Designation: D 2533 – 99

An American National Standard

Standard Test Method for Vapor-Liquid Ratio of Spark-Ignition Engine Fuels[1]

1. Scope*

1.1 This test method covers a procedure for measuring the volume of vapor formed at atmospheric pressure from a given volume of gasoline. The ratio of these volumes is expressed as the vapor-liquid (V/L) ratio of the gasoline at the temperature of the test.

1.2 Dry glycerol can be used as the containing liquid for nonoxygenated fuels.

1.3 Mercury can be used as the containing liquid with both oxygenated and nonoxygenated fuels. Because oxygenates in fuels may be partially soluble in glycerol, gasoline-oxygenate blends must be tested using mercury as the containing fluid.

NOTE 1—Test Method D 4815 can be used to determine the presence of oxygenates in fuels.[2]

1.4 The values stated in both inch-pound and SI units are to be regarded separately as the standard. The units given in parentheses are for information only.

1.5 *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 Section 7 and Note 11.

2. Referenced Documents

2.1 *ASTM Standards:*

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[3]

D 4815 Test Method for Determination of MTBE, ETBE, TAME, DIPE, *tertiary*-Amyl Alcohol and C_1 to C_4 Alcohols in Gasoline by Gas Chromatography[3]

D 5188 Test Method for Vapor-Liquid Ratio Temperature Determination of Fuels (Evacuated Chamber Method)[3]

E 1 Specification for ASTM Thermometers[4]

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 liquid sample charged, at 0°C (32°F).

NOTE 2—This ratio differs from the absolute vapor-liquid ratio because corrections are not made for (*1*) liquid sample expansion with increasing temperature, (*2*) decrease in liquid sample volume by vaporization, and (*3*) dissolved air in the liquid sample.

4. Summary of Test Method [5]

4.1 A measured volume of liquid fuel at 32 to 40°F (0 to 4°C) is introduced through a rubber septum into a glycerol or mercury filled buret. The charged buret is placed in a temperature-controlled water bath. The volume of vapor in equilibrium with liquid fuel is measured as the desired temperature or temperatures and the specified pressure, usually 760 mm Hg. The vapor-liquid ratio (V/L) is then calculated.

4.2 If it is desired to know the temperature corresponding to a given V/L, the vapor-liquid ratio is determined at several temperatures and the selected pressure. The results are plotted and the temperature read at the given V/L.

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[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

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In 1967, this method was adopted as standard without revision.

[2] Test Method D 5188 is applicable to both gasoline and gasoline-oxygenate blends for measuring this property and does not require the use of mercury.

[3] *Annual Book of ASTM Standards*, Vol 05.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] Another procedure for measuring the vapor-liquid ratio of gasoline, which gives equivalent results, and employing the Sunbury Vapor-Liquid Ratio Apparatus, is described in Appendix VII, p. 783, *1964 Annual Book of ASTM Standards*, Part 17.

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

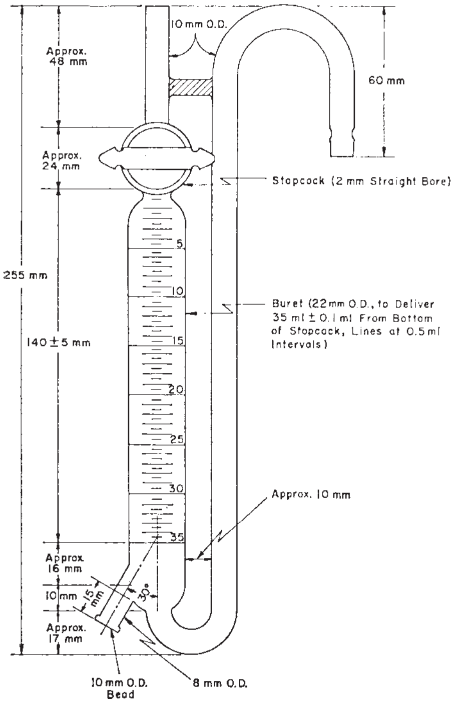

FIG. 1 Vapor-Liquid Ratio Buret

5. Significance and Use

5.1 The tendency of a fuel to vaporize in common automobile fuel systems is indicated by the vapor-liquid ratio of that fuel at conditions approximating those in critical parts of the fuel systems.

6. Apparatus

6.1 *V/L Buret*,[6] constructed of borosilicate glass according to the dimensions shown in Fig. 1. The short bottom arm is closed with a rubber serum bottle stopper that is alcohol resistant; U.S. Army Medical Corps type.

6.2 *Pressure Control Equipment*, including the following:

6.2.1 A 250-mL leveling bulb containing dry glycerol or mercury, attached to the V/L buret by rubber tubing as shown in Fig. 2. The top of the leveling bulb must be fitted with an air-drying device. When mercury is used the drying tube must contain mercury vapor absorbent[7] packed between balls of glass wool. This minimizes the escape of mercury vapor.

6.2.2 Means for measuring the difference in liquid level between the V/L buret and the leveling bulb. A millimetre scale for measuring the heights above a level table top is suitable for use with glycerol. A cathetometer or similar optical leveling device must be used with mercury.

6.2.3 *Barometer*, accurate to 0.5 mm Hg.

6.2.4 A mercury manometer with 1-mm divisions, required only for measurements at pressures appreciably above or below the prevailing atmospheric pressure (see Note 12). The manometer shall be connected with rubber tubing to one arm of a glass T-tube, the other arms of which shall be connected to the top of the glycerol (mercury) leveling bulb and to a stopcock or pinch clamp.

6.3 *Water Bath*, glass, at least 10.5 in. (266.7 mm) deep, stirred and thermostatically controlled, capable of being adjusted to any temperature between 90°F (3°C) and 180°F (82°C) and maintaining the water temperature within ±0.2°F (0.1°C) of the desired temperature.

6.4 *Thermometers*—ASTM Precision Thermometers having a range from 77 to 131°F and 122 to 176°F (25 to 55°C and 50 to 80°C) and conforming to the requirements for Thermometers 64F and 65F (64C and 65C) as prescribed in Specification E 1 or, for the limited ranges they cover, the ASTM Saybolt Viscosity Thermometers having a range from 94 to 108°F, 120 to 134°F, and 134 to 148°F (34 to 42°C, 49 to 57°C, and 57 to 65°C) and conforming to the requirements for Thermometers 18F, 19F, and 20F (18C, 19C, and 20C) as prescribed in Specification E 1.

NOTE 3—Digital temperature indicating devices with thermocouple, resistance, or thermistor probes are also acceptable if the system accuracy is at least 0.2°F (0.1°).

6.5 *Cooling Bath*, containing ice and water at 32 to 36°F (0 to 2°C).

6.6 *Hypodermic Syringe*, 0.5 and 1.0-mL Hamilton syringes with Chaney adaptors, fitted with a No. 20 gage, 2-in. (51-mm) needle.

6.7 *Calibration Accessories*, including an analytical balance accurate to 1 mg or better, a capillary stopcock, and weighing vials. A calibration stopper[3] for the V/L buret stopcock, that is, an extra three-way solid stopcock stopper with an outlet through the end of the stopper, is convenient during calibration.

7. Reagent

7.1 *Acetone*

NOTE 4—**Warning:** Extremely Flammable.

7.2 *Chromic Acid*

NOTE 5—**Warning:** Causes severe burns. A recognized carcinogen. A strong oxidizer. Contact with organic material may cause fire.

7.3 *Glycerol*, at least 99 % pure (1 % maximum water content). Store in a dry screw-capped bottle to prevent absorption of moisture.

7.4 *Mercury*

NOTE 6—**Warning:** Poison. May be harmful or fatal if inhaled.

8. Handling of Samples

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 resulting changes in composition is such as to require utmost precaution and the most meticulous care in handling the samples.

NOTE 7—**Warning:** Extremely flammable, harmful in inhaled or ingested. Skin irritant on repeated contact. Aspiration hazard.

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[6] A V/L buret and calibration stopper that have been found satisfactory for this purpose may be obtained from the Chatas Glass Co., 570 Broadlawn Terrace, Drawer H, Vineland, NJ 08360.

[7] J. T. Baker Resisorb has been found satisfactory for this purpose.

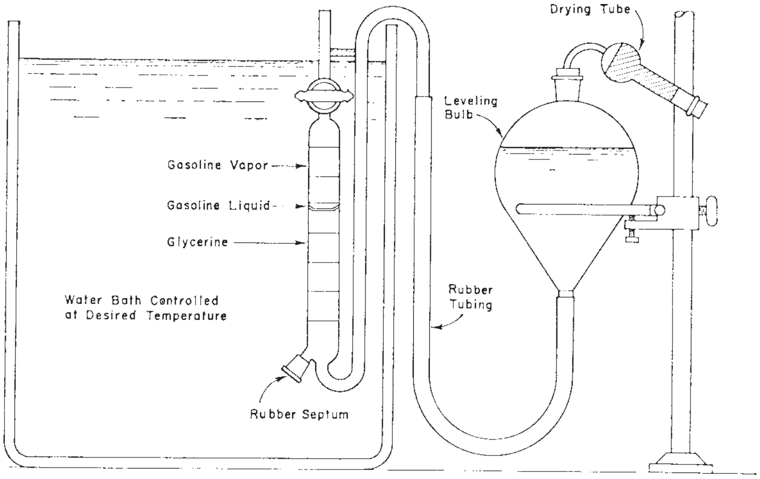

FIG. 2 Arrangement of Apparatus

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 was derived using samples in 1 L (1 qt) containers. However, samples taken in containers of other sizes 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 size 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 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 ice bath or refrigerator 0 to 1°C (32 to 34°F) prior to opening the sample container. Ensure sufficient time to reach this temperature by direct measurement 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 After the sample reaches thermal equilibrium at 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 8). If the sample is contained in a transparent glass container, verify that the container is 70 to 80 % full by suitable means (see Note 8).

NOTE 8—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.

8.3.2 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, of the container capacity, pour out enough sample to bring the container contents to within the 70 to 80 % volume range. Do not return any sample to the container once it has been withdrawn.

8.3.4 Reseal the container, if necessary, and return it 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 of 0 to 1°C, take the container from the cooling bath, 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 cooling bath or refrigerator for a minimum of 2 min.

8.4.2 *Transparent Containers 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. After performing this task, proceed with 8.4.1.

9. Calibration

9.1 Calibrate the V/L buret and the hypodermic syringe and correct the subsequent experimental readings from the calibration curves obtained.

9.2 Fill the hypodermic syringe with air-free distilled water at 77°F (25°C), expel any air bubbles, and depress the plunger exactly to a calibration mark. Discharge the contents, to the bottom of plunger travel, into a weighing vial and weigh. Repeat at 0.2-mL intervals from 0.2 to 1.0 mL and average the results from two or more determinations. Calculate the volumes at 32°F (0°C) from the weights of water (Note 7) and prepare a calibration curve.

NOTE 9—One gram of water at 77°F (25°C) = 1.0036 mL volume at 32°F (0°C) in resistance glass or 1.0038 mL volume at 32°F (0°C) in borosilicate glass.[8]

9.3 Clean the V/L buret thoroughly, rinse with distilled water, attach a capillary stopcock with rubber tubing snug to the side arm, and fill the system completely with air-free distilled water at 77°F (25°C). Calibrate at 5-mL intervals starting from the bottom of the buret stopcock (Note 9), by weighing water drained through the capillary stopcock into weighing vials. Repeat and average for two or more determinations. Calculate the volumes at 122°F (50°C) from the weights of water (Note 10) and prepare a calibration curve.

NOTE 10—Use of the calibration stopper described in 6.1 facilitates setting the water level at the bottom of the stopcock.

NOTE 11—One gram of water at 77°F (25°C) = 1.0042 mL volume at 122°F (50°C) in borosilicate glass.[8]

10. Preparation for Test

10.1 *Cleaning Equipment*—Before assembly, thoroughly clean and dry all the equipment, including burets, hypodermic syringes, leveling bulbs, and connecting tubing. Lubricate the buret stopcock with high-vacuum silicone stopcock grease and attach a spring or rubber band to hold securely in place. Thereafter, to clean the buret between tests, rinse thoroughly by flushing with water (Note 11), then with acetone, and dry with air. Clean the hypodermic syringe and needle with acetone and dry in an air stream.

NOTE 12—If a film is noted in the buret, clean it further with sodium dichromate-sulfuric acid solution. **Warning**—Causes severe burns. A recognized carcinogen. A strong oxidizer. Contact with organic material may cause fire.

10.2 *Filling System With Glycerol (Mercury)*—Connect the leveling bulb to the buret with rubber tubing, fill the bulb with clean, dry glycerol (mercury) at room temperature and attach the air-drying device. If mercury is used, be certain that the drying tube contains mercury vapor absorbent (see 6.2.1). Draw glycerol (mercury) into the buret by applying vacuum to the stopcock, and expel all air bubbles from the tubing and rubber septum attachment. It may be necessary to loosen the rubber septum to release air trapped therein.

10.3 *Preparation of Apparatus for Subsequent Tests*—Due to the partial solubility of certain oxygenates in the glycerol during testing, separate methods are used for subsequent test preparation using glycerol or mercury as the containing fluid.

10.3.1 *Glycerol Containing Fluid*—The glycerol in the buret is replaced between each test. Following the completion of V/L measurements with a fuel, allow the buret to cool until the vapor has condensed and glycerol has refilled the buret. Clamp the tubing between the leveling bulb and the buret as near the buret as possible. Remove the buret from the tubing, open the stopcock and pour the glycerol out of the buret. Fill a clean buret for the next test as described in 10.2. Add new, dry glycerol to the leveling bulb to replace the displaced used glycerol.

10.3.2 *Mercury Containing Fluid*—Following the completion of V/L measurements with a fuel, allow the buret to cool until the vapor has condensed and mercury has refilled the buret. While slowly lowering the leveling bulb, slowly open the stopcock and allow all but a few millilitres of mercury to flow back into the leveling bulb. Apply a pinch clamp to the rubber tubing as near the buret as possible and remove the buret. Attach the rubber tubing to a clean buret, and fill the buret as described in 10.2. Add new mercury to the leveling bulb to replace the displaced mercury as needed.

10.4 *Preparation of Hypodermic Syringe*— Assemble the syringe and needle and insert the needle tip in a small rubber stopper. Cover with drained chipped ice or chill by other means to 32 to 36°F (0 to 2°C).

10.5 *Adjustment of Constant-Temperature Bath*—Adjust the water bath to the desired test temperature and maintain at that temperature ±0.2°F (0.1°C).

11. Procedure

11.1 Read and record the barometric pressure.

11.2 With the V/L buret at room temperature or somewhat above (Note 12) and everything in readiness, carry out the following steps as quickly as possible. Open the chilled sample container, tip it so as to reach the liquid with the hypodermic syringe needle, taking caution to prevent water from reaching the sample and partially fill the syringe. Point the needle upward and dispel the contents to eliminate all air bubbles. Immediately refill the syringe from the sample container and check for air or vapor in the syringe (Note 13).

NOTE 13—Glycerol (mercury) in the buret may be somewhat above room temperature due to warming in the previous test, but should not be so warm as to cause the sample to vaporize when injected.

NOTE 14—Vapor may form if the sample is drawn in too rapidly. If this happens, repeat the sampling with a clean, chilled syringe.

NOTE 15—Use cotton gloves to reduce heat transfer from the hands to the syringe.

11.2.1 Depress the plunger exactly to the mark for the sample size desired, then, taking care not to disturb the plunger position, insert the needle through the rubber septum full length into the V/L buret. Depress the plunger all the way to inject the sample, and withdraw the needle. Use a 1-mL sample if the highest V/L ratio expected for the sample is less than 35. For higher V/L ratios, use a smaller sample sufficient to give 20 to 35 mL of vapor at the highest temperature to be tested.

11.3 Record the volume of sample charged, corrected by means of the calibration specified in 9.1 and 9.2.

11.4 Transfer the charged buret to the water bath set at the desired temperature and position so that the water level comes above the stopcock barrel.

11.5 As vapor forms in the buret, adjust the height of the leveling bulb to give the desired pressure on the sample. If glycerol is used as the confining medium, raise the level of glycerol in the reservoir 10.80 mm above the level of the glycerol in the buret for every mm of mercury that the barometric pressure is below 760 mm Hg; or lower it by a like amount for every mm that the barometric pressure is above 760 mm Hg. If mercury is used as the confining medium, raise the level of the mercury in the reservoir 1.0 mm above the level of the mercury in the buret for every mm of mercury that the

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[8] Meites, L., *Handbook of Analytical Chemistry*, McGraw-Hill Book Co, New York, NY, 1936, pp. 3–237, 3–238.

barometric pressure is below 760 mm Hg; or lower it by a like amount for every mm that the barometric pressure is above 760 mm Hg.

NOTE 16— If the difference between the atmospheric pressure in the laboratory and the pressure for which the V/L measurement is desired is too great for convenient correction by means of the leveling bulb alone, use the mercury-filled manometer described in 6.2.4 to set the pressure. Keep the level of glycerol in the leveling bulb the same as that in the buret and apply pressure or vacuum gently to the air space in the leveling bulb as needed to obtain the desired pressure on the manometer.

NOTE 17—With some narrow boiling gasoline fractions, super heating may occur and no vapors are formed in the buret, even after immersion for as long as 15 min or more. When vaporization takes place, it does so rapidly and sometimes explosively. With these samples, it is recommended that injection be carried out with warm glycerol in the buret, such that a few millilitres of vapor are formed immediately after injection. The temperature of the glycerol is dependent on the sample composition but in general should not be more than 50°F (28°C) above ambient. With mercury as the confining medium this phenomenon has not been observed.

11.6 Without removing the buret from the water bath, shake it sufficiently to agitate the liquid sample, but not so vigorously as to disperse droplets of sample into the glycerol.

NOTE 18—Shaking is not necessary if mercury is used as the confining liquid because the superior heat transfer properties of the mercury will result in rapid thermal equilibrium of the system. With mercury as the confining liquid shaking is discouraged because of the danger of breakage of the glass caused by accidental impact.

11.7 Readjust the height of the leveling bulb, if necessary, to give the desired pressure on the sample. Because of mercury's high density, the use of a cathetometer or similar optical leveling device is necessary to minimize pressure errors.

NOTE 19—Any spilled mercury, and any that may be purged from the equipment, should be placed in an airtight closed vessel. This recovered mercury may be sent to a reprocessor, who can provide shipping instructions. (Names of mercury reprocessors are available from ASTM Headquarters.) To minimize spillage, a catch pan that is large enough to contain all the mercury in case of failure should be placed under the apparatus.

It is useful to have a 1 L vacuum flask available connected to a vacuum source. Introduce a few ounces of a solid mercury vapor absorbent in the flask. Connect one end of a piece of tubing to the top of the flask and insert a glass eyedropper at the other end. Use the eyedropper end to pick up spilled mercury and to remove the spent sample and excess mercury from the top of the burets at the end of the run.

11.8 Read the volume of vapor to the nearest 0.1 mL. Repeat until the volume remains constant for at least 2 min. Record the volume, corrected by means of the calibration specified in 9.1 and 9.3, the bath temperature, and the pressure.

11.9 If the vapor-liquid ratio is also desired at another temperature, either adjust the temperature of the bath accordingly, or transfer the buret to another bath at the desired temperature. Repeat the operations described in 11.5 and 11.8.

NOTE 20—During the cleaning procedure note that small amounts of hydrocarbons can be trapped between the glass and the Hg column. If they are not removed, they may contaminate the next sample. After removal of the spent sample at the end of the run, close the valve at the top of the tube and immerse the tube in the hottest bath available at that moment for about 5 min. Then raise the tube 50 to 75 cm and hold in this position for 5 to 10 s. The trapped hydrocarbons will rise to the top of the tube, from where they can be removed.

12. Calculation

12.1 For each determination calculate the vapor-liquid ratio as follows:

$$\text{Vapor}-\text{liquid ratio} = A/B \tag{1}$$

where:

A = volume of vapor, mL and

B = volume of sample charged, mL.

13. Report

13.1 Report the vapor-liquid ratio(s) to the nearest 0.1 unit and the corresponding temperature in degrees Fahrenheit or Celsius to 0.1°. Also report the pressure in millimetres of mercury if other than 760. If not reported, the pressure is understood to be 760 mm Hg.

13.2 If vapor-liquid ratios have been determined at a series of at least three temperatures, and if values at intermediate temperatures, or temperatures for intermediate V/L ratios are desired, proceed as follows: Plot the experimental results in the form of a curve of vapor-liquid ratio versus temperature, smoothing if necessary, on graph paper which can be read easily to 0.1 V/L and 0.2°F (or 0.1°C). Read from this curve the desired intermediate V/L and temperature values. Report V/L, temperature, and pressure as prescribed in 12.1, but accompany the result with the word "interpolated." Thus, for example, report "V/L = 15.0 interpolated at 127.6°F" or "V/L = 12.3 interpolated at 125.0°F and 665 mm Hg."

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[9] is as follows:

14.1.1 *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 the following values only in one case in twenty:

$$\text{glycerol } 1.0°C\ (1.8°F) \tag{2}$$

$$\text{mercury } 1.4°C\ (2.5°F)$$

14.1.2 *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 the following values only in one case in twenty:

$$\text{glycerol } 1.3°C\ (2.3°F) \tag{3}$$

$$\text{mercury } 1.6°C\ (3.0°F)$$

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.

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[9] Supporting data are available from ASTM Headquarters. Request D02-1244.

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

14.2.2.1 *Containing Fluids*—No significant bias was determined between the methods using glycerol or mercury as the containing fluid with the gasolines without oxygenates.

14.2.2.2 *Glycerol Containing Fluid*—Using the mercury method as a standard, a small but significant bias of + 0.5°C (0.9°F) was found for the gasoline-15 V % MTBE blend data relative to the hydrocarbon-only gasoline data.

14.2.2.3 *Test Methods*—No significant bias was determined between this test method and Test Method D 5188 data.

NOTE 21—The precision and bias data[8] were developed in a 1991 cooperative testing program based on the duplicate analysis of twelve 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).

SUMMARY OF CHANGES

Committee D 02.08 has identified the location of selected changes to this standard since the D 2533–96 that may impact the use of this standard.

(*1*) A new Note 8 has been added after 8.3.1, to provide guidance as to how to verify whether a transparent or non-transparent container is filled from 70 to 80 % by volume of the sample container capacity.

(*2*) In 8.3.4, it has been explicitly stated to reseal the sample container, if necessary.

(*3*) Paragraph 8.4 has been subdivided into sections 8.4.1 (transparent containers only) and 8.4.2 to ensure that transparent and non-transparent containers are opened the same number of times prior to analysis.