Designation: D 5188 – 04a

An American National Standard

Standard Test Method for Vapor-Liquid Ratio Temperature Determination of Fuels (Evacuated Chamber Method)[1]

1. Scope*

1.1 This test method covers the determination of the temperature at which the vapor formed from a selected volume of volatile petroleum product saturated with air at 0 to 1°C (32 to 34°F) produces a pressure of one atmosphere in an evacuated chamber of fixed volume. This test method is applicable to samples for which the determined temperature is between 36 and 80°C (97 and 176°F) and the vapor-liquid ratio is between 8 to 1 and 75 to 1.

NOTE 1—When the vapor-liquid ratio is 20:1, the result is intended to be comparable to the results determined by Test Method D 2533.

NOTE 2—This test method may also be applicable at pressures other than one atmosphere, but the stated precision may not apply.

1.2 This test method is applicable to both gasoline and gasoline-oxygenate blends.

1.2.1 Some gasoline-oxygenate blends may show a haze when cooled to 0 to 1°C. If a haze is observed in 11.5, it shall be indicated in the reporting of results. The precision and bias statements for hazy samples have not been determined (see Note 9).

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

1.4 *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 warnings, see 7.2 and 8.1.1.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 2533 Test Method for Vapor-Liquid Ratio of Spark-Ignition Engine Fuels

D 2892 Test Method for Distillation of Crude Petroleum (15-Theoretical Plate Column)

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 $T_{(V/L = 20)}$, *n*—the equilibrium temperature at which the partial pressure of a sample under test conditions is equal to 101.3 kPa (14.69 psia) and the vapor-liquid ratio is 20.

3.1.2 *vapor-liquid ratio of a fuel*, *n*—the ratio at a specified temperature and pressure of the volume of vapor in equilibrium with liquid to the volume of sample charged, as a liquid, at 0°C (32°F).

4. Summary of Test Method

4.1 A known volume of chilled, air-saturated sample is introduced into an evacuated, thermostatically controlled test chamber of known volume. The sample volume is calculated to give the desired vapor-liquid ratio for the chamber volume in use. After injection, the chamber temperature is adjusted until a stable chamber pressure of 101.3 kPa (14.69 psia) is achieved.

5. Significance and Use

5.1 The tendency of a fuel to vaporize in automotive engine fuel systems is indicated by the vapor-liquid ratio of the fuel.

5.2 Automotive fuel specifications generally include $T_{(V/L = 20)}$ limits to ensure products of suitable volatility performance. For high ambient temperatures, a fuel with a high value of $T_{(V/L = 20)}$, indicating a fuel with a low tendency to vaporize, is generally specified; conversely for low ambient temperatures, a fuel with a low value of $T_{(V/L = 20)}$ is specified.

6. Apparatus

6.1 Apparatus suitable for use shall employ a small volume test chamber incorporating a transducer for pressure measurements and associated equipment for thermostatically controlling the chamber temperature, evacuating the test chamber prior to sample introduction, and cleaning and purging the

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

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[2] 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.

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

chamber following the test. Critical elements of the apparatus shall meet the following specifications:

6.1.1 *Test Chamber*, constructed of stainless steel or aluminum, designed to contain between 5 and 50 mL of liquid plus vapor with a tolerance of ±1 % of the nominal capacity.

NOTE 3—The test chamber volumes of the instruments used for the 1991 cooperative test program to determine the precision and bias statements were between 5 and 15 mL of liquid plus vapor with a tolerance of ±1 % of the nominal capacity.

6.1.2 *Pressure Transducer*, minimum operational range from 1 to 177 kPa (0 to 25.6 psia) with minimum resolution of 0.1 kPa (0.01 psi) and minimum accuracy of ±0.8 kPa (±0.12 psi). The pressure measurement system shall include associated electronics and readout devices to display the resulting pressure reading.

6.1.3 *Heater*, thermostatically controlled heater capable of maintaining the test chamber within 0.1°C of the set temperature for a minimum of 5 min.

NOTE 4—The use of automated instruments that automatically adjust the temperature, after sample injection, to give a pressure of 101.3 kPa (14.69 psia) is allowed.

6.1.4 *Platinum Resistance Thermometer*, used to measure the temperature of the test chamber, having a minimum temperature range of 36 to 80°C, minimum resolution of 0.1°C (0.2°F) and minimum accuracy of ±0.1°C (±0.2°F).

6.2 *Vacuum Pump*, capable of reducing the pressure in the test chamber to less than 0.01 kPa (0.001 psia).

6.3 *Syringes*, (if required for sample introduction), gas-tight, 1- to 20-mL capacity with minimum accuracy and precision of ±1 %.

6.4 *Bath*, iced water or air, for chilling the samples and syringe (if required) to between 0 and 1°C (32 to 34°F).

6.5 *Pressure Measuring Device*, capable of measuring local station pressure 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.

6.5.1 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 6.5.

6.6 *McLeod Vacuum Gage or Calibrated Electronic Vacuum Measuring Device for Calibration*, to cover at least the range from 0.01 to 0.67 kPa (0.1 to 5 mm Hg). The calibration of the electronic vacuum measuring device shall be regularly verified in accordance with the annex section on Vacuum Sensors (A6.3) of Test Method D 2892.

7. Reagents and Materials

7.1 *Purity of Reagents*—Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society[3] where such specifications are available. 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. Reagents used for performance checks (see 10.3) shall be of at least 99 % purity.

7.2 *n* -pentane, (**Warning—**Flammable, health hazard.)

8. Sampling

8.1 *General*:

8.1.1 Conduct bulk sampling to obtain the laboratory samples in accordance with Practice D 4057 except for gasoline-oxygenate blends where water displacement is not used. The extreme sensitivity of $T_{(V/L)}$ 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. (**Warning—**Extremely flammable, harmful if inhaled or ingested. Skin irritant on repeated contact. Aspiration hazard.)

8.1.2 The size of the sample container from which the sample is taken shall be 1 L (1 qt). It shall be 70 to 80 % filled with sample.

8.1.3 The precision statement will be derived using samples in 1-L (1-qt) containers. However, samples taken in containers of other sizes as prescribed in Practice D 4057 can be used if it is recognized that the precision could be affected. In the case of referee testing, the 1-L (1-qt) sample is mandatory.

8.1.4 Perform the $T_{(V/L)}$ determination on the first test specimen withdrawn from the sample container. Do not use the remaining sample in the container for a second $T_{(V/L)}$ determination. If a second determination is necessary, obtain a new sample.

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

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

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

8.3 *Verification of Sample Container Filling*:

8.3.1 With the sample at a temperature of 0 to 1°C, take the container from the cooling bath or refrigerator and wipe dry with absorbent material. If the container is not transparent,

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[3] *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 *Annual 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.

unseal it and using a suitable gage, confirm that the sample volume equals 70 to 80 % of the container capacity (see Note 5). If the sample is contained in a transparent glass container, verify that the container is 70 to 80 % full by suitable means (see Note 5).

8.3.2 Discard the sample if the container is filled to less than 70 % by volume of the container capacity.

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

NOTE 5—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 by comparing the sample container to a like container which has the 70 and 80 % levels clearly marked, has been found suitable.

8.3.4 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 *Transparent Container Only*—Since 8.3.1 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.

8.4.2 With the sample again at a temperature of 0 to 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 vigorously. Return it to the bath or refrigerator for a minimum of 2 min.

9. Preparation of Apparatus

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

9.2 Clean and dry the test chamber to avoid contamination of the test sample.

9.3 Prior to sample introduction, visually determine from the instrument display that the test chamber pressure is stable and does not exceed 0.1 kPa (0.01 psia). If the pressure is not stable or exceeds this value, check that the chamber contains no volatile materials from a previous sample, the seals are satisfactory, the calibration of the transducer is correct, and the apparatus does not leak.

9.4 If a syringe is used for introduction of the sample, chill the syringe to between 0 and 1.0°C (32 to 34°F) in a refrigerator or ice bath before drawing in the sample. The syringe must be kept completely dry during this cooling. Place the clean and dry syringe in a water tight plastic bag and place the sealed bag in the ice bath or refrigerator maintained at 0 to 1.0°C (32 to 34°F).

9.5 Prior to introduction of the test specimen, set the temperature of the test chamber to close to the expected result, if known; or 40°C (104°F), if unknown.

10. Calibration

10.1 Calibrate the pressure transducer at least every 30 days or when needed as indicated by the performance check (see 10.3). The calibration of the transducer is accomplished using two reference points, zero pressure, and ambient barometric pressure.

NOTE 6—Pressure transducers can be sensitive to working temperature. Calibration should be performed at the test chamber temperature most frequently used.

10.1.1 Connect a McLeod gage or a calibrated electronic vacuum measuring device to the vacuum source in line with the test chamber. Apply vacuum to the test chamber. When the vacuum measuring device registers a pressure less than 0.1 kPa (0.8 mm Hg) adjust the indicator to zero or to the actual reading on the vacuum measuring device as dictated by the instrument design or manufacturer's instructions.

10.1.2 Open the test chamber to the atmosphere and observe the indicator reading. If the pressure reading is not equal to the ambient barometric pressure, adjust the indicator span control until the appropriate reading is observed. Ensure that the instrument is set to display the total pressure and not a calculated or corrected value.

10.1.3 Repeat 10.1.1 and 10.1.2 until the zero and barometric pressure read correctly without further adjustments.

10.2 Calibrate the platinum resistance thermometer and indicator used to monitor the temperature of the test chamber at least every six months against a National Institute of Standards and Technology (NIST) traceable thermometer.

10.3 Check the performance of the instrument each day it is in use by running a sample consisting of air saturated *n*-pentane, $T_{(V/L = 20)} = 36.1°C$ (96.9°F). Handle the performance check material in the same manner as a sample (see Sections 8 and 11), except that for pure compounds (see 7.1), multiple test specimens may be taken from the same container over time, provided the test specimen is air-saturated according to the procedure given in 8.4 and the spent test specimens are not reused in whole or in part. If the observed $T_{V/L = 20}$ differs by more than 0.5°C (1.0°F), check the instrument calibration (see 10.1 and 10.2).

NOTE 7—Other pure compounds having vapor pressures within the range of fuels to be tested may be used.

11. Procedure

11.1 Calculate the volume of sample required to give the desired vapor-liquid ratio using Eq 1 (12.1).

11.2 Remove the sample from the cooling bath or refrigerator, dry the exterior of the container with absorbent material, unseal and insert a chilled transfer tube or gas-tight syringe (9.4). Draw a bubble-free aliquot of sample into the syringe or transfer tube and deliver this sample to the test chamber as rapidly as possible. The total time between opening the chilled sample container and inserting/securing the syringe into the sealed test chamber shall not exceed 1 min.

11.3 Perform the analysis in accordance with the manufacturer's instructions for operation of the instrument.

11.4 Record the temperature reading from the instrument to the nearest 0.1°C (0.2°F) after the pressure indicator has remained stable at 101.3 kPa (14.69 psia) for 1 min.

NOTE 8—If the measurement is made at a pressure other than 101.3 kPa (14.69 psia) the pressure must be noted.

11.5 *Verification of Single Phase Sample*—Check the remaining sample from 8.3 for phase separation. If the sample is contained in a transparent container, this observation can be made prior to sample transfer (11.2). If 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).

12. Calculations

12.1 Use the following equation to calculate vapor-liquid ratio:

$$V/L = \frac{V - v}{v} \quad (1)$$

where:
V/L = vapor-liquid ratio
V = volume of test chamber, mL, and
v = volume of sample at 0°C (32°F), mL.

13. Report

13.1 Report $T_{(V/L)}$ temperature to the nearest 0.1°C (0.2°F) and the vapor-liquid ratio without reference to pressure.

13.2 If the sample was observed to be hazy in 11.5, report the test result as in 13.1, followed by the letter H.

NOTE 9—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 10—The inclusion of the letter H in 13.2 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 in accordance with the requirements in 13.2, it is permissible for the laboratory to report the result obtained as in 13.1, along with a statement or annotation that clearly conveys to the data recipient that the sample analyzed was hazy.

14. Precision and Bias

14.1 *Precision*—The precision of this test method for measuring the $T_{(V/L=20)}$ *value of gasoline and gasoline-oxygenate blends as determined by the statistical examination of interlaboratory test results is as follows:*

14.1.1 *Repeatability*— The difference between two 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:

$$0.6°C\ (1.1°F) \quad (2)$$

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

$$0.9°C\ (1.6°F) \quad (3)$$

NOTE 11—The precision data were developed in a 1991 cooperative testing program based on the duplicate analysis of twelve samples of gasolines and gasoline-oxygenate blends by a minimum of five laboratories. The test fuels had a range of oxygenate content (ethanol and MTBE) from 0 to 15 % by volume and a $T_{(V/L=20)}$ range from approximately 37.8°C (100°F) to 60.0°C (140°F).

14.2 *Bias*:

14.2.1 *Absolute*—Since there is no accepted reference material suitable for determining the bias for this test method, no statement on bias is being made.

14.2.2 Based on statistical analyses of the data from the 1991 cooperative testing program (see Note 11), the following relative bias statements were determined for $T_{(V/L=20)}$:

14.2.2.1 *Test Method D 5188 Test Instruments*[3]— No significant bias was determined between the data using the two test instruments.

14.2.2.2 *V/L Test Methods*—No significant bias was determined between the data determined using Test Methods D 5188 and D 2533, using mercury as the containing fluid.

15. Keywords

15.1 gasoline; gasoline-oxygenate blends; petroleum products;$T_{(V/L = 20)}$; vapor-liquid ratio

SUMMARY OF CHANGES

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D 5188–04) that may impact the use of this standard.

(1) Added Test Method D 2892 to the Referenced Documents.
(2) Revised 6.6.
(3) Revised 10.1.1.

Subcommittee D02.08 has identified the location of selected changes to this standard since the last issue (D 5188–99) that may impact the use of this standard.

(1) Updated 10.3 to allow the performance check material to be used more than once from the same container if they are pure compounds (based on the information in 7.1), so that the same material can be used for control chart purposes.

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