
Moisture (Distillation Method)

Purpose: To determine the moisture in all spice by co-distillation with toluene with the exception of capsicums and dehydrated vegetables. (Note 1)

A. Apparatus:

1. Glass distillation apparatus with ground glass joints constructed and assembled as shown in the diagram.
 - a. 500 mL or 1000 mL, round bottom, shortneck flask with a T.S. 24/40 joint.
 - b. West condenser with drip tip, 400 mm in length, with a T.S. 24/40 joint.
 - c. Bidwell-Sterling trap, T.S. 24/40 joint, 5 mL capacity graduated in 0.1 mL intervals.
2. Heat source capable of refluxing solvent in apparatus above. An electric heating mantel supported by a variable speed stirring plate and an egg shape Teflon covered stir bar is recommended. If not using stirring plate add boiling chips.
3. Nylon bristle burette brush, ½ inch in diameter, or a wire loop. It should be long enough to extend through the condenser.
4. Analytical Balance, sensitivity 0.01g.

B. Reagents:

1. Toluene, ACS grade. (Note 1)

C. Preparation of Sample:

1. Use Method 1.0

D. Procedure:

1. Weigh sample to the nearest 0.01 g sufficient to yield 2 - 4 mL of water (about 40 g).
2. Quantitatively transfer to a distillation flask, add sufficient toluene to cover the sample completely and to middle of distillation flask. Add a stir bar or boiling chips.
3. Assemble apparatus to heating mantle as shown in Figure. Fill trap with toluene by pouring through condenser until it just fills the trap and begins to flow into the flask. (Note 2)

4. Bring to a boil and reflux at a rate of about two drops per second until most of the water has been collected in the trap, then increase the reflux rate to about four drops per second.
5. Continue refluxing until two consecutive readings 15 minutes apart show no change (about three hours). Turn off heat and allow to cool to ambient temperature. Dislodge any water held up in the condenser with a brush or wire loop. Rinse carefully with about 5 mL toluene.
6. Read volume of water in trap. (Note 3 & 4)

E. Calculation:

$$1. \quad \text{Moisture \%} = \frac{\text{Volume of Water (mL)}}{\text{Correction Factor}} \times \frac{100}{\text{Weight of sample (g)}}$$

$$\text{Correction Factor} = \frac{\text{mL distilled}}{\text{mL added}}$$

F. Statistics:

Coefficient of Variation:

Capsicum	11%
Basil	8%

G. Notes:

1. Sugars decompose and produce water under prolonged heating in toluene or other solvents with boiling point over 100°C (Fetzer, 1951). Therefore, ASTA recommends Method 2.1 for capsicums, onion, garlic and other spices containing large amounts of sugar. However, if method 2.0 must be used for these materials, hexane should be substituted for toluene. The method typically requires longer distillation time (five hour minimum) and will have greater variability when hexane is used in place of toluene.
2. Inserting a loose cotton or glasswool plug in top of condenser can prevent condensation of ambient moisture.
3. A correction blank for toluene must be conducted periodically by adding 1.00 mL of distilled water to 150 mL toluene in distillation flask and run method as described in D1 through D6 above.

4. Detergents such as Fisher Scientific Company concentrate RBS-2S may be used to clean the traps. It is recommended to rinse the trap with acetone before soaking in the detergent.

H. Reference:

AOAC Official Methods of Analysis (1995) 43.1.03 (941.11) & 43.1.04 (986.21).

JAOAC, 24,667 (1941); 70,391 (1987); 69,834 (1986).

Fetzer, W.R. "Determination of Moisture by Distillation," *Analytical Chemistry* **1951**, 23, 1062-1069)

I. Revision History

07/01/11 Revised Note 1 to clarify reason for use of lower temperature methods and added Fetzer reference. Strengthened recommendation for alternate use of hexane.

