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भारतीय मानक मासौदा

पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ
भाग 147 मोटर गैसोलीन में पोटेन्सियल गम का निर्धारण

(IS 1448 भाग 147 का *पहला पुनरीक्षण*)

Draft Indian Standard

PETROLEUM AND ITS PRODUCTS — TEST METHODS PART 147 DETERMINATION OF POTENTIAL GUM IN MOTOR GASOLINES

(*First Revision of IS 1448 Part 147*)

(ICS 75.080)

Methods of Sampling and Test for Petroleum and related Products of Natural or Synthetic Origin (excluding bitumen) Sectional Committee PCD 01

Last date for receipt of comments
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FOREWORD

(*Formal clause will be added later*)

This standard was published in 1998 and assistance was taken from ASTM D 873-88 ‘Standard test method for oxidation stability of aviation fuels (Potential Residue method)’.

The first revision has been taken up to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

a) Amendment No. 1 has been incorporated.
b) The reference clause has been updated.
c) The term ‘oxidation bomb’ has been substituted with ‘pressure vessel’ to align with current practices.
d) The purity requirements for n-heptane have been specified.
e) Precision data have been updated.

In reporting the result of the test made in accordance with this standard. If the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*).

1 SCOPE

This standard (Part 147) describes the determination of potential gum in motor gasolines.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 170: 2020	Acetone — Specification (*fifth revision*)
IS 1447 (Part 1): 2021	Methods of sampling of petroleum and its products: Part 1 Manual Sampling (*second revision*)
IS 1448	Methods of test for petroleum and its products
(Part 28) : 2008/ ISO 7536: 1994	Petroleum products — Determination of oxidation stability of gasoline — Induction period method (*fourth revision*)
(Part 29) : 2021/ISO 6246 : 2017	Petroleum products — Gum content of fuels — Jet evaporation method (*fourth revision*)

3 OUTLINE OF THE TEST METHOD

The sample is oxidised in a pressure vessel in the presence of oxygen for 4 h under prescribed test conditions. The oxidised gasoline is evaporated and *n*-heptane insoluble portion of evaporation residue is reported as potential gum.

4 TERMINOLOGY

For the purpose of this method, the following definitions shall apply.

4.1 Potential Gum

Deterioration products present at the end of a specific ageing period. These deterioration products exist in solution in the aged fuel and as the toluene acetone soluble portion of the deposits on the glass sample container. The potential gum is obtained as *n*-heptane insoluble portion of evaporation residue obtained by evaporating the aged fuel along with the washing from the glass sample container, under standard conditions of the test.

5 APPARATUS

5.1 For Oxidation of Sample

Oxidation bath, oxidation pressure vessel, glass sample container and cover, pressure vessel accessories, pressure gauge and needle valve, thermometers, same as prescribed in IS 1448 (Part 28).

5.2 For determination of evaporation residue evaporation bath, flow meter, glass beaker, cooling vessel, thermometer, balance, same as prescribed in IS 1448 (Part 29).

6 MATERIALS

6.1 Gum Solvent

A mixture of equal volume of reagent grade toluene and acetone (*see* **IS 170**).

6.2 Air-Filtered

Supplied at a pressure not more than 345 m bar (34.5 kPa).

6.3 *n*-Heptane — minimum 99.7 percent purity

7 SAMPLING

Sampling shall be done in accordance with the procedure for oxidation stability as prescribed in **9.4** of IS 1447 (Part 1).

8 PREPARATION OF APPARATUS

8.1 Oxidation of Sample

8.1.1 Thoroughly clean a glass sample container to remove traces of any adhering material. Immerse the container and its cover for at least 6 h in chromic acid solution. Remove them from the cleaning solution by means of corrosion-resistant steel tongs and handle only with tongs thereafter. Wash thoroughly first with tap water and then with distilled water, and dry in an oven at 100 °C to 150 °C for 1 h. Cool the sample container and covers to room temperature.

8.1.2 Drain any fuel from the pressure vessel and wipe the inside of the pressure vessel and lid, first with a clean cloth moistened with gum solvent and then with a clean dry cloth. Remove, any gum or gasoline from the annular space between the filler rod and stem with gum solvent. Occasionally remove the filler rod and stem and carefully clean both stem and rod. Also clean the needle valve, the quick release air coupling and all lines leading to the pressure vessel. The pressure vessel, the valve and all connecting lines shall be thoroughly dry before each test is started.

8.1.3 If a thermostatically controlled constant temperature oxidation bath is used, adjust the temperature to (100.0 ± 0.1) °C and maintain it within this temperature range for the duration of the test.

8.1.4 If a boiling water oxidation bath is used, adjust the temperature within the range of 99.5 °C to 100.5 °C by the addition of water or a higher boiling liquid such as ethylene glycol. Factors are given in Table 1 to adjust the 4 h ageing time if the bath temperature at the start of the test deviates from 100 °C.

8.2 Assembly of Air-jet Apparatus

Set up the air-jet apparatus as shown in Fig. 1 of IS 1448 (Part 29). With the apparatus at room temperature, adjust the air flow to give a rate of 600 ml/s for the outlet under test. Check the remaining outlets for uniform air flow. Make necessary adjustments to individual outlets if the rate differs by more than (600 ± 90) ml/s.

8.2.1 To put the apparatus in operation, apply heat to the bath and when its temperature reaches 160 °C, introduce air into the apparatus until a rate of (1000 ± 150) ml/s for each outlet is reached. Measure the temperature in each well with the thermometer 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.

Heat the evaporating bath to the operating temperatures given below:

Test conditions of operating evaporation bath

Sample	*Vaporizing medium*	*Operating*	
		temperature, °C	
		Bath	Test well
Motor gasoline	Air	160-165	150-160

Introduce air to apparatus and regulate the flow rate to give (1000 ±150) ml/s at operating temperature.

8.2.2 Wash the beakers, tare as well as sample beakers, with the gum solvent until free from gum.

8.2.3 Rinse thoroughly with water and immerse them for at least 6 h in chromic acid cleaning solution/or suitable detergent solution. Remove the beakers from cleaning solution by means of corrosion resistant steel tong and handle only with tongs thereafter. Wash the beakers thoroughly first with tap water then with distilled water and dry in an oven at 150 °C for at least 1 h. Cool the beakers for 2 h in the cooling vessel placed in the vicinity of the balance. Weigh the tare beaker and the sample beakers to the nearest 0.1 mg.

9 PROCEDURE

9.1 Oxidation of the Sample

9.1.1 Bring the pressure vessel, glass sample container and the fuel to be tested to a temperature of 15 °C to 25 °C. Place the glass sample container in the Pressure Vessel and add (100 ± 1) ml of sample. Cover the sample container, close the pressure vessel, and using a quick release air-coupling, introduce oxygen until a pressure of 689 kPa to 703 kPa is attained. Allow the gas in the pressure vessel to escape slowly through the needle valve at a rate not to exceed 345 kPa/min. Repeat the charging and exhausting of the oxygen once more in order to flush out the air originally present. Introduce oxygen again until a pressure of 689 kPa to 703 kPa is attained and observe for leaks, ignoring an initial rapid drop in pressure (generally not over 41.4 kPa), which can be observed because of the solution of oxygen in the sample. Proceed with the test if the rate of pressure drop does not exceed 13.8 kPa in 10 min, assume the absence of leaks and proceed with the test.

9.1.2 Place the charged pressure vessel in the described oxidation bath, being careful to avoid shaking and record the time of immersion as the starting time. Leave the pressure vessel in the oxidation bath for the 4 h ageing time. If a boiling water bath is used and the temperature at the start of the test varies from 100 °C, adjust the 4 h ageing time by the correction factors given in Table 1.

Table 1 Ageing time Correction Factors

(*Clauses* 8.1.4 *and* 9.1.2)

Sl No.	Temperature, ºC	Correction Factor
(1)	(2)	(3)
i)	99.5	1.06
ii)	99.6	1.04
iii)	99.7	1.03
iv)	99.8	1.02
v)	99.9	1.01
vi)	100.0	1.00
vii)	100.1	0.99
viii)	100.2	0.98
ix)	100.3	0.97
x)	100.4	0.96
xi)	100.5	0.95

NOTE — To obtain the correct ageing time at the operating temperature multiply the 4 h time specified for 100 ºC by the correction factor.

9.1.3 At the completion of the period of oxidation, remove the pressure vessel from the bath and cool rapidly with water while the charging valve is still closed. Release the pressure slowly. Take the pressure vessel apart and remove the sample container.

CAUTION — Release the pressure through the needle valve at a rate not to exceed 345 kPa/min.

9.2 Determination of Potential Gum

9.2.1 Transfer the oxidised fuel from the glass pressure vessel liner to two glass evaporation beaker for the determination of potential gum as given in **9.2.2.**

9.2.2 Add the oxidised fuel to two sample beakers proximately half in each beaker. Place the sample beaker and tare beaker in the evaporation bath. When evaporating samples by means of air, replace the conical jet, maintain the temperature and the rate of air flow of the test. Allow the evaporation for 20 min. Wash the interior of the glass pressure vessel liner twice with 10 ml portions of gum solvent to remove gums from the glass liner. Add the equal amount of washings to sample evaporation beakers and evaporate under the same operating conditions for another 10 min.

9.2.3 At the end of heating period transfer the beakers from the bath to the desiccator and allow to cool at room temperature for 45 min.

9.2.4 To each beaker containing the residue. From oxidised gasoline add 25 ml of *n*-heptane and swirl gently for 30 s. Treat the tare beaker in the same manner. Allow the mixture to settle for 10 min.

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

9.2.6 Repeat the extraction with a second portion of 25 ml of *n*-heptane in the same manner as described in **9.2.4** and **9.2.5**. If the extract is coloured, repeat the extraction with third portion (25 ml) of *n*-heptane.

9.2.7 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.

9.2.8 At the end of the drying period, remove the beakers from the bath, place them in the desiccator and allow to cool in the vicinity of the balance for at least 2 h. Weigh the sample beakers and tare beaker to the nearest 0.1 mg.

10 CALCULATION

Calculate the potential gum of gasoline as follows:

Combine the results obtained separately determined residues in two evaporation beakers as the potential gum.

11 REPORTING

Report the potential gum to the nearest g/m^3 as potential gum, heptane washed.

12 PRECISION

The results of two determinations on the same sample should not differ by more than the amount given below:

Potential gum, glm^3	*Repeatability*	*Reproducibility*
Up to 50	20	30
51 - 100	30	40
101 - 200	40	60