



# Standard Test Method for Vapor Pressure of Petroleum Products (Mini Method—Atmospheric)<sup>1</sup>

This standard is issued under the fixed designation D 5482; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope \*

1.1 This test method covers a procedure for the determination of total vapor pressure of petroleum products using automatic vapor pressure instruments. The test method is suitable for testing samples with boiling points above 0°C (32°F) that exert a vapor pressure between 7 and 110 kPa (1.0 and 16 psi) at 37.8°C (100°F) at a vapor-to-liquid ratio of 4:1. The test method is applicable to gasolines containing oxygenates. No account is made of dissolved water in the sample.

NOTE 1—Because the external atmospheric pressure does not influence the resultant vapor pressure, this vapor pressure is an absolute pressure at 37.8°C (100°F) in kPa (psi). This vapor pressure differs from the true vapor pressure of the sample due to some small vaporization of the sample and dissolved air into the air of the confined space.

1.1.1 Some gasoline-oxygenate blends may show a haze when cooled to 0 to 1 °C. If a haze is observed in 8.5, it shall be indicated in the reporting of results. The precision and bias statements for hazy samples have not been determined (see Note 6).

1.2 This test method is a modification of Test Method D 5191 (Mini Method) in which the test chamber is at atmospheric pressure prior to sample injection.

1.3 This test method covers the use of automated vapor pressure instruments that perform measurements on liquid sample sizes in the range from 1 to 10 mL.

1.4 This test method is suitable for the determination of the dry vapor pressure equivalent (DVPE) of gasoline and gasoline-oxygenate blends by means of a correlation equation (see 13.2). The calculated DVPE is considered equivalent to the result obtained on the same material when tested by Test Method D 4953.

1.5 The values stated in acceptable SI units are regarded as standard. The values given in parentheses are provided for information only.

1.6 *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. (For specific hazard statements, see 7.2 through 7.7.)*

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>2</sup>

D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)<sup>3</sup>

D 5190 Test Method for Vapor Pressure of Petroleum Products (Automatic Method)<sup>3</sup>

D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)<sup>3</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *dry vapor pressure equivalent (DVPE)*—a value calculated by a correlation equation (see 13.2) from the total pressure.

3.1.1.1 *Discussion*—The DVPE is expected to be equivalent to the value obtained on the sample by Test Method D 4953.

3.1.2 *total pressure*—the observed pressure measured in the experiment that is the resultant pressure increase from the initial ambient atmospheric pressure.

## 4. Summary of Test Method

4.1 A known volume of chilled, air-saturated sample is introduced into a thermostatically controlled test chamber, the internal volume of which is five times that of the total test specimen introduced into the chamber. The test chamber is at atmospheric pressure prior to introduction of the sample. After introduction of the sample into the test chamber, the test specimen is allowed to reach thermal equilibrium at the test temperature, 37.8°C (100°F). The resulting rise in pressure in the chamber is measured using a pressure transducer sensor and indicator.

4.2 The measured total vapor pressure is converted to a

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.03.

\*A Summary of Changes section appears at the end of this standard.

DVPE by use of a correlation equation (see 13.2).

## 5. Significance and Use

5.1 Vapor pressure is an important physical property of volatile liquids.

5.2 Vapor pressure is critically important for both automotive and aviation gasolines, affecting starting, warm-up, and tendency to vapor lock with high operating temperatures or high altitudes. Maximum vapor pressure limits for gasoline are legally mandated in some areas as a measure of air pollution control.

## 6. Apparatus

6.1 *Vapor Pressure Apparatus*—The type of apparatus<sup>4</sup> suitable for use in this test method employs a small volume test chamber incorporating a transducer for pressure measurements and associated equipment for thermostatically controlling the chamber temperature.

6.1.1 The test chamber shall be designed to contain between 2 and 50 mL of liquid and vapor and be capable of maintaining a vapor-liquid ratio between 3.95 and 1.00 and 4.05 and 1.00.

6.1.2 The pressure transducer shall have a minimum operational range from 0 to 172 kPa (0 to 25.0 psi) with a minimum resolution of 0.1 kPa (0.01 psi) and a minimum accuracy of  $\pm 0.3$  kPa ( $\pm 0.05$  psi). The pressure measurement system shall include associated electronics and readout devices to display the resulting pressure reading.

6.1.3 A thermostatically controlled heater shall be used to maintain the test chamber at  $37.8 \pm 0.1^\circ\text{C}$  ( $100 \pm 0.2^\circ\text{F}$ ) for the duration of the test.

6.1.4 A platinum resistance thermometer shall be used for measuring the temperature of the test chamber. The minimum temperature range of the measuring device shall be from ambient to  $75^\circ\text{C}$  ( $167^\circ\text{F}$ ) with a resolution of  $0.1^\circ\text{C}$  ( $0.2^\circ\text{F}$ ) and accuracy of  $0.1^\circ\text{C}$  ( $0.2^\circ\text{F}$ ).

6.1.5 The vapor pressure apparatus shall have provisions for introduction of the test specimen into the test chamber and for the cleaning or purging of the chamber following the test.

6.2 *Syringe*, if required, gas tight, 1 to 20 mL capacity with a  $\pm 1\%$ , or better, accuracy and a  $\pm 1\%$ , or better, precision. The capacity of the syringe shall not exceed two times the volume of the test specimen being dispensed, and shall be chosen so as to provide maximum accuracy and resolution for the volume to be injected.

6.3 *Iced-Water Bath or Air Bath*, for chilling the samples and syringe to temperatures between  $0$  and  $1^\circ\text{C}$  ( $32$  and  $34^\circ\text{F}$ ).

6.4 *Pressure Measuring Device*, capable of measuring ambient and above ambient pressures with an accuracy of 0.20 kPa (0.03 psi) or better at the same elevation relative to sea level as the apparatus in the laboratory.

6.4.1 When a mercury manometer is not used as the pressure measuring device, the calibration of the pressure measuring device employed shall be periodically checked

(with traceability to a nationally recognized standard) to ensure that the device remains within the required accuracy specified in 6.4.

6.5 *Pressure Source*, clean, dry compressed gas or other suitable compressed air capable of providing pressure for calibration of the transducer and cleaning of the cell.

NOTE 2—A vacuum source is an alternate means for cleaning of the cell.

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Use chemicals of at least 99 % purity for quality control checks (see Section 11). Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>5</sup> Lower purities can be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

7.2 *Cyclohexane*, (**Warning**—Cyclohexane is flammable and a health hazard).

7.3 *Cyclopentane*, (**Warning**—Cyclopentane is flammable and a health hazard).

7.4 *2,2-Dimethylbutane*, (**Warning**—2,2-dimethylbutane is flammable and a health hazard).

7.5 *2,3-Dimethylbutane*, (**Warning**—2,3-dimethylbutane is flammable and a health hazard).

7.6 *2-Methylpentane*, (**Warning**—2-methylpentane is flammable and a health hazard).

7.7 *Toluene*, (**Warning**—Toluene is flammable and a health hazard).

## 8. Sampling

### 8.1 General Requirements:

8.1.1 The extreme sensitivity of vapor pressure measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution and the most meticulous care in the handling of samples.

8.1.2 Obtain a sample and test specimen in accordance with 10.3 of Practice D 4057, except do not use 10.3.1.8 of Practice D 4057, Sampling by Water Displacement, for fuels containing oxygenates. Use a 1-L (1-qt) sized container filled between 70 to 80 % with sample.

8.1.3 Perform the vapor pressure determination on the first test specimen withdrawn from a sample container. Do not use the remaining sample in the container for a second vapor pressure determination. If a second determination is necessary, obtain a new sample.

8.1.4 Protect samples from excessive temperatures prior to testing. This can be accomplished by storage in an appropriate ice bath or refrigerator.

8.1.5 Do not test samples stored in leaky containers. Discard and obtain a new sample if leaks are detected.

<sup>4</sup> The following instruments have been found satisfactory for use in this test procedure as determined by interlaboratory testing: Herzog Mini Reid Vapor Pressure Model MP970—available from Varlen Instruments, Inc., 2777 Washington Blvd., Bellwood, IL 60104 and ABB Model 4100—available from ABB Process Analytics, Lewisburg, WV.

<sup>5</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

8.1.6 Do not store samples in plastic (polyethylene, polypropylene, and so forth) containers since volatile materials may diffuse through the walls of the container.

8.2 *Sampling Temperature*—Cool the sample container and contents in an ice bath or refrigerator to the 0 to 1°C (32 to 34°F) range prior to opening the sample container. Allow sufficient time to reach this temperature. Verify the sample temperature by direct measurement of the temperature of a similar liquid in a similar container placed in the cooling bath or refrigerator at the same time as the sample.

8.3 *Verification of Sample Container Filling*—With the sample at a temperature of 0 to 1°C, take the container from the cooling bath or refrigerator and wipe dry with an absorbent material. If the container is not transparent, unseal it and using a suitable gage, confirm that the sample volume equals 70 to 80 % of the container capacity (see Note 3). If the sample is contained in a transparent glass container, verify that the container is 70 to 80 % full by suitable means (see Note 3).

8.3.1 Do not perform a vapor pressure test on the sample if the container is filled to less than 70 volume % of the container capacity.

8.3.2 If the container is more than 80 volume % full, pour out enough sample to bring the container contents within the 70 to 80 volume % range. Do not return any sample to the container once it has been withdrawn.

NOTE 3—For non-transparent containers, one way to confirm that the sample volume equals 70 to 80 % of the container capacity is to use a dipstick that has been pre-marked to indicate the 70 and 80 % container capacities. The dipstick should be of such material that it shows wetting after being immersed and withdrawn from the sample. To confirm the sample volume, insert the dipstick into the sample container so that it touches the bottom of the container at a perpendicular angle, before removing the dipstick. For transparent containers, using a marked ruler or comparing the sample container to a like container that has the 70 and 80 % levels clearly marked, has been found suitable.

8.3.3 Reseal the container, if necessary, and return the sample container to the cooling bath or refrigerator.

8.4 *Air Saturation of the Sample in the Sample Container:*

8.4.1 *Non-transparent Containers*—With the sample again at a temperature between 0 and 1°C, take the container from the cooling bath or refrigerator, wipe it dry with an absorbent material, remove the cap momentarily, taking care that no water enters, reseal, and shake it vigorously. Return it to the cooling bath or refrigerator for a minimum of 2 min.

8.4.2 *Transparent Containers*—Since 8.3 does not require that the sample container be opened to verify the sample capacity, it is necessary to unseal the cap momentarily before resealing it so that samples in transparent containers are treated the same as samples in non-transparent containers. After performing this task, proceed with 8.4.1.

8.4.3 Repeat 8.4.1 twice more. Return the sample to the bath or refrigerator until the beginning of the procedure.

8.5 *Verification of Single Phase Samples*—After drawing a test specimen and injecting it into the instrument for analysis, check the remaining sample for phase separation. If the sample is contained in a transparent container, this observation can be made prior to sample transfer. When the sample is contained in a non-transparent container, mix the sample thoroughly and immediately pour a portion of the remaining sample into a

clear glass container and observe for evidence of phase separation. A hazy appearance is to be carefully distinguished from separation into two distinct phases. The hazy appearance shall not be considered grounds for rejection of the fuel. If a second phase is observed, discard the test and the sample. Hazy samples may be analyzed (see Report section).

## 9. Preparation of Apparatus

9.1 Prepare the instrument for operation in accordance with the manufacturer's instructions.

9.2 Prepare the sample introduction accessories, if required, in accordance with the manufacturer's instructions.

9.3 Chill the sample syringe to between 0 and 4.5°C (32 and 40°F) in a refrigerator or ice bath. Avoid water contamination of the syringe reservoir by suitably sealing the outlet of the syringe during the cooling process.

9.4 Clean and dry the test chamber in accordance with the manufacturer's instructions. With the test chamber sealed, observe that the instrument display is stable and does not exceed  $0.00 \pm 0.20$  kPa ( $0.00 \pm 0.03$  psi).

9.5 If in doubt of the cleanliness of the cell, refer to the cleaning procedure in the manufacturer's instructions, or if the display does not return to zero, refer to the calibration procedure in the manufacturer's instructions.

9.6 Verify that the temperature of the test chamber is within the required range from  $37.8 \pm 0.1$ °C ( $100 \pm 0.2$ °F).

## 10. Calibration

### 10.1 *Pressure Transducer:*

10.1.1 Check the calibration of the transducer in accordance with the manufacturer's instructions on a monthly basis or when needed as indicated from the quality control checks (see Section 11). The calibration of the transducer shall be checked using two reference points, ambient barometric pressure, and a pressure above ambient pressure, determined by the operator, which is at least 80 % of the expected maximum pressure encountered during the test.

10.2 *Temperature Measuring Device*—Check the calibration of the temperature measuring device used to monitor the temperature of the test chamber at least every six months against a thermometer traceable to a national standard.

## 11. Quality Control Checks

11.1 Use a verification fluid with known volatility as an independent check against the instrument calibration each day the instrument is in use. For pure compounds (see 7.1), multiple test specimens may be taken from the same container over time, provided the pure compound is air saturated in accordance with the procedure given in 8.4, and the spent test specimens are not re-used, in whole or in part. If the observed vapor pressure differs from the reference value by more than 1.0 kPa (0.15 psi), then check the instrument calibration (Section 9).

11.2 Some possible materials and their corresponding vapor pressures, as found in ASTM DS4B,<sup>6</sup> include: cyclopentane,

<sup>6</sup> "Physical Constants of Hydrocarbon and Non-Hydrocarbon Materials," available from ASTM Headquarters. Order PCN 28-003092-12.

68.3 kPa (9.91 psi); 2,2-dimethylbutane, 68.0 kPa (9.86 psi); 2,3-dimethylbutane, 51.1 kPa (7.41 psi); 2-methylpentane, 46.7 kPa (6.77 psi); and toluene, 7.1 kPa (1.03 psi).<sup>7</sup>

NOTE 4—It is recommended that at least one type of control sample used in 10.1 be representative of the samples regularly tested by the laboratory. The total vapor pressure measurement process (including operator technique) can be checked periodically by performing this test method on previously prepared samples from one batch of product, in accordance with the procedure described in 8.1.2. Samples should be stored in an environment suitable for long term storage without sample degradation. Analysis of results from these quality control samples can be carried out using control chart techniques<sup>8</sup> or other statistically equivalent techniques.

## 12. Procedure

12.1 Remove the sample from the cooling bath or refrigerator, dry the exterior of the container with absorbent material, uncap, and insert a chilled syringe. Draw a bubble-free aliquot of sample into the gas tight syringe, and deliver this test specimen to the test chamber as rapidly as possible. The total time between opening the chilled sample container and securing the syringe into the test chamber shall not exceed 1 min.

12.2 Follow the manufacturer's instructions for injection of the test specimen into the test chamber and for operation of the instrument to obtain a vapor pressure result for the test specimen.

12.3 If the instrument is capable of calculating the dry vapor pressure equivalent automatically, ensure that the equation in 13.2 is used.

## 13. Calculation

13.1 Record the vapor pressure reading from the instrument to the nearest 0.1 kPa (0.01 psi). For instruments that do not automatically record a stable pressure value, manually record the pressure indicator reading every minute to the nearest 0.1 kPa. When three successive readings agree to within 0.1 kPa, record the result to the nearest 0.1 kPa (0.01 psi).

13.2 Calculate the DVPE using Eq 1. Ensure that the instrument reading used in this equation corresponds to the total pressure and has not been corrected by an automatically programmed correction factor. Use the variable pertaining to the type of equipment utilized.

$$DVPE, \text{ kPa (psi)} = (0.965 X) + A \quad (1)$$

where:

$X$  = measured total vapor pressure in kPa (psi),

$A$  = 0.538 kPa (0.078 psi) for HERZOG Model SC 970, and

$A$  = 1.937 kPa (0.281 psi) for ABB Model 4100.

NOTE 5—The correlation equations were derived from data obtained in a 1991 interlaboratory cooperative test program.<sup>8</sup> The equations correct for the relative bias between the measured vapor pressure and the dry vapor pressure obtained in accordance with Test Method D 4953, Procedure A.

13.3 The calculation described in Eq 1 can be accomplished automatically by the instrument, if so equipped, and in such cases the user shall not apply any further correction.

## 14. Report

14.1 Report the following information:

14.1.1 Report the dry vapor pressure equivalent to the nearest 0.1 kPa (0.01 psi) without reference to temperature.

14.1.2 If the sample was observed to be hazy in 8.5, report the test result as in 14.1.1, followed by the letter "H".

NOTE 6—The precision and bias statements have not been determined for hazy samples, since these types of samples have not been evaluated as part of an interlaboratory study.

NOTE 7—The inclusion of the letter "H" in 14.1.2 is intended to alert the data recipient that the sample analyzed was hazy. In the event a laboratory has a computer system that is incapable of reporting alphanumeric results according to the requirements in 14.1.2, it is permissible for the laboratory to report the result obtained as in 14.1.1, along with a statement or annotation that clearly conveys to the data recipient that the sample analyzed was hazy.

## 15. Precision and Bias

15.1 The precision of this procedure as determined by the statistical examination of interlaboratory test results is as follows: The following precision data were developed in a 1991 interlaboratory cooperative test program.<sup>9</sup> Participants analyzed sample sets comprised of blind duplicates of 14 types of hydrocarbons and hydrocarbon-oxygenate blends. The oxygenates used were ethanol and MTBE. The oxygenate content ranged nominally from 0 to 15 % by volume and the vapor pressure ranged nominally from 14 to 100 kPa (2 to 15 psi). A total of 60 laboratories participated. Some participants performed more than one test method, using separate sample sets for each. Thirteen sample sets were tested by this test method using two different instruments, 26 sample sets were tested by Test Method D 4953, 13 by Test Method D 5190, and 27 by Test Method D 5191. In addition, six sets were tested by modified Test Method D 5190.

15.1.1 *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, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

1.31 kPa (0.19 psi) for HERZOG Model SC 970

1.79 kPa (0.26 psi) for ABB Model 4100

15.1.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following value only in one case in twenty:

2.69 kPa (0.39 psi) for HERZOG Model SC 970

4.14 kPa (0.60 psi) for ABB Model 4100

15.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedures, bias cannot be determined.

<sup>7</sup> "Manual on Presentation of Data and Control Chart Analysis," ASTM MNL 7, Sixth Edition, 1990, Section 3.

<sup>8</sup> The vapor pressure values cited were obtained from Phillips Petroleum Company, Bartlesville, Oklahoma or the Table of Physical Constants, Gas Processors Association—Standard 2145.

<sup>9</sup> The results of this test program are filed at ASTM Headquarters. Request RR:D02-1286.

15.3 *Relative Bias*—Statistically significant relative biases were observed in the 1991 interlaboratory cooperative test program<sup>8</sup> between the total pressure obtained using instruments described in this test method and the dry vapor pressure obtained using Test Method D 4953, Procedure A. These biases are corrected by applying Eq 1.

NOTE 8—The bias and precision information provided for the ABB apparatus are applicable only for the instruments manual mode of operation, for a nominal vapor pressure range of 13.8 to 82.68 kPa (2 to 12 psi).

15.4 *Reproducibility Between Methods*—Just as the reproducibility supplies a 95 % confidence level on the difference between measurements by two different laboratories using the same method, there exists an equivalent reproducibility describing the 95 % confidence level on the difference between measurements by two laboratories using different methods.

15.4.1 A statistically based method for calculation of reproducibility between different methods was developed as listed below:

$$R_m = \sqrt{\frac{1}{2} \left[ R_1^2 \left( 1 + \frac{1}{n_1} \right) + R_2^2 \left( 1 + \frac{1}{n_2} \right) \right]} \quad (2)$$

where:

$R_1, R_2$  = the reproducibility figures for each method under consideration, methods one and two, respectively, and

$n_1, n_2$  = the number of labs whose data was used to calculate  $R_1$  and  $R_2$ , methods one and two, respectively. For this test method the number of labs is six.

## 16. Keywords

16.1 dry vapor pressure equivalent; gasoline; hydrocarbon-oxygenate blends; mini method-atmospheric; petroleum products; vapor pressure

## SUMMARY OF CHANGES

Committee D02.08 has identified the location of selected changes to this standard since the last issue (D5482–99) that may impact the use of this standard.

(J) Paragraph 11.2, removed cyclohexane as a quality control check since it is solid at the test temperature of 0°C.

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