AOAC Official Method 950.02

Animal Feed

Preparation of Sample

Grind sample to pass sieve with circular openings 1 mm diameter and mix thoroughly. If sample
cannot be ground, reduce to as fine condition as possible. Do not grind molasses feed.

AOAC Official Method 962.09

Fiber (Crude) in Animal Feed and Pet Food

Ceramic Fiber Filter Method

First Action 1962

Final Action 1971

Revised First Action 1982

AOCS-AOAC Method

A. Principle

Crude fiber is loss on ignition of dried residue remaining after digestion of sample with 1.25% H₂SO₄ and 1.25% NaOH solutions under specific conditions. Method is applicable to grains, meals, flours, feeds, fiber-bearing material, and pet foods from which fat can be extracted to leave workable residue.

B. Reagents

(a) Sulfuric acid solution.----.0255+/- .005N. 1.25 g H₂SO₄/100 ml. Concentration must be checked by titration.

(b) Sodium hydroxide solution. --0.3 13 +/- 0.005N. 1.25 g NaOH/100 ml. free, or nearly so, from Na₂CO₃. Concentration must be checked by titration.
Prepared ceramic fiber. -Place 60 g ceramic fiber (Cerafiber, 8 lb/cu ft, E.J. Bartell Co., 700 Powell Ave., SW, Renton, WA 98055) in blender, add 800 ml H2O, and blend 1 min at low speed.

Determine blank by treating ca 2 g (dry weight) of prepared ceramic fiber with acid and alkali as in determination. Correct crude fiber results for any blank, which should be negligible (ca 2 mg).

(d) Alcohol.-95% or reagent alcohol, methanol, or isopropanol.

(e) Antifoam.-Dow Corning Corp. Antifoam A compound diluted 1 + 4 with mineral spirits or petroleum ether, or H2O-diluted Antifoam B Emulsion (1 + 4). Do not use Antifoam Spray.

(f) Bumping chips or granules.-Broken Alundum crucibles or equivalent granules (RR Alundum 90 mesh, Norton Co., I New Bond St., Worcester, MA 01606) are satisfactory.

C. Apparatus

(a) Digestion apparatus.-With condenser to fit 600 ml beaker, and hot plate adjustable to temperature that will bring 200 ml H2O at 25° to rolling boil in 15 +/- 2 min. (Available from Labconco Corp., 8811 Prospect Ave., Kansas City, MO 64132.)

(b) Ashing dishes,-Silica, Vitreosil 70 x 16 mm; or porcelain, Coors Ceramics Co., 600 9th St., Golden, CO 80401, No.60230, or equivalent.

(c) Desiccator.-With efficient desiccant such as 4-8 mesh Drierite (CaCl2 is not satisfactory).

(d) Filtering device.-With No.200 type 304 or 316 stainless steel screen (W.S.Tyler Inc., 8570 Tyler Blvd., Mentor, OH 44060), easily washed free of digested residue. Either Oklahoma State filter screen (see Figure 962.09A; available from Labconco Corp.) or modified California plastic Buchner (see Figure 962.09B; consists of 2 piece polypropylene plastic funnel manufactured by Nalge Co., 75 Panorama Creek Dr, P0 Box 20365, Rochester, NY 14602, Cat. No. 42110-0700, 70 mm [without No.200 screen], or equivalent [also available from Labconco Corp.]. Seal screen to filtering surface of funnel, using small-tip soldering iron).

(e) Suction filter.-To accommodate filtering devices. Attach suction flask to trap in line with aspirator or other source of vacuum with valve to break vacuum.

(f) Liquid preheater. -For preheating H2O, 1.25% H2SO4, and 1.25% NaOH solutions to bp of H2O. Convenient system, shown in Figure 962.09C. consists of sheet Cu tank with 3 coils of 3/8" (10 mm) od Cu tubing. 12.5' (3.8 m) long. Solder inlets and outlets where tubing passes through tank walls. Connect to reflux condenser and fill with H2O. Keep H2O boiling with two 750 watt thermostatically controlled hot plates. Use Tygon for inlet leads to reservoirs of H2O, acid. and
alkali; use gum rubber tubing for outlets. Capacity of preheater is adequate for 60 analyses in 8 h.

D. Preparation of Sample

Reduce sample (riffle is suitable) to 100 g and place portion in sealed container for H₂O determination. Immediately determine H₂O. Grind remainder to uniform fineness. (Weber mill with screen 0.033-.040" [No. 18 or 20], Micro mill [Hosokawa Micron Powder Systems. Inc., 10 Chatham Rd, Summit, NJ 07901] with screen 1/25-1/16" [No. 18-No. 12], and Wiley mill with 1 mm [No. 18] screen give comparable fineness.) Since most materials lose moisture during grinding, determine H₂O on ground sample at same time sample is taken for crude fiber determination.

Determination

Extract 2 g ground material with ether or petroleum ether (initial boiling temperature. 35-38°, dry-flask end point. 52-60°, >95% distilling <54°, and <60% distilling <40°. specific gravity at 60°F. 0.630-0.660; evaporation residue <0.002% by weight). If fat is <1% extraction may be omitted. Transfer to 600 mL beaker, avoiding fiber contamination from paper or brush. Add ca 1.5-2.0 g dry weight of prepared ceramic fiber, 200 ml. Boiling 1.25% H₂SO₄ and 1 drop diluted antifoam. (Excess antifoam may give high results; use only if necessary to control foaming.) Bumping chips or granules may also be added. If extremely fine materials are being analyzed and filters are to be precoated with a filter mat. Prepare two beakers of ceramic fiber mixture for each sample as follows: Add 1.5 g dry weight of prepared ceramic fiber to each 100 ml beaker, then add 60-75 ml 0.255N sulfuric acid to each beaker and allow to soak until precoat step. Place beaker on digestion apparatus with preadjusted hot plate and boil exactly 30 min., rotating beaker periodically to keep solids from adhering to sides. Remove beaker, and filter as in (a) or (b).

(a). Using Oklahoma filter screen (1) (Extremely fine materials only.) Skip to step (2) if no filter precoating is necessary. Precoat the filter screen as follows: Attach Oklahoma filter screen to vacuum flask. Turn on suction. Mix well the 60-75 ml 0.225N sulfuric acid and 1.5 g ceramic fiber mixture previously prepared. Insert the screen into beaker keeping face of screen just under the surface of liquid until all liquid is removed. Without breaking suction, proceed to step (2).

(2) Turn on suction and insert screen (precoated with ceramic fiber if extremely fine materials are being analyzed) into beaker, keeping face of screen just under surface liquid until all liquid is removed. Without breaking suction or raising filter, add 50-75 ml boiling H₂O. (Work rapidly to keep mat from becoming dry.) Remove filter from beaker and drain all H₂O from line by raising above trap level. Return mat and residue to beaker by breaking suction and blowing back. Add 200 ml boiling 1.25% NaOH and boil exactly 30 min. Remove beaker.

(3) (Extremely fine materials only) Skip to step (4) if no filter precoating is necessary. Precoat the filter screen using the second beaker of ceramic mixture as described in E(a)(1)
(4) Filter as in E(a)(2). Without breaking suction, wash with 25 ml boiling 1.25% H2SO4 and three 50 ml portions boiling H2O. Drain free of excess H2O by raising filter. Lower filter into beaker and wash with 25 ml alcohol. Drain line, break suction, and remove mat by blowing back through filter screen into ashing dish. Proceed as in E(c).

(b) Using California Buchner (1) (Extremely fine materials only.) Skip to step (2) if no filtering precoating is necessary. Precoat the filter screen as follows: Attach California buchner to vacuum flask funnel must be level. Do not turn on vacuum (Note: California buchner may be held level above vacuum flask if vacuum connot be turned off. Mix well the 60-75 ml 0.225N sulfuric acid and 1.5 g ceramic fiber mixture previously prepared. Pour ceramic fiber/acid slurry into California Buchner. Allow to settle 5-10 s. Turn on min. vacuum, just sufficient to form ceramic fiber "pad".

(2) Filter contents of beaker through Buchner (precoated with ceramic fiber if extremely fine materials are being analyzed.) rinse beaker with 50-75 ml boiling H2O and wash through Buchner. Repeat with three 50 ml portions H2O and such dry. Remove mat and residue by snapping bottom of Buchner against white top while covering stem with thumb or forefinger and replace in beaker. Add 200 ml. Boiling 1.25% NaOH and boil exactly 30 min. Remove beaker.

(3)(Extremely fine materials only) Skip to step (4) if no filter precoating is necessary. Precoat the filter screen using the second beaker of ceramic mixture as described in E(b)(1)

(4) Filter as in E(b)(2) Wash with 25 ml boiling 1.25% H2SO4 three 50 ml portions H2O and 25 ml alcohol. Remove mat and residue; transfer to ashing dish.

(c) Treatment of residue Dry mat and residue 2 hr. at 130 +/- 2° Cool in desiccator and weigh. Ignite 30 min at 600 +/- 15°. Cool in desiccator and reweigh.

\[ \% \text{Crude fiber in ground sample} = C = \frac{(\text{Loss in weight on ignition} - \text{loss in weight of ceramic fiber blank}) \times 100}{\text{weight sample}} \]

\[ \% \text{Crude fiber on desired moisture basis} = C \times \frac{(100 - \% \text{ moisture desired})}{(100 - \% \text{ moisture in sample})} \]

*Report results to 0.1%*