# TECHNICAL SPECIFICATION

## ISO/TS 11007-2

Second edition 2023-02

Petroleum products and lubricants —
Determination of rust-prevention
characteristics of lubricating
greases —

Part 2:

Method withwater wash-out

Produits pétroliers et lubrifiants — Détermination des caractéristiques antirouille des graisses lubrifiantes —

Partie 2: Méthode avec lavage à l'eau
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#### **Foreword**

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see <a href="www.iso.org/directives">www.iso.org/directives</a>).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see <a href="https://www.iso.org/iso/foreword.html">www.iso.org/iso/foreword.html</a>.

This document was prepared by Technical Committee \$50/TC 28, Petroleum and related products, fuels and lubricants from natural or synthetic sources.

This second edition cancels and replaces the first edition (ISO/TS 11007-2:2021) which has been technically revised.

The main changes are as follows:

- normative references have been updated;
- 6.2 has been updated.

A list of all parts in the ISO 11007 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

#### Introduction

ISO 11007-1 specifies a method for the evaluation of the corrosion protection properties of lubricating grease in the presence of non-flowing water; the test bearing is immersed partially in the test fluid.

In some industries (e.g. wet sections of paper mills, roll neck bearings of rolling mills) the bearings are submitted to the flow of water, rolling emulsions, paper treatment liquors, etc. In case of seal damage of the bearings, the corrosion inhibitors present in the grease may be potentially washed out, hence impairing the corrosion protection properties.

This document describes a procedure using the flow of test fluid (wash-out) instead of the non-flow conditions described in ISO 11007-1.

This test method is commonly known as Emcor (Emulsion corrosion) test in the industry.

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

STANDARDSISO.COM. Cick to view the full Port of ISOF The precision of the method described in this document has not yet been determined by an interlaboratory study.

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# Petroleum products and lubricants — Determination of rust-prevention characteristics of lubricating greases —

#### Part 2:

### Method with water wash-out

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 in the presence of a flow of an aqueous test fluid.

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, under wash out conditions.

#### 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 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

DIN 630, Rolling bearings - Self aligning ball bearings - Double row with cylindrical or tapered bore

#### 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 <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <a href="https://www.electropedia.org/">https://www.electropedia.org/</a>

#### 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 bearings in accordance with DIN 630, such as SKF 1306K/236725<sup>1)</sup>, with steel cage, specially inspected, protected with a waxy type corrosion inhibitor (no preservative with chemical rust inhibitor) and packed.

NOTE It is possible that a chemical rust inhibitor containing preservative is not completely eliminated during the washing procedure, thus leaving some corrosion protection and introducing bias in the test.

- **6.2 Test rig**, as specified in Annex A.
- **6.3 Peristaltic pump**, capable of delivering at a constant flow rate of  $2,10 \text{ ml/min} \pm 0,2 \text{ ml/min}$  (approximately 3 000 ml for the whole test which equals to 1 000 ml for every 8 h rotation of the test rig).

NOTE Multi-channel peristaltic pumps exist, up to 24 channels, allowing to feed the necessary number of plummer blocks.

- **6.4 Oven,** capable of maintaining a temperature of 90 °C ± 2 °C to dry the bearing.
- **6.5 Dentist's mirror**, no magnification.
- **6.6 Pipette,** conforming to ISO 648, capacity of 10 ml.
- **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.

<sup>1)</sup> SKF 1306K/236725 is an example of a suitable product available commercially. This information is given for convenience of the user of this document and does not constitute an endorsement of ISO of this product.

#### 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[3] or mineral spirit according to ASTM D235<sup>[2]</sup> (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,** a mass fraction of 3,2% (1,65 mol/l).

NOTE The ammonia solution is equivalent to a tenfold dilution of 35 % ammonia solution ( $\rho$  = 880 kg/m3) 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 ( $\underline{7.4}$ ) mixed with one volume of ammonia solution ( $\underline{7.5}$ ).
- **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. 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  $(\underline{6.8})$  for all subsequent handling. 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 (preservant or preservation fluid).

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 in 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 (6.4) 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 (6.5). 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  $\pm$  0,1 ml using Formula (1):

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

where

- $\rho$  is the density of the grease, expressed in kg/m<sup>3</sup>;
- 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 locknuts. 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.

- 9.2 Run the rig immediately after assembly for 30 min at 8,7 rad/s  $\pm$  0,5 rad/s (83 min<sup>-1</sup>  $\pm$  5 min<sup>-1</sup>) to uniformly distribute the grease.
- NOTE While 80 min<sup>-1</sup> (8,4 rad/s) is the preferred rig speed, instruments are available that run at 80 min<sup>-1</sup> and 85 min<sup>-1</sup>. These two speeds have been demonstrated to give equivalent results. Hence the specification is  $83 \text{ min}^{-1} \pm 5 \text{ min}^{-1}$  (8,7 rad/s  $\pm$  0,5 rad/s) as given in Annex A.
- **9.3** Adjust the peristaltic pump flow rate of test fluid to  $2,10 \text{ ml/min} \pm 0,2 \text{ ml/min}$  (approximately 1 000 ml for every 8 h rotation of the test rig).
- 9.4 Connect the output of the peristaltic pump (6.3) to the inlet hole of the plummer block?
- **9.5** Tilt the assembly of the plummer block by 1,5° to favour the flow out of the test fluid.
- **9.6** Run the rig according to the following steps.
- a) Run the rig for  $8 h \pm 10 min$ . Stop the rig and the flow of test fluid and allow to stand for  $16 h \pm 10 min$ .
- b) Run the rig for a further  $8 \text{ h} \pm 10 \text{ min}$ , applying the test fluid flow. Stop the rig and the flow of test fluid and allow to stand for  $16 \text{ h} \pm 10 \text{ min}$ .
- c) Run the rig for a further  $8 \text{ h} \pm 10 \text{ min}$ , applying the test fluid flow. Stop the rig and the flow of test fluid, and allow to stand for  $108 \text{ h} \pm 2 \text{ h}$ .
- 9.7 Carry out 9.3 to 9.5 with minimum delay. As soon as the shaft of the rig begins to rotate [9.6 a], press the V-ring (face) seals up against the plummer blocks using the special tool (see Figure A.1, key reference 7).

#### 10 Dismantling the apparatus

- **10.1** Remove the top halves of the plummer blocks. Lift the shaft and bearings onto 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 locknut 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  $(\underline{7.6})$  and dry using a cloth  $(\underline{6.7})$ . 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<sup>2</sup>) as specified in <u>Table 1</u>.

Table 1 — Evaluation of rust or etch spots

Rating	Degree of rusting	
0	No corrosion	
1	Not more than three small spots, each one 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. These bands shall not be confused 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 <u>Annex B</u>. In 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

At the time of publication of this document, no precision data are available for this test method.

#### 13 Test report

The test report shall contain at least the following information:

- a) a reference to this document, i.e. ISO/TS 11007-2:2023;
- b) the type and complete identification of the product tested;
- c) the result of the inspection (see <u>Clause 11</u>);
- 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)

### 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 adapter sleeves and locknuts (3) of polyamide plastic. The bearings (2) are located in plummer blocks (1) which are polyamide plastic. The plummer blocks are provided with an inlet nozzle (8), to inject the test fluid and exhaust pipe (9). The rig is mounted on a machined steel plate (6), approximately 275 mm x 985 mm x 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. To favour the flow of test fluid, the steel plate is tilted 1,5°.

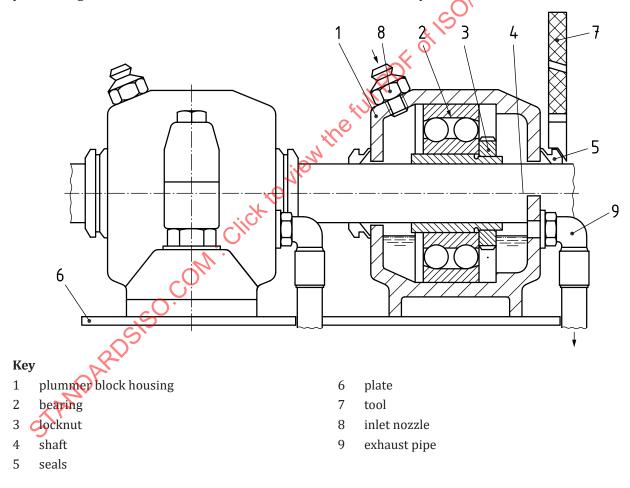


Figure A.1 — Suitable test rig apparatus

#### 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<sup>-1</sup>) or 9,0 rad/s (85 min<sup>-1</sup>). The specification of 8,7 rad/s  $\pm$  0,5 rad/s (83 min<sup>-1</sup>  $\pm$  5 min<sup>-1</sup>) 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.

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