MOISTURE (Oven)

PRINCIPLE

Moisture in a weighed sample is removed by heating in an oven under specified conditions of time, temperature and vacuum and the residual dry substance weighed (Note 1). The weight loss is calculated as moisture.

SCOPE

This method has a procedure applicable to corn starch, corn sugar, corn syrup, dextrin, and feedstuffs derived from corn (Note 2, Note 3). The method also has a procedure for all corn syrups.

SPECIAL APPARATUS

1. Oven: Use a good quality vacuum oven having uniform heat distribution and capable of retaining vacuum for several hours after the pump is shut off. (The oven shelves may be soldered to the wall retaining sleeve to aid in heat transfer to the sample dishes.)

2. Vacuum Pump: A vacuum supply such as a laboratory vacuum pump capable of maintaining an oven pressure during operation not in excess of 100 Torr is necessary.

3. Drying Train: A drying tower filled with indicating "Drierite" is attached to the air inlet of the oven. The tower is connected in series to a gas scrubber containing concentrated sulfuric acid.

4. Moisture Dishes: Metal dishes, preferably aluminum, approximately 2 inches in diameter and equipped with covers, are recommended.

5. Aluminum Desicoolers (Fisher Scientific Company): Dishes are 3 inches high and 3.5 inches in diameter. Remove handle from cover and close the opening with a rivet or by other means. Remove the inner tray from the Desicooler.

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6. Stirrers: Pyrex test tubes, 100 x 13 mm, are equipped with extensions which are made with stainless steel rods (8 x 180 mm). The rods are fitted near one end with two rubber rings cut from tubing with the rings being so spaced on the rod that when it is inserted into the test tube a snug fit is obtained at the top and bottom of the test tube.

7. Weighing Bottles: 45 mL glass weighing bottles (40 x 50 mm) with cap style ground stoppers.

REAGENTS

Hyflo Super-Cel, a Celite Diatomite Filter Aid sold by Johns-Manville Products Corporation, Lompoc, CA (Note 4): Wash a large quantity of the filter aid by percolation on a Buchner funnel with purified water acidified with hydrochloric acid (1 mL concentrated hydrochloric acid per liter of purified water). Continue washing until the effluent is acid to litmus. Then wash with purified water until the effluent pH is 4 or above. Air dry the washed filter aid for storage.

When the air dried filter aid is to be used, dry a quantity overnight in an air oven at 105 °C and keep in a closed container.

PROCEDURE

If required, grind sample (Note 5) completely through a laboratory cutting mill to 20 mesh or finer, taking precautions to prevent significant loss of moisture, and mix thoroughly.

METHOD PARAMETERS TABLE

Sample Type	Sample Weight (g)	Vacuum Oven Temperature	Vacuun Oven Pressure	Time in Vacuum Oven	Notes
Dextrin	4-6	120°C	100 Torr	4 hours	1,2,4
Corn Starch	4-6	120°C	100 Torr	4 hours	1,2,4
Corn Syrup	7-10	70°C, 100°C	100 Torr	20 hours	
Dextrose,hydrate	4-6	70°C	100 Torr	2 hours	1
Dextrose,anhydrous	9-11	70°C	100 Torr	2 hours	1
Feedstuffs	4-6	80°C	100 Torr	20 hours	1-4

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Weigh accurately, to the nearest 0.0001 g, the specified amount of sample into a predried, cooled and tared moisture dish. Place dish and cover (cover removed) in vacuum oven operating at the specified temperature and maintain at the specified pressure for the specified time.

While sample is drying, bleed a small stream of air through the drying train and oven.
Shut off the vacuum line and slowly fill the oven with air drawn through the drying train. Open oven, quickly close dish with cover, place in desiccator until cool (30 minutes are usually sufficient) and weigh.

For corn syrups:

Add approximately 30 g of previously dried filter aid to two or more of the sample dishes. One of these dishes is used as a blank and the others are used for samples (Note 6). Place stirrers (test tubes) without the extension rods in the sample dishes. Place sample dishes and the blank (covers removed) in the vacuum oven at 100 °C and at a pressure not in excess of 25 torr. While the dishes are drying, bleed a small stream of air through the oven and drying train. After 5 hours, shut off the vacuum and fill the oven slowly with air drawn through the drying train. Open the oven, quickly close the sample dishes and the blank, place in a desiccator and cool to room temperature (Note 7). Release the closure of the sample dishes and the blank momentarily before weighing. Record the weights to the nearest milligram.

Weigh accurately 7 to 10 g of sample (4 to 7 g dry substance) in a 45 mL (40 x 50 mm) weighing bottle equipped with a cap style ground stopper (Note 8). Add 10 mL of warm purified water and stir thoroughly with a small glass rod. Pour the diluted sample onto the filter aid in the sample dish and complete the transfer of the sample quantitatively with three 5 mL portions of warm purified water. Insert the steel extension rod into the stirring tube in sample dish and stir until the sample is homogeneously dispersed throughout the filter aid (Note 9). Remove

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the rod, leaving the stirring tube in the dish and place the blank and sample dishes (with covers off) in the vacuum oven adjusted to operate at 70 °C for corn syrups over 58 D.E. and at 100 °C for corn syrups of 58 D.E. and below (Note 10).

After 5 hours in the vaccum oven, using conditions described previously, remove sample dishes from the oven, insert the stirring extension rods into the stirring tubes and rework the filter aid until a fine powder free of lumps is obtained (Note 11).

Return dishes containing samples to the oven and heat for 15-16 additional hours using conditions described previously.

Shut off the vacuum line and slowly fill the oven with air drawn through the drying train. Open the oven, quickly place covers on the blank and sample dishes and place in desiccators. Cool blank and sample dishes to room temperature (Note 7) and weigh as before. Record blank weight change (Note 6).

CALCULATION

$$\% \text{ Moisture} = \frac{(\text{Sample Wt. (g)} - \text{Dry Sample Wt. (g)}) \times 100}{\text{Sample Wt. (g)}}$$

NOTES AND PRECAUTIONS

1. Gravity convection and forced-draft air ovens are sometimes substituted for the vacuum oven. However, moisture values are generally 0.1 to 0.3 percent lower than those obtained in a vacuum oven.

2. This procedure is not applicable to highly-modified starches which show evidence of decomposition (usually discoloration) under the conditions specified. When analyzing samples containing volatiles in addition to water, the results should be expressed as "loss on drying" rather than moisture.

3.

MOISTURE (Oven) — continued

Many corn-derived by-product feedstuffs contain heat-labile components. Prolonged exposure to elevated temperatures may cause decomposition or interaction with other components and effect loss of water not present originally as sorbed moisture.
For this reason, temperature, time and pressure details of this procedure must be followed rigorously to obtain the desired precision and accuracy. This method produces results in good agreement with those obtained by the azeotropic distillation procedure using benzene (Method G-14).
CRA Analytical Method G-17 is recommended for higher moisture feed**.**

4. Do not substitute a commercial "acid-washed" diatomaceous earth filter aid for the Hyflo Super-Cel. Erroneous results may be obtained when using other filter aids.

5. Samples with particles larger than 20 mesh or samples containing hard granular pellets should be ground.

6. A significant change in weight of the blank between the initial and final weighing indicates insufficient drying of the filter aid and the tests should be repeated.

7. The dishes must be cooled in the desiccator to room temperature prior to removing them for weighing. This may require 1 hour or more when several Desicoolers are in the same desiccator.

8. Use of the weighing bottle with cap style ground stopper avoids evaporation losses which might be encountered during weighing of the sample. Quickly add the approximate weight of sample to the tared weighing bottle, close with the stopper and obtain accurate weight of sample. Alternatively, for routine analyses a 100 mL metal weighing scoop may be used. The weighing should be made as quickly as possible to minimize any weight loss by evaporation of the water contained in sample during transfer and weighing of the sample.

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9. The success of the determination depends upon the thoroughness with which the sample is incorporated in the filter aid. Consequently, time required for thoroughness in mixing at this stage is well spent.

10. All fructose-containing syrups should be dried at 70 °C regardless of the D.E. of the syrup. The D.E. of the syrups or sugars may be determined by using Method E-26, Standard Analytical Methods of the Member Companies of the Corn Refiners Association, Inc. A preliminary dry substance level for the purpose of calculating the D.E. may be obtained by drying the sample at 70 °C. If the D.E. of the sample is below 58, the true dry substance level should be determined by drying at 100 °C.

11. For routine determination of moisture or dry substance, the initial removal of water (5 hour drying period) may be accomplished in a vacuum oven at the specified temperature, using a water aspirator or other laboratory vacuum supply capable of maintaining a pressure of 50 to 75 torr.

METHOD HISTORY

Combined the Moisture (Oven) methods for Corn Starch (Unmodified) (B-38), Dextrin (D-38), Corn Syrup (E-42), Corn Syrup (E-44), Corn Sugar (F-34) and Feedstuffs (G-16) on 11-09-2010.

Corn Starch (Unmodified), Moisture (Oven) (B-38), Date of Acceptance 11-26-1956, Revised 4-28-1987.

Dextrin, Moisture (Oven) (D-38), Date of Acceptance 6-21-1974, Revised 10-08-1996.

Corn Syrup, Moisture (Oven) (E-42), Date of Acceptance 4-05-1954, Revised 10-17-1989.

Corn Syrup, Moisture (Oven) (E-44), Date of Acceptance 11-26-1956, Revised 10-17-1989.

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Corn Sugar, Moisture (Oven) (F-34), Date of Acceptance 6-08-1959, Revised 9-30-1997.

Feedstuffs, Moisture (Oven) (G-16), Date of Acceptance 11-18-1957, Revised 10-20-1987.