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

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



Reference number
ISO 11007:1997(E)

Foreword

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International Standard ISO 11007 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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

WARNING - The use of this International Standard may involve hazardous materials, operations and equipment. 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 standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the rust-prevention characteristics of lubricating grease in the presence of an aqueous test fluid.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 15:1981, *Rolling bearings - Radial bearings - Boundary dimensions - General plan.*

ISO 648:1977, *Laboratory glassware - One-mark pipettes.*

ISO 3696:1987, *Water for analytical laboratory use - Specification and test methods.*

ISO 3838:1983, *Crude petroleum and liquid or solid petroleum products - Determination of density or relative density - Capillary-stoppered pycnometer and graduated bicapillary pycnometer methods.*

ISO 7120:1987, *Petroleum products and lubricants - Petroleum oils and other fluids - Determination of rust-preventing characteristics in the presence of water.*

3 Principle

A test portion of grease is run in a specialized and carefully cleaned test rig under prescribed conditions for a total of 24 h, in three separate 8 h periods, after introduction of an aqueous test fluid. At the end of the test period, the apparatus is dismantled and the condition of the outer ring track of the bearing is examined for corrosion and rated on an arbitrary scale.

4 Reagents and materials

During the analysis, use only reagents of recognized analytical grade.

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

NOTE 1 White spirit to British Standard BS 245 or mineral spirit to ASTM D 235 (all classes) is suitable (see annex B).

4.2 Water, conforming to grade 3 of ISO 3696.

NOTE 2 Water as defined in 4.2 is used as a wash solvent but water quality in the test fluids (4.3) is critical, and for these a different grade is specified.

4.3 Test fluid. Use one of the following types:

- water conforming to grade 2 of ISO 3696, or
- synthetic sea water conforming to the specification given in ISO 7120, or

- c) an 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 3 Other water types can 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.

4.4 Propan-2-ol.

4.5 Ammonia solution, 3,2 mol/l concentration.

NOTE 4 The ammonia solution is equivalent to a tenfold dilution of 35 % ammonia solution ($\rho = 880 \text{ kg/m}^3$) with water conforming to grade 3 of ISO 3696. This ammonia concentration is critical to the results of the test.

4.6 Solvent rinse solution, consisting of nine volumes of propan-2-ol (4.4) mixed with one volume of ammonia solution (4.5).

4.7 Silver nitrate solution, 0,1 mol/l concentration.

4.8 Cloth, absorbent, lint-free.

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

4.10 Filter paper, any suitable absorbent grade.

5 Apparatus

5.1 Test rig, power-operated with all the test bearings mounted on a single shaft. The rig shall consist of a steel shaft coated with polyamide, and polyamide plummer blocks.

NOTE 5 A suitable rig is detailed in annex A¹⁾

5.2 Test bearing: Use a double row self-aligning ball bearing (30 mm x 72 mm x 19 mm), conforming to 1306K of ISO 15, with a steel cage, specially inspected and packed to prevent rust. In cases of dispute, use the test bearing specified in note 6 as the referee bearing.

NOTE 6 Precision has only been evaluated on SKF 1306K/236 725 bearings.

5.3 Dentist's mirror, no magnification.

5.4 Pipette, conforming to ISO 648, capacity 10 ml.

5.5 Oven, capable of maintaining a temperature of $90 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$.

6 Preparation of apparatus

6.1 Remove all traces of grease from previous tests by wiping the plummer blocks. Wash the plummer blocks in solvent rinse solution (4.6) followed by water (4.2). Dry thoroughly using a cloth (4.8).

Where previous tests have been carried out with either synthetic sea water or salt water, pour a few drops of silver nitrate solution (4.7) 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 (4.2) and repeat this washing until a withdrawn sample is clear.

6.2 Wear protective gloves (5.4) for all subsequent handling. Use two new bearings for each test.

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

NOTE 7 An electric pen, grounded (earthed) on the outer ring surface, is a suitable means of marking.

6.4 Wash the bearings in wash solvent (4.1) at $50 \text{ }^\circ\text{C}$ to $65 \text{ }^\circ\text{C}$, 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 (note 1) is the referee solvent.

NOTE 8 The complete removal of the rust-preventative is critical to the precision of this International Standard. Failure to remove the material wholly will invalidate results.

6.5 Transfer the bearings from the wash solvent to the solvent rinse solution (4.6) to remove any solvent remaining. Rinse the bearings and rotate the outer ring slowly relative to the inner ring, whilst the bearing is immersed in freshly-made solvent rinse solution heated to a minimum of $65 \text{ }^\circ\text{C}$.

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.

¹⁾ A list of suppliers of suitable test rigs is available from ISO/TC 28 Secretariat.

6.6 Remove the bearings from the solvent rinse solution and place on a filter paper (4.10) or cloth (4.8) to drain thoroughly. Place the bearings in the oven (5.5) until completely dry. This requires a minimum of 15 min.

6.7 Remove the bearings from the oven and allow to cool 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.

6.8 Inspect the outer ring tracks using a dentist's mirror (5.3). If etch spots or corrosion are evident, reject the bearing.

6.9 Determine the mass of grease, m , in grams, equivalent to $10,5 \text{ ml} \pm 0,1 \text{ ml}$ using the following equation:

$$m = \rho V$$

where

ρ is the density of the grease, in kilograms per cubic metre;

V is the volume, $= 10,5 \text{ ml} = 0,0105 \text{ l}$

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.

Weigh the clean bearing, then distribute $10,5 \text{ ml} \pm 0,1 \text{ ml}$ 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.

6.10 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.

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

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

7 Procedure

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

NOTE 9 The precision data was generated under these ambient conditions.

7.1 Run the rig for 30 min at $8,7 \text{ rad/s} \pm 0,5 \text{ rad/s}$ ($83 \text{ rev/min} \pm 5 \text{ rev/min}$) immediately after assembly to distribute the grease evenly.

NOTE 10 Whilst 80 rev/min ($8,4 \text{ rad/s}$) is the preferred rig speed, instruments are available that run at 80 rev/min and 85 rev/min . These two speeds have been demonstrated to give equivalent results. Hence the specification is $83 \text{ rev/min} \pm 5 \text{ rev/min}$ ($8,7 \text{ rad/s} \pm 0,5 \text{ rad/s}$) as given in annex A.

7.2 Remove the top halves of the plummer blocks and introduce 10 ml of the chosen test fluid (4.3) into each side of each plummer block using the pipette (5.4) (i.e. a total of 20 ml). Refit the top halves of the plummer blocks and screw down finger-tight.

7.3

- Run the rig for $8 \text{ h} \pm 10 \text{ min}$. Stop the rig and allow to stand for $16 \text{ h} \pm 10 \text{ min}$.
- Run the rig for a further $8 \text{ h} \pm 10 \text{ min}$. Stop the rig and allow to stand for $16 \text{ h} \pm 10 \text{ min}$.
- Run the rig for a further $8 \text{ h} \pm 10 \text{ min}$. Stop the rig and allow to stand for $108 \text{ h} \pm 2 \text{ h}$.

7.4 Carry out 7.2 and 7.3 with minimum delay. As soon as the shaft of the rig begins to rotate (7.3 a), press the V-ring (face) seals up against the plummer blocks using the special tool [shown as (7) in figure A.1].

8 Dismantling the apparatus

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

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

- unscrew the sleeve nut one or two revolutions;
- 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;
- pull the bearing, seals and sleeve off the shaft.

8.3 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.

8.4 Rinse the outer ring of the bearing in the cleaning solvent rinse solution (4.6), and dry using a cloth (4.8). Examine the bearing immediately.

9 Inspection

9.1 Examine the outer ring track for rust or etch spots and evaluate the surface area (3 680 mm²) as specified in table 1. Ignore any staining through which the underlying metal surface is clearly visible.

Table 1 - Evaluation of rust or etch spots

Rating	Degree of rusting
0	No corrosion
1	Not more than three small spots each just sufficient to be visible to the naked eye and less than 1 mm in 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

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.

NOTE 11 To assist in estimating percentage corrosion area, a transparent grid divided into suitable squares can be used.

NOTE 12 The various ratings are illustrated in figure 1. In all cases, the area referred to represents the whole of the track.

9.2 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.

10 Expression of results

Report the two individual ratings (taking into account the requirements of 9.2) and the test fluid (4.3) used.

11 Precision

The precision of the method, as obtained by statistical examination of inter-laboratory test results, is given in 11.1 and 11.2.

11.1 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 normal and correct operation of the test method, exceed the values shown in table 2 only in one case in 20.

11.2 Reproducibility. The difference between two single and independent results, obtained by different operators working in different laboratories on nominally identical test material would, in the normal and correct operation of the test method, exceed the values shown in table 2 only in one case in 20.

Table 2 - Precision data

Test fluid	Repeatability	Reproducibility
Water as specified in 4.3 a)	1	1
Synthetic sea water as specified in 4.3 b)	1	2
Sodium chloride solution as specified in 4.3 c)	1	2

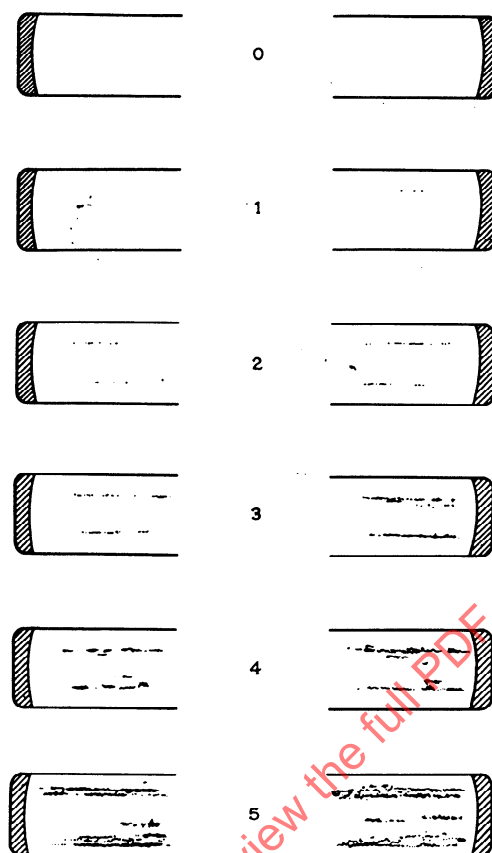


Figure 1 - Various ratings illustrating degree of corrosion

12 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) the type and complete identification of the product tested;
- c) the results of the test (see clause 10);
- d) any deviation, by agreement or otherwise, from the standard procedures specified;
- e) the date of the test.

Annex A (informative)

Suitable test rig apparatus

A.1 Dynamic anti-rust test rigs

Figure A.1 shows part of a standard rig (Drawing No. SKF 1516600) which is capable of accommodating up to eight self-aligning ball bearings fitted with adaptor sleeves and sleeve nuts (3) of polyamide plastic. The bearings (2) are located in plummer blocks (1) which are polyamide plastic, according to drawing number SKF 720315. 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 seals¹⁾

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 rev/min) or 9,0 rad/s (85 rev/min). The specification of 8,7 rad/s $\pm$ 0,5 rad/s (83 rev/min $\pm$ 5 rev/min) covers these two standard speeds (see note 9).

A.3 General

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

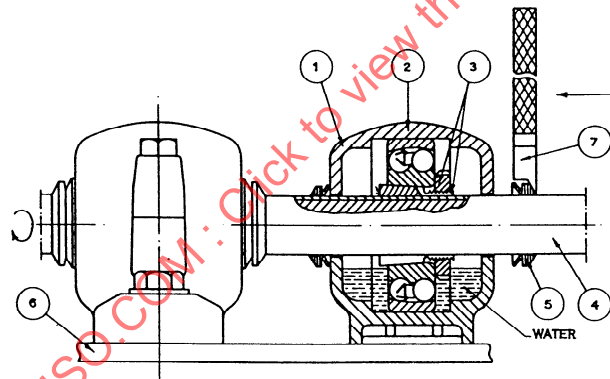


Figure A.1 - Suitable test rig

1) The SKF designations given above are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Annex B (informative)

Bibliography

B.1 The references and sources of the specifications for the wash solvent alternatives (4.1) are given in B.1.1 and B.1.2.

B.1.1 White spirit

BS 245:1976, *Specification for mineral solvents (white spirit and related hydrocarbon solvents) for paints and other purposes*.

Obtainable from British Standards Institution, 389 Chiswick High Road, London, W4 4AL, UK.

B.1.2 Mineral spirit

ASTM D 235:1992, *Standard specification for mineral spirits (petroleum spirits) (hydrocarbon dry cleaning solvent)*.

Obtainable from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, U.S.A.

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