

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

A weighed sample is diluted with water and absorbed on a filter paper coil to provide a large surface area for drying. The moisture is then removed by heating under high vacuum and, after cooling, the residual dry substance is weighed. The weight loss is calculated as moisture.

SCOPE

The method is applicable to all corn syrups and, with minor modification, to refined dextrose.

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: 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 filled with concentrated sulfuric acid.
4. Weighing Bottles: 50 x 60 mm, high form, with ground glass stopper
5. Filter Paper Coil: The coil consists of a strip of Whatman No. 1 filter paper, 1.75 x 20 inches, with a pH less than 7 (Note 1), and a corrugated strip of phosphorbronze (or equivalent), 0.5 x 17.5 inches, No. 36 B & S gauge, made by running the strip through meshed gears producing turns at 3 mm intervals.

MOISTURE (Oven-Filter Paper) — continued

Prepare the filter paper coil by rolling a strip of filter paper onto the corrugated separator, the paper being longer and wider than the separator, in order that the ends and outer layers of the coil will consist of filter paper. The coil is held together by wrapping with a short length of fine copper wire, twisting the ends and leaving about 0.25 inch for handling the coil.

PROCEDURE

Place coil in weighing bottle and dry 1 hour in vacuum oven at 100 °C, cool 30 minutes in desiccator and weigh (Note 2).

Remove coil and weigh accurately about 1 g of sample in weighing bottle. Add 2 mL of purified water to the sample, warm and mix by swirling to form a dilute solution. Introduce paper coil and allow it to absorb the sample solution; take up last trace by shaking coil across bottom of weighing bottle (Note 3). Place cover at an angle on top of bottle containing sample, to allow free escape of moisture, and place in an air oven at 70 °C for 30 minutes. Transfer to vacuum oven operating at the specified temperature (Note 4) and maintain at a pressure not in excess of 25 torr for 7 hours. While 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 bottle with cover, place in desiccator until cool (30 minutes usually sufficient) and weigh.

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. The filter paper must show a pH less than 7 when an indicator is applied to it. This precaution is sufficient when corn syrup is dried because it contains a small amount of residual acid and it is fairly well buffered. However, when pure dextrose solutions are dried, it often becomes necessary to

MOISTURE (Oven-Filter Paper) — continued

acidify the solution with an acid such as lactic in order to prevent decomposition.

2. The weighing bottle must always be closed when transferred from oven to desiccator, to prevent adsorption of moisture by either the filter paper or dried sample, both of which are quite hygroscopic.
3. The weighing bottle should not be placed in the vacuum oven until the filter paper has thoroughly absorbed the sample solution, as evidenced by the disappearance of excess where the edge of the filter paper touches the bottom of the weighing bottle. Normally, 30 minutes should be allowed for absorption.
4. Use an oven temperature of 70 °C for drying all corn syrups having a D.E. above 58, and all fructose containing syrups. Use an oven temperature of 100 C for all other syrups. The D.E. of the syrup may be determined by using procedure E-26, Standard Analytical Methods of the Member Companies of the Corn Refiners Association, Inc.