AOAC Official Method 993.21
Total Dietary Fiber in Foods and Food Products with ≤2% Starch
Non-Enzymatic-Gravimetric Method
First Action 1993
Final Action 1996

(Applicable to determination of ≥10% total dietary fiber in foods and food products with ≤2% starch, dry weight basis.)

See Table 993.21 for the results of the interlaboratory study supporting the acceptance of the method.

A. Principle

Dried fruit, vegetable, or isolated fiber sources are suspended in H₂O and incubated 90 min at 37°C to solubilize sugars and other water-soluble components. Water-soluble fiber components are then precipitated with ethanol. Residue is washed sequentially with 78% ethanol, 95% ethanol, and acetone and then dried at 105°C. One duplicate is analyzed for crude protein, the other for ash. Total dietary fiber (TDF) is calculated as weight of residue less weight of protein and ash.

B. Apparatus

(a) Analytical balance.—Capable of weighing to 0.1 mg.
(b) Air oven.—Capable of maintaining 105 ± 0.5°C.
(c) Beakers.—250 mL.
(d) Desiccator.—Containing mixture of colorless and indicating desiccant (Drierite is suitable).
(e) Filtering flask.—1 L capacity.
(f) Fritted crucible.—Porosity No. 2 (coarse ASTM 40–60 μm). Wet 0.5 g filter aid and evenly distribute by swirling with 78% ethanol; then apply vacuum to form even mat. Heat crucible containing filter aid in muffle furnace 1 h at 525°C, let cool in desiccator, and weigh before use.
(g) Incubator or water bath.—Capable of maintaining 37 ± 0.5°C.
(h) Muffle furnace.—Capable of achieving 525°C.

C. Reagents

(a) Ethanol.—(1) 95% (without any organic additive). (2) 78%. Dilute 207 mL H₂O with 95% ethanol to 1 L.
(b) Acetone.
(c) Analytical filter aid.—Acid-washed diatomaceous silica, ca 97.5% SiOH, ca 5% retained on 150 mesh screen [Celite®, Fisher Scientific, 711 Forbes Ave, Pittsburgh, PA, USA; or C.A.F.A. (Celite Analytical Filter Aid), Manville Products Corp., PO Box 5108, Denver, CO, USA, is suitable].

D. Determination

Accurately weigh to nearest 0.1 mg duplicate 500 mg freeze-dried, ground (≤30 mesh) test portions or homogenized (by food processor) wet test portions (containing ca 0.5 g dry matter) into separate 250 mL beakers. Add 25 mL (or volume necessary to bring wet test portion to 25 mL) H₂O to each beaker; sonicate or gently stir suspensions until test portions are thoroughly wet, i.e., no clumps remain. Scrape down any particles on inside wall of beaker with rubber policeman, and rinse walls with 1–2 mL H₂O. Cover beakers with Al foil and let stand 90 min without stirring in 37°C incubator or water bath.

Add 100 mL 95% ethanol to each beaker and let stand 1 h at room temperature (25 ± 2°C). Collect residue under vacuum in preweighed crucible containing filter aid. If and when filtration becomes very slow, use closed-end Luer needle, or any small pointed object, to gently scratch matted test portion without disturbing filter aid. Positive pressure may also be used if available.

Wash residue with 20 mL 78% ethanol, 2× with 10 mL 95% ethanol, and 1× with 10 mL acetone. Final rinsing with acetone should be done in fume hood, collecting acetone wash in separate filtering flask for proper disposal. Dry crucible containing residue ≥2 h at 105°C. Cool crucibles ≥2 h in desiccator and weigh to nearest 0.1 mg.

Ash residue from one duplicate 5 h at 525°C. Cool crucible ≥2 h in desiccator and weigh to nearest 0.1 mg.

Analyze residue from remaining duplicate for crude protein by Kjeldahl nitrogen determination, see 992.15 (see 12.1.07) or 993.21, using %N × 6.25.

E. Calculations

Calculate TDF (%) as follows:

\[
TDF, \% = \frac{100 \times W_r - [(P + A) / 100] \times W_s}{W_s}
\]

where \(W_r\) = mg residue, \(P = \%\) protein in residue, \(A = \%\) ash in residue, and \(W_s\) = mg test portion.

Reference: J. AOAC Int. 77, 687(1994).
Revised: March 1997