

## 45.4.08

**AOAC Official Method 993.19**  
**Soluble Dietary Fiber**  
**in Food and Food Products**  
**Enzymatic–Gravimetric Method (Phosphate Buffer)**  
**First Action 1993**  
**Final Action 1996**

(Applicable to determination of soluble dietary fiber [SDF] in vegetables, fruit, and cereal grains; and to determination of total dietary fiber [TDF] in conjunction with 991.42 [see 32.1.16], Insoluble Dietary Fiber [IDF] in Food and Food Products.)

See Tables 993.19A and B for the results of the interlaboratory study supporting acceptance of the method.

**A. Principle**

Duplicate test portions of dried foods, fat-extracted if >10% fat, are gelatinized with heat-stable  $\alpha$ -amylase and then enzymatically digested with protease and amyloglucosidase to remove protein and starch. IDF is removed by filtering and washing residue with water. SDF in filtrate is precipitated by adding 95% ethanol to filtrate. Precipitate is filtered and washed with 78% ethanol, 95% ethanol, and acetone, dried, and weighed. One duplicate is analyzed for protein, and second is incinerated at 525 °C to determine ash. SDF is weight of residue minus weight of protein and ash.

**B. Apparatus**

See 991.42B (see 32.1.16).

**C. Reagents**

See 991.42C (see 32.1.16) with following change:

(j) *Celite*.—Medium grade (acid-washed).

**D. Enzyme Purity**

See 991.42D (see 32.1.16).

**E. Preparation of Test Portions**

Analyze dry foods without pretreatment whenever possible. Dry-mill to 0.3–0.5 mm mesh. Homogenize and freeze-dry wet foods before milling. If high fat content (>10%) prevents proper milling, defat with three 25 mL portions of petroleum ether/g food before milling. Determine residual moisture in milled foods by drying overnight in 70 °C vacuum oven or 5 h in 105 °C air oven. Record weight loss due to fat and/or water, and make appropriate correction to final % TDF and SDF. (Note: For foods high in sugars that cannot be dried by lyophilization, extract test portion 3 each with 10 volumes 85% methanol to remove sugars before milling or lyophilization, which may interfere in determination.)

**F. SDF Determination**

Proceed as in 991.42F (see 32.1.16), from beginning up through “Wash residue . . . 2 times with 10 mL acetone” in paragraph 5.

Adjust weight of combined filtrate and water washings to 100 g with H<sub>2</sub>O. Add 4 volumes (400 mL) 95% ethanol, preheated to 60°C. Let precipitate form at room temperature 60 min.

Tare crucible containing Celite to nearest 0.1 mg; then wet and redistribute Celite bed in crucible, using stream of 78% ethyl alcohol from wash bottle. Apply suction to crucible to draw Celite onto frittered glass as even mat.

Filter precipitate mixture and wash residue successively with three 20 mL portions of 78% ethyl alcohol, two 10 mL portions of 95% ethyl alcohol, and two 10 mL portions of acetone.

Proceed as in 991.42F (see 32.1.16), starting with “Break surface film . . .” in paragraph 5 through end of 991.42F (see 32.1.16).

**G. Calculations**

Determination of blank:

$$B = \text{blank, mg} = \text{weight residue} - P_B - A_B$$

where weight residue = average of residue weights (mg) for duplicate blank determinations; and  $P_B$  and  $A_B$  = weights (mg) of

**Table 993.19A. Interlaboratory study results for soluble dietary fiber in foods by enzymatic-gravimetric method (phosphate buffer)**

Food	No. of labs	Average SDF, %	$s_r$	$s_R$	RSD <sub>r</sub> , %	RSD <sub>R</sub> , %	HorRat
Apricots	8	11.20	0.42	0.91	3.78	8.11	2.9
Carrots	9	11.53	0.53	1.11	4.59	9.61	3.5
Chick peas	10	1.21	0.23	0.34	19.52	28.28	7.3
Onions	8	4.13	0.91	1.00	21.93	24.12	7.5
Raisins	8	7.95	0.67	0.67	8.41	8.41	2.9
Sugar beet fiber	10	20.65	0.80	1.35	3.88	6.52	2.6

**Table 993.19B. Interlaboratory study results for total dietary fiber in foods by enzymatic-gravimetric method (phosphate buffer)**

Food	No. of labs	Average TDF, %	$s_r$	$s_R$	RSD <sub>r</sub> , %	RSD <sub>R</sub> , %
Apricots	10	24.63	0.74	0.97	3.03	3.95
Carrots	9	23.25	0.49	0.79	2.10	3.38
Chick peas	10	14.33	0.85	1.00	5.93	7.01
Onions	9	16.13	0.95	1.02	5.88	6.33
Raisins	10	30.28	0.81	2.05	2.69	6.78
Sugar beet fiber	10	66.07	1.15	1.59	1.74	2.41

protein and ash, respectively, determined in first and second blank residues.

Calculate SDF as follows:

$$\text{SDF, \%} = \frac{\text{weight residue } P + A + B}{\text{weight test portion}} \times 100$$

where weight residue average of weights (mg) for duplicate test portion determinations;  $P$  and  $A$  = weights (mg) of protein and ash,

respectively, in first and second sample residues; and weight test portion = average of 2 test portion weights (mg) taken.

Calculate TDF as follows:

$$\text{TDF, \%} = \text{SDF} + \text{IDF [from 991.42 (see 32.1.16)]}$$

Reference: [J. AOAC Int. 77, 690\(1994\)](#).

*Revised: June 2000*