**Table III. Percentage Adsorption and Elution of Dicamba and DSA from $C_{18}$ Columns When Eluted with 2 mL of Methanol**

| process | 10 ppb dicamba | 1 ppm dicamba | 10 ppb DSA | 1 ppm DSA |
|---|---|---|---|---|
| adsorption[a] | 98.8 ± 0.3[b] | 98.3 ± 0.5 | 95.8 ± 0.2 | 91.9 ± 0.2 |
| elution | 95.5 ± 7.2 | 98.5 ± 5.2 | 57.4 ± 18.6 | 77.3 ± 16.3 |

[a] Columns were conditioned with methanol prior to adsorption; 100-mL samples of water adjusted to pH 1 were used. [b] Data represent the mean of four replicates ± SD.

dicamba analysis were desired. In order to improve the recovery, different salt solutions were examined. Table II shows the results of the study with the amino columns when 2 mL of 1 M $K_2HPO_4$ was used as the eluting solvent. Dicamba recovery was 92.7–93.9%. Recovery of DSA ranged from 62.1 to 78.6%, much better than when 1 M NaCl was used.

Table III shows the percentage adsorption and elution of dicamba and DSA from $C_{18}$ SEP-PAK cartridges. Because the pH had to be adjusted to 1 prior to adsorption, some column packing eluted with the samples. $C_{18}$ SEP-PAK cartridges adsorb many organic compounds and thus are not as selective as the ion-exchange cartridges.

**HPLC Analysis.** The use of the amino ion-exchange column as a means of solid-phase extraction of dicamba and DSA eliminates the normal organic solvent partitioning of the aqueous phase, and subsequent derivatization, which is used with the gas chromatographic procedures, is time-consuming, and requires expensive environmentally hazardous solvents. The use of solid-phase concentrators was adaptable to multiple samples, and we routinely extracted 12 water samples at the same time. We could detect 2 ng of dicamba or DSA with our detector (25-μL injection), which corresponds to 1.6 ppb when 100 mL of water was used. It is a simple procedure; it does not require expensive chemicals other than PIC reagent A and is easily automated. We have used this method for analysis of over 600 water samples.

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# Extraction of Soluble Dietary Fiber

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The influence of extraction conditions on the solubility of dietary fiber was examined in four cereals (wheat, rye, barley, oats) and four vegetables (potato, carrot, lettuce, pea). The extraction conditions examined were (a) pH 5.0 acetate buffer at 96 °C for 1 h and 60 °C for 4 h during starch degradation, (b) water at 38 °C for 2 h, (c) pH 1.5 HCl/KCl buffer at 38 °C for 2 h, and (d) pretreatment with absolute ethanol at 96 °C for 1 h and extraction with water at 38 °C for 2 h. Although extraction at high temperature gave in general the highest values for soluble fiber, and extraction in acidic buffer the lowest, the yield and composition of soluble fiber varied considerably with extraction conditions and food sample. The use of standardized and physiologically more appropriate extraction conditions is proposed.

Many of the beneficial effects of dietary fiber, such as reduced postprandial glucose response and blood cholesterol, have been attributed mainly to the activity of soluble fibers (Jenkins, 1980; Kay and Truswell, 1980). Thus, many methods for determining fiber allow the extraction and subsequent quantification of a soluble component. Such extraction procedures were often designed to fit conveniently into an analytical procedure rather than to correspond to actual physiological conditions and commonly are preceded by the gelatinization of starch at 96 °C for up to 1 h and further incubation at lower temperatures for up to 16 h (Theander and Åman, 1979; Englyst et al., 1982; Asp et al., 1983; Theander and Westerlund, 1986). This led Åman and Graham (1987) to suggest extraction of mixed-linked β-glucans at body temperature

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Table I. Content of Total Dietary Fiber Components (mg/g of Dry Matter)

| | nonstarch polysaccharide residue | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| sample | rhamnose | arabinose | xylose | mannose | galactose | glucose | uronic acids | Klason lignin | total fiber |
| wheat | tr | 22.8 | 36.8 | 4.5 | 4.7 | 35.9 | 4.3 | 7.2 | 116.2 |
| rye | 1.0 | 30.3 | 50.9 | 6.1 | 5.8 | 39.1 | 4.2 | 12.2 | 149.6 |
| barley | tr | 22.3 | 45.6 | 5.4 | 3.5 | 78.7 | 5.4 | 11.5 | 172.4 |
| oats | 1.1 | 19.7 | 94.1 | 4.4 | 6.7 | 141.0 | 11.7 | 61.6 | 340.3 |
| potato | 1.8 | 3.9 | 1.6 | 3.2 | 15.6 | 30.7 | 17.0 | 5.1 | 78.9 |
| carrot | 7.8 | 19.4 | 4.5 | 8.4 | 33.0 | 82.2 | 91.7 | 9.0 | 256.0 |
| lettuce | 8.6 | 12.4 | 10.6 | 9.4 | 21.5 | 85.0 | 95.3 | 17.5 | 260.3 |
| pea | 3.9 | 38.1 | 10.5 | 4.0 | 8.0 | 129.8 | 34.1 | 4.9 | 233.3 |

(38 °C), either at the neutral pH of the small intestine or under acidic conditions (pH 1.5) to prevent solubilization of fiber by endogenous food enzymes. As some fiber components, especially furanosidic sugar residues, may be partially hydrolyzed at this pH (Asp et al., 1983), endogenous enzyme activity may alternatively be prevented by pretreating samples with ethanol at 96 °C. However, little is known about the influence of extraction conditions on the solubility of dietary fiber, and thus this study was designed to examine the effect of the four extraction conditions described on the yield of soluble fiber in different cereals and vegetables.

## MATERIALS AND METHODS

**Samples and Sample Preparation.** The wheat (cv. Holme) and rye (cv. Kungs II) were supplied by Weibulls AB, the barley (cv. Kristina) was supplied by Svalöv AB, and the oats (feed oats, cv. unknown) were supplied by the Feed Mill at this university. Frozen peas were purchased from a local supermarket. The potatoes (cv. Maria), lettuce, and carrots were grown near Uppsala during 1985 and were quickly washed, sliced, and frozen after harvesting. Vegetables were freeze-dried, and all eight foods were ground in a Tecator cyclone sample mill to pass a 0.5-mm screen prior to analysis.

**Isolation of Total Fiber.** Total dietary fiber was prepared essentially as described in method C of Theander and Westerlund (1986). Samples (300–400 mg) were suspended in acetate buffer (5 mL, 0.1 M, pH 5.0) with a thermostable $\alpha$-amylase (100 $\mu$L; Termamyl 120 L, Novo A/S, Copenhagen) and incubated in closed tubes at 96 °C for 1 h. Following the addition of amyloglucosidase (400 $\mu$L; Boehringer Mannheim), the samples were further incubated at 60 °C for 4 h. Soluble fibers were precipitated by the addition of absolute ethanol to a final ethanol concentration of 80%, and total fiber was recovered by centrifugation (2000$g$, 20 min) after the mixture was allowed to stand at 4 °C overnight. The pellet was washed by resuspension and centrifugation with 80% aqueous ethanol (2 × 25 mL) and acetone (25 mL) and left to dry at room temperature.

**Extraction of Soluble Fiber.** Four different methods were employed for the extraction of soluble fiber. In the first, samples (300–400 mg) were suspended in 10 mL of acetate buffer (0.1 M, pH 5.0) and starch was enzymatically degraded as described above, i.e. at 96 °C for 1 h and 60 °C for 4 h. After cooling, the samples were centrifuged (2000$g$, 20 min) and the pellets washed with a further 10 mL of acetate buffer. The combined supernatants were made up to 25 mL, and soluble fibers were recovered, by centrifugation, in duplicate 10-mL aliquots following the addition of absolute ethanol to a final ethanol concentration of 80%. One duplicate was used for the determination of neutral nonstarch polysaccharide residues and the other for uronic acid residue quantification.

In the second method, designed to correspond more closely to physiological conditions in the small intestine, soluble fibers were extracted by incubating samples (300–400 mg) with 10 mL of distilled water at 38 °C for 2 h [see Åman and Graham (1987)]. After centrifugation, the pellet was washed with a further 10 mL of water; the supernatants were combined and made up to 25 mL. Duplicate 10-mL aliquots were treated with absolute ethanol to a final ethanol concentration of 80%, and uronic acids were determined on one of the fiber pellets recovered on centrifugation. The other pellet was suspended in acetate buffer, starch was enzymatically degraded as described above, and the fibers, recovered by centrifugation after precipitation in 80% aqueous ethanol, were analyzed for neutral nonstarch polysaccharide residues.

The last two methods were designed to prevent fiber solubilization by endogenous food enzyme activity (Åman and Graham, 1987). Method 3 was similar to method 2, except that soluble fibers were extracted with 10 mL of HCl/KCl buffer (0.1 M, pH 1.5) at 38 °C for 2 h. Method 4 was also as described for method 2, except that samples were pretreated with 10 mL of absolute ethanol at 96 °C for 1 h prior to extraction of soluble fibers with water at 38 °C for 2 h.

**Analysis of Fiber.** Total and soluble fiber preparations were treated with 12 M sulfuric acid (30 °C, 1 h) and hydrolyzed by autoclaving in 0.4 M sulfuric acid (125 °C, 1 h) with internal standard myoinositol added (Theander and Westerlund, 1986). The insoluble residue—Klason lignin—was recovered by filtration. Duplicate aliquots of the filtrate were reduced with potassium borohydride and acetylated with acetic anhydride by the 1-methylimidazole catalyst method (Blakeney et al., 1983; Theander and Westerlund, 1986). The alditol acetates formed were quantified on a Varian 3700 gas–liquid chromatograph fitted with an OV-225 capillary column (10 m × 0.22 mm (i.d.); helium gas flow ~40 cm/s; 200 °C). Uronic acid content was determined by decarboxylation (Theander and Åman, 1979).

**Statistical Analysis.** All soluble fibers were analyzed in triplicate, and means and pooled standard error of means (SEM) are reported. Differences between means were evaluated by the General Linear Models procedure of Statistical Analysis System (1982).

## RESULTS AND DISCUSSION

**Total Fiber.** The total fiber contents (sum of nonstarch polysaccharides and Klason lignin; Theander and Åman, 1979) of the samples analyzed varied from 79 (potato) to 340 (oats) mg/g (Table I) and are similar to published data (Theander and Åman, 1979; Nyman, 1985; Theander and Westerlund, 1976; Åman, 1987). Glucose residues, mainly cellulose and mixed-linked $\beta$-glucans, were a major constituent of the cereal grains, while contents of xylose and arabinose residues (from arabinoxylans) were also high (Theander and Åman, 1979). In the vegetables, glucose residues from cellulose and xyloglucans (Selvendran, 1987) were again predominant, with pectic residues including galacturonic acid (Theander and Åman, 1979), galactose, and arabinose also present in significant quantities. With the exception of the oats, Klason lignin contents were

**Table II. Content of Soluble Fiber Components Extracted under Four Different Conditions (mg/g of Dry Matter)[a]**

| extraction conditions | soluble nonstarch polysaccharide residue | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | rhamnose | arabinose | xylose | mannose | galactose | glucose | uronic acids | total |
| wheat | | | | | | | | |
| buffer (pH 5.0), 96 °C, 60 °C | 0.3[a] | 3.7[a] | 5.8[a] | 1.4[a] | 3.4[ab] | 9.6[a] | 2.2[a] | 26.5[a] |
| water, 38 °C | 0.1[a] | 2.6[b] | 3.4[a] | 0.9[b] | 2.7[ac] | 1.7[b] | 0.8[b] | 12.2[b] |
| buffer (pH 1.5), 38 °C | 0.1[a] | 2.7[b] | 3.8[a] | 0.7[b] | 2.6[c] | 1.3[b] | 0.3[bc] | 11.6[b] |
| ethanol/water, 96/38 °C | 0.2[a] | 2.5[b] | 4.0[a] | 1.4[a] | 3.5[b] | 2.1[b] | 0.1[c] | 13.7[b] |
| SEM | 0.03 | 0.2 | 0.3 | 0.1 | 0.1 | 0.4 | 0.1 | 0.9 |
| rye | | | | | | | | |
| buffer (pH 5.0), 95 °C, 60 °C | 0.2[a] | 9.6[a] | 17.7[a] | 2.7[a] | 2.5[a] | 7.2[a] | 2.6[a] | 42.4[a] |
| water, 38 °C | 0.1[b] | 7.2[b] | 13.5[b] | 1.1[b] | 1.9[b] | 2.5[b] | 0.7[b] | 27.1[b] |
| buffer (pH 1.5), 38 °C | 0.1[b] | 7.6[b] | 13.9[b] | 1.1[b] | 1.9[b] | 2.0[b] | 0.6[b] | 27.2[b] |
| ethanol/water, 96/38 °C | 0.2[a] | 5.8[c] | 11.1[c] | 1.7[c] | 3.0[c] | 2.6[b] | 0.1[c] | 24.4[b] |
| SEM | 0.01 | 0.1 | 0.2 | 0.02 | 0.05 | 0.3 | 0.07 | 0.5 |
| barley | | | | | | | | |
| buffer (pH 5.0), 96 °C, 60 °C | 0.3[a] | 2.6[a] | 3.4[a] | 1.6[a] | 2.0[a] | 29.4[a] | 3.1[a] | 42.4[a] |
| water, 38 °C | 0.2[a] | 2.5[ab] | 3.6[a] | 0.9[b] | 1.7[b] | 23.2[b] | 0.7[b] | 32.7[b] |
| buffer (pH 1.5), 38 °C | 0.2[a] | 1.8[bc] | 2.5[b] | 0.8[c] | 1.7[b] | 11.2[c] | 0.6[b] | 18.7[c] |
| ethanol/water, 96/38 °C | 0.2[a] | 1.6[c] | 2.6[b] | 1.3[d] | 2.6[c] | 8.8[c] | 0.1[b] | 17.1[c] |
| SEM | 0.02 | 0.1 | 0.1 | 0.02 | 0.04 | 0.4 | 0.1 | 0.6 |
| oats | | | | | | | | |
| buffer (pH 5.0), 96 °C, 60 °C | 0.3[a] | 1.7[a] | 2.0[a] | 1.3[a] | 2.6[ab] | 25.7[a] | 2.4[a] | 36.0[a] |
| water, 38 °C | 0.3[a] | 1.6[a] | 2.1[a] | 0.9[b] | 1.9[b] | 36.3[b] | 0.7[b] | 43.7[a] |
| buffer (pH 1.5), 38 °C | 0.3[a] | 1.5[ab] | 1.8[ab] | 0.6[c] | 1.9[b] | 19.7[c] | 0.8[b] | 26.6[b] |
| ethanol/water, 96/38 °C | 0.3[a] | 1.3[b] | 1.6[b] | 1.2[ab] | 2.9[a] | 16.1[c] | 2.6[a] | 26.0[b] |
| SEM | 0.02 | 0.1 | 0.1 | 0.05 | 0.1 | 1.3 | 0.1 | 1.4 |
| potato | | | | | | | | |
| buffer (pH 5.0), 96 °C, 60 °C | 1.0[a] | 1.9[a] | 0.6[a] | 1.4[a] | 9.6[a] | 2.7[a] | 9.6[a] | 26.9[a] |
| water, 38 °C | 0.1[b] | 0.4[b] | 0.4[b] | 0.7[b] | 1.6[b] | 0.7[b] | 1.6[b] | 5.5[b] |
| buffer (pH 1.5), 38 °C | 0.1[b] | 0.5[b] | 0.4[b] | 0.6[b] | 1.7[b] | 0.8[b] | 1.1[b] | 5.2[b] |
| ethanol/water, 96/38 °C | 0.3[c] | 0.4[b] | 0.6[a] | 0.7[b] | 2.7[b] | 1.2[b] | 3.2[c] | 9.0[c] |
| SEM | 0.02 | 0.03 | 0.02 | 0.03 | 0.2 | 0.2 | 0.1 | 0.3 |
| carrot | | | | | | | | |
| buffer (pH 5.0), 96 °C, 60 °C | 4.2[a] | 11.3[a] | 0.7[ab] | 1.3[a] | 21.4[a] | 2.2[a] | 60.9[a] | 101.9[a] |
| water, 38 °C | 0.3[b] | 1.9[b] | 0.5[b] | 0.9[b] | 5.5[b] | 1.1[b] | 4.2[b] | 14.4[b] |
| buffer (pH 1.5), 38 °C | 0.3[b] | 2.8[b] | 0.6[b] | 1.0[b] | 5.3[b] | 1.2[b] | 3.4[b] | 14.5[b] |
| ethanol/water, 96/38 °C | 0.8[b] | 2.1[b] | 0.8[a] | 1.0[b] | 6.5[b] | 1.6[ab] | 20.9[c] | 33.7[c] |
| SEM | 0.1 | 0.2 | 0.03 | 0.04 | 0.4 | 0.1 | 1.4 | 1.7 |
| lettuce | | | | | | | | |
| buffer (pH 5.0), 96 °C, 60 °C | 1.4[a] | 3.8[a] | 0.7[ab] | 1.0[a] | 8.6[a] | 1.5[a] | 32.1[a] | 49.2[a] |
| water, 38 °C | 0.2[b] | 3.1[b] | 0.5[c] | 1.2[a] | 8.4[a] | 1.5[a] | 6.8[bc] | 21.7[b] |
| buffer (pH 1.5), 38 °C | 0.2[b] | 2.6[c] | 0.6[bc] | 1.2[a] | 6.9[b] | 1.3[a] | 6.0[b] | 18.8[b] |
| ethanol/water, 96/38 °C | 0.5[b] | 1.6[d] | 0.8[a] | 1.0[a] | 4.7[c] | 1.6[a] | 8.8[c] | 19.1[b] |
| SEM | 0.05 | 0.1 | 0.02 | 0.04 | 0.1 | 0.1 | 0.3 | 0.5 |
| pea | | | | | | | | |
| buffer (pH 5.0), 96 °C, 60 °C | 0.6[a] | 5.1[a] | 1.4[a] | 1.6[a] | 4.4[a] | 2.7[a] | 10.5[a] | 26.3[a] |
| water, 38 °C | 0.3[b] | 2.2[b] | 0.8[b] | 0.8[b] | 2.5[b] | 1.0[b] | 4.2[b] | 11.7[b] |
| buffer (pH 1.5), 38 °C | 0.2[b] | 1.5[b] | 0.6[b] | 0.6[b] | 1.9[b] | 0.7[b] | 2.0[c] | 7.5[c] |
| ethanol/water, 96/38 °C | 0.3[b] | 1.7[b] | 0.9[b] | 0.9[b] | 2.5[b] | 1.2[b] | 3.3[b] | 10.7[b] |
| SEM | 0.04 | 0.2 | 0.04 | 0.06 | 0.1 | 0.1 | 0.2 | 0.4 |

[a] Means and pooled standard error of means (SEM) of triplicate analyses. For any sample, values within a column that do not share a common superscript differ significantly ($P < 0.001$).

generally about 10 mg/g; the value for oats, 62 mg/g, lies within the range found in Swedish oats (54–128 mg/g; Åman, 1987).

**Soluble Fiber.** The soluble fiber preparations were completely hydrolyzed with 0.4 M sulfuric acid; i.e., no Klason lignin was recovered. As for total fiber, glucose, xylose, and arabinose were the main soluble nonstarch polysaccharide residues in all cereals (Table II). While arabinoxylans were the major soluble fiber in rye, glucose residues, probably mainly from mixed-linked β-glucans, predominated in the extracts from barley and oats. In cereal grains the soluble fiber is found mainly in the endosperm, while brans contribute most of the insoluble fiber (Nyman, 1985). Of the grains examined, the percent solubility of nonstarch polysaccharides was in general highest for rye (mean 22%) and lowest for oats (mean 13%); however, the oats had on average the highest absolute content of soluble fibers (mean 33 mg/g). In the vegetable samples, uronic acid, galactose, and arabinose were the main soluble fiber residues. The carrot (14–102 mg/g) and lettuce (19–49 mg/g) samples had higher contents of soluble fibers than the potatoes (5–27 mg/g) and peas (8–26 mg/g).

The variability of the extraction methods was not significantly influenced by sample, and average coefficients of variation for all methods ranged from 2.6% in the rye to 7.1% in the carrots (mean 4.4%). No between-method differences were apparent, with coefficients of variation for methods 1–4 of 4.3%, 4.2%, 4.7%, and 4.6%, respectively.

**Comparison of Extraction Methods.** Extraction of soluble fibers following starch degradation, i.e. 1 h at 96 °C and 4 h at 60 °C, gave significantly ($P < 0.001$) higher values than the other three methods employed for all samples except the oats. Treatment at higher temperatures, for example at 125 °C in an autoclave, can lead to an even greater solubilization of fiber (Asp et al., 1983). In the oats, and to a lesser extent the barley, extraction with water at 38 °C for 2 h gave higher values for soluble glucose residues, and thus soluble fiber, than did extraction with pH 1.5 buffer or following treatment with ethanol (96 °C, 1 h). Thus, as shown by Åman and Graham (1987),

endogenous β-glucanase activity in barley and oats can greatly influence the solubility of β-glucans under certain extraction conditions.

It is notable that, for the potato and carrot, extraction following starch degradation at high temperature gave approximately 6-fold higher values than extraction with water at 38 °C. The exceptional susceptibility of the fibers in these two samples to solubilization at high temperature led to a significantly ($P < 0.001$) increased solubility of fiber in water at 38 °C following pretreatment by boiling in ethanol (Table II). With the exception of the potato and carrot samples, the values for soluble fiber extracted by the two methods designed to prevent endogenous enzyme activity, i.e. in acidic buffer or pretreatment with ethanol, were very similar. That extraction in acidic buffer gave numerically, and sometimes significantly, lower soluble fiber values than extraction in water at the same temperature for all samples could result from reduced solubility at lower pH or decreased endogenous enzyme activity (Åman and Graham, 1987).

The composition, as well as yield, of soluble fiber was also effected by extraction conditions. For example, the glucose to arabinose ratios in the soluble fiber in barley varied between 5.5 (method 4) and 11.3 (method 1), while the xylose to arabinose ratios remained around 1.4. Indeed the xylose to arabinose ratios in the soluble fiber were about 1.5 for all cereals, irrespective of extraction method.

The present study establishes that the determination of soluble dietary fiber is very dependent on the extraction method employed. It is also apparent that the relationship between different extraction methods depends on the sample under analysis. Which extraction method is most closely correlated to the physiological activity of soluble fiber is not known, although the solubility of nonstarch polysaccharides can increase greatly during passage through the small intestine (Graham et al., 1986). However, extraction by methods more relevant to physiological conditions may be more suitable than the high-temperature procedures most commonly employed.

**Registry No.** Klason lignin, 8068-04-0.

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# Kinetic Study of Maillard Reactions in Milk Powder by Photoacoustic Spectroscopy

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Application of photoacoustic spectroscopy (PAS) to the analysis of milk or dairy products is reported. We have studied the kinetic aspects of the Maillard browning reactions in milk powder tablets. The reaction rate has been assessed by using the spectral ratio $I_{335}/I_{280}$ (i.e., the PA intensity of the Maillard reaction products to that of proteins). The molar energy and enthalpy of activation were found to be 115 and 113 kJ mol$^{-1}$ at 298 K, respectively. We discuss the relevance of the application of photoacoustic methodology in dairy research and the milk industry.

The nonenzymatic browning of Maillard reaction (Maillard, 1916) is very important in many respects such as food processing and storage, cataract formation, and diabetes. The primary reaction in Maillard browning is thermal condensation of an amino compound (i.e., protein amino acids, especially lysine) (Candiano et al., 1985) with the carbonyl group of a sugar in the open-chain form, probably to form a Schiff base, thereby reducing the amount of lysine available for nutrition. Loss of palatability often occurs. On the other hand, maple syrup owes its fine flavor and color to browning as the distinctive caramel and butterscotch flavors of dairy products derive from the browning of milk. This nonenzymatic browning of food products, which is different from caramelization, heat-induced browning of sugars in the absence of amino

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