

INTERNATIONAL STANDARD

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6246

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Petroleum products — Gum content of light and middle distillate fuels — Jet evaporation method

*Produits pétroliers — Teneur en gommages des distillats légers et
moyens — Méthode d'évaporation au jet*



Reference number
ISO 6246:1995(E)

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 6246 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 6246:1981), which has been technically revised.

Annex A forms an integral part of this International Standard.

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Petroleum products — Gum content of light and middle distillate fuels — Jet evaporation method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems 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.

1 Scope

This International Standard specifies a method for 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, and at the time of test.

For non-aviation fuels, a procedure for the determination of the heptane-insoluble portion of the residue is also described.

CAUTION — This method is not intended for the testing of gasoline components, particularly those with a high percentage of low-boiling unsaturated compounds, as they may cause explosions during evaporation.

NOTE 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 content can cause induction-system deposits and sticking of intake valves, and in most instances it can be assumed that low gum content will ensure absence of induction-system difficulties. The user should, however, realize that the test is not of itself correlative to induction-system deposits. The primary purpose of the test, 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 non-volatile 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.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3170:1988, *Petroleum liquids — Manual sampling*.

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 4788:1980, *Laboratory glassware — Graduated measuring cylinders*.

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series*.

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series*.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 existent gum: Evaporation residue of aviation fuels, without any further treatment.

3.2 evaporation residue: Material that remains after controlled heating under a flow of air or steam.

3.3 gum content, unwashed (non-aviation fuel): Evaporation residue of the product under test, without any further treatment.

3.4 gum content, solvent-washed (non-aviation fuel): Residue remaining after the evaporation residue has been washed with heptane and the washings discarded.

4 Principle

A measured test portion of fuel is evaporated under controlled conditions of temperature and flow of air or steam. The resulting residue is weighed, and may be subject to further treatment by solvent washing and further weighing.

5 Reagents and materials

During the analysis, unless otherwise stated, use only reagents specified in ISO 6353-2 and ISO 6353-3 if listed there; if not, use reagents of recognized analytical grade. Water where specified, shall be of a quality equivalent to grade 3 of ISO 3696.

5.1 Heptane, $\text{CH}_3(\text{CH}_2)_5\text{CH}_3$.

5.2 Toluene, $\text{C}_6\text{H}_5\text{CH}_3$.

5.3 Acetone, CH_3COCH_3 .

5.4 Gum solvent, a mixture of equal volumes of toluene (5.2) and acetone (5.3).

5.5 Air supply, filtered, at a gauge pressure of not greater than 34,5 kPa.

5.6 Steam supply, free of oily residue and at a pressure of not less than 34,5 kPa.

6 Apparatus

6.1 Balance, capable of weighing to an accuracy of 0,1 mg.

6.2 Beakers, 100 ml of capacity, tall form, as illustrated in figure 1, individually permanently marked.

NOTE 2 It is expedient to arrange the beakers in sets, the number in each set being the number of beaker wells in the evaporating bath. The lowest-mass beaker in each set should be reserved for use as the tare.

6.3 Cooling vessel, tightly covered, for cooling the beakers before weighing.

NOTE 3 If a desiccator is used, a drying agent is not recommended.

6.4 Evaporation bath, either a solid metal block bath or a liquid bath, electrically heated and constructed in accordance with the general principles shown in figure 1, having wells and jets for two or more beakers.

The rate of air/steam flow at the temperature of test from each outlet jet when fitted with the conical adaptors shall be $1\,000\text{ ml/s} \pm 150\text{ ml/s}$. A liquid bath, if used, shall be filled to within 25 mm of the top with a suitable liquid. Maintain the bath temperature either by means of thermostatic controls or by refluxing liquid of suitable composition.

6.5 Flow indicator, capable of indicating a total flow of air or steam equivalent to $1\,000\text{ ml/s}$ for each outlet jet.

6.6 Sintered glass filter funnel, capacity 150 ml, with a maximum pore diameter between 150 μm and 250 μm .

6.7 Steam superheater, gas-fired or electrically heated, capable of delivering to the bath inlet the required amount of steam at $232\text{ }^\circ\text{C} \pm 3\text{ }^\circ\text{C}$.

6.8 Two thermometers, conforming to the requirements in annex A.

6.9 Graduated cylinders, capacity 50 ml or 100 ml and 2 litres, conforming to the requirements of ISO 4788.

6.10 Forceps, stainless steel, spade-ended.

6.11 Oven, capable of being maintained at $150\text{ }^\circ\text{C} \pm 2\text{ }^\circ\text{C}$.

Dimensions in millimetres

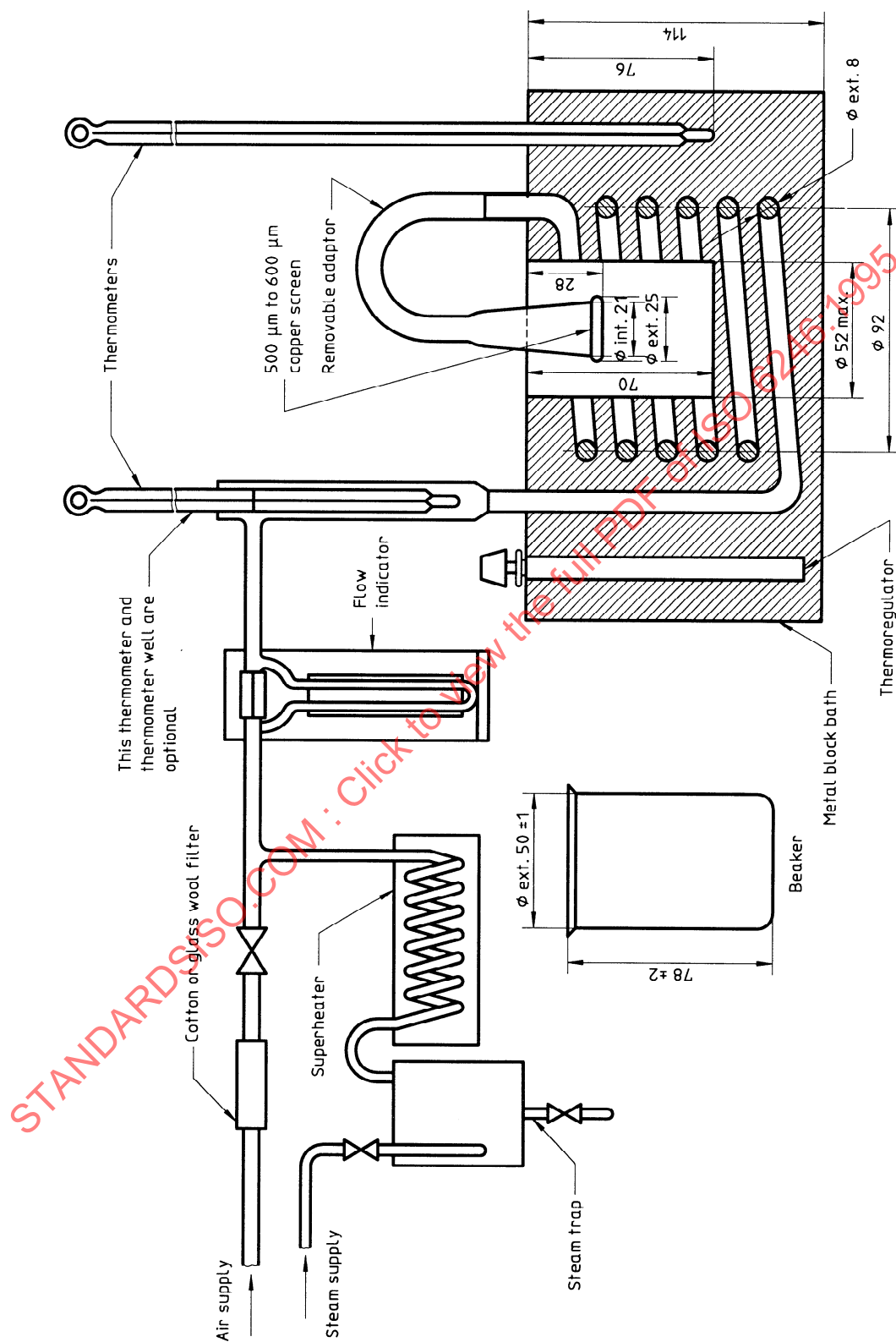


Figure 1 — Apparatus for determining gum content by jet evaporation

7 Assembly of air-jet apparatus

7.1 Assemble the air-jet apparatus as shown in figure 1. With the apparatus at room temperature, adjust the flow of air to a rate of 600 ml/s at one of the outlets, with the remaining conical adaptors in position. Check the other outlets individually under the same conditions for uniform air flowrate within the range of 600 ml/s $\pm$ 90 ml/s.

NOTE 4 A total reading on a flow indicator (calibrated under ambient conditions) corresponding to 600 ml/s $\pm$ 90 ml/s at each outlet will ensure a flowrate of 1 000 ml/s $\pm$ 150 ml/s at a temperature of 155 °C $\pm$ 5 °C, provided that the pressure at the outlet of the flow indicator is not greater than 34,5 kPa.

7.2 In order to set the apparatus in operation, heat the bath until the temperature reaches 162 °C, and then introduce air into the apparatus until the reading established in accordance with 7.1 is obtained on the flow indicator. Measure the temperature in each well with the thermometer (6.8) placed with the bulb resting on the bottom of a beaker (6.2) in the well. Do not use any well having a temperature that differs from 155 °C by more than 5 °C for standard tests.

8 Assembly of steam-jet apparatus

8.1 Assemble the steam-jet apparatus as shown in figure 1.

8.2 In order to set the apparatus in operation, heat the bath until the temperature reaches 232 °C. Operate the superheater, and slowly admit dry steam into the apparatus until a flowrate of 1 000 ml/s $\pm$ 150 ml/s per outlet is obtained. Regulate the temperature of the bath within the range 239 °C $\pm$ 7 °C and that of the superheater to provide a well temperature of 232 °C $\pm$ 3 °C. Measure the temperature with the thermometer (6.8) placed with the bulb resting on the bottom of a beaker (6.2) in the well. Do not use any well having a temperature that differs from 232 °C by more than 3 °C for standard tests.

8.3 Calibrate the flowmeter by successively condensing the steam flow from each outlet and weighing the total quantity of water recovered. To accomplish this, attach a copper tube to a steam outlet jet and extend the tube into a 2 litre cylinder (6.9) 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 flowrate, S , as follows:

$$S = (m_0 - m_1) 1\,000/kt$$

where

s is the flowrate of the steam, in millilitres per second, at 232 °C;

m_0 is the mass, in grams, of the cylinder with the condensed steam;

m_1 is the mass, in grams, of the cylinder and ice;

k is the mass (0,434 g) of 1 000 ml of steam at 232 °C at atmospheric pressure;

t is the condensing time, in seconds.

8.4 Adjust the apparatus to give a steam flowrate of 1 000 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 per second, record the flowmeter reading and use this setting for subsequent testing.

9 Samples and sampling

Samples for testing by the procedures described in this International Standard shall be taken by the procedure described in ISO 3170, ISO 3171 or an equivalent national standard.

10 Procedure

10.1 Wash the beakers (6.2) including the tare, with the gum solvent (5.4) until free of gum. Rinse thoroughly with water and immerse in detergent cleaning solution. The type of detergent and conditions for its use shall be established in each laboratory. The criterion for satisfactory cleaning shall be a matching of the quality of that obtained with chromic acid cleaning solution on used beakers (fresh chromic acid, 6 h soaking period, rinsing with water and drying). Remove the beakers from the cleaning solution by means of forceps (6.10) and handle only with forceps thereafter. Wash the beakers thoroughly, first with tap water and then with grade 3 water, and dry in the oven (6.11), controlled at 150 °C, for at least 1 h. Cool the beakers for at least 2 h in the cooling vessel (6.3) placed in the vicinity of the balance (6.1).

NOTE 5 For the comparison of cleaning efficiency, visual appearance and loss in mass on heating the glassware under test conditions may be used. Detergent cleaning avoids

the potential hazards and inconvenience related to handling corrosive chromic acid solution. The latter remains as the reference cleaning practice and as such may function as an alternative to the preferred procedure of cleaning with detergent solutions.

WARNING — Chromic acid is a health hazard. It is toxic, a recognized carcinogen as it contains Cr(VI) compounds, highly corrosive and potentially hazardous in contact with organic materials. When using chromic acid cleaning solution, eye protection and protective clothing are essential. Never pipette the cleaning solution by mouth. After use, do not pour cleaning solution down the drain, but neutralize it with great care owing to the concentrated sulfuric acid present, and dispose of it in accordance with standard procedures for toxic laboratory waste (chromium is highly dangerous to the environment).

Non-chromium-containing, strongly oxidizing acid cleaning solutions are also highly corrosive and potentially hazardous in contact with organic materials, but do not contain chromium which has special disposal problems.

10.2 Select the required conditions for testing aviation and motor gasolines or aircraft turbine fuel from table 1 and set the apparatus in operation following the procedures of 7.2 or 8.2 as appropriate. If an external preheater is used, regulate the temperature of the (vaporizing) medium to give the specified test well temperature.

Table 1 — Test conditions

Sample type	Vaporizing medium	Operating temperature °C	
		Bath	Test well
Aviation and motor gasoline	Air	160 to 165	150 to 160
Aircraft turbine fuel	Steam	232 to 246	229 to 235

10.3 Weigh the test beakers against the tare beaker to the nearest 0,1 mg. When a single-pan balance is used, weigh the tare beaker as a blank. Record the mass of each beaker.

10.4 If suspended or settled solid matter is present, mix the contents of the sample container thoroughly. Immediately filter a quantity of the sample, at atmospheric pressure, through the sintered glass funnel (6.6). Treat the filtrate as specified in 10.5 to 10.7 inclusive.

10.5 By means of the graduated cylinders (6.9), add 50 ml of the sample to each beaker except the tare, using one beaker for each of the fuels to be tested.

Place the filled beakers, and the tare, in the evaporation bath (6.4). The elapsed time between placing the first and last beakers in the bath shall be as short as possible. When evaporating samples by means of air, replace the conical adaptor as each individual beaker is placed in the bath. When using steam, allow the beakers to heat for 3 min before replacing the conical adaptor, which shall be preheated in the steam stream prior to attaching to the outlet. Centre the conical adaptors above the centre of the liquid. Take care to avoid splashing when introducing the jet of air or vapour, as this could cause gum values to be in error.

Maintain the temperature and rate of flow and allow the test portions to evaporate for 30 min. Samples tested simultaneously shall have similar evaporation characteristics.

NOTE 6 In certain cases, duplicate testing is advisable to determine precision.

10.6 At the end of the heating period, transfer the beakers from the bath to the cooling vessel (6.3). 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 mass of the beakers.

10.7 Segregate beakers containing residues from products for which the heptane-insoluble portion of the gum is also required to be determined, and return remaining beakers for cleaning and re-use.

10.8 To each of the beakers segregated in 10.7, together with the tare beaker, add 25 ml of heptane and swirl gently for 30 s. Allow the mixture to stand for 10 min.

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 25-ml portion of the heptane, as described in 10.8 and 10.9. Repeat the extraction a third time if the extract is coloured.

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

10.12 At the end of the drying period, remove the beakers from the bath, place them in the cooling vessel (6.3), and allow them to cool in the vicinity of the balance for at least 2 h. Weigh and record the mass of the beakers.

11 Calculation of gum content

11.1 Calculate the gum content, A , from the formula:

$$A = 2\,000 [(m_1 - m_3) - (m_2 - m_4)]$$

where

- A is the gum content, expressed as milligrams per 100 ml;
- m_1 is the mass, in grams, of the sample beaker plus residue;
- m_2 is the mass, in grams, of the tare beaker after treatment;
- m_3 is the mass, in grams, of the empty sample beaker;
- m_4 is the mass, in grams, of the tare beaker before treatment.

11.2 If a double-pan balance is used, with the weighings made against the tare before and after treatment, use the formula:

$$A = 2\,000 (m_5 - m_6)$$

where

- $m_5 = m_1 - m_2$, in grams;
- $m_6 = m_3 - m_4$, in grams.

12 Expression of results

12.1 For aviation fuels, express the results, to the nearest 1 mg/100 ml, as existent gum.

12.2 For non-aviation fuels, express the results, to the nearest 1 mg/100 ml, as either gum content (unwashed) or gum content (solvent-washed), or both.

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

13 Precision

The precision, as obtained by statistical examination of interlaboratory test results, is given in 13.1 and 13.2 and illustrated graphically in figures 2 to 4.

13.1 Repeatability, r

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 normal and correct operation of the test method, exceed the value below only in one case in 20:

$r = 1,71 + 0,095x$ for existent gum (aviation gasoline);

$r = 0,588\,2 + 0,249x$ for existent gum (aviation turbine fuel);

$r = 0,42x$ for gum content (unwashed);

$r = 0,882 + 0,274\,6x$ for gum content (solvent-washed);

where x is the average of the results being compared.

13.2 Reproducibility, R

The difference between two test results independently obtained by different operators operating in different laboratories on nominally identical test material would, in the normal and correct operation of the test method, exceed the value below only in one case in 20:

$R = 2,60 + 0,126x$ for existent gum (aviation gasoline);

$R = 2,941 + 0,279\,4x$ for existent gum (aviation turbine fuel);

$R = 1,333x$ for gum content (unwashed);

$R = 1,06 + 0,556\,7x$ for gum content (solvent-washed);

where x is the average of the results being compared.

NOTE 7 The precision values given above for gum content, both unwashed and solvent-washed, were obtained on motor gasoline samples only, and therefore do not necessarily reflect the values obtainable on other product types.

14 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) the type and complete identification of the product tested;
- c) the result of the test (see clause 12);
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

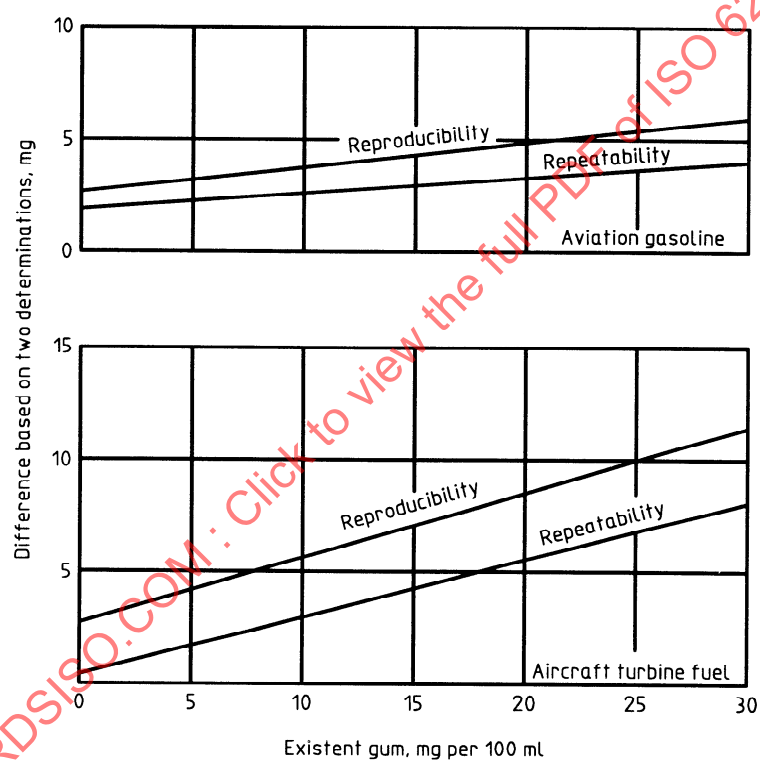


Figure 2 — Precision for existent gum