

MOISTURE (Oven-Filter Aid)**PRINCIPLE**

A weighed sample is diluted with water and dispersed on a diatomaceous earth to provide a large surface area for drying. The moisture is then removed by heating under high vacuum and the residual dry substance weighed. The weight loss is then calculated as moisture.

SCOPE

The method is applicable to all corn syrups.

SPECIAL APPARATUS

1. Oven: Use a good quality vacuum oven, with 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: Use a laboratory vacuum pump capable of maintaining an oven pressure during operation not in excess of 25 torr.
3. Drying Train: Attach a drying tower filled with indicating DRIERITE to the air inlet of the oven. Connect the tower in series to a gas scrubber containing concentrated sulfuric acid.
4. Aluminum Desiccators (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 Desiccator.
5. 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.

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6. 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 1): 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

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 2). 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 3). 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 4). 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 5). 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 6).

After 5 hours in the vacuum 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 7).

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 3) and weigh as before. Record blank weight change (Note 2).

CALCULATION

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

$$\% \text{ Moisture} = 100\% - \text{Dry Substance}$$

NOTES AND PRECAUTIONS

1. 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.
2. 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.
3. 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.

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4. 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.
5. 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.
6. 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.
7. 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.