MOISTURE (Azeotropic Distillation)

PRINCIPLE

Moisture is removed from the sample by distillation 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 all unmodified starches, most modified starches and many starch products (Note 1).

SPECIAL APPARATUS

Recommended equipment for azeotropic distillation is available from several sources (see sketch).

1. Distilling Flask: KIMAX or PYREX short neck, flat bottom boiling flask, 500 mL capacity, with standard-taper 24/40 neck (e.g., Cat. No. F4020-500 or F4022-500, Baxter Scientific Products, 1430 Waukegan Rd., McGaw Park, Illinois 60085)

2. 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)

3. 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.

4. 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.,

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Cat. No. H2402-1, Baxter Scientific Products, 1430 Waukegan Rd., McGaw Park, Illinois 60085)

5. 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

Grind samples containing coarse particles; taking precautions to prevent a significant change in moisture content.

Dry distilling flask overnight in an air oven at 100 °C. Weigh accurately about 30 g of sample and transfer to the distilling flask (Note 3). Add approximately 200 mL of toluene and place the flask and its contents on the hot plate-stirrer. Insert a predried stirring bar; attach moisture trap and condenser supported by a suitable rack (Notes 4 and 5). Place a loose-fitting test tube over the upper end of the condenser. Start stirrer and adjust speed to a rate that keeps 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 (8 hours usually sufficient). 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 procedure substituting about 3 g of water, weighed accurately, for the sample.

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CALCULATION

$$\% \text{ Moisture} = \frac{(\text{Water in Trap, mL} + \text{Blank, mL}) \times 100}{\text{Sample Wt. (g)}}$$

Blank = Water added (g) – Water Recovered (mL) (Note 6)

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

4. 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.

   Condenser: Clean in a similar manner.

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5. 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.

6. 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 ignored in the calculation. The blank should not exceed 0.06 mL which represents 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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