

# Standard Test Method for Depentanization of Gasoline and Naphthas<sup>1</sup>

This standard is issued under the fixed designation D2001; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 This test method covers the removal of pentanes and lighter hydrocarbons from gasolines, naphthas, and similar petroleum distillates to prepare samples suitable for the determination of hydrocarbon types in accordance with Test Method D2789. In addition, this test method determines the volume percent of bottoms remaining after depentanization.

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

1.3 This standard does not purport to address all of the safety concerns, 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.

## 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D1250 Guide for Use of the Petroleum Measurement Tables D2789 Test Method for Hydrocarbon Types in Low Olefinic Gasoline by Mass Spectrometry

## 3. Summary of Test Method

3.1 A 50-mL sample is distilled into an overhead ( $C_5$  and lighter) fraction and a bottoms ( $C_6$  and heavier) fraction. The volume of bottoms is measured and the volume percent, based on the charge, is calculated.

#### 4. Significance and Use

4.1 The presence of pentane and lighter hydrocarbons in gasolines, naphthas, and similar petroleum distillates interferes in Test Method D2789. Pentane and lighter hydrocarbons are separated by this test method so that the depentanized residue

can be analyzed and so the pentane and lighter hydrocarbons can be analyzed by other methods, if desired.

4.2 Under the conditions specified in the test method some  $C_5$  and lighter hydrocarbons remain in the bottoms, and some  $C_6$  and heavier hydrocarbons carry over to the overhead. Expressed as volume percent of charge, the amounts are typically 2 % or less, which is considered adequate for the purpose designated under Scope. It should be recognized, however, that when expressed as volume percent of overhead or of bottoms the percentages can be higher, making this test method unsuitable for any purposes not designated under Scope.

### 5. Apparatus

5.1 *Depentanization Apparatus*, as shown in Fig. 1, consisting of the following parts:

- 5.1.1 Distillation Column,
- 5.1.2 Reflux Condenser Head,
- 5.1.3 Light-Ends Trap,
- 5.1.4 Receiver, graduated, 12.5 mL, and
- 5.1.5 *Thermometer*, <sup>3,4</sup> 10 to 79°C (50 to 175°F).
- 5.2 Column Packing—Two types are required:

5.2.1 *Heli-Pak Column Packing*, <sup>4.5</sup> 1.27 by 2.54 by 2.54 mm (Size B, 0.050 by 0.100 by 0.100 in.).

5.2.2 Heli-Pak Column Packing,  $^{4,5}$  Size C, 4.4 by 4.4 m (0.090 by 0.175 by 0.175 in.), or Cannon Protruded Metal Packing,  $^{4,6}$  4.0 by 4.0 mm (0.16 by 0.16 in.).

5.3 *Distillation Flask*, round-bottom, 100 mL, with  $^{24}4_{0}$  standard-taper female joint.

5.4 Distillation Flask Heating Mantle, Glas-Col, spherical, for 100-mL flask.

5.5 *Variable Transformer*, for regulating power to heating mantle.

<sup>4</sup> If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,<sup>1</sup> which you may attend.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.04.0C on Liquid Chromatography.

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<sup>&</sup>lt;sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>&</sup>lt;sup>3</sup> The sole source of supply of the apparatus known to the committee at this time is Thermometer No. ME510-1 available from Metro Scientific Inc., 11 Willow Park Center, East Farmingdale, NY 11735.

<sup>&</sup>lt;sup>5</sup> The sole source of supply of the apparatus known to the committee at this time is Reliance Glass Works Inc., Gateway Rd., PO Box 825, Bensenville, IL 60106.

<sup>&</sup>lt;sup>6</sup> The sole source of supply of the apparatus known to the committee at this time is Cannon protruded metal packing available from Scientific Development Co., Box 795, State College, PA.

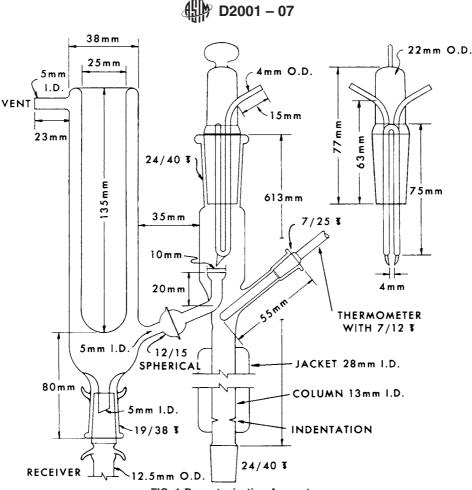


FIG. 1 Depentanization Apparatus

5.6 *Water Cooling Bath*—If a supply of chilled water for the reflux condenser is not available, a satisfactory means for supplying chilled water can be provided by circulating tap water through coiled copper tubing immersed in an ice-water bath.

#### 6. Preparation of Apparatus

6.1 Fill the fractionating column with packing as follows: Place about 25 mm (1 in.) of the Heli-Pak column packing, size C or the Cannon packing on the indentations at the bottom of the column. This packing assures ample free space to prevent flooding at the bottom of the column. Fill the column to within 25 mm (1 in.) of the top of the jacket with Heli-Pak column packing, size B. (The performance of this packing is not sensitive to the exact manner of pouring into the column.)

6.2 Assemble and connect the column, head, light-ends trap, and the receiver. Mount the assembly on a suitable support.

6.3 Insert the thermometer through the side-arm at the top of the column.

NOTE 1—The calibration of the thermometer at the cut temperature should be checked by refluxing a pure compound. Such a calibration can show a need for a correction.

6.4 Immerse the receiver in a Dewar flask containing a mixture of solid carbon dioxide (**Warning**—Extremely cold.) in a suitable liquid. 1,1,2 Trichloro Ethene is recommended. (**Warning**—Health hazard.)

6.5 Circulate water, chilled to a temperature between 4 and  $10^{\circ}$ C (40 and 50°F), through the reflux condenser.

#### 7. Procedure

7.1 Lubricate the joint at the bottom of the column with a hydrocarbon-insoluble grease, such as silicone, starch-glycerol, or metallic soap. Place the heating mantle around the flask. With a 50-mL graduated cylinder, measure 50 mL of the sample. Determine its temperature, and transfer it to the flask. Connect the flask immediately to the column.

7.2 Connect the mantle, through the variable transformer, to the power supply.

7.3 Apply heat to the contents of the flask at a uniform rate.

NOTE 2—With the apparatus prepared as described in Section 6, a setting of 30 on the transformer usually provides a satisfactory initial heating rate; voltage is increased as required.

7.4 As soon as drops of liquid reflux form at the column head, adjust the position of the condenser so that one drip-tip feeds to the take-off line, and the other back to the (**Warning**— Extremely flammable liquid.) column. The reflux condenser remains in this position throughout the distillation to provide about a 1 + 1 reflux ratio.

NOTE 3—Low-boiling light ends will condense on the light-ends trap and collect in the receiver before drops of liquid reflux form at the column head.



7.5 The distillation rate should not exceed 30 to 40 drops/30 s from each tip of the reflux condenser.

7.6 Continue the distillation until the thermometer indicates a temperature of  $49^{\circ}$ C ( $120^{\circ}$ F). When this temperature is reached, shut off the heat and drop the lower portion of the heating mantle. Turn the reflux condenser so that drops from both drippers are returned to the column. Direct a stream of air on the top surface of the flask. Perform the above steps in rapid succession.

7.7 Allow the column to drain and the contents of the flask to cool for 30 min.

7.8 Remove the flask from the column, and carefully transfer the contents to the same graduated cylinder which was used to measure the charge. Measure the volume of the bottoms fraction and determine its temperature.

## 8. Calculation

8.1 If the temperatures of the distillation charge and bottoms differ by more than  $11^{\circ}C$  (20°F), correct the measured volumes to volumes at  $15^{\circ}C$  (60°F) by Guide D1250.

8.2 Calculate the volume of bottoms in percent as follows:

Volume of bottoms, percent = 
$$(A/B) \times 100$$
 (1)

where:

A = volume of bottoms, mL and

B = volume of the charge, mL.

#### 9. Precision and Bias

9.1 The following criteria should be used for judging the acceptability of results (95 % confidence) (Note 4):

9.1.1 *Repeatability*—The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed 2 volume % only in one case in twenty.

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, in the normal and correct operation of the test method, exceed 4 volume % only in one case in twenty.

9.2 *Bias*—The quantities determined are defined by the conditions used in this empirical test method, and a statement of bias is therefore not appropriate.

Note 4—Precision data are based on results obtained by five laboratories testing four samples in duplicate. The samples contained  $C_5$ -and-lighter fractions covering a range of 5 to 25 volume %.

## 10. Keywords

10.1 depentanization; fractionations; hydrocarbon types; pentanes

## SUMMARY OF CHANGES

Subcommittee D02.04 has identified the location of selected changes to this standard since the last issue (D2001-92(2002)) that may impact the use of this standard.

(1) Removed references to Test Method D1319.

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