IS 17943 : 2022

ई 20 रेफरेंस ईंधन — निर्जल इथेनॉल और मोटर गैसोलीन का सम्मिश्रण — विशिष्टि

E20 Reference Fuel — Admixture of Anhydrous Ethanol and Motor Gasoline — Specification

ICS 43.060.01, 75.160.20

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भारतीय मानक ब्यूरो

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum, Lubricants and their Related Products of Synthesis or Biological Origin Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Ethanol is considered an alternative fuel source that possesses environment friendly characteristics associated with its use. IS 15464 'Anhydrous Ethanol for use in Automotive Fuel' specifies the requirement of denatured ethyl alcohol only which need to be used as admixture with motor gasoline to make E5, E10 and E20 blends which can be used in suitably designed positive ignition engines. The requirements of E20 fuel is specified in IS 17021: 2018 'E20 Fuel — Admixture of Anhydrous Ethanol and Gasoline — As Fuel for Spark Ignited Engine Powered Vehicles — Specification'. This standard describes the specification of E20 fuel for regular usage by consumer and to be dispensed for use in suitably compatible vehicles.

However, as per the CMVR requirement (rule No. 115), for new vehicles type approval and conformity of production tests, test agencies and OEMs shall use reference fuel. The reference fuel specification parameters have closer tolerance limits, essential for the above-mentioned regulatory tests consistency and has additional parameters which are not part of the IS 17021: 2018.

Hence, this new standard is derived based on available global references of reference fuel, by expert committee comprising OEMs and test agencies.

The usage of this standard is limited to the above-mentioned purpose, application and not to be referred as an alternate for commercial manufacturing of E20 fuel for consumer usage and dispensing in the retail outlets.

Title of the ISO standards or other standards referred in table are given in Annex C. The composition of the Committee responsible for the formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2: 2022 'Rule for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Title

characteristics of motor and

characteristics of motor fuels

Research method (second)

Determination of oxidation

period

Petroleum Products — Gum

evaporation method (fourth

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revision)

Petroleum

Induction

revision)

Determination

pressure —

Determination

(second revision)

stability of

(fourth revision)

aviation fuels —

method (second revision)

Indian Standard

E20 REFERENCE FUEL — ADMIXTURE OF ANHYDROUS ETHANOL AND MOTOR GASOLINE — SPECIFICATION

IS No.

2018/ISO 5163

2018/ISO 5164

2008/ISO 7536

(Part 26):

(Part 27):

(Part 28):

(Part 29):

(Part 39):

(Part 159):

2012/ISO 3007

: 2017

: 1999

2021/ISO 6246

: 2014

: 2014

: 1994

1 SCOPE

- 1.1 This standard prescribes requirements, methods of sampling and test methods for E20 reference fuel, an admixture of anhydrous ethanol at 20 percent by volume and ethanol free motor gasoline at 80 percent volume, for use in Positive-Ignition Engines Type Approval (TA) and Conformity of Production (COP) tests by authorized test agencies and OEMs.
- **1.2** 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 specification to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 REFERENCES

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

IS No.	Title	2018/ISO 20884 : 2011	content of automotive fuels — Wavelength-dispersive X-Ray fluorescence spectrometry
1447 (Part 1): 2021	Methods of sampling of petroleum and its products: Part 1 Manual Sampling (second revision)	(Part 160) : 2017/ ISO 20846 : 2011	Determination of sulphur content of automotive fuels — Ultraviolet fluorescence method
1448	Methods of test for petroleum and its products	(Part 161): 2017/ISO	Determination of low concentration of sulphur in
(Part 10/Sec 1): 2012/ISO 3015: 2012	Cloud point and pour point, Section 1 Determination of cloud point (second revision)	13032 : 2012	automotive fuels — Energy-dispersive X-Ray fluorescence spectrometric method
(Part 15): 2004/ISO 2160: 1998	Petroleum products — Corrosiveness to copper — Copper strip test (third revision)	(Part 167): 2018/ISO 12185: 1996	Determination of density — Oscillating U-tube method
(Part 18): 2020	Distillation of petroleum products (third revision)	(Part 181) : 2020/ISO 22854 : 2016	Determination of hydrocarbon types and oxygenates in automotive motor gasoline and in ethanol (E85) automotive

IS No.	Title
	fuel — Multidimensional gas chromatography method
(Part 182) : 2020/ISO 12937 : 2000	Petroleum products — Determination of water — Coulometric Karl Fischer titration method
2796 : 2017	Motor gasoline — Specification (sixth revision)
4117 : 2008	Alcohol denaturants — Specification (second revision)
15464 : 2022	Anhydrous ethanol for use as blending component in motor gasoline — Specification (<i>first revision</i>)

3 TERMINOLOGY

For the purposes of this standard, the following definitions shall apply.

3.1 Ethanol

Ethanol is a pure organic chemical, otherwise known as hydroxyethane, corresponding to the constitution CH_3CH_2OH and molecular formula C_2H_5OH .

3.2 Ethyl Alcohol (Absolute Alcohol)

Ethyl alcohol (absolute alcohol) is a clear, colorless and homogeneous liquid, consisting essentially of ethanol admixed with not more than 0.5 percent by volume of water.

3.3 Anhydrous Ethanol

Anhydrous ethanol is essentially ethyl alcohol, which is minimum 99 percent purity and having a boiling point of 78.5°C without any denaturant. In this standard, anhydrous ethanol refers to the ethyl alcohol which is denatured, as given in IS 15464.

3.4 Denaturant

Denaturant is a substance completely miscible in ethyl alcohol and of such a character that while its addition makes the material or any aqueous dilution of it unpleasant and unwholesome for potable purposes. Its presence does not render anhydrous ethanol, either as such or blended with motor gasoline, unsuitable for use in automobile engines. The denaturants and its concentration shall be as prescribed in IS 4117 and or prescribed by central and state regulators.

3.5 Motor Gasoline

Refined petroleum distillate free from un-dissolved water, foreign matters and visible impurities and shall conform to the requirement of BS VI compliant specification of IS 2796.

4 REQUIREMENTS

4.1 Description

E20 reference fuel is admixture of 20 percent anhydrous ethanol conforming to IS 15464 with 80 percent ethanol free motor gasoline fuel complying to IS 2796, volume by volume. E20 reference fuel shall comply with the requirements given in the col 3 of the Table 1.

NOTE — In case anhydrous ethanol and motor gasoline conforming to IS 15464 and IS 2796 respectively are not available, E20 reference fuel may be blended by mixing 20 percent anhydrous ethanol conforming to EN 15376 with 80 percent alcohol free motor gasoline conforming to EN 228, volume by volume. However, final product shall comply with the requirements given in Table 1.

4.2 Suitable stabilizing agents and additives (*see* **4.3.1** to **4.3.3**) in appropriate concentration are permitted to be added to E20 reference fuel to improve the stability. Organic oxygenates, if required, used as blending components and/or stabilizing agents shall be as specified in **4.4** and the amount of such oxygenates shall comply with the limiting requirements as specified in **4.4.2**.

4.3 Stabilizing Agents and Additives

4.3.1 Corrosion Inhibitors

Suitable doses of corrosion inhibitors have to be necessarily added to the E20 reference fuel to prevent corrosion, which may be caused due to hygroscopic nature of ethanol added in gasoline.

4.3.2 Antioxidants

Suitable antioxidants may be added to motor gasoline in sufficient concentration so as to ensure oxidation stability of E20 reference fuel. Some antioxidants of proven chemistry are listed below (This is not an exhaustive list):

- a) N, N'-disecondary-butyl-paraphenylene diamine;
- b) 2,4-dimethyl-6-tert butyl phenol;
- c) 4-methyl-2,6-ditertiary-butylphenol;
- d) N, N'-di-isopropyl-paraphenylenediamine;
- e) N-butyl-p-aminophenol; and
- f) Mixture of N,N'-disecondary-butylparaphenylene diamine and disalicylidene-N-methyldipropylene triamine.

4.3.3 Metal Deactivators

Suitable metal deactivators in sufficient concentration may be added in motor gasoline to make E20 reference fuel compatible with the vehicle fuel system components. Some metal deactivators of proven chemistry are listed below (this is not an exhaustive list):

- a) N,N'-disalicylidene propylene diamine;
- b) N,N'-disalicylidene ethylene diamine; and
- c) N,N'-disalicylidene methylaminedipropylene triamine

4.3.4 Multi-functional Additives

Motor gasoline multifunctional additives (MFA), other than those mentioned in **4.3.1** and **4.3.3** such as detergent/dispersant, dehazer, defoamant additives and additives containing metals such as iron, manganese (MMT) shall not be used.

4.4 Organic Oxygenates

4.4.1 Permitted Components

The following components may be used either individually or as mixtures as blending components or as stabilizing agents to prevent phase separation of the E20 reference fuel:

Ethers — Methyl tertiary butyl ether (MTBE), tertiary amyl methyl ether (methoxy-2-methyl butane) TAME, ethyl tertiary butyl ether (2-ethoxy-2-methyl propane) ETBE and other ethers (R-O-R) with final boiling point not exceeding 210°C and with molecules containing five or more carbon atoms.

4.4.2 *Content*

When tested, the content of the individual components listed in Table 1 shall comply with the limiting requirements as given against the respective components in col 3, for the E20 reference fuel.

In E20 reference fuel, the individual components defined in **4.4.1** or their mixtures are permitted but the total oxygen content shall not exceed the requirements mentioned in Table 1, Sl No. (xvii).

4.4.3 Subject to the effect of the added denaturant, anhydrous ethanol shall comply with the

requirements for general purposes prescribed for ethyl alcohol (*see* IS 15464).

4.5 Phosphorous — In order to protect automotive catalyst system, phosphorous containing additive shall not be used in E20 reference fuel and no external addition of other metal additives including lead is permitted.

5 PACKING AND MARKING

5.1 Packing

5.1.1 The material shall be packed in suitable containers prescribed by Petroleum and Explosives Safety Organization (PESO) from time to time.

5.2 Marking

- **5.2.1** The material shall be supplied in accordance with the marking and shipping regulations laid down by Petroleum and Explosives Safety Organization (PESO) from time to time.
- **5.2.2** Each container shall also be marked with the following information:
 - a) Name and grade of the material;
 - b) Indication of the source of manufacture, initials or trade-mark, if any;
 - c) Volume of the contents, in litres;
 - d) Year of manufacture or packing; and
 - e) Any other statutory requirements.

5.3.1 BIS Certification Marking

The container may also be marked with the Standard Mark.

5.3.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standard Act*, 2016 and the Rules and Regulations made thereunder. The details of conditions under which the license for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING AND TEST METHODS

- **6.1** Representative samples of the material shall be drawn as prescribed in IS 1447 (Part 1).
- **6.2** Tests shall be conducted by the test method(s) as prescribed in col 4 of Table 1.

Table 1 Requirement for E20 reference fuel for use in positive ignition engines TA and COP Tests

(Clause 4.1, 4.4.2 and 6.2)

Sl No.	Characteristics	Requirements	Method of test, Ref to Parts of IS 1448/ISO/ASTM/EN
(1)	(2)	(3)	(4)
i)	Density at 15°C, kg/m ³	743 to 760	Part 167 ³ /ISO 12185
ii)	Distillation		Part 18 ³ /ISO 3405
	a) Initial boiling point, °C	To report	-
	b) percent evaporated at 70°C (E70 °C), percent v/v	34 to 46	-
	c) percent evaporated at 100°C (E100°C), percent v/v	64 to 72	-
	d) percent evaporated at 150°C (E150°C), percent v/v, Min	88 to 95	-
	e) Final Boiling Point, °C, Max	165 to 195	-
	f) 10 percent by volume recovered at, °C	To report	-
	g) 50 percent by volume recovered at, °C	To report	-
	h) 90 percent by volume recovered at, °C	To report	-
	j) Residue, percent by volume, Max	2	-
iii)	Research Octane Number (RON), Min	98	Part 27 ³ /ISO 5164
iv)	Motor Octane Number (MON), Min	88	Part 26 ³ /ISO 5163
v)	Gumcontent (solvent washed), g/m³, Max	40	Part 29 ³ /ISO 6246
vi)	Total Sulphur, mg/kg, Max	10	Part 159/Part 160 ³⁾ /ISO 20846
vii)	Lead Content (as Pb), g/l, Max	0.005	EN 237
viii)	Reid vapour pressure at 37.8°C, kPa, <i>Max</i>	60 to 65	Annex A ³⁾ /EN 13016-1
ix)	Vapour Lock Index (VLI)	To report	Calculation (VLI = $10 \times RVP + 7 \times E70$)
x)	Benzene content, percent by volume, Max	1	Part 181 ³ /EN 238/ISO 22854/EN 12177
xi)	Copper strip corrosion (3 h at 50°C)	Class 1	Part 15 ³ /ISO 2160
xii)	Water tolerance of gasoline-alcohol blends, temperature for phase separation, °C, <i>Max</i>	(-)15	Annex B
xiii)	Induction period, minutes, Min	480	Part 28 ³⁾ /ISO 7536

Sl No.	Characteristics	Requirements	Method of test, Ref to Parts of IS 1448/ISO/ASTM/EN
(1)	(2)	(3)	(4)
xiv)	Olefin content, percent by volume	6 to 13	Part 181 ³⁾ /ISO 22854
xv)	Aromatic content, percent by volume	20 to 30	Part 181 ³⁾ /ISO 22854
xvi)	Saturates, percent by volume	To report	Part 181 ³⁾ /ISO 22854
xvii)	Oxygen content, percent by mass	7 to 7.4	Part 181 ³⁾ /ISO 22854
xviii)	Ethanol content, percent <i>v/v</i>	19 to 20	Part 181 ³⁾ /ISO 22854
xix)	Water content, percent by volume, Max	0.050	Part 182 ³ /ISO 12937
xx)	Net heating value, MJ/kg	To report	D240
xxi)	Hydrogen, percent by mass	To report	D5291
xxii)	Carbon, percent by mass	To report	D5291
xxiii)	Carbon/hydrogen ratio	To report	Calculated from D5291
xxiv)	Carbon/oxygen ratio	To report	Calculated from D5291
xxv)	Phosphorus content, g/l, Max	0.001	D3231

NOTES

- f 1 All the test methods referred in this standard include a precision statement. The Interpretation of results shall be based on test method and precision data of test method whenever applicable. In case of dispute the procedure described in ISO 4259 shall be used.
- 2 The dry vapour test method and water tolerance test method given in Annex A and B shall be respectively followed.
- 3 In case of disputes, this method shall be the referee method.
- **4** MTBE, ETBE or TAME if added, need to be reported.
- 5 No addition of detergents, dispersants, silicon, chlorine containing additives, materials and metallic additives are allowed in the reference fuel.

ANNEX A

[Table 1, Sl No. (viii)]

TEST METHOD FOR VAPOUR PRESSURE OF GASOLINE-ALCOHOL BLEND (DRY METHOD)

A-1 GENERAL

This test method covers the determination of the absolute vapour pressure of gasoline and gasoline-oxygenate blends.

NOTE — Because the external atmospheric pressure is counteracted by the atmospheric pressure initially present in the air chamber, the 'vapour pressure' is an absolute pressure at 37.8°C in kilopascals. This vapour pressure differs from the true pressure of the sample due to some small sample vaporization and the presence of air in the confined space.

A-2 SUMMARY OF TEST METHOD

The fuel chamber of the vapour pressure apparatus is filled with the chilled sample and connected to the air chamber at 37.8°C. The apparatus is immersed in a bath at 37.8°C and is shaken periodically until a constant pressure is observed on the gauge attached to the apparatus. The gauge reading, suitably corrected, is reported as the vapour pressure.

A-3 SIGNIFICANCE AND USE

The method mentioned in IS 1448 (Part 39) cannot be used to determine the vapour pressure of gasoline oxygenate blends which contain water-extractable oxygenates because the fuel sample comes into contact with water. This test method is a modification of IS 1448 (Part 39) where contact with water has been eliminated.

A-4 APPARATUS

The construction of the required apparatus is described in **3** of IS 1448 (Part 39).

A-5 REAGENTS

A-5.1 Purity of Reagents

Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the relevant Indian Standard where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

A-5.2 Acetone

WARNING — Extremely flammable. Irritating to skin, eyes and mucous membranes.

A-5.3 Naphtha

NOTE — Naphtha (tag closed cup flash point below 6° C)

WARNING — Extremely flammable. Harmful if inhaled. Skin irritant on repeated contact. Aspiration hazard.

A-6 HANDLING OF SAMPLES

A-6.1 The extreme sensitivity of vapour pressure measurements to losses through evaporation and the resulting changes in composition, require the utmost precaution and the most meticulous care in the handling of samples. This is applicable to all samples for vapour pressure determinations.

A-6.2 Sample in accordance with IS 1447 (Part 1) except that water displacement must not be used.

A-6.3 Sample Container Size

The size of the sample container from which the vapour pressure sample is taken as 1 l. It will be 70 to 80 percent filled with the sample.

A-6.4 Precautions

A-6.4.1 Determine vapour pressure as the first test run on a sample. Do not withdraw more than one sample from the sample container for this test.

A-6.4.2 Protect samples from excessive heat prior to testing.

A-6.4.3 Do not test samples in leaky containers. Discard them and obtain new samples.

A-6.4.4 Discard samples that have separated into two phases and obtain new samples (*see* Note 2 under **A-8.3**).

A-6.5 Sample Handling Temperature

In all case, cool the sample container and contents to 0 to 1°C before the container is opened. To ensure sufficient time to reach this temperature, directly measure the temperature of a similar liquid in a like container placed in the cooling bath at the same time as the sample.

A-7 PREPARATION FOR TEST

A-7.1 Verification of Sample Container Filling

With the sample at a temperature of 0 to 1°C, take the container from the cooling bath, wipe dry with an absorbent material, unseal it, and examine its ullage. The sample content, as determined by use of a suitable gauge, must be equal to 70 to 80 percent of the container capacity.

A-7.1.1 Discard the sample if its volume is less than 70 percent of the container capacity.

A-7.1.2 If the container is more than 80 percent full, pour out enough sample to bring the container contents within the 70 to 80 percent range. Under no circumstances, may any sample poured out, be returned to the container.

A-7.2 Air Saturation of Sample in Sample Container

A-7.2.1 With the sample again at a temperature of 0 to 1°C take the container from the cooling bath, wipe it dry with an absorbent material, unseal it momentarily, taking care to prevent water entry, reseal it, and shake it vigorously. Return it to the bath for a minimum of 2 min.

A-7.2.2 Repeat **A-7.2.1** twice more. Return the sample to the bath and keep it there until the beginning of the procedure (**A-8**).

A-7.3 Preparation of Fuel Chamber

Observe the apparatus preparation procedure of **A-8.5**, then store the stoppered fuel chamber and the sample transfer connection in a refrigerator or icewater bath for a sufficient time to allow the chamber and the connection to reach a temperature of 0 to 1°C. If an ice-water bath is used, keep the chamber upright and not immersed over the top of the coupling threads. The transfer connection is inserted over the top of the coupling threads. The transfer

connection is inserted into a plastic bag to keep it completely dry during cooling.

A-7.4 Preparation of Air Chamber

Observe the apparatus preparation procedure of **A-8.5**. Connect the gauge to the air chamber and close the lower opening securely with a dry No. 6 rubber stopper. Make sure the stopper is inserted far enough to securely close the vent hole in the air chamber connection. Immerse the air chamber to at least 25 mm above its top in the water bath until the fuel chamber has been filled with the sample as described in **A-8.1**.

A-8 PROCEDURE

A-8.1 Sample Transfer

With everything in readings, remove the chilled sample container from the bath, dry it with absorbent material, uncap it, and dry and insert the chilled transfer apparatus (*see* Fig. 1). Quickly place the chilled fuel chamber, in an inverted position, over the sample delivery tube of the transfer apparatus. Invert the entire system rapidly so that the fuel chamber is upright, with the end of the delivery tube touching the bottom of the fuel chamber. Fill the fuel chamber to the overflowing. Withdraw the delivery tube from the fuel chamber while allowing the sample to continue flowing up to the moment of complete withdrawal.

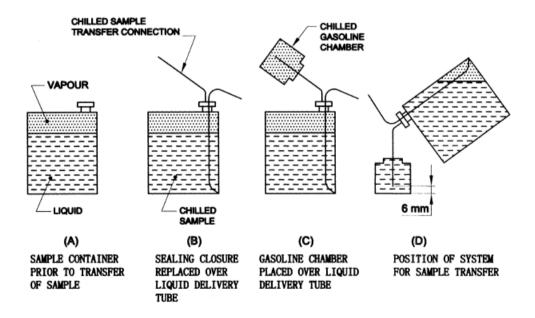


Fig. 1 Simplified Sketches Outlining Method of Transferring Samples to Gasoline Chamber From Open-Type Containers

A-8.1.1 Caution

Make provision for suitable collection and disposal of the overflowing fuel to avoid fire hazard.

A-8.2 Assembly of Apparatus

Immediately remove the air chamber from the water bath and immediately dry the exterior of the chamber with absorbent material giving particular care to the connection between the air chamber and the fuel chamber. Remove the stopper after drying and immediately couple the two chambers. Not more than 10 s shall be consumed in coupling the two chambers.

NOTE — When the air chamber is removed from the water bath, dried and the stopper is removed, connect it to the fuel chamber without undue movements through the air, which could promote exchange of room temperature air with the 37.8°C air in the chamber.

A-8.3 Introduction of Apparatus into Bath

Turn the assembled vapour pressure apparatus upside down to allow the sample in the fuel chamber to run into the air chamber. With the apparatus still inverted, shake it vigorously eight times in a direction parallel to the length of the apparatus. With the gauge end up, immerse the assembled apparatus in the bath, maintained at $37.8 \pm 0.1^{\circ}\text{C}$ in an inclined position so that the connection of the fuel and air chambers is below the water level and may be carefully examined for leaks 25 mm above the top of the air chamber. Observe the apparatus for leakage throughout the test. Discard the test at any time a leak is detected.

NOTES

- 1 Liquid leaks are more difficult to detect than vapour leaks, and because the coupling between the chambers is normally in the liquid section of the apparatus give the coupling particular attention.
- 2 After the apparatus has been immersed in the bath check the remaining sample for phase separation. If the sample is contained in a glass container, this observation can be made prior to sample transfer (see A-8.1). If the sample is contained in a non-transparent container, shake the sample vigorously for 5 s and then immediately pour a portion of the remaining sample into a clear glass container. Immediately after shaking this sample again for 5 s, observe the sample for phase separation. If this sample is not clear and bright, and free of a second phase, discard the test and the sample.

A-8.4 Measurement of Vapour Pressure

After the assembled vapour pressure apparatus has been immersed in the bath for at least 5 min, tap the pressure gauge lightly and observe the reading. Withdraw the apparatus from the bath and repeat **A-8.3**.

At intervals of not less than 2 min, perform **A-8.3** until a total of not less than five shakings and gauge

readings have been made continue thereafter, if necessary, until the last two consecutive gauge readings are constant, indicating equilibrium attainment.

These operations normally require 20 to 30 min. Read the final gauge pressure to the nearest 0.25 kPa for gauges with intermediate graduations of 0.5 kPa or less than to the nearest 0.5 kPa for gauges with graduations of 1.0 to 2.5 kPa and record the values as the 'uncorrected vapour pressure' of the sample. Without undue delay remove the pressure gauge and, without attempting to remove any liquid which may be trapped in the gauge, check its reading against that of the manometer while both are subjected to a common steady pressure which is no more than 1.0 kPa different from the recorded 'uncorrected vapour pressure'. If a difference is observed between the gauge and manometer readings, the difference shall be added to or subtracted from the 'uncorrected vapour pressure' recorded for the sample being tested, and the resulting value shall be recorded as the vapour pressure of the sample.

NOTES

- 1 Cooling the assembly prior disconnecting the gauge will facilitate disassembly and reduce the amount of hydrocarbon vapour released into the room.
- 2 Verification of sample integrity Disconnect the air chamber from the fuel chamber. Drain the sample from the air and fuel chambers as completely as possible into a dry (8 oz) 240 ml clear glass bottle. Seal the bottle and shake it vigorously for 5 s. If the sample is clear, bright, and free of a second phase, note this observation and record the test is valid. If the sample is not clear and bright and free of second phase. Immerse the bottle in the 37.8°C water bath upto about 25 mm above the top of the sample level for 15 min in order to heat the sample to the test temperature. Remove the sample from the water bath and immediately shake it vigorously for 5 s and observe the sample. If the sample is not clear, bright and free of second phase, note this observation and record that the test is not valid because of phase separation. A fuel that is not clear, bright and free of second phase at this point of the test, it indicates that the fuel was contacted with sufficient water to exceed the water tolerance of the fuel during the test procedure. Water can most likely get into the test chambers during preparation of the fuel and air chambers (see A-7.3 and see A-7.4) or assembly of the air and fuel chambers (see A-8.2), or both, specially if water baths are used for these procedures.

A-8.5 Preparation of Apparatus for Next Test

Thoroughly purge the air chamber of residual sample by filling it with warm water above 32°C and allowing it to drain. Repeat the purging atleast five times. After disconnecting the pressure gauge from its manifold connection with the manometer, remove trapped centrifugal thrusts. This may be accomplished in the following manner: hold the gauge between the palms of the hands with the right hand on the face side and the threaded connection of the gauge forward. Extend the arms forward and

upward at an angle of 45° with the coupling of the gauge pointing in the same direction. Swing the arms downward through an arc of about 135° so that the centrifugal force aids gravity in removing the trapped liquid. Repeat this operation three times to expel all liquid. Purge the pressure gauge by directing a small jet of air into its Bourdon tube for atleast 5 min. Rinse both chambers and the sample transfer connection several times with petroleum naphtha, then several times with acetone, then blow dry using dried air. Stopper the fuel chamber and place it in the refrigerator or ice-water bath for the next test.

NOTE — If the purging of the air chamber is done in a bath, be sure to avoid small and unnoticeable films of floating sample by keeping the bottom and top opening of the chamber closed as they pass through the water surface.

A-9 PRECAUTIONS

A-9.1 Gross errors can be obtained in vapour pressure measurements, if the prescribed procedure is not followed carefully. The following list emphasizes the importance of strict adherence to the precautions given in the procedure.

A-9.1.1 Checking the Pressure Gauge

Check all gauges against a manometer after each test in order to ensure high precision of results (*see* **A-8.4**). Read all gauges while the gauges are in a vertical position and after tapping them lightly.

A-9.1.2 Shake the container vigorously to ensure equilibrium of the sample with the air in the container (*see* **A-7.2**).

A-9.1.3 Checking for Leaks

Check the apparatus before and during each test for both liquid and vapour leaks.

A-9.1.4 Sampling

Because initial sampling and the handling of samples will greatly affect the final results, employ the utmost precaution and the most meticulous care to avoid losses through evaporation and even slight changes in composition (*see* **A-6.5** and **A-8.1**). In no case shall any part of the apparatus itself be used as the sample container prior to actually conducting the test.

A-9.1.5 Purging the Apparatus

Thoroughly purge the pressure gauge, the fuel chamber and the air chamber to be sure they are free of residual sample. [This is most conveniently done at the end of the previous test (*see* **A-8.5**)]. It is important to remove all water from the apparatus before cooling the gasoline chambers and heating the air chamber. In high-humidity conditions be alert

for and avoid condensation on the transfer connection and interior walls of the apparatus.

A-10 REPORT

A-10.1 Reporting Results

Report to the nearest 0.25 kPa or 0.5 kPa the gauge result observed in A-8.4, after correcting for any difference between the gauge and manometer, as the vapour pressure in kilopascals without reference to temperature.

A-11 PRECISION AND BIAS

A-11.1 Precision

The following criteria should be used for judging the acceptability of results (95 percent confidence).

A-11.1.1 *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 following value only in one case in twenty.

Component Gauge	Repeatability
(Range)	kPa
0 to 100 kPa	4.90

A-11.1.2 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 following value only in one case in twenty.

Component Gauge	Reproducibility
(Range)	kPa
0 to 100 kPa	7.790

A-11.2 Bias

There being no criteria for measuring bias in these test-product combinations, no statement of bias can be made.

ANNEX B

[Table 1 Sl No. (xii)]

TEST METHOD FOR WATER TOLERANCE (PHASE SEPARATION) OF GASOLINE-ALCOHOL BLEND

B-1 GENERAL

This test method determines the ability of gasolineoxygenate blends to retain water in solution or in a stable suspension at the lowest temperature to which they are likely to be exposed in use.

B-2 SUMMARY OF TEST METHOD

The sample of fuel is cooled at a controlled rate to its expected use temperature and is periodically observed for both haze and phase separation. The apparatus as given in 4 of IS 1448 (Part 10/Sec 1) or a dry ice-isopropyl alcohol bath may be used. A maximum cooling rate of 2°C/mm is specified because phase separation in gasoline-oxygenate blends has a relatively long but unpredictable induction period.

B-3 SIGNIFICANCE AND USE

- **B-3.1** Some oxygenate-containing fuels, and gasoline-alcohol blends in particular have a very limited ability to retain water in solution or in stable suspension, and if the amount of water in the blend exceeds this limit, the fuel will separate into a lower oxygenate-rich aqueous phase and an upper oxygenate-lean hydrocarbon phase. The most important factor governing the ability of a specific fuel to retain water without such separation is its temperature. This method is intended to determine the maximum temperature at which the fuel will separate. The temperatures represent the maximum temperatures above which the fuel must not separate into two distinct phases.
- **B-3.2** Note that in this test, actual separation of the sample into two distinct phases is the criterion for failure. The following are indications of phase separation.
- **B-3.2.1** The formation of droplets large enough to be detected by the unaided eye. They may be either clinging to sides of the container or collect on the bottom.
- **B-3.2.2** The formation of two layers separated by either a common boundary, or a layer of emulsion. Formation of haze without one of these indications of separation is not cause for rejection.

B-4 APPARATUS

- **B-4.1 Test Container** Any glass container of about 100 ml capacity may be used. This container may be marked at the level of 40 ml.
- **B-4.2 Thermometers** A thermometer must be provided for each container, mounted to pass through the stopper, with the requirements as follows:
 - a) Range -80 to +20°C,
 - b) Graduations 1°C, and
 - c) Immersion 76 mm.
- **B-4.3 Viton Rubber Stopper** To fit sample container bored centrally for the test thermometer.
- **B-4.4 Cooling Bath** May be of similar dimensions to those specified in **4** of IS 1448 (Part 10/Sec 1) and provided with a jacket disk and gasket (as specified in the apparatus section) filled with an equal-volume mixture of water and 'Permanent' antifreeze and provided with refrigeration coils capable of reducing its temperature to 40°C. Alternatively, a dry ice-isopropyl alcohol bath may be used.

B-5 SAMPLING AND HANDLING

- **B-5.1** Draw samples in accordance with IS 1447 (Part 1), except water displacement shall not be used.
- **B-5.2** Draw the samples in steel cans that have been solvent washed.
- **B-5.3** Store the samples in refrigerator (2 to 7°C) whenever not actually transferring sample.
- **B-5.4** Because gasoline-alcohol blends are hygroscopic as well as volatile, minimize contact with the atmosphere by keeping sample containers tightly closed except when transferring sample.

B-6 PROCEDURE

- **B-6.1** Warm the sample to 15°C and shake, to redissolve any water that may have settled out at the refrigerator temperature.
- **B-6.2** Cool the test container to 10 to 15°C. Carry out steps **B-6.3** through **B-6.5** as promptly as possible to minimize vaporization losses and absorption of water from the atmosphere.

- **B-6.3** Rinse out the cooled test container with some of the sample to be tested. Drain.
- **B-6.4** Pour about 40 ml of the sample into the test container. The precise amount is not critical, but it must be enough to submerge the thermometer bulb adequately, without being so much as to require an excessive amount of cooling time. If the sample has separated, as defined in **B-3.2**, terminate the test.
- **B-6.5** Seal the test container with the rubber stopper. Locate the thermometer bulb approximately at the centre of the fuel sample.
- **B-6.6** Cool the sample by intermittent immersion in or circulation of the coolant. The sample is not to be swirled or shaken while in the cooling bath. Starting at a cooling bath temperature not higher than 10°C or 16°C above the test temperature, cool the sample at a maximum rate of 2°C/min until phase separation occurs, or the test temperature is reached.
- **B-6.7** At 2°C intervals, remove the test container from the cooling bath and shake vigorously for 5 to 10 s. Wipe the exterior of the sample container with a towel moistened with isopropyl alcohol to remove any condensation, and observe the condition of the

- sample for no more than 5 s against a light coloured illuminated background.
- **B-6.8** It is likely that the sample will get hazy prior to actual phase separation as defined in **B-3.2**. Record the sample temperature at the first indication of haze (when cooling); and the temperature when the haze disappears (warming).
- **B-6.9** Record the temperature of phase separation (*see* **B-3.2**). Then allow the sample to warm at ambient temperature. Shake the sample vigorously after a temperature rise of 2°C, and observe. Record a 'warming' phase recombination temperature, and the haze disappearance temperature. Average these 'cooling' and 'warming' temperatures to determine the actual phase separation and haze point temperatures. Repeat this process for improved accuracy.

B-7 REPORT

- **B-7.1** Report the following information.
- **B-7.1.1** Report the averaged haze point and phase separation temperatures found in **B-6.9.**

ANNEX C

BIBLIOGRAPHICAL REFERENCES

International/ Other Standard No	Title
ISO 2160 : 1998	Petroleum products — Corrosiveness to copper — Copper strip test
ISO 3405 : 2019	Petroleum and related products from natural or synthetic sources — Determination of distillation characteristics at atmospheric pressure
ISO 5163 : 2014	Petroleum products — Determination of knock characteristics of motor and aviation fuels — Motor method
ISO 5164 : 2014	Petroleum products — Determination of knock characteristics of motor fuels — Research method
ISO 6246 : 2017	Petroleum products — Gum content of fuels — Jet evaporation method
ISO 7536 : 1994	Petroleum products — Determination of oxidation stability of gasoline — Induction period method
ISO 12185 : 1996	Crude petroleum and petroleum products — Determination of density — Oscillating U-tube method
ISO 12937 : 2000	Petroleum products — Determination of water — Coulometric Karl Fischer titration method
ISO 20846 : 2019	Petroleum products — Determination of sulfur content of automotive fuels — Ultraviolet fluorescence method
ISO 22854 : 2016	Liquid petroleum products — Determination of hydrocarbon types and oxygenates in automotive-motor gasoline and in ethanol (E85) automotive fuel — Multidimensional gas chromatography method
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EN 15376 : 2014	Automotive fuels — Ethanol as a blending component for petrol — Requirements and test methods

ANNEX D

(Foreword)

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