FAT, CRUDE (Hexane Extractables)

PRINCIPLE

Lipids are recovered by simultaneous milling and extraction with hexane in a sealed ball mill vial.

SCOPE

The method is applicable to feedstuffs, corn germ and other components derived from the milling of corn (Note 1).

SPECIAL APPARATUS

1. Mill: Model 8000 Spex Mixer/Mill, equipped with a Cat. No. 8001 hardened steel vial, or equivalent, for grinding, and both 12 mm and 6 mm steel balls.

2. Filter Funnel: Gelman Cat. No. 4201 (47 mm) or Cat. No. 4230 (47 mm), or equivalent.

3. Filter Paper: Whatman Cat. No. 7195-004, a cellulose nitrate 5 micron filter which is hexane compatible.

4. Bath: Steam-heated water bath in a well ventilated hood.

5. Oven: Forced air or convection oven, operating at 100 °C.

REAGENTS

1. n-Hexane: Reagent Grade or HPLC Grade (Note 2)

PROCEDURE

Grind a 50-100 g sample to a uniform particle size (10 mesh) in a grinder or cutting mill (Note 3). Mix the sample thoroughly and weigh accurately about 4-5 g into the steel grinding vial. Add one 12 mm and two 6 mm steel balls and 25 mL of n-hexane. Seal the vial and mill in the Mixer/Mill for 15 minutes. Prior to opening, cool vial in a cold water bath until vial cools to touch.

FAT, CRUDE (Hexane Extractables) — continued

Place a filter paper in the funnel and filter the milled sample slurry into a clean and dry vacuum flask (Note 4). Quantitatively transfer the residual oil from the steel milling vial and cap with the aid of three 10 mL portions of hexane, and wash residual oil from the sample with another three 10 mL portions of hexane. Transfer the filtered hexane and oil miscella into a dried and tared 150 mL beaker. Rinse the vacuum flask residue into the beaker with small portions of hexane.

Evaporate the solvent on a steam-heated water bath under the hood (Note 5). Place the beaker with residue in the air oven and dry it 1 hour at 100 °C (Note 6). Remove the beaker and cool in a desiccator. Weigh the beaker and calculate the dry extract weight.

CALCULATION

$$\%\ \text{Crude Fat (as is)} = \frac{\text{Dry Extract Wt. (g)} \times 100}{\text{Sample Wt. (g)}} \quad \text{(Note 7)}$$

NOTES AND PRECAUTIONS

1. The material for extraction should be at or below equilibrium moisture. Wet materials should be dried by a method which does not result in fat oxidation.

2. Hexane is a very flammable solvent and all work should be performed in a well ventilated area with no open flames. Always insure that the hood is functioning properly before use.

3. High oil content samples should be ground in a cutting mill (e.g., Brinkman Model ZM-1); low oil content samples may be ground in a cutting mill or attrition mill.

4. Alternatively, the flask can be weighed and used for solvent evaporation as described later, if desired. Also, take care that no hexane is aspirated out of the flask on low oil samples.

5. Care must be taken when evaporating hexane. If the hexane is heated at too high a rate, the evaporation will occur with occasional violent boiling which will result in the loss of crude fat or oil.

FAT, CRUDE (Hexane Extractables) — continued

6. Prolonged evaporation or drying of the extract at elevated temperatures may cause fat oxidation and high results.

7. It is customary to report the result as percent oil when analyzing corn germ samples.

METHOD HISTORY

Feedstuffs, Fat, Crude (Hexane Extractables), (G-11) Date of Acceptance 4-19-1989, Revised 10-08-2009.