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

This standard is issued under the fixed designation D 381; 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 test method was issued as a joint ASTM-IP standard in 1965.
This method has been adopted for use by government agencies to replace Method 3302 of Federal Test Method Standard No. 791b.

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

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 hazard statements, see 6.4, 7.4, and 9.1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products[2]

E 1 Specification for ASTM Thermometers[3]

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specification[4]

2.2 *IP Standard:*

Standard Methods for Analysis and Testing of Petroleum Products[5]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *existent gum*—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*—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*—the evaporation residue of the product or component under test, without any further treatment.

4. Summary of Test Method

4.1 A measured quantity of fuel is evaporated under controlled conditions of temperature and flow of air or steam. 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.

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

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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 D02.14 on Stability and Cleanliness of Liquid Fuels.

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In the IP, this test method is under the jurisdiction of the Standardization Committee.

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

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

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] Available from Institute of Petroleum, 61 New Cavendish St., London, WIG 7AR, England.

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 1—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 D 381 – 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 to 600 micron 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 *Flowmeter*, capable of metering a flow of air or steam equivalent to 1000 mL/s for each outlet.

NOTE 2—Alternatively, a pressure gage may be used to meter the flow of air or steam equivalent to 1000 ± 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 to 246°C.

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

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

6.10 *Forceps*, stainless steel, spade ended.

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 ± 90 mL/s for the outlet under test. Check the remaining outlets for uniform air flow.

NOTE 3—A flowmeter reading of 600 mL/s for each outlet on a flowmeter calibrated at room temperature and atmospheric pressure will ensure delivery of 1000 ± 150 mL/s at the temperature of 155 ± 5°C, provided the back pressure of the flowmeter is less than 1 kPa. Typically, the conical adapter shown in Fig. 1 at each outlet is temporarily removed to allow a flowmeter to be easily connected to the outlet port being checked, so that the proper air flow rate specified in 8.1 can be set to within the required limits.

8.2 Apply heat to the evaporation bath (see 6.4) until the temperature of the bath is between 160 and 165°C. Introduce air into the apparatus at a rate indicated on the flowmeter (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

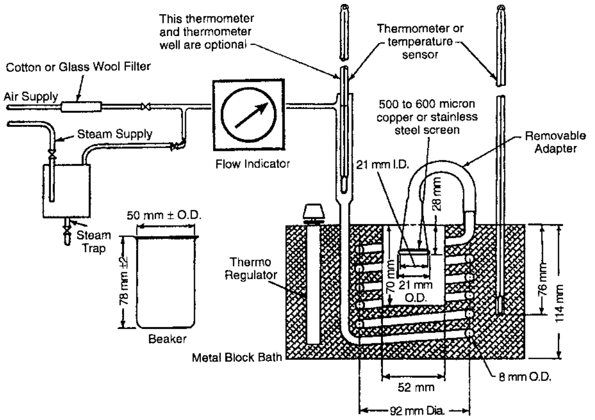

FIG. 1 Apparatus for Determining Gum Content by Jet Evaporation

outside the range from 150 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 ± 150 mL/s for each outlet is reached. Regulate the temperature of the bath to a range from 232 to 246°C to provide a well temperature of 232 ± 3°C. Measure the temperature with the specified thermometer, placed with the bulb 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.

9.3 Calibrate the flowmeter or pressure gage by successively condensing the steam flow from each outlet and weighing the water condensate. One way to accomplish this, 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 then 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. Weigh the cylinder. The gain in mass represents the amount of steam condensed. Calculate the steam rate as follows:

$$R = (M - m)1000/kt \tag{1}$$

where:

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.

9.4 Adjust the flow to give a steam rate of 1000 mL/s for the outlet under test. Check the remaining outlets for uniform steam flow. Make necessary changes to individual outlets if the rate varies by more than 150 mL of steam/s. With all outlets adjusted to deliver 1000 ± 150 mL of steam/s, record the flowmeter or pressure gage reading and use this setting for subsequent testing.

10. Procedure

10.1 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.

10.1.1 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.

10.1.2 Remove the beakers from the cleaning solution by means of stainless steel forceps and handle only with forceps 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.

10.2 Select the operating conditions, corresponding to the aviation 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.

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

10.4 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 12.3). Treat the filtrate as described in 10.5-10.7.

10.5 Measure a 50 ± 0.5 mL test specimen in a graduated cylinder (see 6.9), and transfer it to a weighed beaker (see 6.2). 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 to replace the conical jet as each individual beaker is placed in the bath. When using steam, allow the beakers to heat for 3 to 4 min before using forceps 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 (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 ± 0.5 min.

NOTE 4—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.

10.6 At the end of the heating period, remove the conical jets using forceps, or other suitable means, and transfer the

TABLE 1 Schedule of Test Conditions

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

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 10.3. Record the masses.

10.7 Segregate the beakers containing the residues from motor gasolines for finishing as described in 10.8 through 10.12. The remaining beakers may be returned for cleaning and reuse.

10.7.1 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.

10.8 For non-aviation fuels that have unwashed results that are <0.5 mg/100 mL (see 10.6, Section 11, and 12.2) it is not necessary to perform the washing steps identified in this section, as well as in those that follow (10.9 to 10.12) since the washed gum value will always be ≤ 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 ± 1 min. Treat the tare beaker in the same manner.

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

10.10 Repeat the extraction with a second portion of approximately 25 mL of heptane as described in 10.8 and 10.9. Repeat the extraction a third time if the extract is colored.

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

10.12 At the end of the drying period, remove the beakers from the evaporation bath using forceps, 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 10.3. Record the masses.

11. Calculation

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

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

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

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

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

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

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 10.6 for the sample beaker plus residue, g,
C = mass recorded in 10.12 for the sample beaker plus residue, g,
D = mass recorded in 10.3 for the empty sample beaker, g,
X = mass recorded in 10.3 for the tare beaker, g,
Y = mass recorded in 10.6 for the tare beaker, g, and
Z = mass recorded in 10.12 for the tare beaker, g.

12. Report

12.1 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 D 381. Round figures in accordance with Practice E 29 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."

12.2 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 D 381. Round figures in accordance with Practice E 29 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 10.8).

12.3 For all fuels, if the filtration step (see 10.4) has been carried out before evaporation, the expression filtered shall follow the numerical value.

13. Precision and Bias

13.1 The precision, as obtained by statistical examination of interlaboratory test results, is given in 13.1.1 and 13.1.2, and illustrated graphically in Fig. 2. Analysis details are in the research report.

13.1.1 *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.

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

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

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

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

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

13.1.2 *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:

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

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

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

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

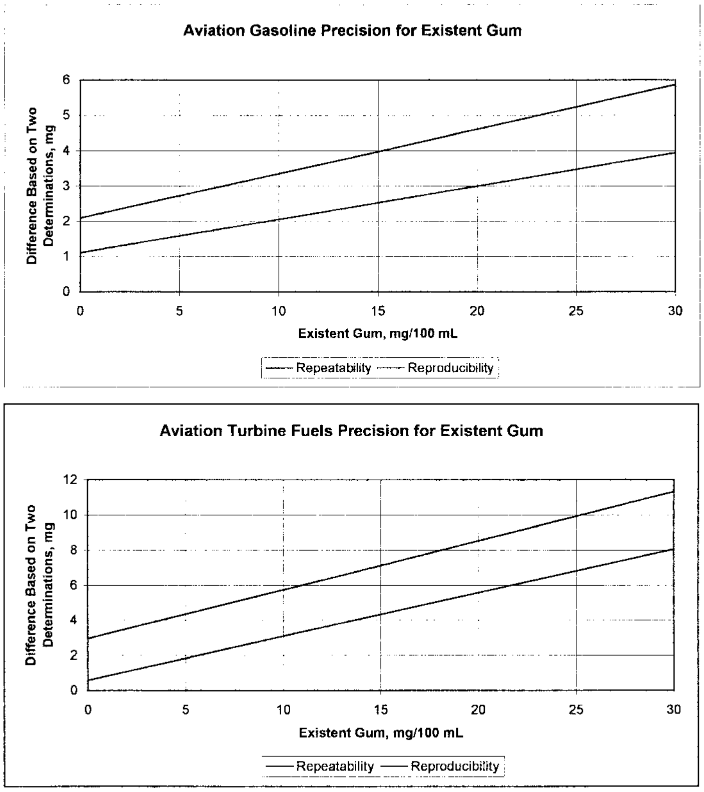

FIG. 2 Precision for Existent Gum

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

NOTE 5—The precision values given above for solvent washed and unwashed gum content were obtained on finished motor gasolines, including those containing alcohol and ether type oxygenates as well as deposit control additives as determined in a 1997 interlaboratory study (Research Report RR:D02–1466). The precision values for the solvent washed and unwashed gum content are based on samples containing between 0 to 15 mg/100 mL and 0 to 50 mg/100mL gum content, respectively.

13.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in Test Method D 381 for measuring existent gum (solvent washed or unwashed gum), bias has not been determined.

14. Keywords

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

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