भारतीय मानक

Indian Standard

IS 1448 (Part 194) : 2023
ISO 11007-1 : 2021

पेट्रोलियम और उसके उत्पाद — परीक्षण पद्धतियाँ

भाग 194 लुब्रिकेटिंग ग्रीस की जंग रोकथाम विशेषताओं का निर्धारण — डाईनेमिक वेट कंडीशंस

Petroleum and its Products — Methods of Test

Part 194 Determination of Rust — Prevention Characteristics of Lubricating Greases-Dynamic Wet Conditions

ICS 75.100

भारतीय मानक ब्यूरो
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October 2023 **Price Group 5**

Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee, PCD 01

NATIONAL FOREWORD

This Indian Standard which is identical to ISO 11007-1 : 2021 'Petroleum products and lubricants — Determination of rust-prevention characteristics of lubricating greases — Part 1: Dynamic wet conditions' issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendation of the Methods for Sampling and Test for Petroleum and Related Products of Natural or Synthetic Origin (Excluding Bitumen) Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

The text of ISO standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'; and

b) Comma (,) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

This standard is one of the series of Indian Standards on 'Methods of test for petroleum and its products'.

In this adopted standard, reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards, which are to be substituted in their respective places, are listed below along with their degree of equivalence for the editions indicated:

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 15 Rolling bearings — Radial bearings — Boundary dimensions, general plan	IS 5669 : 2019/ISO 15 : 2017 Rolling bearings — Radial bearings — Boundary dimensions, general plan (*second revision*)	Identical
ISO 648 Laboratory glassware — Single-volume pipettes	IS 1117 : 2018/ISO 648 : 2008 Laboratory glassware — Single — Volume pipettes (*second revision*)	Identical
ISO 1998-1 Petroleum industry — Terminology — Part 1: Raw materials and products	IS 4639 (Part 1) : 2000/ISO 1998-1 : 1998 Petroleum industry terminology: Part 1 Raw materials and products (*first revision*)	Identical

(*Continued on third cover*)

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Introduction

ISO 6743-9 classifies greases according to their conditions of use. The complete designation of a grease comprises of the ISO sign, the letter L (lubricant, industrial oils and related products), the letter X for the grease family, four symbols related to the conditions of use, and the NLGI consistency number. The third symbol is related to the ability of the grease to ensure satisfactory lubrication in presence of water and provide an adequate corrosion protection level.

The symbol 3 is a combination of the rust protection level evaluated using this document and of the level of resistance to water contamination using the water washout test according to ISO 11009.

The purpose of this document is to prescribe a method for the evaluation of the corrosion protection properties of lubricating grease in dynamic wet conditions. This test method is commonly known as Emcor [1)] test in the industry.

Greases are specified in ISO 12924.

A rolling bearing grease may be not suitable to lubricate plain bearings or gears.

1) Emcor stands for Emulsion corrosion.

Indian Standard

PETROLEUM AND ITS PRODUCTS — METHODS OF TEST

PART 194 DETERMINATION OF RUST — PREVENTION CHARACTERISTICS OF LUBRICATING GREASES-DYNAMIC WET CONDITIONS

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the document, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document specifies a method for the determination of the rust prevention characteristics of lubricating grease under dynamic wet conditions.

This test method is used to assess the ability of a grease to prevent corrosion in rolling bearings operated in presence of water, synthetic sea water or any industrial aqueous pollutant.

NOTE For the purposes of this document, the term "% (*m*/*m*)" is used to represent the mass fraction.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 15, *Rolling bearings — Radial bearings — Boundary dimensions, general plan*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1998-1, *Petroleum industry — Terminology — Part 1: Raw materials and products*

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

ISO 3838, *Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary-stoppered pyknometer and graduated bicapillary pyknometer methods*

ISO 7120, *Petroleum products and lubricants — Petroleum oils and other fluids — Determination of rust-preventing characteristics in the presence of water*

ISO 23572, *Petroleum products — Lubricating greases — Sampling of greases*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1998-1 and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
corrosion
chemical reaction on metal surfaces, when (rolling bearing) steel is in contact with moisture, e. g. water or acid, that causes oxidation of surfaces

4 Principle

A test portion of grease is run in a specialized and carefully cleaned test rig under specified conditions, after introduction of an aqueous test fluid (distilled water, synthetic sea water, sodium chloride solution, user-specified solutions), for a period of approximately a week with a predetermined sequence of running and stopping. At the end of the test period, the apparatus is dismantled and the condition of the outer ring track of the bearing is examined and rated according to the degree of corrosion.

5 Sampling

Unless otherwise specified, sampling shall be conducted in accordance with ISO 23572. The sample shall be evaluated on a representative portion. Any drum, barrel, tanker compartment or any type of container delivered to the end user may be sampled and analysed at the discretion of the purchaser.

6 Apparatus and materials

6.1 Test bearings, double-row self-aligning ball bearings conforming to 1306 K of ISO 15 with a steel cage or polyamide cage, specially inspected and packed to prevent rust.

6.2 Test rig, as specified in Annex A.

6.3 Oven, capable of maintaining a temperature of 90 °C ± 2 °C to dry the bearing.

6.4 Dentist's mirror, no magnification.

6.5 Pipette, conforming to ISO 648, capacity of 10 ml.

6.6 Tweezers, to hold the bearings.

6.7 Cloth, absorbent, lint free.

6.8 Protective gloves, smooth, clean polyvinyl chloride (PVC), or polyethylene, or nitrile, or latex.

6.9 Filter paper, any suitable absorbent grade.

6.10 Tong, stainless steel or aluminium with covered forceps.

7 Reagents

Use only reagents of recognized analytical grade.

7.1 Wash solvent, consisting of low-sulfur, low aromatic, low volatility hydrocarbon.

NOTE White spirit according to BS 245 or mineral spirit according to ASTM D235 (all classes) are suitable.

7.2 Water, conforming to grade 3 of ISO 3696, as wash solvent.

7.3 Test fluid, use one of the following types:

a) water conforming to grade 2 of ISO 3696, or

b) synthetic sea water conforming to the specification given in ISO 7120, or

c) a 0,5 mol/l solution of sodium chloride prepared using water conforming to grade 2 of ISO 3696, at a pH of 8,0 to 8,2, by titration with sodium hydroxide solution.

NOTE Water quality of water used as a test fluid is critical and therefore is a different grade from that specified in 7.2.

Other water types may be used in this test, although the precision when using other water types has not been determined. It is recommended that the pH of other water types is determined before use.

7.4 Propan-2-ol.

7.5 Ammonia solution, 3,2 % (*m*/*m*) (1,65 mol/l).

NOTE The ammonia solution is equivalent to a tenfold dilution of 35 % ammonia solution (ρ = 880 kg/m^3) with water conforming to grade 3 of ISO 3696. This ammonia concentration is critical to the results of the test.

7.6 Solvent rinse solution, consisting of nine volumes of propan-2-ol (7.4) mixed with one volume of ammonia solution (7.5).

7.7 Silver nitrate solution, 0,1 mol/l.

8 Preparation of the apparatus

8.1 Remove all traces of grease from previous tests by wiping the plummer blocks housing (see A.1). Wash the plummer blocks housing and all other parts in solvent rinse solution (7.6) followed by water (7.2). Dry thoroughly using a cloth (6.7).

Where previous tests have been carried out with either synthetic sea water [7.3.b)] or salt water [7.3.c)], pour a few drops of silver nitrate solution (7.7) into the plummer-block. Withdraw a sample of the silver nitrate solution using a clean pipette, and inspect for milkiness or a white precipitate. If a white precipitate appears, rinse the plummer-block with hot water (7.2) and repeat this washing until a withdrawn sample is clear.

8.2 Wear protective gloves (6.8) or use tweezers (6.6) for all subsequent handling. Do not touch the bearings with bare fingers. Use two new bearings for each test.

8.3 Number the bearings on the outside diameter of the outer ring but do not use acid etching.

A “vibro pen” or etching marker may be used.

8.4 Wash the bearings in wash solvent (7.1) at 50 °C to 65 °C, to remove the rust-preventative.

Repeat the wash using fresh portions of hot wash solvent until the rust-preventative has been completely removed. In cases of dispute, white spirit (see NOTE to 7.1) is the referee solvent.

IMPORTANT — The complete removal of the rust-preventative is critical to the precision of this method. Failure to remove the material wholly will invalidate results.

8.5 Transfer the bearings from the wash solvent to the solvent rinse solution (7.6) to remove any solvent remaining. Rinse the bearings and rotate the outer ring slowly relative to the inner ring, while the bearing is immersed in freshly made solvent rinse solution heated to a minimum of 65 °C.

CAUTION — The washing temperatures specified are significantly higher than the closed flash point of the solvent. Therefore, carry out the washing operations in a well-ventilated hood where no flames or other ignition sources are present.

8.6 Remove the bearings from the solvent rinse solution and place on a filter paper (6.9) or cloth (6.7} to drain thoroughly. Place the bearings in the oven (6.3) until they are completely dry. This requires a minimum of 15 min.

8.7 Remove the bearings from the oven and allow cooling to ambient temperature. Examine the surfaces to ensure that each bearing is corrosion-free and free-turning.

WARNING — Take care not to spin the bearings after cleaning and drying.

8.8 Inspect the outer ring tracks using a dentist's mirror (6.4). If etch spots or corrosion are evident, reject the bearing.

8.9 Determine the mass of grease, m, in grams, equivalent to 10,5 ml ± 0,1 ml using Formula (1):

$$m = \rho \cdot V \tag{1}$$

where

ρ is the density of the grease, in kg/m^3;

V is the volume = 10,5 ml = 0,010 5 l.

If the density of the grease is not known, it shall be determined using a pycnometer method such as that given in ISO 3838, or another standardized procedure.

Distribute 10,5 ml ± 0,1 ml of grease evenly in each test bearing, as determined by the mass gain of the bearing. Take particular care to ensure that the outer raceway is completely coated.

8.10 Place the adaptor sleeves, bearings and V-ring seals in position on the shaft and finger-tighten the sleeve nuts. Carry out the operation with the shaft suitably supported on the work bench.

8.11 Place the shaft complete with greased bearings in position in the rig, taking care that the bearings are central in the plummer-blocks.

8.12 Place the top halves of the plummer-blocks in position and finger-tighten the locking screws.

9 Test procedure

9.1 Carry out duplicate determinations at an ambient temperature of 15 °C to 25 °C.

NOTE The precision data was generated under these ambient conditions.

9.2 Run the rig for 30 min at 8,7 rad/s ± 0,5 rad/s (83 min^{-1} ± 5 min^{-1}) immediately after assembly to distribute the grease evenly.

NOTE While 80 min^{-1} (8,4 rad/s) is the preferred rig speed, instruments are available that run at 80 min^{-1} and 85 min^{-1}. These two speeds have been demonstrated to give equivalent results. Hence the specification is 83 min^{-1} ± 5 min^{-1} (8,7 rad/s ± 0,5 rad/s) as given in Annex A.

9.3 Remove the top halves of the plummer-blocks and introduce 10 ml of the chosen test fluid (7.3) into each side of each plummer-block using the pipette (6.5) or a syringe (i.e. a total of 20 ml). Refit the top halves of the plummer blacks and screw down finger-tight.

9.4 Run the rig according to the following steps.

a) Run the rig for 8 h ± 10 min. Stop the rig and allow to stand for 16 h ± 10 min.

b) Run the rig for a further 8 h ± 10 min. Stop the rig and allow to stand for 16 h ± 10 min.

c) Run the rig for a further 8 h ± 10 min. Stop the rig and allow to stand for 108 h ± 2 h.

9.5 Carry out 9.3 with minimum delay. As soon as the shaft of the rig begins to rotate [9.4 a)], press the V-ring (face) seals up against the plummer-blocks using the special tool [shown as (7) in Figure A.1].

10 Dismantling the apparatus

10.1 Remove the top halves of the plummer-blocks. Lift the shaft and bearings on to a suitable support on the work bench.

10.2 Remove the bearings and V-ring seals from the shaft in the following manner:

a) Unscrew the sleeve nut one or two revolutions.

b) Tap the end face of the inner ring of the bearings lightly using a suitable tool such as a hammer and drift, and release the bearings from the tapered adaptor.

c) Pull the bearing, seals and sleeve off the shaft.

10.3 Wash the outer ring of the bearing out and lever a sufficient number of the balls out of the cage pockets, at diametrically opposite sides of the cage, thus allowing the cage to be dismantled.

10.4 Rinse the outer ring of the bearing in the cleaning solvent rinse solution (7.6). and dry using a cloth (6.7). Examine the bearing immediately.

11 Inspection

11.1 Examine the entire outer ring raceway for rust or etch spots and evaluate the surface area (3 680 mm^2) as specified in Table 1.

Table 1 — Evaluation of rust or etch spots

Rating	Degree of rusting
0	No corrosion
1	Not more than three small spots each such sufficient to be visible to the naked eye and less than 1 mm diameter
2	Small areas of corrosion covering up to 1 % of the surface
3	Areas of corrosion covering between 1 % and 5 % of the surface
4	Areas of corrosion covering between 5 % and 10 % of the surface
5	Areas of corrosion covering more than 10 % of the surface

Ignore any staining through which the underlying metal surface is clearly visible.

Under certain circumstances, a pair of parallel bands may be observed, the colour of which changes with viewing angle: do not confuse these bands with rust, which appears black when viewed from all angles.

To assist in estimating percentage corrosion area, a transparent grid divided into suitable squares may be used.

NOTE The various ratings are illustrated in Annex B. ln all cases, the area referred to represents the whole of the track.

11.2 If the two ratings do not agree within the precision given for repeatability, carry out two further determinations. If the two further determinations still do not agree within the precision for repeatability, record both results as the worst degree of rusting obtained, i.e. the highest numerical rating obtained.

12 Precision

12.1 General

The precision of the method, as obtained by statistical examination of interlaboratory test results, is given in 12.2 and 12.3.

12.2 Repeatability, *r*

The difference between two independent results obtained using this method for test material considered to be the same in the same laboratory, by the same operator using the same equipment within short intervals of time, in the normal and correct operation of the method that is expected to be exceeded with a probability of 5 % due to random variation, is shown in Table 2.

12.3 Reproducibility, *R*

The difference between two independent results obtained using this method for test material considered to be the same in different laboratories, where different laboratory means a different operator, different equipment, different geographic location, and under different supervisory control, in the normal and correct operation of the test that is expected to be exceeded with a probability of 5 % due to random variation, is shown in Table 2.

Table 2 — Precision data

Test fluid	Repeatability	Reproducibility
water [as in 7.3 a)]	1	1
synthetic sea water [as in 7.3 b)]	1	2
sodium chloride solution [as in 7.3 c)]	1	2
NOTE Precision is not established for any other type of test fluid.		

13 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 11007-1:2021;

b) the type and complete identification of the product tested;

c) the result of the inspection (see Clause 11);

d) the nature of the test fluid;

e) any deviations from the procedure;

f) any unusual features observed;

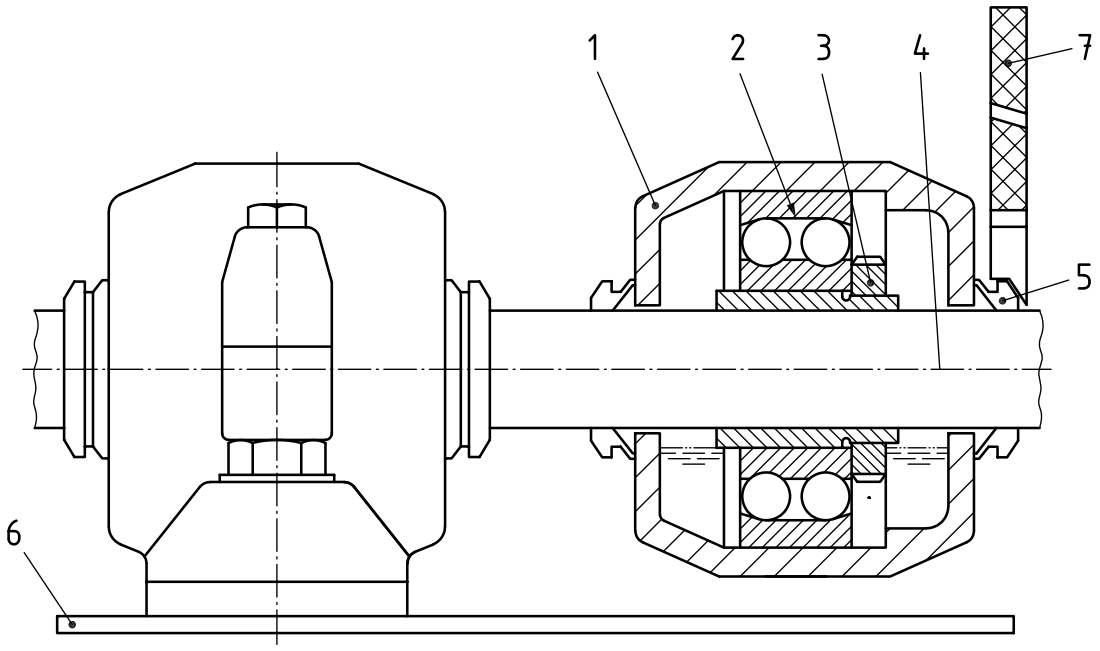
g) the date of the test.

Annex A
(normative)

Suitable test rig apparatus

A.1 Dynamic anti-rust test rig

Figure A.1 shows part of a standard rig which is capable of accommodating up to eight self-aligning ball bearings fitted with adaptor sleeves and locknuts (3) of polyamide plastic. The bearings (2) are located in the plummer block housing (1) which are polyamide plastic. The rig is mounted on a machined steel plate (6), approximately 275 mm × 985 mm × 10 mm. The shaft (4), 25 mm in diameter, is coated with polyamide plastic. Two V-ring (face) seals (5) per plummer block are required, together with a tool (7) for correct positioning of the seals.



Key

1	plummer block housing	5	seals
2	bearing	6	plate
3	locknut	7	tool
4	shaft		

Figure A.1 — Suitable test rig apparatus

A.2 Electric motor

Use any suitable type of electric motor. The motor shall be fitted with reduction gear and flexible coupling to drive the shaft directly at 8,4 rad/s (80 min^{-1}) or 9,0 rad/s (85 min^{-1}). The specification of 8,7 rad/s ± 0,5 rad/s (83 min^{-1} ± 5 min^{-1}) covers these two standard speeds (see 9.2, NOTE).

A.3 Other

A stand to hold the shaft on the work bench is also required and an automatic timing device is recommended.

Annex B
(informative)

Various ratings illustrating the degree of corrosion

The shaded area in Figure B.1 represents the outer ring raceway. Illustrations of the degree of corrosion that are given in Table 1 are given in Figure B.2.

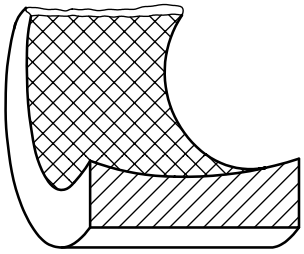


Figure B.1 — Partial outer ring of double-row self-aligning bearing

Only one half of the outer ring is shown in projected view, however the entire raceway surface shall be inspected (3 680 mm^2).

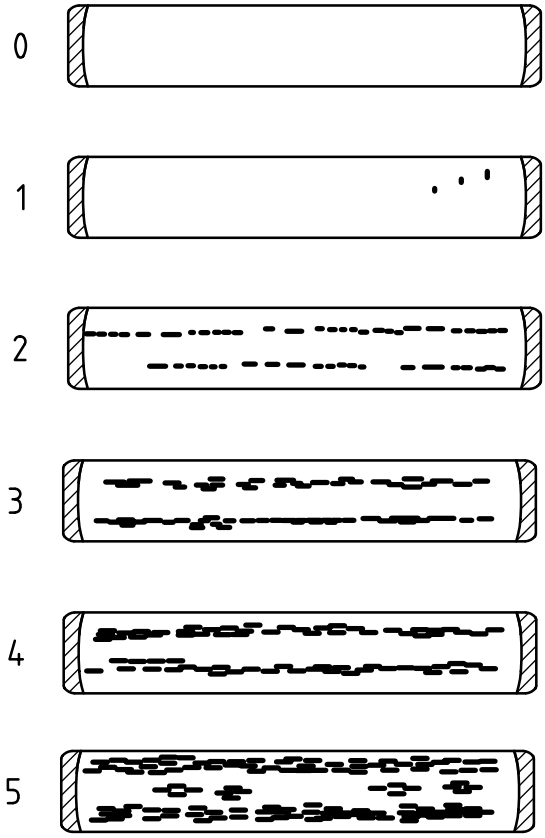


Figure B.2 — Various ratings illustrating the degree of corrosion

Bibliography

[1] ISO 6743-9, *Lubricants, industrial oils and related products (class L) — Classification — Part 9: Family X (Greases)*

[2] ISO/TS 11007-2, *Petroleum products and lubricants — Determination of rust-prevention characteristics of lubricating grease — Part 2: Method with water wash-out*

[3] ISO 11009, *Petroleum products and lubricants — Determination of water washout characteristics of lubricating greases*

[4] ISO 12924, *Lubricants, industrial oils and related products (Class L) — Family X (Greases) — Specification*

[5] ASTM D235, *Standard Specification for Mineral Spirits (Petroleum Spirits) (Hydrocarbon Dry Cleaning Solvent)*

[6] BS 245, *Specification for mineral solvents (white spirit and related hydrocarbon solvents) for paints and other purposes*

(*Continued from second cover*)

International Standard	*Corresponding Indian Standard*	*Degree of Equivalence*
ISO 3696 Water for analytical laboratory use — Specification and test methods	IS 1070 : 1992 Reagent grade water — specification (*third revision*)	Not equivalent
ISO 3838 Crude petroleum and liquid or solid petroleum products — Determination of density or relative density Capillary — stoppered pyknometer and graduated bicapillary pyknometer methods	IS 1448 (Part 32) : 2019/ISO 3838 : 2004 Methods of test for petroleum and its products: Part 32 Crude petroleum and liquid or solid petroleum products — Determination of density or relative density — Capillary stoppered pyknometer and graduated bicapillary pyknometer methods (*third revision*)	Identical
ISO 7120 Petroleum products and lubricants — Petroleum oils and other fluids — Determination of rust-preventing characteristics in the presence of water	IS 1448 (Part 96) : 2019/ISO 7120 : 1987 Methods of test for petroleum and its products: Part 96 Petroleum products and lubricants — Petroleum oils and other fluids — Determination of rust-preventing characteristics in the presence of water (*first revision*)	Identical
ISO 23572 Petroleum products — Lubricating greases — Sampling of greases	IS 1447 (Part 5) : 2023/ISO 23572 : 2020 Methods of sampling of petroleum and its products: Part 5 Sampling of greases	Identical

In reporting the result of a test or analysis 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*)’.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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