MOISTURE (Azeotropic Distillation)

PRINCIPLE

Moisture is removed from the ground corn by distillaton as an azeotrope with toluene. The water is collected in a suitable trap and its volume is measured at a known temperature.

SCOPE

This method is applicable to corn of the various types and moisture contents found in commercial channels (Note1).

SPECIAL APPARATUS

1. Mill: Intermediate Wiley cutting mill (available from Fisher Scientific, Cat. No. 08-338, 08-338-1 or 08-338-3) with necessary accessories. The mill as received should be changed as follows: Drill a ¼ inch hole in the center of the lid of the hopper. Prepare a plunger of hard wood 6 inches long, 2 inches of the length being ½ inch in diameter and the remainder being a little less than ¼ inch, provided with a collar or other means to prevent the plunger falling into the knife blades.

   Other recommended equipment is available from several sources (see sketch).

2. Distilling Flask: KIMAX or PYREX short neck, flat bottom boiling flask, 500 mL capacity, with standard-taper 24/40 neck (e.g., Cat. No. F-4020-500 or F-4022-500, Baxter Scientific Products, 1430 Waukegan Rd., McGaw Park, Illinois 60085)

3. Moisture Trap: Bidwell & Sterling, PYREX, 5 mL capacity, with 0.1 mL graduations (Note 2), and with standard-taper 24/40 joints for connecting the distilling flask and reflux condenser (e.g., Cat. No. 7705, Ace Glass Incorporated, P.O. Box 688, 1430 Northwest Blvd., Vineland, New Jersey 08360)

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4. Condenser: PYREX, West-type with drip tip and 40 cm jacket. The bottom end has a standard-taper 24/40 joint for attaching the moisture trap; the upper end extends 3 inches beyond the jacket to allow a test tube closure during distillation.

5. Hot Plate-Stirrer: The unit should provide a maximum temperature (plate surface) of 800 °F and a stirring range of 250 to 1000 rpm. Stepless individual control of both temperature and stirring rate is necessary. (e.g., Cat. No. H2402-1, Baxter Scientific Products, 1430 Waukegan Rd., McGaw Park, Illinois 60085)

6. Stirring Bar: TEFLON coated, octagonal with molded pivot ring, 5/16 inch by 2 inches long (e.g., Cat. No. S-8311-9, Baxter Scientific Products, 1430 Waukegan Rd., McGaw Park, Illinois 60085)

REAGENTS

1. Toluene: Reagent Grade

PROCEDURE

If the moisture content is above 20%, it is necessary to predry before grinding. Fill previously dried and tared moisture dishes (with covers) with approximately 100 g of grain and weigh to the nearest 0.1 g. Place dishes (covers removed) in a warm well-ventilated place (on top of a heated oven) protected from dust so that the grain will dry reasonably fast and reach an approximate air-dried condition in 14-24 hours. Reweigh the dishes and calculate the grams of moisture lost.

Treat predried samples and those containing originally less than 20% moisture as follows:

Weigh accurately about 100 g of corn (Note 3) into a glass-stoppered weighing bottle. Transfer the corn to the Wiley mill hopper (Note 4) and replace the lid. Fix in position the 20 mesh screen, connect to a glass receiving bottle and tightly screw the glass plate to the face of the mill. Place a clean sheet of paper under the bottle in order to recover any falling particles.

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After the motor has been turned on, raise the wooden plug which seals the hopper and grind the sample completely.

The raising and lowering of this plug facilitates the steady flow of kernels. No kernels should be allowed to enter the mill unless the motor is on; otherwise the mill will become plugged.

Upon completion of the grinding, carefully disassemble the mill. Brush any dust in the hopper into the mill with a camel's-hair brush. Brush the dust accumulated on the rim of the glass plate onto the paper. Then clean the mill completely taking care to insure that all particles between the knife blades are removed. Transfer all ground material to the weighing bottle and weigh. Determine the weight lost in grinding (Note 5).

Dry a distilling flask overnight in an air oven at 100 °C. Weigh accurately about 25 g of well-mixed, ground sample and transfer to the distilling flask (Note 6). Add approximately 200 mL of toluene and place the flask and its contents on the hot plate-stirrer. Insert a predried stirring bar, and attach moisture trap and condenser supported by a suitable rack (Notes 7 and 8). Place a loose fitting test tube over the upper end of the condenser. Start stirrer and adjust speed to a rate that keeps the sample suspended but avoids splashing sample suspension on distilling flask walls. Apply heat so that distillation occurs at a rate providing about 2 drops of condensate per second to fall from the condenser tip. After 1 hour, increase distillation rate to give about 4 drops of condensate per second.

Continue the distillation until the volume of water in the trap ceases to increase (48 hours recommended). Wash down the condenser with about 10 mL of toluene and continue the distillation for 30 minutes.

Disconnect the trap and immerse the graduated portion containing the distillate in a water bath at 20 °C until the trap contents reach the bath temperature (15 minutes). Read the volume of water in the trap, estimating to the nearest hundredth of a milliliter.

Determine a blank by the above procedures substituting about 3 g of water, weighed accurately, for the sample.

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CALCULATION

For samples predried: (Note 9)

$$\% \text{ Moisture} = \frac{[(\text{mL } H_2O \text{ in Trap + Blank})\frac{C}{D} + (B - C) + (A - B)] \times 100}{A}$$

For samples not predried:

$$\% \text{ Moisture} = \frac{[(\text{mL } H_2O \text{ in Trap + Blank})\frac{C}{D} + (B - C)] \times 100}{B}$$

Blank = Water Added (g) – Water Recovered (mL) (Note 10)

Where:

A	=	Sample weight (g) to be predried
B	=	Sample weight (g) after predrying or to be ground
C	=	Sample weight (g) after grinding
D	=	Sample weight (g) taken for distillation
A—B	=	Moisture loss (g) during predrying
B—C	=	Moisture loss (g) during grinding

NOTES AND PRECAUTIONS

1. At the time of its development during the 1940s, the azeotropic distillation procedure was considered to be the most accurate for the determination of moisture in products tending to be heat labile. Consequently, the method has been and is now employed as a referee technique to establish conditions for moisture measurement by simplified methods used for control analysis.

2. Traps may be calibrated by adding weighed quantities of water at 1 mL intervals and layering with toluene to give the meniscus proper shape.

3. Because subsequent milling results in moisture and mechanical losses which should be maintained relatively small and constant, a 100 g sample should be ground although this amount is not required for later analysis.

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4. The Wiley mill is of the shearing type and very little heating occurs during grinding. However, to prevent moisture loss when grinding a series of samples, allow the mill to cool between samples.

5. The grinding loss will vary from 0.2 to 0.4%, the amount depending on the moisture in the corn and the skill of the operator.

6. The weight of sample selected for analysis depends on the moisture content and should provide between 3 and 5 mL of water.

7. The traps and condensers must be scrupulously clean and dry. A recommended procedure for their preparation follows:

   Traps: Thoroughly scrub with hot water containing a good detergent, using a small tube brush to scrub the graduated portion. Flush thoroughly with purified water. At this point the water should drain uniformly from the inner surface of the trap. Then close the bottom standard-taper joint with a cap, fill the trap with hot cleaning solution and allow to stand for 2 hours.

   Empty the trap, rinse thoroughly with purified water, fill with 1% sodium hydroxide solution, and allow to stand 15 minutes. Remove the sodium hydroxide solution and rinse the trap at least 5 times with purified water. Invert trap at an angle under an infrared lamp to drain and dry.

   Condensers: Clean in a similar manner.

8. Before assembling the apparatus (see sketch), it is advisable to place a number of marks on the standard-taper joints with a soft lead pencil. This will reduce the tendency of the joints to freeze. To insure against moisture vapor loss through the standard-taper joint of the distilling flask, the joint may be lubricated with C.P. xylene.

9. The entire predried sample is ground.

10. Because a correction for the density of water in the trap would be less than the precision with which the volume can be read, such a correction can be

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ignored in the calculation. The blank should not exceed 0.06 mL which represents the solubility of water in the quantity of toluene specified.

REFERENCES

1. Fetzer, W. R., Determination of Moisture by Distillation. Anal. Chem.*1951*, 23 (8), 1062 – 1069 (and references cited therein).

2. Sair, L. and Fetzer, W. R., The Determination of Moisture in the Corn Wet Milling Industry, Parts I-VI, Cereal Chem., *1942*, 19 (5), 633 - 720.

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