Objective

These methods determine moisture content as loss in weight of a sample when heated under specified conditions. The results are in close agreement with those obtained by Method 44-40.01 (vacuum-oven). The methods are applicable to flour, semolina, bread, all kinds of grains and cereal products, and food products (except those that are sugar coated). These methods are not recommended for feeds and feedstuffs when fat determination is to be made on dried samples.

Apparatus

1. Laboratory mill, equipped with 18- or 20-mesh screen and 4-oz receiving bottle; should grind without undue exposure to atmosphere and without appreciable heating.
2. Oven (either gravity-convection or mechanical-convection), capable of being maintained at 130° (±1°) uniformly throughout oven and provided with good ventilation. Thermometer shall be so situated in oven that tip of bulb is level with top of moisture dishes but not directly over any dish.
3. Oven for drying corn and soybeans, meeting requirements of oven in item 2 except that it is maintained at 103° (±1°). Testing at 130° is not recommended for corn and soybeans.
4. Moisture dishes having diameter of about 55 mm and height of about 15 mm. They should be of heavy-gauge aluminum with slightly tapered sides and provided with tightly fitting slip-in covers that are designed to fit snugly under dishes when they are placed in oven. Both dish and cover should be identified by same number. Before using, dry for 1 hr at 130°, cool in desiccator, and obtain tare weight. See Note 1.
5. Airtight desiccator containing activated alumina, molecular sieves (type 4A or 4A X W), or other equally suitable desiccant. See Note 2.
6. Balance, accurate to at least 1 mg.

Procedure

One-stage

For samples containing less than 13% moisture, except soybeans, for which a level of 10% is applicable; also for flour, cornmeal, farina, and semolina except for grinding.
1. Grind 30- to 40-g sample in mill. Mix rapidly with spoon or spatula and transfer immediately a 2- to 3-g portion to each of two or more tared moisture dishes. Cover and weigh dishes at once. Subtract tare weights and record weight of sample. Clean mill between samples.
2. Uncover dishes and place them on shelf of oven; place covers under dishes. Insert shelf in oven at level of thermometer bulb. Heat for exactly 60 min after oven recovers its temperature See Note 3.
Moisture—Air-Oven Methods (continued)

3. Remove shelf and dishes from oven, cover rapidly (using rubber finger insulators), and transfer to desiccator as quickly as possible. Weigh dishes after they reach room temperature (45–60 min usually). Determine loss in weight as moisture. See equation 1. Replicate determinations must check within 0.2% moisture; otherwise repeat determination.

Two-stage

For samples containing 13% or more moisture (10% for soybeans), loss of moisture incident to grinding is likely to be excessive; hence, the following two-stage procedure should be used.

1. Fill two or more tared moisture dishes nearly full with representative portions of unground sample. Cover and weigh dishes. Subtract tare weights and record weight of sample.

2. Uncover dishes and place covers under dishes. Place tins in well-ventilated place (preferably on top of heated oven protected from dust) so that sample will dry reasonably fast and reach approximate air-dry condition. This will usually be accomplished in 14–16 hr when top of heated oven is used, or approximately 60 hr when room temperature is used for this preliminary drying. In all cases, moisture content should be reduced to less than 10%, ideally to 7–8% in air-drying stage.

3. Cover dishes containing air-dried samples and weigh them soon after they cool to room temperature. Determine loss in weight and record it as moisture loss due to air-drying, using equation 1.

4. Using air-dried sample, follow one-stage procedure described above, starting with grinding step. Calculate total moisture content by using equation 3.

Air-oven, for corn and soybeans, at 103°

Place approximately 15 g of representative portion of unground sample in each of two or more tared moisture dishes. Weigh covered dishes and contents. Subtract weight of each dish from total weight and record result as weight of sample. Put covers under dishes and heat for 72 hr in oven regulated at 103 ± 1°. Dishes should be placed on single shelf with bulb of oven thermometer as close as possible to them. At end of heating period, remove shelf containing dishes, cover dishes immediately, and place in desiccator. Weigh dishes when they reach room temperature. Determine loss in weight as moisture by using equation 1.

Replicate determinations should check within 0.2% moisture.

Air-oven, for flax, at 103°

Proceed as above for corn and soybeans, except use 5- to 7-g sample and 4-hr oven time.
Moisture—Air-Oven Methods (continued)

Bread, two-stage

Air-dry and grind sample as directed in Method 62-05.01. Using 2- to 3-g portion of ground air-dried sample, follow one-stage procedure described above. Calculate total moisture loss by using equation 2. To obtain percent of total solids in fresh loaf, subtract percent total moisture from 100.

Calculation

Equation 1 (one-stage and 103° air oven):

\[
\% \text{ Moisture} = \frac{A}{B} \times 100
\]

in which \( A \) = moisture loss in grams, \( B \) = original weight of sample.

Equation 2 (two-stage):

\[
\% \text{ Total solids} = \frac{Y \times Z}{X}
\]

where \( X \) = weight of original sample used for air-drying, \( Y \) = weight of sample after air-drying, \( Z \) = percent total solids in prepared ground sample (total solids = 100% – percent moisture at assay).

Equation 3:

\[
\% \text{ Total moisture} = A + \frac{(100 - A)B}{100}
\]

where \( A \) = percent moisture loss on air-drying, \( B \) = percent moisture loss as determined by oven-drying.

Notes

1. Tare weight will usually remain constant within a few tenths of mg for approximately 1 year if dishes are dumped and then carefully wiped clean with a soft cloth.
2. Silica gel and anhydrous CaCl₂ are not suitable desiccants.
3. Oven should regain temperature within 15–20 min after insertion of a full load (24 moisture dishes). If oven requires a longer time to recover, it should not be used.

References

Moisture—Air-Oven Methods (continued)


