Steam Volatile Oil in Cassia

Purpose: To determine the amount of water insoluble steam volatile oil in cassia (see Note 8).

Principle: Volatile oil is collected by steam distillation in a receiver trap with a small reservoir of xylene and measured volumetrically.

A. Apparatus:

1. One liter, T.S. 24/40 short neck, round-bottom flask.
2. Electric heating mantle for one liter flask.
3. Variable voltage transformer to regulate the heating mantle.
4. Volatile oil trap, Clevenger, 5 mL, or distilling receivers crankcase oil, 5.0 x 0.1 mL subdivisions, ASTM standard D322.
5. West condenser with T.S. 24/40 drip tip.
6. 2.00 mL (TD) volumetric pipette.
7. Balance, top loader, readable to 0.01 g.

B. Reagents:

1. Xylenes, ACS reagent grade.
2. Sodium chloride, technical grade minimum

C. Preparation of Sample:

1. Use Method 1.0.

D. Procedure:

1. Cleaned trap (Note 3) is rinsed with acetone and then rinsed well with water.
2. Weigh 35 g sample to nearest 0.01 g and quantitatively transfer to the one liter flask.
3. Add 500 mL of 10% sodium chloride solution. (Note 4)
4. Add small amount of water to the trap followed by 2.00 mL xylenes using the volumetric pipette.

5. Assemble the apparatus, using a small amount of non-volatile stopcock grease on the ground joints. (Note 5)

6. Heat the flask and maintain a distillation rate of 30 drops/minute for 5 hours after boiling begins. (Note 6)

7. Cool the trap to 20°C by placing it in a suitable water bath and hold until the oil layer is clear. Read the trap to the nearest 0.05 mL (Note 9). If the trap is read at some temperature other than 20°C, this fact should be noted in the report. The volume of the oil is obtained by deducting the blank for xylenes. (Note 2)

E. Calculation:

$$\text{Volatile oil, \% (v/w)} = \frac{\text{Vol. of oil (mL)} - \text{Vol. of xylene in blank (mL)}}{\text{Wt. of sample (g)}} \text{X } 100$$

ASTA recommends the use of a maximum allowable variation (MAV) decision procedure for acceptance or rejection of cassia on volatile oil content (Note 8).

F. Statistics:

Coefficient of Variation or relative standard deviation = ±16%. The coefficient of variation is an important factor to consider when setting tolerance limits for product acceptability.

G. Notes:

1. Traps should be calibrated for volumetric accuracy by adding water to the trap and then adding xylenes from a class A buret.

2. A xylenes blank must be run for each trap by performing the analysis as specified, omitting the cassia. This should be done in triplicate and an average taken. This calibration procedure should be performed once a year. The volume of the blank must be subtracted from the volume of volatile oil collected in the analysis.

3. Alkaline laboratory detergents that are designed to remove organic material and decontaminate glassware may be used to clean the traps. It is recommended to rinse the trap with acetone after use and then soak in the detergent solution for at least 12 hours followed by water rinsing. Make sure the trap is clean by adding some distilled water into the trap to see that the water flows down the wall of the trap smoothly. If not, repeat the cleaning until it does.

4. Salt solution is used to provide a higher boiling temperature than water.

5. The neck of the trap should be insulated with glass wool, aluminum foil or equivalent to reduce refluxing at the neck.

6. If foaming occurs at the beginning of the distillation, heat should momentarily be removed or the flask swirled vigorously. The water used to cool the condenser should be cold enough to keep the reflux ring lower than 1/3 of the full condenser height.

7. Percent VO calculated with this method is based upon total mass of sample and, therefore, will be theoretically lower than values obtained by ISO method 6571 ("Spices, condiments and herbs–determination of volatile oil content"), which expresses percent VO as the volume of oil per dry mass of herb or spice. Other small differences in results between these methods could result from differences in trap design, sample grinding, distillation time and conditions for reading volumetric results.

8. Maximum allowable variation (MAV) decision procedure for volatile oil: The arithmetic average of the five subsample volatile oil results shall be at or above the volatile oil specified in the contract. No single result shall be less than 90% of the contract value and no more than two subsamples shall lie between 90% of the contract value and the contract value.

9. AOAC Official Method 962.17 refers to reading to the nearest 0.01ml but the ASTA method sub-committee considers that reading to the nearest 0.05ml is more suitable for the accuracy of the typical glassware used in the Spice Industry and unifies all ASTA methods for the measurement of VO.

H. Reference:

AOAC Official Methods of Analysis 962.17.

I. Revision History

3/22/10 Revised formula to show correction for use of xylene. Clarified the importance of the coefficient of variation. Changed Note 2 to calibrate traps with xylene instead of toluene and to use a class A buret rather than National Bureau of Standards certified buret. Revised Note 4 to specify use of alkaline detergent and provide visual inspection of cleanliness. Added Note 5 to explain reason for salt solution. Revised Note 7 with instruction to keep condenser from overheating. Added Note 8 to explain differences between this method and ISO method 6571.

10/2013 Added Principle section. Added definition of the maximum allowable variation (MAV) decision procedure for acceptance or rejection of cassia on volatile oil content (Note 8). See "Raw Cassia and Cinnamon – Sampling and Testing Program," *ASTA Technical Bulletin* for additional background on MAV.

Revised the reading accuracy (procedure section 7) to the nearest 0.05mL to match the accuracy of the typical glassware used in the Spice Industry and unify all ASTA methods for the measurement of VO. Clarified equipment description in section A-4, removed note 1 and re-numbered the note section accordingly.