International Standard 6246

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Petroleum products — Motor gasoline and aviation fuels — Determination of existent gum — Jet evaporation method

Produits pétroliers — Essence automobile et essence aviation — Détermination des gommes actuelles — Méthode d'évaporation au jet

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6246 was developed by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in May 1980.

It has been approved by the member bodies of the following countries :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Brazil	Israel	Sweden
Bulgaria	Italy	Switzerland
Canada	Japan	Turkey
China	Korea, Rep. of	United Kingdom
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Egypt, Arab Rep. of	Peru	USSR
France	Poland	Venezuela

No member body expressed disapproval of the document.

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Petroleum products — Motor gasoline and aviation fuels — Determination of existent gum — Jet evaporation method

1 Scope and field of application

1.1 This International Standard specifies a method for the determination of the existent gum in motor gasoline, aviation gasoline, volatile distillates used in their preparation and aircraft turbine fuel, at the time of test.

1.2 The determination of the unwashed gum content of motor gasoline is also specified.

1.3 The true significance of this method for determining gum in motor gasoline is not firmly established. It has been proven that high gum can cause deposits on induction manifolds, carburettors and intake valves as well as sticking of intake valves, and in most instances it can be assumed that low gum will ensure absence of induction-system difficulties. It should, however, be realized 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 and under the conditions of the test, which are not as severe as those encountered in practical use. As 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.

2 References

ISO 3967, *Petroleum distillates and liquid hydrocarbons — Determination of density or relative density — Bingham pyknometer method.* [1]

ISO 5661, *Petroleum products — Hydrocarbon liquids — Determination of refractive index, refractive dispersion and specific optical dispersion.* [1]

3 Definitions

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

3.1 existent gum : The evaporation residue of aviation gasoline or aircraft turbine fuel or the heptane-insoluble portion of the evaporation residue of motor gasoline.

3.2 unwashed gum : The evaporation residue of motor gasoline consisting of existent gum and non-volatile additive components.

4 Principle

A measured test portion of fuel is evaporated under controlled conditions of temperature and flow of air or steam. For aviation gasoline and aircraft 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 *n*-heptane and the results reported as milligrams per 100 ml.

5 Materials

5.1 Air, supply of filtered air at a gauge pressure not more than 35 kPa *.

5.2 Gum solvent, a mixture of equal volumes of toluene and acetone.

5.3 ***n*-Heptane,** knock test grade, conforming to the requirements given in annex A.

5.4 Steam, supply of steam free of oily residue and at a gauge pressure not less than 35 kPa *.

6 Apparatus

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

6.2 Beakers, of 100 ml capacity, as illustrated in figure 1.

Arrange the beakers in sets, the number in each set depending upon the number of beaker wells in the evaporating bath. Permanently mark each beaker in the set with an identifying number or letter, reserving the lowest mass beaker for use as a tare.

1) At present at the stage of draft.

* 35 kPa = 0,35 bar

6.3 Cooling vessel, desiccator or other type of tightly covered vessel for cooling the beakers before weighing. The use of 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. The bath shall have wells and outlets for two or more beakers. The rate of flow from each outlet when fitted with the conical adapters shall be 1 000 ± 150 ml/s. A liquid bath, if used, shall be filled to within 25 mm of the top with a suitable liquid. Temperature may be maintained by means of thermostatic controls or by refluxing liquids of suitable composition.

6.5 Flow meter, capable of metering a total flow of air or steam equal to $n \times 1\,000$ ml/s, where n is the number of heating wells in the apparatus.

6.6 Sintered glass filtering funnel, of capacity 150 ml, range of maximum pore diameter between 150 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 ± 3 °C.

6.8 Thermometer, conforming to the essential requirements set out in annex B.

6.9 Graduated cylinders, of capacity 50 ml.

6.10 Stainless steel forceps.

6.11 Oven, capable of being controlled at 150 ± 2 °C.

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 so as to obtain, at each outlet, a flow of 600 ml/s, controlling this value by a suitable regulator outside the apparatus.

Make the necessary adjustments in order that the flow at each outlet shall lie between 510 and 690 ml/s; once these adjustments have been made, note the total flow rate indicated by the flow meter.

NOTE — A total reading on the flow meter corresponding to 600 ± 90 ml/s at each outlet will ensure, using a flow meter calibrated under ambient conditions, a flow of 1 000 ± 150 ml/s at a temperature of 155 ± 5 °C, provided that the pressure at the outlet of the flow meter is not greater than 35 kPa *.

7.2 In order to set the apparatus in operation, heat the bath until the temperature reaches 162 °C; then introduce air into the apparatus until the reading established in accordance with 7.1 is obtained on the flow meter.

Measure the temperature in each well with the thermometer (6.8), placed with the bulb resting on the bottom of the beaker in the well. Any well having a temperature that differs by more than 5 °C from 155 °C is not suitable for standard tests.

8 Assembly of steam-jet apparatus

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

8.2 To set the apparatus in operation, heat the bath; when the temperature reaches 232 °C, operate the superheater and slowly admit superheated steam until a flow rate of 1 000 ± 150 ml/s per outlet is obtained. In order to do this, adjust the admission of steam so as to reproduce the total flow meter reading established in carrying out the preliminary procedures specified in 8.3 and 8.4 and without changing the other adjustments which were then made. Regulate the temperature of the bath within the range of 239 ± 7 °C and that of the superheater to provide a well temperature of 232 ± 3 °C. Measure the temperature with the thermometer (6.8), placed with the bulb resting on the bottom of a beaker in the well. Any well having a temperature that differs by more than 3 °C from 232 °C is not suitable 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 and extend the tube into a 2 litre 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 R as follows :

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

where

R is the steam rate, in millilitres per second, of the steam 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 flow rate so as to obtain 1 000 ml/s at the outlet under test; this value should be controlled as specified in 8.3. Check, in the same way, that the remaining outlets have a uniform flow. Make the adjustments required so that the flow rates do not differ by more than 150 ml/s from the specified rate. When all the outlets have been adjusted to deliver 1 000 ± 150 ml/s of steam, note the flow meter reading and use this setting for preparing the apparatus as in 8.2.

* 35 kPa = 0,35 bar

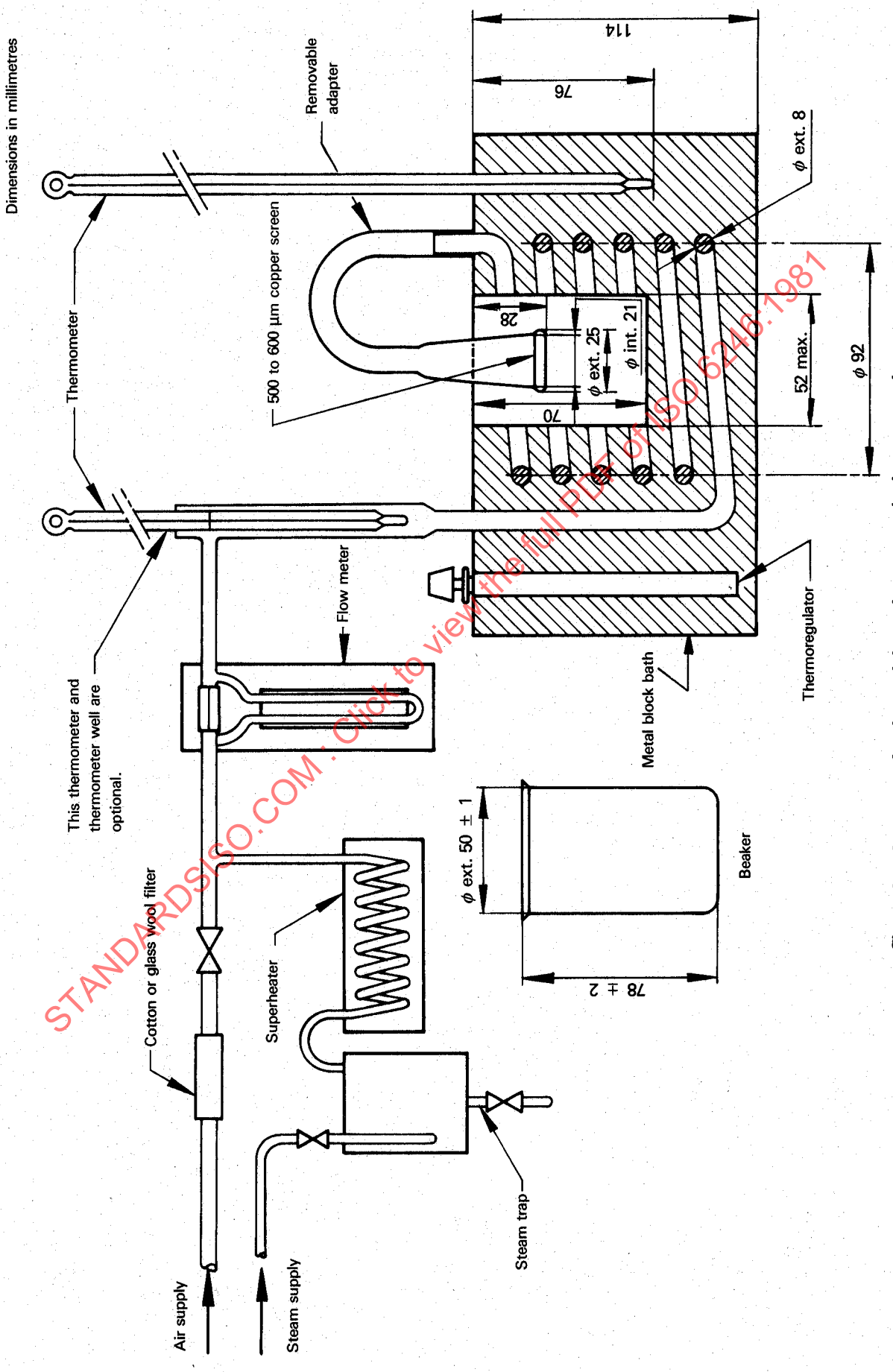

Figure 1 — Apparatus for determining existent gum by jet evaporation

9 Procedure

9.1 Wash the beakers (6.2), including the tare, with the gum solvent (5.2) until free of gum. Rinse thoroughly with water and immerse in detergent cleaning solution. Remove the beakers from the cleaning solution by means of the stainless steel forceps (6.10) and handle only with the forceps thereafter. Wash the beakers thoroughly, first with tap water and then with distilled 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 — The type of detergent and conditions for its use need to be established in each laboratory. The criterion for satisfactory cleaning should 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 distilled water, and drying). For this comparison 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.

9.2 Select the required conditions for 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 prescribed test well temperature.

9.3 Weigh the test beakers against the tare beaker to the nearest 0,1 mg. When a single-pan type balance is used, weigh the tare beaker as a blank.

9.4 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 9.5 to 9.7 inclusive.

9.5 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 surface of the liquid. 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.

NOTES

1 Care should be taken to avoid splashing when introducing the jet of air or vapor. Splashing may cause existent gum values to be in error.

2 In certain cases duplicate testing may be advisable. Duplicate tests are required to determine precision.

9.6 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 9.3.

9.7 Segregate any beakers containing the residues from motor gasolines and carry out the procedure specified in 9.8 to 9.12 inclusive. The remaining beakers may be returned for cleaning and re-use.

NOTE — If retained samples of the original finished gasoline are available for reference testing, qualitative evidence of motor gasoline contamination may be obtained by weighing the residue at this point. This reference testing is essential, as motor gasoline may contain deliberately added materials that are non-volatile. If evidence of contamination is obtained, further investigation is indicated.

9.8 To each of the beakers containing the residues from motor gasolines, add 25 ml of the *n*-heptane (5.3) and swirl gently for 30 s. Allow the mixture to stand for 10 min. Treat the tare beaker in the same manner.

9.9 Decant and discard the *n*-heptane solution, taking care to prevent the loss of any solid residue.

9.10 Repeat the extraction with a second 25 ml portion of the *n*-heptane, as specified in 9.8 and 9.9. Repeat the extraction a third time if the extract is coloured.

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

9.12 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 the beakers in accordance with 9.3.

Table 1 — Test conditions

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

10 Expression of results

10.1 Method of calculation

10.1.1 Calculate the existent gum content of aircraft fuels as follows :

10.1.1.1 Weighings made with a double-pan balance

$$A = 2\,000\,(m_1 - m_3)$$

10.1.1.2 Weighings made with a single-pan balance

$$A = 2\,000\,(m_1 - m_3 + m_4 - m_5)$$

10.1.2 Calculate the existent gum content of motor gasoline as follows :

10.1.2.1 Weighings made with a double-pan balance

$$A = 2\,000\,(m_2 - m_3)$$

10.1.2.2 Weighings made with a single-pan balance

$$A = 2\,000\,(m_2 - m_3 + m_4 - m_6)$$

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

10.1.3.1 Weighings made with a double-pan balance

$$U = 2\,000\,(m_1 - m_3)$$

10.1.3.2 Weighings made with a single-pan balance :

$$U = 2\,000\,(m_1 - m_3 + m_4 - m_5)$$

where

A is the existent gum content, in milligrams per 100 ml;

U is the unwashed gum content, in milligrams per 100 ml;

m_1 is the mass, in grams, recorded in 9.6 for the sample beaker plus residue;

m_2 is the mass, in grams, recorded in 9.12 for the sample beaker plus residue;

m_3 is the mass, in grams, recorded in 9.3 for the empty sample beaker;

m_4 is the mass, in grams, recorded in 9.3 for the tare beaker;

m_5 is the mass, in grams, recorded in 9.6 for the tare beaker;

m_6 is the mass, in grams, recorded in 9.12 for the tare beaker.

Express the results to the nearest milligram per 100 ml, as existent or unwashed gum by the jet evaporation method. Round off the figures in accordance with a recommended practice for indicating which places of figures are to be considered significant in specified limiting values.

After the numerical gum value, designate by the word "filtered" that extraneous material has been removed as provided for in 9.4.

10.2 Precision

The precision of the method, as obtained by statistical examination of interlaboratory test results, is as follows :

10.2.1 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 shown in figures 2 and 3 for repeatability only in one case in twenty.

10.2.2 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 values shown in figures 2 and 3 for reproducibility only in one case in twenty.

11 Test report

The test report shall contain at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard;

c) the result of the test, expressed in accordance with 10.1;

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.

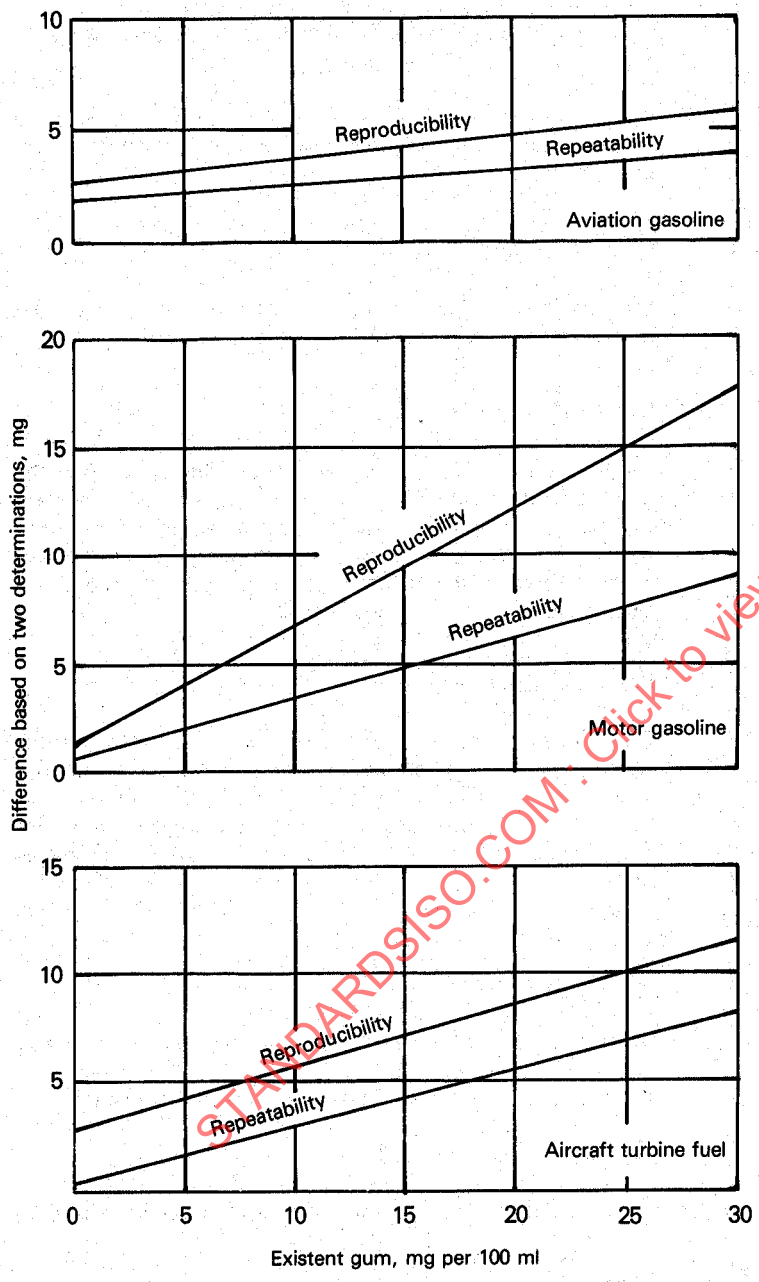

Figure 2 — Precision for existent gum

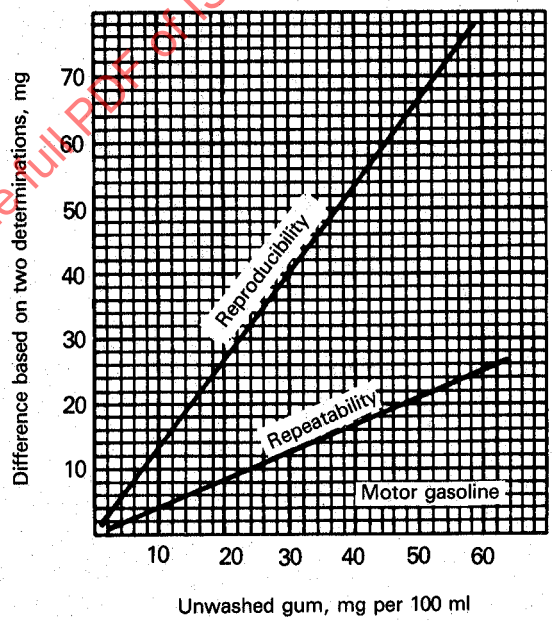

Figure 3 — Precision for unwashed gum

Annex A

Requirements for *n*-heptane

(Forms part of this International Standard.)

Density at 20 °C, g/ml (ISO 3967)	0,683 80 ± 0,000 15
Refractive index, n_D^{20} (ISO 5661)	1,387 70 ± 0,000 15
Freezing point, °C (ASTM D 1015)	−90,710 min.
Distillation[1)]	
50 % recovered, °C	98,427 ± 0,025
Temperature difference between the 20 % and 50 % distillation points, °C	0,020 max.

These requirements are, except for omission of the limit for lead, identical with those of knock rating grade *n*-heptane.

The reference to the ASTM test method is given for information only.

1) For equipment and method see Research Paper No. 2079, *Journal of Research*, National Bureau of Standards, 1950, **44**, 309-310.