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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*



Reference number
ISO 11009:2000(E)

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 11009 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

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

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International 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 evaluating the resistance of a lubricating grease to washout by water from a bearing, when tested at 38 °C and 79 °C under specified laboratory conditions.

It is not to be considered the equivalent of service evaluation tests.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For a dated reference, subsequent amendments to, or revisions of, the publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For an undated reference, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Principle

The grease is packed in a ball bearing. The bearing is then inserted in a housing with specified clearances, and rotated at $63 \text{ rad/s} \pm 3 \text{ rad/s}$. Water, controlled at 38 °C or 79 °C, impinges on the bearing housing at a rate of $5 \text{ ml/s} \pm 0,5 \text{ ml/s}$. The amount of grease washed out in 60 min is taken as a measure of the resistance of the grease to water washout.

4 Reagents and materials

4.1 Water, conforming to the requirements of grade 3 of ISO 3696.

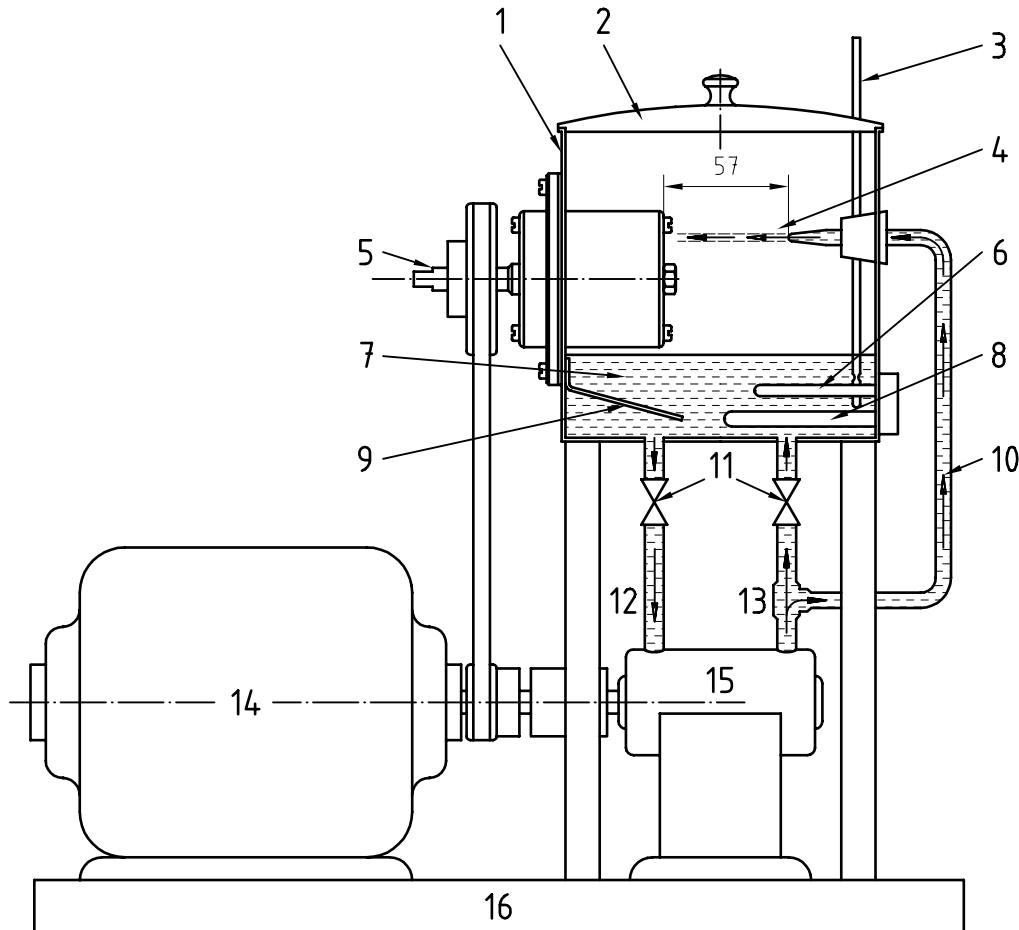
4.2 Cleaning solvent, consisting of a low sulfur, low aromatic, low volatility hydrocarbon.

NOTE White spirit according to British Standard BS 245 [1] or mineral spirit according to ASTM D235 [2] (all classes) are suitable.

5 Apparatus¹⁾

5.1 **Water washout apparatus**, comprising a housing (5.3) with a spindle supported by two ball bearings (5.2) and a water-injection pump, which are rotated by means of an electrical motor(s), a water reservoir and a vane system to regulate the water flow rate through an injection nozzle, and a water heating system (5.4). Suitable apparatus is shown in Figure 1.

Dimensions in millimetres



Key

- | | |
|---|-----------------------------|
| 1 Tank size: approx. 150 mm × 150 mm × 150 mm | 8 Heater |
| 2 Cover | 9 Baffle |
| 3 Thermometer | 10 Supply |
| 4 Water-jet flow rate: 5 ml/s ± 0,5 ml/s through 1 mm φ capillary | 11 Flow-rate control valves |
| 5 Rotation speed of ball bearings: 63 rad/s ± 3 rad/s | 12 Return |
| 6 Thermostat | 13 Bypass |
| 7 750 ml (min.) of distilled water in reservoir below the bearing housing | 14 Motor |
| | 15 Fluid pump |
| | 16 Base |

Figure 1 — Apparatus for determining water washout characteristics of lubricating greases

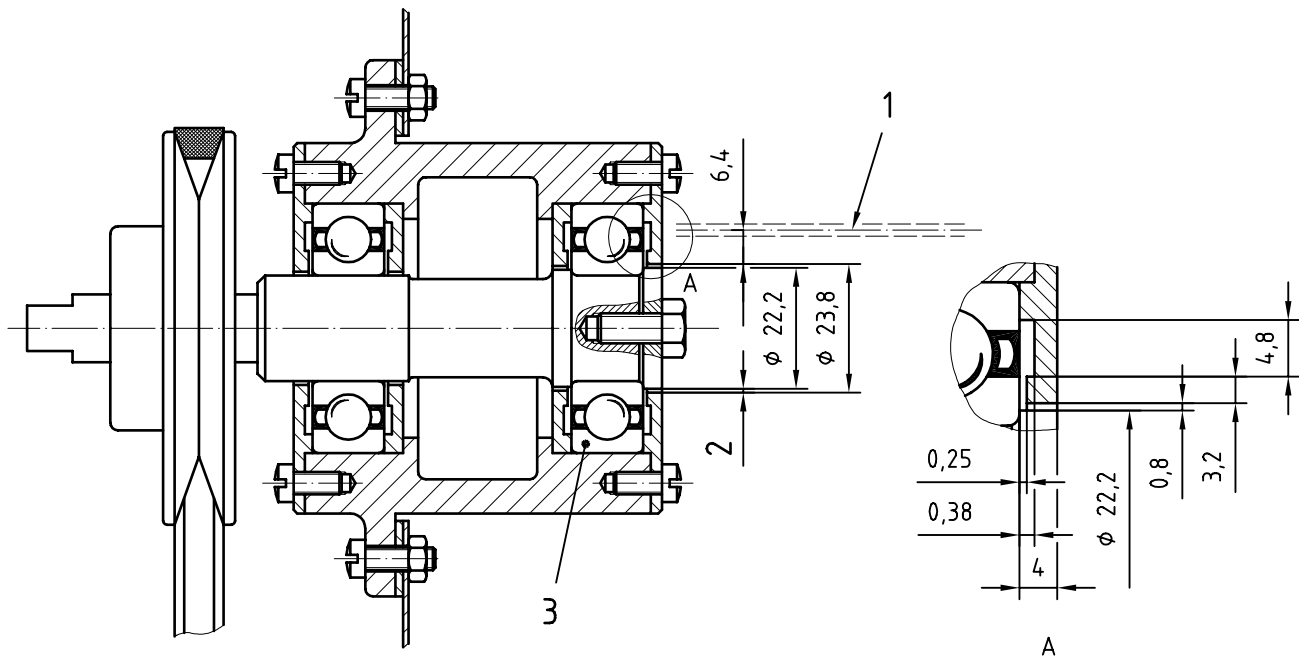
¹⁾ This apparatus is available from: the Precision Scientific Co., 3737 Cortland St., Chicago, IL, USA, Labline Inc., 3072-82 W. Grant Ave., Chicago, IL-60622, USA, as catalogue No. 4165; and Stanhope-Seta, Park Close, Englefield Green, Egham, Surrey, TW20 0XD, UK as catalogue No. 19160. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

5.2 Ball bearing, size 6204 C3H.²⁾

NOTE 6204 C4 bearings may be used. However, precision has only been determined using SKF 6204 C3H bearings.

5.3 Bearing housing and shield, with dimensions as shown in Figure 2.

Dimensions in millimetres



Key

- 1 Centreline of capillary, ϕ 1 mm
- 2 Annular opening, 0,8 mm wide
- 3 Test bearing of type 6204 C3H

Figure 2 — Typical cross-section of thrust-bearing assembly

5.4 Heating source, to maintain the water temperature at $38\text{ }^{\circ}\text{C} \pm 1,7\text{ }^{\circ}\text{C}$ or $79\text{ }^{\circ}\text{C} \pm 1,7\text{ }^{\circ}\text{C}$.

NOTE A suitable temperature can be obtained by the use of immersion heaters, steam coils, or infrared heat lamps, in conjunction with transformers or thermostats.

5.5 Thermometer or thermocouple, of adequate range, to determine the temperature of the water reservoir at $\pm 1\text{ }^{\circ}\text{C}$.

5.6 Timer, indicating tenths of a second.

5.7 Watch glasses, of sufficient size to accommodate a test bearing and shield.

5.8 Graduated cylinder, of 100 ml capacity.

5.9 Oven, natural convection type, capable of maintaining the specified drying temperature at $\pm 3\text{ }^{\circ}\text{C}$.

5.10 Balance, capable of weighing to 1 mg.

²⁾ 6204 C3H bearings are available from local suppliers or ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA, at nominal cost by specifying PCN 12-433360-12.

6 Sampling

- 6.1 Each test will require a quantity of grease sufficient to fill two test bearings (approximately 4 g each).
- 6.2 A minimum of 15 g of sample shall be supplied.
- 6.3 Examine the sample for any indication of non-homogeneity such as oil separation, phase changes, or gross contamination. If any abnormal conditions are found, obtain a new sample.

7 Preparation of apparatus

Clean the reservoir and water passages by flushing with water (4.1). Wipe off any oil scum which has been deposited on the surfaces of the reservoir. Clean the test bearing with cleaning solvent (4.2). Ensure that the water flow rate can be maintained within the specified limits for a 60 min test run.

8 Procedure

- 8.1 Carry out the test in duplicate. Pack the tared bearing with $4,00 \text{ g} \pm 0,05 \text{ g}$ of test grease. 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.
- 8.2 Add a minimum of 750 ml of preheated water (4.1) 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 or with a deflecting metal shield, until equilibrium temperature is attained. When the test is to be carried out at the higher temperature of $79 \text{ }^\circ\text{C} \pm 1,7 \text{ }^\circ\text{C}$, 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.
- 8.3 When the water reaches the specified temperature, adjust the by-pass valve to give a water-flow rate, through the rubber tubing and into the graduated cylinder (5.8), of $5 \text{ ml/s} \pm 0,5 \text{ ml/s}$. Determine the flow rate from the volume of liquid flowing into the cylinder over a period of 10 s, as measured with the timer (5.6). Remove the rubber tubing from the discharge tube, and adjust the water jet so that it impinges on the shield 6,4 mm above the annular opening.
- 8.4 Start the test and continue operation for $60 \text{ min} \pm 5 \text{ min}$ from the moment when the rotational speed of the bearing reaches $63 \text{ rad/s} \pm 3 \text{ rad/s}$.
- 8.5 Shut off the motor and the heating source, if used. Remove the test bearing and shields and place them on a tared watch glass (5.7). The shields shall be separated from the bearing and placed with their inner faces upward to expose the wet grease to the air.
- 8.6 Dry the bearing and shields for 15 h at $77 \text{ }^\circ\text{C} \pm 6 \text{ }^\circ\text{C}$ (see the note) in the oven (5.9), and then weigh to the nearest 0,01 g to determine the grease loss. The grease remaining on the shields, and any leakage occurring during the drying period, should not be considered as grease lost.

NOTE Some mass loss can be experienced for greases containing low-viscosity oils because of oil evaporation during drying. The drying temperature should be increased to $93 \text{ }^\circ\text{C} \pm 3 \text{ }^\circ\text{C}$ for greases containing high-viscosity oils, to facilitate removal of water during the time period specified.

9 Calculation

Calculate the loss in mass by water washout, w , expressed as a mass fraction, using the following equations.

$$\Delta m_e = m_2 - m_1$$

$$\Delta m_a = m_3 - m_1$$

$$w = 100 \times \frac{\Delta m_a - \Delta m_e}{\Delta m_e}$$

where

m_1 is the mass of the bearing and shields, in grams;

m_2 is the mass of the grease, the bearing and shields before testing, in grams;

m_3 is the mass of the grease, the bearing and shields after testing, in grams.

10 Expression of results

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 Precision

11.1 General

The precision, as determined by statistical examination of interlaboratory test results, is given in 11.2 and 11.3.

11.2 Repeatability, r

The difference between two 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 values in only one case in twenty.

$$\text{At } 38 \text{ }^\circ\text{C} \quad r = 0,8 (X + 2)$$

$$\text{At } 79 \text{ }^\circ\text{C} \quad r = 0,6 (X + 4,6)$$

where X is the average of two results given as mass fractions.

11.3 Reproducibility, R

The difference between two single and independent test 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 values in only one case in twenty.

$$\text{At } 38 \text{ }^\circ\text{C} \quad R = 1,4 (X + 2)$$

$$\text{At } 79 \text{ }^\circ\text{C} \quad R = 1,1 (X + 4,6)$$

where X is the average of two results given as mass fractions.

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 procedure specified;
- e) the date of the test.

Bibliography

- [1] BS 245:1976, *Specification for mineral solvents (white spirit and related hydrocarbon solvents) for paints and other purposes.*³⁾
- [2] ASTM D 235-95, *Standard Specification for Mineral Spirits (Petroleum spirits) (Hydrocarbon Dry Cleaning Solvent).*⁴⁾

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- 3) Obtainable from British Standards Institution, British Standards House, 389 Chiswick High Road, London, W4 4AL, UK.
 - 4) Obtainable from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA.

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