Standard Test Method for Distillation of Plant Spray Oils

This standard is issued under the fixed designation D 447; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (e) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the volatility of plant spray oils by means of distillation. Its primary purpose is to establish the classification of a spray oil by determining the fraction distilled at specified temperatures. Both a manual method and an automatic method are specified.

1.2 In cases of dispute, the referee test method is the manual test method.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.4 This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific hazard statements, see Note 3.

2. Referenced Documents

2.1 ASTM Standards:
D 850 Test Method for Distillation of Industrial Aromatic Hydrocarbons and Related Materials
D 1078 Test Method for Distillation Range of Volatile Organic Liquids
E 133 Specification for Distillation Equipment
E 220 Method for Calibration of Thermocouples by Comparison Techniques

3. Summary of Test Method

3.1 A 100 mL sample is distilled in a 125-mL flask at a rate of 4 to 6 mL/min. The temperature is recorded at 5 mL distilled intervals.

4. Significance and Use

4.1 To obtain optimum persistence with minimal damage to fruit and foliage, a plant spray oil should possess appropriate volatility characteristics, as indicated by distillation. A narrow range, for example, 55°C, ensures uniform evaporation, while the proper level of initial and final boiling points prevents either too rapid or undesirably prolonged evaporation of the oil.

5. Apparatus

5.1 All items listed in 5.1.1 to 5.1.7 shall conform to Specification E 133. All of the following references are to Specification E 133:

5.1.1 Distilling Flask, Flask B (125 mL).
5.1.2 Condenser and Cooling Bath, Section 5 and Figs. 1 and 2 of Specification E 133.
5.1.3 Heater, Section 7 and Figs. 1 and 2 of Specification E 133.
5.1.4 Flask Support, Board C [51 mm (2.0-in.) hole]. An additional board, which will completely cover the top of the shield, is split and recessed to fit the neck of the flask.
5.1.5 Graduated Cylinder, Graduate B, 100 mL, as shown in Fig. 4 of Specification E 133. The cylinder must have graduations at the 5 mL level and from 90 to 100 mL in 1-mL increments. For automatic apparatus, the cylinder shall conform to the physical specifications described in this section, with the exception of the graduations.
5.1.5.1 For automatic apparatus, the level follower/recording mechanism of the apparatus will have a resolution of 0.1 mL, with an accuracy of ±1 mL. The calibration of the assembly should be confirmed according to the manufacturer's instructions at regular intervals. The typical calibration procedure involves the verification of the output with the receiver containing 5 and 100 mL of material, respectively.
5.1.6 Temperature Sensor:
5.1.6.1 ASTM Thermometer 8C (8F) as prescribed in Section 10 of Specification E 133.

NOTE 1—Thermometers heated to high temperatures, in the range required for spray oil distillations, sometimes develop stresses that may affect the accuracy of calibration. It is recommended that, when thermometers vary from the standard thermometer when checked at any convenient temperature, the thermometers be allowed to rest at room temperature for at least 24 h to relieve stresses.

5.1.6.2 Temperature measurement systems using thermocouples or resistance thermometers must exhibit the same temperature lag and accuracy as the equivalent mercury in glass thermometers. Confirmation of the calibration of these temperature sensors is to be made on a regular basis. This can be accomplished as described in Method E 220, potentiometrically by the use of standard precision resistance, depending on the type of probe. Another technique is to distill pure toluene in accordance with Test Method D 850 and compare the temperature indicated with that shown by the above mentioned mercury in glass thermometers when carrying out a manual test under the same condition.

NOTE 2—Toluene is shown in reference manuals as boiling at 110.6°C under the conditions of Test Method D 1078, which uses a partial immersion thermometer. Because this test method uses total immersion thermometers, the results will be lower. The approximate value for an 8C thermometer is 110.0°C.
5.1.7 Water Bath—A suitable water bath maintained at boiling temperature, in which the cylinder can be immersed to the top graduation mark.

6. Reagents and Materials

6.1 Petroleum Ether, with a boiling range from 35 to 60°C, or IP Petroleum Spirit 40/60. (Warning—See Note 3).

Note 3: Warning—Extremely flammable. Skin irritant on repeated contact. Aspiration hazard.

7. Procedure

7.1 Swab the condenser tube to remove any liquid remaining from the previous determination. A piece of soft cloth attached to a cord or copper wire is satisfactory for this purpose.

7.2 Measure 100 mL of the sample at 21 to 27°C into the 100-mL cylinder, and pour it into the distilling flask, allowing the cylinder to drain for 30 s. Hold or support the cylinder at the top graduation mark in a boiling water bath for approximately 1 min, then allow the cylinder to drain again into the flask for 30 s. Rinse the cylinder with petroleum ether, blow dry, and place the same cylinder at the bottom of the vapor outlet tube in such a position that the condenser tube extends approximately 25 mm into the graduate but not below the 100-mL mark. Do not place cylinder in contact with the tip of condenser tube.

7.3 Check the thermometer for accuracy at any convenient temperature, and then install the thermometer, provided with a cork, in the neck of the flask so that the bulb is in the center of the neck, and the top of the bulb (that is, the end of the enamel backing) is level with the inside of the bottom of the vapor outlet tube at its highest point. Place the flask on the 51 mm (2.0 in.) hole in the flash support with the vapor tube parallel to the condenser, and not flow down the side. Adjust the flask so that it fits the vertical-edged hole in the flash support snugly. The vapor tube should be parallel to the condenser tube and extend into it not less than 25 mm nor more than 51 mm. Cover the shield with the split board, which should fit the neck of the flask closely.

7.4 Maintain the temperature of the condenser bath at 57 to 63°C throughout the distillation. Heat the flask at a uniform rate, so regulated that the first drop of condensate falls from the condenser in not less than 10 nor more than 15 min; record the temperature when this occurs. Continue the distillation, adjusting the heat so as to maintain a rate of not less than 4 nor more than 6 mL/min. Permit the oil to fall directly into the graduate, and not flow down the side.

7.4.1 Too much stress cannot be placed on the necessity for maintaining the distillation rate. It must at all times be within the 4 to 6 mL/min specified, preferably as close to 5 mL/min as possible. If the rate falls outside of these limits at any point, discard any results and repeat the determination. The distillation never "catches up," and all subsequent points will be in error.

7.5 Record the temperature as each 5 mL or other specified amounts of distillate are collected; also record the volume of condensate at any specified temperature, these volumes being uncorrected for temperature. Continue as described until 95 mL have been collected in the receiver, unless cracking occurs sooner. Cracking will be observed by an increase in the distilling rate, with erratic thermometer readings; an effort to adjust the rate will usually result in a decided drop in temperature reading.

7.6 Record the barometric pressure prevailing during the distillation.

8. Calculation

8.1 Correct the observed temperature readings, if the barometric pressure varies from 101.3 kPa (760 mm Hg) by more than 1.3 kPa (10 mm Hg), by means of either of the following two equations:

\[
\begin{align*}
C_c &= 0.0009 \left(101.3 - P_1\right) \left(273 + t_o\right) \\
C_c &= 0.00012 \left(760 - P_2\right) \left(273 + t_o\right)
\end{align*}
\]

where:

- \(C_c\) = correction to be added to the observed vapor temperature, \(t_o\)
- \(P_1\) = barometric pressure, kPa, prevailing at the time of test, and
- \(P_2\) = barometric pressure, mm Hg, prevailing at the time of test.

Note 4—The calculated correction factors within the temperature range encountered in the distillation, as shown in Table 1, will affect the temperature recorded at any given volume by more than 0.4°C when the barometric pressure varies from 101.3 kPa by more than 1 kPa.

8.2 Correct the temperature readings from 8.1 in accordance with the thermometer calibration. Report the final corrected initial boiling point and temperature of each 5% distilled or other specified amounts.

8.3 When the percentage distilled at a certain temperature is required and the barometric pressure varies from 760 mm Hg by more than 100 mm, use the temperature corrected to 760 mm Hg prior to the distillation.

Note 5—Instead of correcting the temperature prior to the distillation, it is permissible to plot the final corrected temperatures against the corresponding percentages and report the indicated percentage at the required temperature from the curve.

9. Precision and Bias

9.1 Precision—The precision of the manual test method as determined by the statistical examination of interlaboratory test results is as follows:

9.1.1 Repeatability—The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:
Thermometer reading at specified:

<table>
<thead>
<tr>
<th>% Recovered</th>
<th>Repeatability, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>2.2</td>
</tr>
<tr>
<td>50</td>
<td>1.7</td>
</tr>
<tr>
<td>90</td>
<td>2.2</td>
</tr>
</tbody>
</table>

Percent recovered at 335.6°C 2%

9.1.2 Reproducibility—The difference between two, single and independent results obtained by different operators working in different laboratories on identical test material would, in the long run, and in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

<table>
<thead>
<tr>
<th>% Recovered</th>
<th>Reproducibility, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>5.0</td>
</tr>
<tr>
<td>50</td>
<td>3.9</td>
</tr>
<tr>
<td>90</td>
<td>5.0</td>
</tr>
</tbody>
</table>

Percent recovered at 335.6°C 4%

9.2 The precision of the test method using automatic equipment has not been determined.

9.3 Bias:

9.3.1 Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, a statement on bias cannot be estimated.

9.3.2 The bias between the manual and the automatic test method has not been determined.

10. Keywords

10.1 distillation; plant spray oils; spray oils; volatility

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