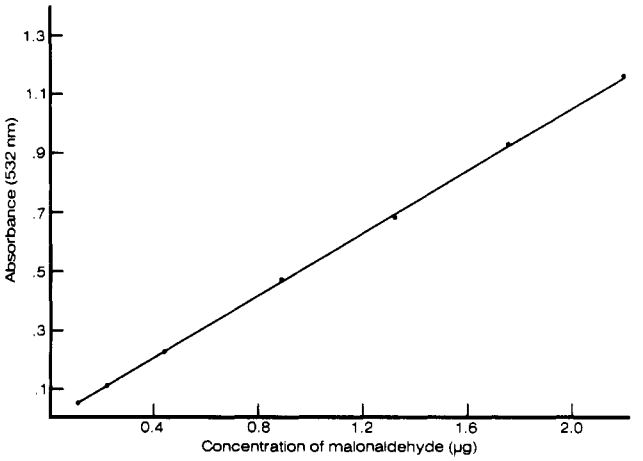

Figure 6. Concentration curve for the 1,1,3,3-tetramethoxypropane (TMP) standard solubilized in water as determined by the TBA assay.

Recommended Procedures. Perhaps the most standard method for measuring MA in meat is the distillation method of Tarladgis et al. (1960). The minimum recommendation is that BHT or other appropriate antioxidant should be added during the distillation procedure. Also, for all variations of the TBA assay, BHT or other antioxidant should be added before the critical heating step of the assay procedure. Addition of antioxidants during the homogenization, distillation, or extraction step of any procedure for TBA assay is recommended. However, in aqueous extracts and homogenates, hydrophobic BHT might not be as effective in protecting fat from peroxidation as it is in organic extractions where it and lipids are both freely soluble.

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Registry No. BHT, 128-37-0; malonaldehyde, 542-78-9.

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A Comparison between Detergent and Nondetergent Analyses of Dietary Fiber in Human Foodstuffs, Using High-Performance Liquid Chromatography To Measure Neutral Sugar Composition

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Dietary fiber analysis of wheat bran, peas, apples, and a food composite, using the modified neutral detergent fiber (NDF) method and a nondetergent method, has been examined. In the latter method, starch hydrolysis was enhanced by using ultrasonics for gelatinization and a heat-stable α-amylase. Neutral sugar compositions, determined by high-performance liquid chromatography, of the insoluble fractions were similar, although, with the exception of wheat bran, neutral detergent extracted significantly more arabinans than did water. NDF contents of the foodstuffs were marginally lower than the corresponding nondetergent fiber contents, due to lower concentrations of uronic acids and lignin in the NDF residues. Water-soluble fractions recovered by the nondetergent method represented only a small portion of the dry matter of the foodstuffs studied. However, for apples and food composite the fraction occupied about 25% of the total dietary fiber content.

Increasing evidence (Spiller and Kay, 1980) that dietary fiber (DF) has an important physiological role in human nutrition has resulted in a considerable increase in the number of published methods for measuring DF in human foodstuffs. Recently, many of the principal approaches were evaluated in an international collaborative study (James and Theander, 1981). The range of results obtained in this study show that uniform and accurate measurement of DF is intrinsically difficult and is complicated further by the wide variety of approaches currently being used (Asp and Johansson, 1981; Furda, 1981; Robertson and Van Soest, 1981; Southgate, 1981; Theander and Aman, 1981).

Direct analysis of the carbohydrate components in DF is necessary in order to conduct meaningful physiological studies and to compare the DF constituents of different foods. Colorimetric (Southgate, 1981) and gas–liquid chromatographic (Englyst, 1981; Theander and Aman, 1981) methods are commonly used to measure carbohydrates in DF. However, recent technological developments in carbohydrate analysis by high-performance liquid chromatography (HPLC) (McGinnis and Fang, 1980) have made it feasible to quantitate the major carbohydrate components in DF from human foodstuffs (Slavin and Marlett, 1983) by using this comparatively simple and rapid technique.

The modified neutral detergent fiber (NDF) method (American Association of Cereal Chemists, 1977; Robertson and Van Soest, 1981) has been used for food fiber analysis, although the method has been critized because it is gravimetric and only provides empirical information. This laboratory recently reported that measurement of the neutral sugars in acid hydrolysates of NDF residues by HPLC overcomes this disadvantage. The NDF method, however, does not recover the water-soluble fiber fraction that has been associated with physiologically significant functions (Anderson, 1980; Jenkins, 1980; Kay and Truswell, 1980). In contrast, the nondetergent procedure of

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