Steam Volatile Oil (Lee and Ogg Method)

Purpose: To determine the amount of water insoluble steam volatile oil (see Note 7).

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 traps as shown in Fig. 1.
   (a) Lighter-than-water designed for oils with densities less than that of water.
   (b) Heavier-than-water designed for oils with densities greater than water. (Note 1)

5. West condenser, 400 mm in length, with T.S. 24/40 drip tip.

B. Reagents:

1. Antifoam agent (Dow Corning Antifoam B or equivalent containing no volatile oil).

2. Sodium alkylbenzene sulfonate

3. Xylene, ACS grade.

C. Preparation of Sample:

1. Use method 1.0.

D. Procedure:

1. Weigh a sample that will yield 0.5 - 1.5 mL of oil and transfer quantitatively to the one liter flask.

2. Add about 500 mL water and place the flask in the heating mantle (See Note 2 & 3 for cassia).

3. Assemble the apparatus as shown in Fig. 2, using the proper trap.

4. Heat the flask with occasional shaking and maintain a distillation rate of 1 to 1½ drops per second. If foaming occurs, reduce the voltage, and increase the temperature gradually until the proper distillation is obtained. (Note 4)

5. Distill until two consecutive readings taken at half hour intervals show no change of oil volume in the trap. (Note 5 & 6)

6. Cool to the room temperature, allow to stand until the oil layer is clear and read the volume of the oil collected, estimated to the nearest 0.05 mL. If the calculated volume of oil is below 0.5 mL or above 1.5 mL, the test should be repeated with appropriate adjustment to the amount of sample used (see Step 1 above).

E. Calculation:

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

When xylene is used (Note 3), calculate the volatile oil by:

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

F. Statistics:

Coefficient of Variation (relative standard deviation):

Nutmeg	±2.5%
Caraway	±3.5%
Allspice	±2.1%

The coefficient of variance 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 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. In the analysis of cassia, under procedure, step 2, add 500 mL 10% sodium chloride solution instead of water to raise the boiling point of the pot. Note, however, Method 16.0 is specified (and preferred) for the determination of steam volatile oil in cassia.

3. When the density of the volatile oil is nearly one, as for example clove oil, the samples are best handled by distillation in the lighter-than-water trap. Prior to distillation, add 0.6 mL of xylene to the graduated trap. 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.

4. If foaming occurs, use a non-volatile anti-foaming agent such as specified in B. Reagents. 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.

5. If the water and oil do not separate sharply, allow to stand or centrifuge until separation is obtained.

6. If difficulty is encountered in holding the oil in the trap as in the determination of celery seed oil, add a concentrated aqueous solution of sodium alkylbenzene sulfonate dropwise through the condenser tube. Three to five drops are usually sufficient. Distillation must be continued for 10 minutes after the addition of the detergent.

7. Percent VO calculated with this method is based upon <u>total</u> 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 <u>dry</u> 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 and reference cited.

I. Revision History

3/22/10 Added instruction to repeat measurement if result falls outside of specified test range (Proc. Step 6). Added formula to explain how to correct for use of xylene. Clarified the importance of the coefficient of variation. Revised Note 1 to specify trap volume calibration and cleaning procedure (replaced use of chromic acid with alkaline detergent). Revised Note 2 to explain reason for salt solution. Revised Note 3 to include trap xylene calibration. Revised Note 4 with instruction to keep condenser from overheating. Added Note 7 to explain differences between this method and ISO method 6571.

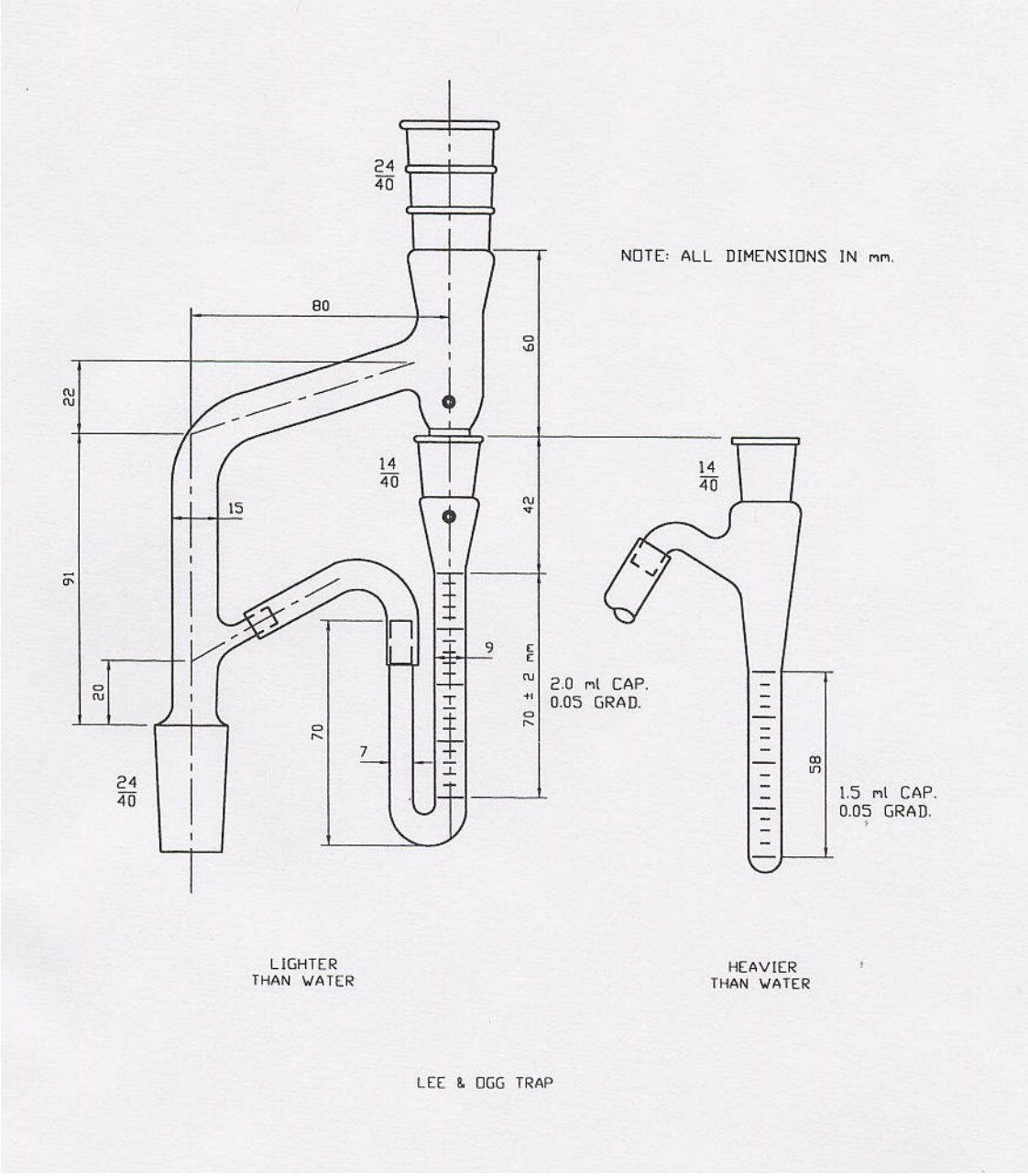

Figure 1. Volatile oil traps for ASTA method 5.1.

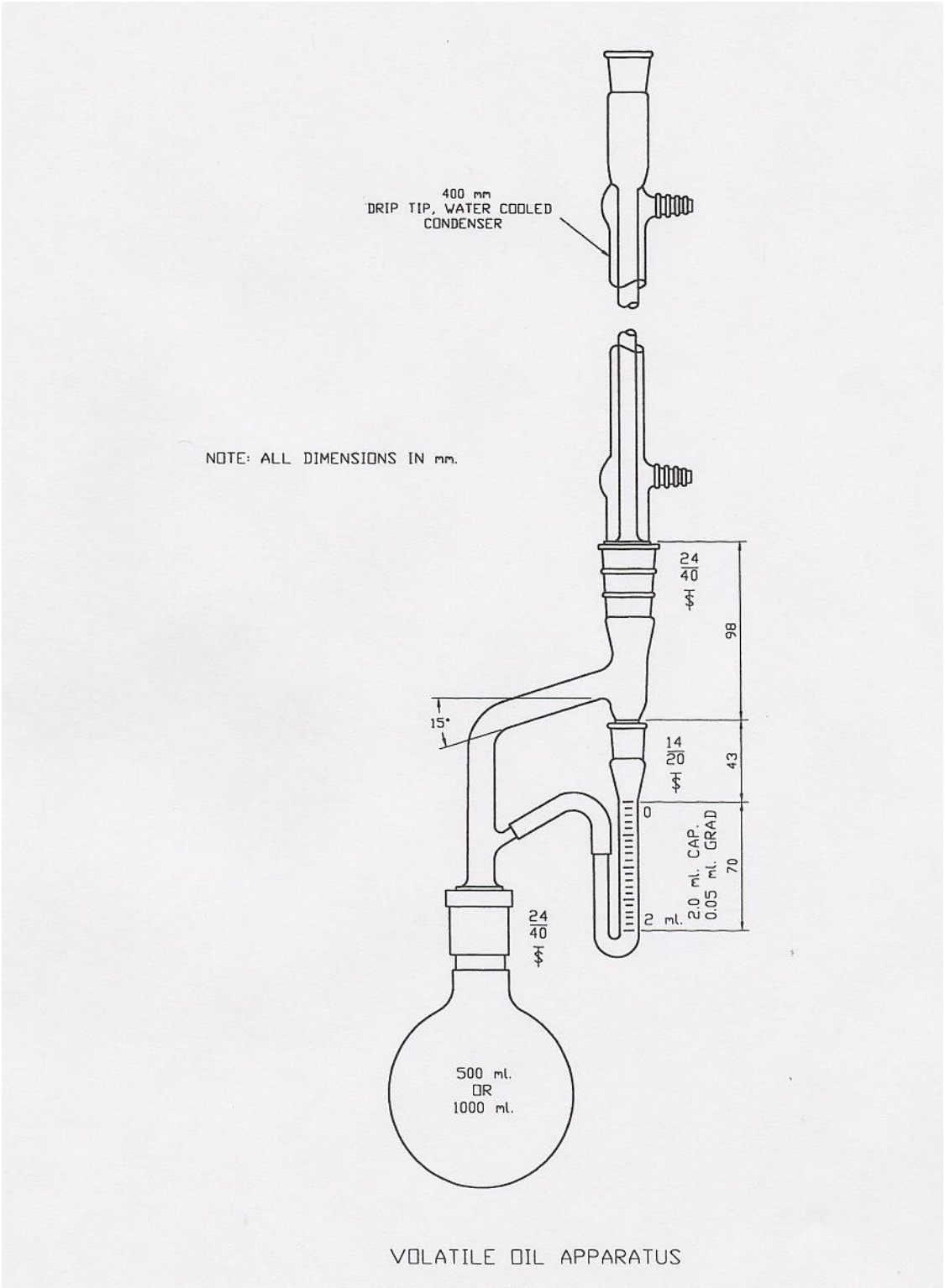

Figure 2. Apparatus assembled for steam distillation (ASTA method 5.1).