INTERNATIONAL STANDARD

ISO 11009

Second edition

Petroleum products and lubricants — Determination of water washout characteristics of lubricating greases

Produits pétroliers et lubrifiants — Détermination de la résistance au délavage à l'eau des graisses lubrifiantes

PROOF/ÉPREUVE

Reference number
ISO 11009:2021(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

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 www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 11009:2000), which has been technically revised.

The main changes compared to the previous edition are as follows:

— addition of a rating of the appearance of the grease and of the water after test;

— addition of a note concerning the application of the test results to determine the symbol 3 “water contamination and rust protection“ of ISO 12924 grease specifications.

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 www.iso.org/members.html.

Introduction

ISO 6743-9[1] 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 ISO 11007[2] and the level of resistance to water contamination using this document.

Greases are specified in ISO 12924[3].

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

Petroleum products and lubricants — Determination of water washout characteristics of lubricating greases

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 evaluating the resistance of a lubricating grease to washout by water from a bearing, when tested at 38 °C or 79 °C under specified laboratory test conditions.

This test method estimates the resistance of greases to washout from ball bearings under specified conditions. No formal correlation with field service has been established so far.

This document is used for development and specification purposes.

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

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

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 apply.

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

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

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

4 Principle

A test portion of grease is packed in a ball bearing inserted in a housing with specified clearances. The bearing is rotated at 63 rad/s ± 3 rad/s (600 min^{-1} ± 30 min^{-1}). Water, controlled at a specified test temperature, impinges on the bearing housing from a nozzle with a diameter of 1 mm, at a rate of 5 ml/s ± 0,5 ml/s. The distance between the end of the nozzle and the shield of the housing is 57 mm. The water jet is set to strike the bearing 6 mm ± 2 mm above the upper edge of the shield. The amount of grease washed out in 60 min ± 5 min is a measure of the resistance of the grease to water washout.

5 Sampling

Unless otherwise specified, sampling of bearing greases shall be carried out in accordance with the relevant procedure described in 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

6.1 Test apparatus, consisting of a bronze housing, with a spindle supported by two deep groove ball bearings (type 6204) and a water injection pump, actuated by an electrical motor, as well as a water reservoir, a vane system allowing to adjust the water flow rate through an injection nozzle, and a water heating device, recommended to be capable of maintaining the water temperature at the test temperature at ± 1,7 °C.

See complete description in Annex A.

6.2 Bearing, type 6204 bearing with steel cage (in accordance with ISO 15) – C3 clearance.

6.3 Graduated cylinder, 100 ml to measure the flow rate of fluid.

6.4 Timer, accurate to 1/10 s.

6.5 Watch glass, with sufficient size to contain the test bearing and the shields.

6.6 Balance, which can be read to the nearest 1 mg.

6.7 Oven, with sufficient size capable of maintaining the specified drying temperature to ± 2 °C.

6.8 Desiccator, of sufficient size with drying agent.

6.9 Thermometer or **temperature probe,** with a precision to 1 °C to control the temperature of the water in the reservoir.

7 Materials

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[6] or mineral spirit according to ASTM D235[4] (all classes) are suitable.

7.2 Water, conforming to grade 3 of ISO 3696.

8 Preparation of the apparatus

The water reservoir, the piping and the pump, following their degree of soiling, are first cleaned with the solvent (7.1), then by water (7.2) circulation through the pump. The oil traces deposited onto the walls of the water reservoir, on the baffles shall be thoroughly wiped. The bearings, shafts and shields shall be dismantled and carefully cleaned with the solvent and dried. The housing shall be carefully wiped with a lint free rag.

In addition, the washer fixed at the end of the rotating shaft shall be very well centred to ensure the 0,8 mm circular clearance with the shield. For that purpose, use a fitting screw, with a non-threaded part crossing the washer, the diameter of the non-threaded part being equal to that of the hole in the middle of the washer. See Figure A.2.

9 Operating procedure

9.1 Carry out the test in duplicate. Fill the tared bearing (6.2) with 4,00 g ± 0,05 g of test grease, equally distributed over the bearing. Insert the bearing and tared shields into the housing, and assemble the unit in the test apparatus. Record the mass of the bearing, shields and grease, to the nearest 0,01 g.

9.2 Add a minimum of 750 ml of preheated water (7.2) to the reservoir, but keep the water level below the bearing housing by means of a piece of tubing attached to the capillary discharge tube (key 4 on Figure A.1) or with a deflecting metal shield, until equilibrium temperature is attained. When the test is to be carried out at the higher temperature of 79,0 °C ± 1,7 °C, bring the water to the specified temperature by a previously selected source of heat. If the rig has one motor driving both the pump and test bearing, remove the belt from the test-bearing spindle pulley during the water warm-up time.

9.3 When the water reaches the specified temperature, adjust the by-pass valve to give a water-flow rate of 5 ml/s ± 0,5 ml/s through the piece of tubing fixed on the capillary discharge tube and into the graduated cylinder (6.3). Determine the flow rate from the volume of liquid flowing into the cylinder over a period of 10 s, as measured with the timer (6.4). Remove the piece of tubing from the capillary discharge tube, and adjust the water jet so that it impinges on the shield 6,4 mm above the annular opening.

9.4 Start the test and continue operation for 60 min ± 5 min from the moment when the rotational speed of the bearing reaches 63 rad/s ± 3 rad/s.

9.5 Shut off the motor and the heating source, if used. Remove the test bearing and shields and place them on a tared watch glass (6.5). The shields shall be separated from the bearing and placed with their inner faces upward to expose the wet grease to the air.

9.6 Dry the bearing and shields for 15 h at 77 °C ± 6 °C (see NOTE) in the oven (6.7), and then weigh to the nearest 0,01 g to determine the grease mass loss. The grease remaining on the shields, and any leakage occurring during the drying period, should not be considered as grease mass loss.

NOTE In some cases, a mass increase and not a mass loss, can be noted. The origin can be building up of stable grease – water emulsion, from which the water cannot be evaporated in the foreseen drying conditions.

The drying temperature should be increased to 93 °C ± 3 °C for greases containing high-viscosity oils, to facilitate removal of water during the time period specified.

9.7 Important mass loss can be encountered with grease containing very fluid base oils, that can partially evaporate under the specified drying conditions. In that case, it is advisable to carry out a blank test. The blank test is performed packing the bearing with the prescribed grease quantity as indicated in 9.1; then the bearing is placed in the test apparatus and rotated during 5 min to distribute the grease evenly in the bearing, but without applying the water circulation. Then operate as indicated in 9.6.

10 Calculations

The mass loss of grease by water washout, w, expressed in % (m/m), is calculated using Formulae (1), (2) and (3):

$$\Delta m_e = m_2 - m_1 \quad (1)$$

$$\Delta m_a = m_3 - m_1 \quad (2)$$

$$w = \frac{100 \cdot (\Delta m_e - \Delta m_a)}{\Delta m_e} \quad (3)$$

where

Δm_e is the mass of grease before test, expressed in g;

Δm_a is the mass of grease after test, expressed in g;

m_1 is the mass of the bearing and shields, expressed in g;

m_2 is the mass of grease, bearings and shields before test, expressed in g;

m_3 is the mass of grease, bearing and shields, after test, expressed in g.

Report the mass loss, w, in % (m/m) rounded to the nearest whole percent.

11 Expression of results

11.1 Report the average of duplicate tests as the percentage mass of grease washed out at the test temperature, and indicate the temperature at which the bearing assembly and grease were dried. Round the mass percentage to the nearest whole percent.

11.2 Carry out a visual examination, using the following observations as applicable:

11.2.1 Aspect of the grease after test:

— appearance unchanged;

— grease emulsified;

— grease hardened;

— grease softened;

— grease leaking out from the bearing.

11.2.2 Aspect of the water after test:

— water clear;

— water with a milky appearance;

— water with supernatant oil.

