Steam Volatile Oil in Spices

Purpose: To determine the amount of steam volatile oil in spices. (Based upon AOAC method 962.17. See Note 3.)

A. Apparatus:

1. Erlenmeyer flask, 2 L. with a T.S. 24/40 ground joint.
2. Stirring hotplate suitable for 2,000 mL flask size with variable voltage transformer.
3. Volatile oil trap, clevenger, 5 mL, without stopcock, or distilling receivers crankcase oil, 5.0 x 0.1 mL subdivisions, ASTM standard D322.
4. West condenser, 400 mm length with drip tip and T.S. 24/40 ground joints.
5. Brinkmann, Retsch centrifugal grinding mill model ZM-1 or an equivalent cold grinder.
6. Pipet, 2 mL, TD.
7. Stirring bars, Teflon-coated, egg-shaped.
8. Pipet filler.
9. Cylinder, corning graduated metric cylinder, 2,000 mL.
10. Funnels, stainless steel.
11. Tubing, tygon tubing Norton, I.D. in 5/16 or equivalent.
12. Refrigerated circulating bath.
13. Insulating tape.

B. Reagents:

1. Xylene, ACS grade.
2. Sodium chloride. (NaCl)
3. Antifoam B, Dow Corning or equivalent.

C. Preparation of Sample:

1. Use Method 1.0. Ginger and Turmeric samples must be cracked before grinding.

D. Procedure:

1. Weight sample as specified in Table 1 and transfer quantitatively to the flask.

2. Add about 1,200 mL of water or 10% sodium chloride solution (for higher boiling temperature) as specified in Table 1 and add 2-3 drops of antifoam. Insert egg-shaped stir bar.

3. Place the trap (Note 1) on the flask using a small amount of non-volatile stopcock grease on ground joints. Fill the collection arms with water or 10% NaC1 as specified in Table 1. For allspice, anise and cloves, it is necessary to add trap additives to the graduated portion.

4. Accurately deliver 2 mL of xylene using a 2 mL pipet and verify added quantity using trap calibration (Note 2). Repeat the analysis if part of the xylene passes back into the flask during distillation.

5. Connect to the condenser using a small amount of non-volatile stopcock grease and connect the condenser to the refrigerated circulating bath at 20° C. Insulate the neck of the trap and flask with insulating tape.

6. Heat the flask on the stirring hot plate with constant stirring. Adjust the trap and condenser so that drops flow down the wall of the trap and do not drop directly on the surface of the liquid. Gently swirl the flask several times during initial heating until the sample is at a rolling boil to eliminate scorching of the sample on the walls. Maintain a reflux rate of 5 to 15 drops per 10 seconds. 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. Distill for eight (8) hours or until two (2) consecutive readings taken at 1 hour intervals show no change of oil volume in the trap. If the separation of oil is not satisfactory, add a drop or two of dishwashing liquid through the condenser or a few mL of a saturated sodium chloride to the trap.

7. Cool at 20° C either by allowing to stand in air or immersing the trap in a water bath. If the trap is read at some temperature other than 20° C, this fact should be noted in the report. Read the volume of oil collected to the nearest 0.01 mL.

E. Calculations:

$$\text{Volatile oil \% (v/w)} = \frac{\text{Vol. of oil (mL) 20°C}}{\text{Wt. of sample (g)}} \times 100$$

When 2 mL of xylene is used (Note 2), calculate the volatile oil by:

$$\frac{\text{Vol. of oil (mL) - vol. of xylene in blank}}{\text{Wt. of sample (g)}} \times 100$$

Wt. of sample (g)

F. Statistics:

See AOAC Official Method 962.17 and references therein.

G. Notes:

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

2. Calibrate each trap for the retention of xylene during distillation by running three blanks and taking the average volume. Repeat the procedure one time per year. This volume must be subtracted from the volume of volatile oil collected from the distillation as shown in the calculations.

3. 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.

H. Reference:

AOAC Official Methods of Analysis 962.17.

I. Revision History

3/22/10 Updated references to AOAC method 962.17 and revised Apparatus section to make trap description consistent with AOAC method. Revised Proc. Step 2 to explain reason for salt solution. Revised Proc. Step 6 with instruction to keep condenser from overheating. Revised Note 1 to specify trap volume calibration and cleaning procedure (replaced use of chromic acid with alkaline detergent). Added Note 3 to explain differences between this method and ISO method 6571.

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TABLE 1

SPICE	SAMPLE WEIGHT (g)	DISTILLATION SOLVENT	TRAP ADDITIVES
Allspice	40	10% sodium chloride	2 mL xylene
Anise	50	10% sodium chloride	2 mL xylene
Basil	50	Water	----------
Bay Leaves	60	Water	----------
Caraway	50	Water	----------
Cardamom	50	Water	----------
Celery Seed	50	Water	----------
Cloves	10	10% sodium chloride	2 mL xylene
Coriander	70	Water	----------
Cumin	50	Water	----------
Dill Seed	50	Water	----------
Fennel	50	Water	----------
Fenugreek	50	Water	----------
Ginger	50	10% sodium chloride	----------
Marjoram	40	Water	----------
Nutmeg	30	Water	----------
Mace	20	Water	----------
Oregano	40	Water	----------
Black Pepper	50	Water	----------
White Pepper	40	Water	----------
Rosemary	50	Water	----------
Sage	50	Water	----------
Savory	40	Water	----------
Tarragon	40	Water	----------
Thyme	50	Water	----------
Turmeric	50	Water	----------