

FIBER, CRUDE

PRINCIPLE

Crude fiber is determined gravimetrically after chemical digestion and solubilization of other materials present. The fiber residue weight is then corrected for ash content after ignition.

SCOPE

The method is applicable to corn and other grains, feedstuffs containing vegetable materials, flours and meals.

SPECIAL APPARATUS

1. Digestion Apparatus: A multi-unit assembly with rheostat- controlled electric heaters, and condensers to fit 600 mL beakers, designed specifically for crude fiber determinations, is recommended. Heaters must be adjustable to the temperature that will bring 200 mL of water at 25 °C to a rolling boil in 15 ± 2 minutes (Note 1).
2. Filtering Device: A California Modified Buchner Funnel, two-piece polyethylene, with a 200 mesh stainless steel screen, is recommended.
3. Muffle Furnace: Equipped with a pyrometer and capable of operating at temperatures up to 600 °C
4. Drying Oven: Forced draft or convection air oven, operating at 130 ± 2 °C

REAGENTS

1. Sodium Hydroxide Solution, 0.312 *N* (1.25%): Prepare and standardize against potassium acid phthalate using phenolphthalein indicator.
2. Sulfuric Acid Solution, 0.255 *N* (1.25%): Prepare and standardize against a standard sodium hydroxide solution using phenolphthalein indicator.

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3. Prepared Ceramic Fiber: Place 60 g of ceramic fiber (Lab Safety Supply Co., P. O. Box 1368, Janesville, WI 53545, (Cat. No. 1740 M, or equivalent)) in a blender, add 800 mL of purified water, and blend 1 minute at low speed. Ten mL of well-mixed suspension contains about 0.75 g of fiber.
4. Alcohol: Methyl, isopropyl, or 95% ethyl alcohol
5. Antifoam: Dow Corning Antifoam A Emulsion diluted 1 + 4 with water

PROCEDURE

Grind about 50 g of sample through a laboratory cutting mill to 20 mesh or finer, and mix thoroughly (Note 2). Determine moisture content of the ground sample by the "Standard" toluene distillation method - A-12 (Note 3), or alternate procedure giving equivalent results.

Weigh accurately about 2 g of sample and transfer to a 9 cm hard filter paper (Note 4) supported on a filter cone in a 60 ° funnel. Extract with three 25 mL portions of ether and apply vacuum until sample is dry (Note 5). Transfer extracted sample quantitatively by brushing into a 600 mL beaker of the fiber digestion apparatus. Add 20 mL of well-mixed ceramic fiber suspension (containing about 1.5 g of fiber - dry weight), 200 mL of boiling 1.25% sulfuric acid solution, and 1 drop of diluted antifoam (Note 6). Place beaker on digestion apparatus with preadjusted heater and boil exactly 30 minutes, rotating beaker periodically to keep solids from adhering to sides.

Remove beaker and filter contents through California Buchner funnel precoated with about 0.75 g of ceramic fiber - dry weight; rinse beaker with 50-75 mL of boiling water, and wash through funnel. Repeat with three 50 mL portions of water, and suck dry. Return fiber mat with residue to beaker by blowing back through funnel. Add 200 mL of boiling 1.25% sodium hydroxide solution, return to heater and boil exactly 30 minutes. Remove beaker and filter as before. Wash with 25 mL of boiling 1.25% sulfuric acid solution, three 50 mL portions of water, and 25 mL of alcohol. Remove mat and residue, and transfer to ashing dish.

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Dry fiber mat and residue at 130 ± 2 °C for 2 hours. Cool in a desiccator (Note 7) and weigh. Ignite at 600 ± 15 °C to constant weight (30 minutes usually sufficient). Cool in desiccator and weigh.

Run a blank determination on the prepared ceramic fiber using the same quantity of fiber and the same amounts of acid and alkali as in the determination (Note 8).

CALCULATION

% Crude Fiber (dry basis) =

$$= \frac{(\text{Dry Residue Wt. (g)} - \text{Ignited Residue Wt. (g)} - \text{Blank Wt. Loss (g)}) \times 100 \times 100}{\text{Sample Wt. (g)} \times \text{Sample Moisture, (\%)}}$$

NOTES AND PRECAUTIONS

1. A 750 mL Erlenmeyer flask with a reflux condenser and laboratory hot plate or gas burner may be substituted for commercially available fiber digestion apparatus.
2. If moisture content is above 20%, it is advisable to pre-dry sample prior to grinding. Place sample to be ground in open dish (but protected from dust or other contamination) in a warm, well-ventilated place so that the grain will dry reasonably fast and reach an approximate air-dried condition in from 14 to 24 hours. Moisture loss need not be recorded since moisture content of the ground sample will be determined.
4. When moisture content of the ground sample is determined by the "Standard" toluene distillation method, the grinding loss is disregarded. The moisture calculation simplifies to:

$$\% \text{ Moisture} = \frac{(\text{mL H}_2\text{O in Trap} + \text{Blank}) \times 100}{\text{Sample Wt. (g)}}$$

5. Schleicher & Schuell No. 597 filter paper, or equivalent, is recommended.
6. If fat content is less than 1%, the extraction may be omitted.

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7. Excess antifoam gives high results; use only if necessary to control foaming. Boiling chips or beads may also be added.
8. Use an efficient desiccant such as 4-8 mesh Drierite (calcium chloride is not satisfactory).
9. Normally, the blank should be no more than 1-2 mg.

REFERENCES

1. D. O. Holst, J. Assoc. Off. Anal. Chem. (Vol. 65, No. 2, 1982), pp. 265-269
2. Official Methods of Analysis, Assoc. Off. Anal. Chem., 1984 Supplement, Secs. 7.066-7.069, pp. 160-161

METHOD HISTORY

Combined the Crude Fiber methods for Corn (A-8) and Feedstuffs (G-12) on 4-15-2011.

Corn, Crude Fiber (A-8), Date of Acceptance 10-24-1958, Revised 10-10-1985.

Feedstuffs, Crude Fiber (G-12), Date of Acceptance 10-24-1958, Revised 10-10-1985.