Designation: D381 – 12 (Reapproved 2017)

Standard Test Method for Gum Content in Fuels by Jet Evaporation[1]

This standard is issued under the fixed designation D381; 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 (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of the existent gum content of aviation fuels, and the gum content of motor gasolines or other volatile distillates in their finished form, (including those containing alcohol and ether type oxygenates and deposit control additives—see Note 7 for additional information) at the time of test.

1.2 Provisions are made for the determination of the heptane insoluble portion of the residue of non-aviation fuels.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3.1 The accepted SI unit of pressure is the Pascal (Pa); the accepted SI unit for temperature is degrees Celsius.

1.4 **WARNING**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website—http://www.epa.gov/mercury/faq.htm—for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law.

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 warning statements, see 6.4, 7.4, and 9.1.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1655 Specification for Aviation Turbine Fuels

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

E1 Specification for ASTM Liquid-in-Glass Thermometers

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

2.2 *Energy Institute Standard:*[3]

IP Standard Methods for Analysis and Testing of Petroleum Products

IP 540 Determination of the existent gum content of aviation turbine fuel – jet evaporation method

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *existent gum, n*—the evaporation residue of aviation fuels, without any further treatment.

3.2 For non-aviation fuels, the following definitions apply.

3.3 *solvent washed gum content, n*—the residue remaining when the evaporation residue (see 3.4) has been washed with heptane and the washings discarded.

3.3.1 *Discussion*—For motor gasoline or non-aviation gasoline, solvent washed gum content was previously referred to as existent gum.

3.4 *unwashed gum content, n*—the evaporation residue of the product or component under test, without any further treatment.

4. Summary of Test Method

4.1 When testing either aviation or motor gasoline, a 50 mL ± 0.5 mL quantity of fuel is evaporated under controlled

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.14 on on Stability, Cleanliness and Compatibility of Liquid Fuels.

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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.

[3] Available from Energy Institute, 61 New Cavendish St., London, W1G 7AR, U.K., http://www.energyinst.org.

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

TABLE 1 Schedule of Test Conditions

Sample Type	Vaporizing Medium	Operating Temperature	
		Bath	Test Well
Aviation and motor gasoline	air	160 °C to 165 °C	150 °C to 160 °C
Aviation turbine fuel	steam	232 °C to 246 °C	229 °C to 235 °C

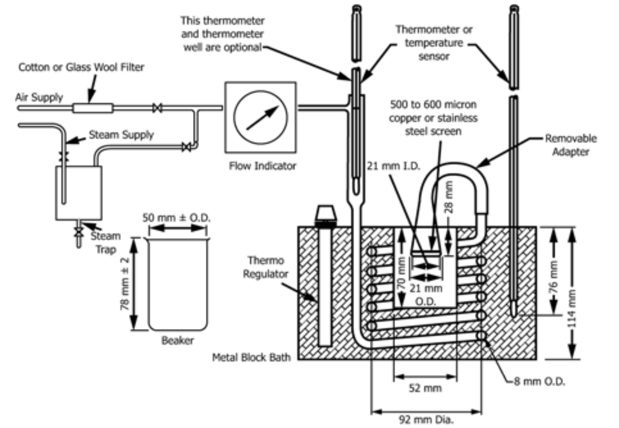

FIG. 1 Apparatus for Determining Gum Content by Jet Evaporation

conditions of temperature and flow of air (see Table 1). When testing aviation turbine fuel, a 50 mL ± 0.5 mL quantity of fuel is evaporated under controlled conditions of temperature and flow of steam (see Table 1). For aviation gasoline and aviation turbine fuel, the resulting residue is weighed and reported as milligrams per 100 mL. For motor gasoline, the residue is weighed before and after extracting with heptane and the results reported as milligrams per 100 mL.

NOTE 1—Specification D1655 allows the existent gum of aviation turbine fuels to be determined by either Test Method D381 or IP 540, with Test Method D381 identified as the referee test method. Test Method D381 specifically requires the use of steam as the evaporating medium for aviation turbine fuels, whereas IP 540 allows either air or steam as the evaporating medium for aviation turbine fuels.

5. Significance and Use

5.1 The true significance of this test method for determining gum in motor gasoline is not firmly established. It has been proved that high gum can cause induction-system deposits and sticking of intake valves, and in most instances, it can be assumed that low gum will ensure absence of induction-system difficulties. The user should, however, realize that the test method is not of itself correlative to induction-system deposits. The primary purpose of the test method, as applied to motor gasoline, is the measurement of the oxidation products formed in the sample prior to or during the comparatively mild conditions of the test procedure. Since many motor gasolines are purposely blended with nonvolatile oils or additives, the heptane extraction step is necessary to remove these from the evaporation residue so that the deleterious material, gum, may be determined. With respect to aviation turbine fuels, large quantities of gum are indicative of contamination of fuel by higher boiling oils or particulate matter and generally reflect poor handling practices in distribution downstream of the refinery.

6. Apparatus

6.1 *Balance,* capable of weighing test specimens to the nearest 0.1 mg.

6.2 *Beakers,* of 100 mL capacity, as illustrated in Fig. 1. Arrange the beakers in sets, the number in each set depending upon the number of beaker wells in the evaporating bath. Mark each beaker in the set, including the tare beaker, with an identifying number or letter.

6.3 *Cooling Vessel*—A tightly covered vessel, such as a desiccator without desiccant, for cooling the beakers before weighing.

NOTE 2—The use of a desiccant could lead to erroneous results.

6.4 *Evaporation Bath* (**Warning—**If a liquid-filled evaporation bath is used, care must be taken that the flash point of the liquid used is at least 30 °C higher than the highest bath temperature expected.) Either a solid metal block bath or a liquid bath, electrically heated, and constructed in accordance with the general principles shown in Fig. 1 may be used. (Although all dimensions are given in SI units, older baths conforming to Test Method D381 – 94, or earlier, are suitable.) The bath should have wells and jets for two or more beakers. The rate of flow from each outlet when fitted with the conical adapters with 500 µm to 600 µm copper or stainless steel screens should not differ from 1000 mL ⁄s by more than 15 %. A liquid bath, if used, shall be filled to within 25 mm of the top with a suitable liquid. Temperature shall be maintained by means of thermostatic controls or by refluxing liquids of suitable composition.

6.5 *Flow Indicator,* as illustrated in Fig. 1, such as a flowmeter, capable of metering a flow of air or steam equivalent to 1000 mL ⁄s for each outlet.

NOTE 3—Alternatively, a pressure gage may be used to meter the flow of air or steam equivalent to 1000 mL ⁄s ± 150 mL ⁄s for each outlet.

6.6 *Sintered Glass Filtering Funnel,* coarse porosity, 150 mL capacity.

6.7 *Steam*—Supply by suitable means capable of delivering to the bath inlet the required amount of steam at 232 °C to 246 °C.

6.8 *Temperature Sensor,* liquid-in-glass thermometer conforming to the requirements in the specification(s) for ASTM 3C/IP73C, found in Specification E1, or another temperature measuring device or system, or both, of at least equivalent accuracy and precision over a temperature range from –5 °C to 400 °C.

6.9 *Graduated Cylinders,* with spout, capable of measuring 50 mL ± 0.5 mL.

6.10 *Handling Equipment,* forceps (stainless steel, spade ended) or tongs (stainless steel) for use in handling the beakers and conical jets, as required by this test method.

7. Materials

7.1 *Air*—Supply of filtered air at a pressure not more than 35 kPa.

7.2 *Gum Solvent*—A mixture of equal volumes of toluene and acetone.

7.3 *Heptane*—Minimum purity of 99.7 %.

7.4 *Steam*—Supply of steam free of oily residue and at a pressure not less than 35 kPa. (**Warning—**If a steam superheater is used, there may be exposed hot surfaces on the steam superheater. Avoid contact with exposed skin by use of protective equipment as required.)

8. Assembly of Air-Jet Apparatus

8.1 Assemble the air-jet apparatus as shown in Fig. 1. With the apparatus at room temperature, adjust the air flow to give a rate of 600 mL ⁄s ± 90 mL ⁄s for the outlet under test. Check the remaining outlets for uniform air flow.

NOTE 4—A rate of 600 mL ⁄s ± 90 mL ⁄s from each outlet, at room temperature and atmospheric pressure, will ensure delivery of 1000 mL ⁄s ± 150 mL ⁄s at the temperature of 155 °C ± 5 °C for each outlet. It is recommended to follow the manufacturers' instructions to verify total flow/s (600 mL ⁄s air flow × number of outlets = total flow/s) and uniformity from each outlet.

8.2 Apply heat to the evaporation bath (see 6.4) until the temperature of the bath is between 160 °C and 165 °C. Introduce air into the apparatus at a rate indicated on the flow indicator (see 6.5) from the exercise carried out in 8.1. Measure the temperature in each well with the temperature sensor (see 6.8) placed with the bulb or sensor tip resting on the bottom of the beaker in the well. Do not use any well having a recorded temperature outside the range from 150 °C to 160 °C.

9. Assembly of Steam-Jet Apparatus

9.1 Assemble the steam-jet apparatus as shown in Fig. 1. (**Warning—**The sample and solvent vapors evaporated during the performance of this test procedure can be extremely flammable or combustible and hazardous from the inhalation standpoint. The evaporation bath must be provided with an effective exhaust hood to control such vapors and reduce the risk of thermal explosion.)

9.2 To place the apparatus in operation, apply heat to the bath. When the temperature reaches 232 °C, slowly introduce dry steam into the system until a rate of 1000 mL ⁄s ± 150 mL ⁄s for each outlet is reached (see 10.2). Regulate the temperature of the bath to a range from 232 °C to 246 °C to provide a well temperature of 232 °C ± 3 °C. Measure the temperature with the temperature sensor, placed resting on the bottom of a beaker in one of the bath wells with the conical adapter in place. Any well having a temperature that differs by more than 3 °C from 232 °C is not suitable for standard tests.

10. Calibration and Standardization

10.1 *Air Flow:*

10.1.1 Verify or calibrate the air flow to ensure all outlets meet the 600 mL ⁄s ± 90 mL ⁄s air flow requirement as measured at room temperature and atmospheric pressure. Refer to the instrument manufacturer instructions for specific guidance on performing the air flow calibration procedure. Note the setting of the flow indicator device for use with air and use this setting for subsequent tests.

10.1.1.1 One way to calibrate the air flow is to use a calibrated flow indicator device, such as a flowmeter, separate from the device specified in 6.5, to check the air flow rate at each outlet directly at room temperature and atmospheric pressure. To obtain accurate results, ensure that the back pressure of the flowmeter is less than 1 kPa.

10.1.1.2 Alternatively, another way to calibrate the air flow is to measure and adjust as appropriate the total air flow rate (mL/s) supplied to the outlets. The total air flow rate equals the expected air flow rate at each outlet times the number or outlet positions (for example, instrument has 5 positions and a total air flow rate measurement of 3000 mL ⁄s, indicating an expected air flow rate of 600 mL ⁄s at each outlet). Once verifying the total flow supplied to the outlets is at the appropriate rate, perform uniformity checks by comparing the relative air flow rates at each outlet position versus the requirements in 10.1.1.

10.2 *Steam Flow:*

10.2.1 Verify or calibrate the steam flow to ensure all outlets meet the 1000 mL ⁄s ± 150 mL ⁄s steam flow requirement. Refer to the instrument manufacturer instructions for specific guidance on performing the steam flow calibration procedure. Note the setting of the flow indicator device for use with steam and use this setting for subsequent tests.

10.2.1.1 One way to calibrate the steam flow, is to attach a copper tube to a steam outlet and extend the tube into a 2 L graduated cylinder that has been filled with crushed ice and water that has been previously weighed. Exhaust the steam into the cylinder for approximately 60 s. Adjust the position of the cylinder so that the end of the copper tube is immersed in the water to a depth of less than 50 mm to prevent excessive back pressure. After the appropriate time has elapsed, remove the copper tube from the cylinder and weigh the cylinder. The gain in mass represents the amount of steam condensed. Calculate the steam rate (mL/s) as follows:

$$R = (M - m)1000/kt \quad (1)$$

where:

R = steam rate (mL/s),
M = mass of graduated cylinder with condensed steam, g,
m = mass of graduated cylinder and ice, g,
k = mass of 1000 mL of steam at 232 °C at atmospheric pressure = 0.434 g, and
t = condensing time, s.

11. Procedure

11.1 Wash the beakers, including the tare, with the gum solvent until free of gum. Rinse thoroughly with water and immerse in a mildly alkaline or neutral pH laboratory detergent cleaning solution.

11.1.1 The type of detergent and conditions for its use need to be established in each laboratory. The criterion for satisfactory cleaning shall be a matching of the quality of that obtained with chromic acid cleaning solutions on used beakers (fresh chromic acid, 6 h soaking period, rinsing with distilled water and drying). For this comparison visual appearance and mass loss on heating the glassware under test conditions may be used. Detergent cleaning avoids the potential hazards and inconveniences related to handling corrosive chromic acid solutions. The latter remains as the reference cleaning practice and as such may function as an alternate to the preferred procedure-cleaning with detergent solutions.

11.1.2 Remove the beakers from the cleaning solution by means of stainless steel forceps or tongs (see 6.10) and handle only with forceps or tongs thereafter. Wash the beakers thoroughly, first with tap water and then with distilled water, and dry in an oven at 150 °C for at least 1 h. Cool the beakers for at least 2 h in the cooling vessel placed in the vicinity of the balance.

11.2 Select the operating conditions, corresponding to the aviation and motor gasoline or aircraft turbine fuel under test, from the data given in Table 1. Heat the bath to the prescribed operating temperature. Introduce air or steam to the apparatus and adjust the total flow to that established in 8.1 or 9.2. If an external preheater is used, regulate the temperature of the vaporizing medium to give the prescribed test well temperature.

11.3 Weigh the tare and test beakers to the nearest 0.1 mg. Record the masses.

11.4 If suspended or settled solid matter is present, mix or shake the contents of the sample container thoroughly using an appropriate method. At atmospheric pressure, immediately filter a quantity of the sample through a sintered-glass funnel of coarse porosity (see 13.3). Treat the filtrate as described in 11.5 – 11.7.

11.5 Measure a 50 mL ± 0.5 mL test specimen in a graduated cylinder (see 6.9), and transfer it to a weighed beaker (see 6.2). (See Practice D4057 regarding sampling.) Use one beaker for each test specimen to be tested, and fill each beaker except the tare. Place the filled beakers and the tare in the evaporation bath, keeping the elapsed time between placing the first and last beaker in the bath to a minimum. When using air as the evaporation medium in an evaporation bath without a mechanical means to raise and lower the conical jets, use forceps or tongs (see 6.10) to replace the conical jet as each individual beaker is placed in the bath. When using steam, allow the beakers to heat for 3 min to 4 min before using forceps or tongs to replace the conical jet (or lowering the conical jets by mechanical means), which shall be preheated either in the steam stream by placing on the steam outlet and positioning the jet between the beaker wells or on top of the hot evaporation bath prior to attaching to the outlets. Using forceps or tongs (if needed), center each conical jet above the surface of the liquid, and start the flow of air or steam, adjusting it to the specified rate. Maintain the temperature and rate of flow, and allow the test specimen to evaporate for 30 min ± 0.5 min.

NOTE 5—When introducing the flow of air or steam, care should be taken to avoid splashing of the test specimen as this may lead to erroneous low results.

11.6 At the end of the heating period, remove the conical jets using forceps, tongs (see 6.10), or other suitable means, and transfer the beakers from the bath to the cooling vessel. Place the cooling vessel in the vicinity of the balance for at least 2 h. Weigh the beakers in accordance with 11.3. Record the masses.

11.7 Segregate the beakers containing the residues from motor gasolines for finishing as described in 11.8 through 11.12. The remaining beakers may be returned for cleaning and reuse.

11.7.1 Qualitative evidence of motor gasoline contamination can be obtained by weighing the residue at this point if retained samples of the original finished gasoline are available for reference testing. This reference testing is essential since motor gasoline may contain deliberately added materials that are nonvolatile. If evidence of contamination is obtained, further investigation is indicated.

11.8 For non-aviation fuels that have unwashed results that are <0.5 mg/100 mL (see 11.6, Section 12, and 13.2) it is not necessary to perform the washing steps identified in this section, as well as in those that follow (11.9 to 11.12) since the washed gum value will always be less than or equal to the unwashed gum value. If the unwashed results are not <0.5 mg/100 mL, to each of the beakers containing the residues from non-aviation fuels, add approximately 25 mL of heptane and swirl gently for 30 s. Allow the mixture to stand for 10 min ± 1 min. Treat the tare beaker in the same manner.

11.9 Decant and discard the heptane solution, taking care to prevent the loss of any solid residue.

11.10 Repeat the extraction with a second portion of approximately 25 mL of heptane as described in 11.8 and 11.9. If, after the second extraction, the extract remains colored, repeat the extraction a third time. Do not perform more than three extractions (Note 6).

NOTE 6—Further extractions (after the third extraction) are not to be performed, since portions of insoluble gum may be removed due to mechanical action, which could lead to lower solvent washed gum contents being determined.

11.11 Place the beakers, including the tare, in the evaporation bath maintained at 160 °C to 165 °C and, without replacing the conical jets, allow the beakers to dry for 5 min ± 0.5 min.

11.12 At the end of the drying period, remove the beakers from the evaporation bath using forceps or tongs (see 6.10), place them in a cooling vessel, and allow them to cool in the vicinity of the balance for at least 2 h. Weigh the beakers in the same manner as described in 11.3. Record the masses.

12. Calculation

12.1 Calculate the existent gum content of aviation fuels as follows:

$$A = 2000(B - D + X - Y) \quad (2)$$

12.2 Calculate the solvent washed gum content of motor gasoline as follows:

$$S = 2000(C - D + X - Z) \quad (3)$$

12.3 Calculate the unwashed gum content of motor gasoline as follows:

$$U = 2000(B - D + X - Y) \quad (4)$$

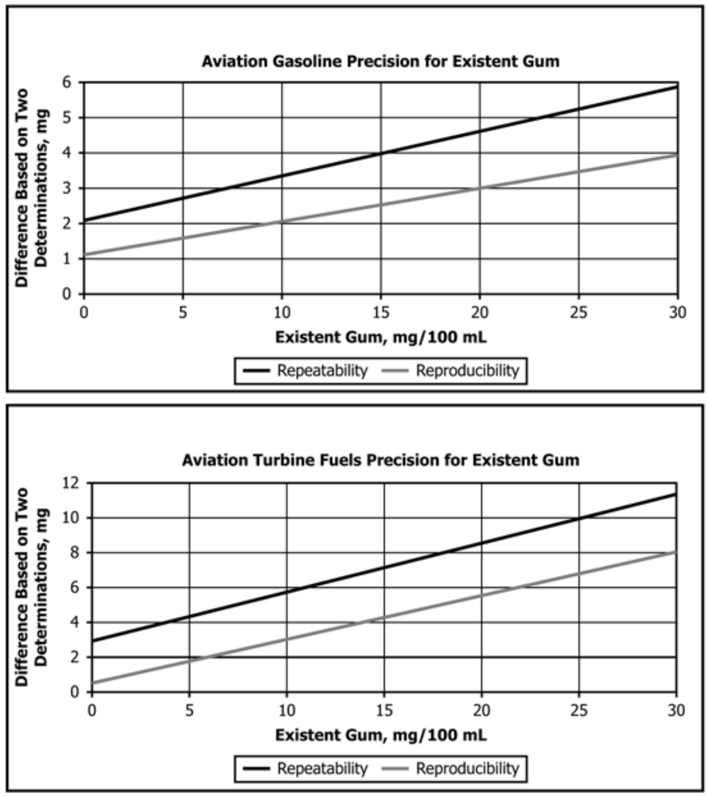

FIG. 2 Precision for Existent Gum

where:

A = existent gum content, mg/100 mL,
S = solvent washed gum content, mg/100 mL,
U = unwashed gum content, mg/100 mL,
B = mass recorded in 11.6 for the sample beaker plus residue, g,
C = mass recorded in 11.12 for the sample beaker plus residue, g,
D = mass recorded in 11.3 for the empty sample beaker, g,
X = mass recorded in 11.3 for the tare beaker, g,
Y = mass recorded in 11.6 for the tare beaker, g, and
Z = mass recorded in 11.12 for the tare beaker, g.

13. Report

13.1 For aviation fuels with existent gum contents ≥1 mg/100 mL, express the results to the nearest 1 mg/100 mL as existent gum content by Test Method D381. Round figures in accordance with Practice E29 or Appendix E of IP Standard Methods for Analysis and Testing of Petroleum and Related Products. For results <1 mg/100 mL, report as "<1 mg/100 mL."

13.2 For non-aviation fuels with either solvent washed or unwashed gum content values ≥0.5 mg/100 mL, express the results to the nearest 0.5 mg/100 mL as either solvent washed gum or unwashed gum content, or both, by Test Method D381. Round figures in accordance with Practice E29 or Appendix E of IP Standard Methods for Analysis and Testing of Petroleum and Related Products. For results <0.5 mg/100 mL, report as "<0.5 mg/100 mL." If the unwashed gum content is <0.5 mg/100 mL, the washed gum may also be reported as "<0.5 mg/100 mL" (see 11.8).

13.3 For all fuels, if the filtration step (see 11.4) has been carried out before evaporation, the expression filtered shall follow the numerical value.

14. Precision and Bias[4]

14.1 The precision, as obtained by statistical examination of interlaboratory test results, is given in 14.1.1 and 14.1.2, and illustrated graphically in Fig. 2. Analysis details are in the research report.

14.1.1 *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 values below only in one case in twenty.

$$r = 1.11 + 0.095x \text{ for existent gum (aviation gasoline),} \quad (5)$$

$$r = 0.5882 + 0.2490x \text{ for existent gum (aviation turbine fuel),} \quad (6)$$

$$r = 0.997x^{0.4} \text{ for unwashed gum content (unwashed), and} \quad (7)$$

$$r = 1.298x^{0.3} \text{ for solvent washed gum content (washed)} \quad (8)$$

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1466. Contact ASTM Customer Service at service@astm.org.

where: x is the average of the results being compared.

14.1.2 *Reproducibility*—The difference between two test results independently obtained by different operators operating in different laboratories on nominally identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values below only in one case in twenty:

$$R = 2.09 + 0.126x \text{ for existent gum (aviation gasoline),} \quad (9)$$

$$R = 2.941 + 0.2794x \text{ for existent gum (aviation turbine fuel),} \quad (10)$$

$$R = 1.928x^{0.4} \text{ for unwashed gum content} \quad (11)$$

$$R = 2.494x^{0.3} \text{ for solvent washed gum content} \quad (12)$$

where: x is the average of the results being compared.

NOTE 7—The precision values given above for solvent washed and unwashed gum content were obtained on fourteen (14) finished motor gasolines, which included two samples containing 10 % by volume ethanol and five (5) samples containing 15 % by volume methyl tertiary butyl ether (MTBE), as well as deposit control additives as determined in a 1997 interlaboratory study.[4] The precision values for the solvent washed and unwashed gum content are based on samples containing between 0 mg/100 mL to 15 mg/100 mL and 0 mg/100 mL to 50 mg/100 mL gum content, respectively.

14.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in Test Method D381 for measuring existent gum (solvent washed or unwashed gum), bias has not been determined.

15. Keywords

15.1 aviation fuels; existent gum; motor gasoline; solvent washed gum; unwashed gum