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Petroleum products and lubricants — Determination of the mechanical stability of greases in presence of water

Produits pétroliers et lubrifiants — Détermination de la stabilité mécanique des graisses en présence d'eau

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

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Introduction

Numerous standards exist to evaluate the influence of water on grease properties. These standards are described in Annex A.

The purpose of this document is to describe a method for the evaluation of the effect of water on the working stability of a grease, by comparing the penetrations of the grease worked without water and the grease worked in presence of water. This allows to predict the mechanical behaviour of the grease, the risk of leakages due to excessive softening, the risk of lubricating properties losses by excessive hardening or complete solubilization.

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Petroleum products and lubricants — Determination of the mechanical stability of greases in presence of water

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1 Scope

This document specifies a method for evaluating the change of consistency of a grease when it is submitted to working and prolonged working in presence of 10 % of water.

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 2137:2020, *Petroleum products and lubricants — Determination of cone penetration of lubricating greases and petrolatum*

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

No terms and definitions are listed in this document.

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/

4 Principle

A specified mass of grease is blended with a specified quantity of water, then worked, either 60 strokes or 100 000 strokes in a grease worker. The penetration measured after this working is compared to the penetration measured after working 60 strokes or 100 000 strokes in absence of water. The difference of the penetrations recorded with and without water yields the water resistance of the grease.

5 Significance of the test

This test method estimates the softening or hardening tendency of a grease when submitted to prolonged working in presence of water. No formal correlation with field service has been established.

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6 Sampling

Sampling of the bearing greases shall be carried out 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.

7 Materials

Only materials of recognized analytical grade shall be used.

7.1 Equipment to perform penetrability measurement, as specified in ISO 2137. Use only the full-scale cone (or the optional cone) and the full-scale worker.

7.2 Cup, flat bottom with a diameter of approximately 200 mm, with handles.

7.3 Spatula, made from non-abrasive material (wood, plastic).

7.4 Balance, which can be read to the nearest 0,1 g.

7.5 Mechanical stirrer, low speed, (electric kitchen) with a suitable mixing bowl.

8 Reagents

Only reagents of recognized analytical grade shall be used.

8.1 Water, conforming to grade 2 of ISO 3696.

8.2 Synthetic sea water, in accordance with ISO 7120.

9 Operating procedure

9.1 Determine the $P_{60\ dry,}$ (penetration after 60 strokes without water) in accordance with ISO 2137:2020, 8.2 after working 60 strokes.

9.2 Determine the penetration, $P_{60\ wet}$. (penetration after 60 strokes in presence of water).

9.2.1 Weigh in the cup (7.2) 450 g of grease and 50 g of water (8.1 or 8.2).

9.2.2 Using the spatula (7.3), blend the grease and the water until completely homogenized.

When the water is incorporated to the grease, fill the worker and proceed as in 9.1 to determine the penetration after working 60 strokes.

NOTE In the case where the blend is not homogeneous, or the water is difficult to incorporate, it is possible to perform the blend using a mechanical stirrer (7.5) the rotating speed of which being chosen so as to avoid water splashing on the walls of the cup and not to provoke too important agitation.

9.3 Determine the penetration $P_{100\ 000\ dry,}$ (penetration after 100 000 strokes without water) as specified in ISO 2137:2020, 8.3, after working 100 000 strokes.

9.4 Determine the penetration $P_{100\ 000\ wet,}$ (penetration after 100 000 strokes in presence of water) on the sample from penetrability $P_{60\ wet}$ as specified in ISO 2137:2020, 8.3, after working 100 000 strokes.

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10 Calculation

Calculate:

$P_{60\,\text{wet}} - P_{60\,\text{dry}}$ which indicates how grease consistency is affected by water ingress (when >0, the grease softens, when <0, the grease hardens).

$P_{100\,000\,\text{wet}} - P_{100\,000\,\text{dry}}$ which indicates how the grease can continue to operate after water ingress.

$P_{100\,000\,\text{wet}} - P_{60\,\text{dry}}$ which gives information about the effect of prolonged working in presence of water.

$P_{100\,000\,\text{wet}} - P_{60\,\text{wet}}$ which gives information of the effect of water ingress on the mechanical stability.

11 Expression of the result

Report the results in 1/10 of millimetres:

$P_{60\,\text{dry}}$, $P_{60\,\text{wet}}$, $P_{100\,000\,\text{dry}}$, $P_{100\,000\,\text{wet}}$

$P_{60\,\text{wet}} - P_{60\,\text{dry}}$, $P_{100\,000\,\text{wet}} - P_{100\,000\,\text{dry}}$, $P_{100\,000\,\text{wet}} - P_{60\,\text{dry}}$, $P_{100\,000\,\text{wet}} - P_{60\,\text{wet}}$

12 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 12921:2024;

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

c) the nature of the water used (distilled or synthetic sea water);

d) the result (see Clause 11);

e) any deviations from the procedure;

f) any unusual features observed;

g) the date of the test.

13 Precision

13.1 General

The precision has been evaluated for $P_{100\,000\,\text{wet}} - P_{100\,000\,\text{dry}}$ and for $P_{100\,000\,\text{wet}} - P_{60\,\text{dry}}$ by examining interlaboratory test results according to ISO 4259-1[16]. The precision for $P_{100\,000\,\text{wet}} - P_{100\,000\,\text{dry}}$, distilled and salt water, for $P_{100\,000\,\text{wet}} - P_{60\,\text{dry}}$ as determined by statistical examination of interlaboratory test results according to ISO 4259-1, is given in 13.2 and 13.3.

13.2 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 1.

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13.3 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 1.

Table 1 — Precision of the test

Penetration	*r*	*R*
$P_{100\,000\,\text{wet}} - P_{60\,\text{dry}}$ distilled water	12	40
$P_{100\,000\,\text{wet}} - P_{60\,\text{dry}}$ synthetic sea water	22	53
$P_{100\,000\,\text{wet}} - P_{60\,\text{dry}}$	20	26

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Annex A
(informative)

Existing standards to evaluate the influence of water on grease properties

Table A.1 shows the various methods currently used to determine the effect of water on the properties of grease.

Table A.1 — Behaviour of grease in presence of water

Behaviour of grease in presence of water		
Property evaluated	**ISO standard**	**Other standard**
Resistance to wash out	ISO 11009[1]	IP 215[4], ASTM D1264[5], ASTM D4049[6], DIN 51807-1[7], DIN 51807-2[8]
Corrosion protection	ISO 11007-1[2]	ASTM D1743[10], IP 220[11], DIN 51802[9], ASTM D6138[12]
	ISO/TS 11007-2[3]	
Resistance to prolonged working in presence of water	ISO 12921	ASTM D7342[13], NF T60-626[14]
Rolling stability in presence water		ASTM D8022[15]

ISO 11009[1] describes a method to evaluate the resistance of grease to washout from a bearing in laboratory conditions. ASTM D1264[5], DIN 51807-2[8] and IP 215[4] use a similar procedure.

DIN 51807-1[7] is a static test.

ASTM D4049[6] indicates the ability of a grease to adhere to a metallic surface when subjected to a water spray.

ISO 11007-1[2] and ISO/TS 11007-2[3] evaluate the ability of a grease to protect bearings against corrosion in dynamic conditions and in presence of wash out respectively. DIN 51802[9] is equivalent to ISO 11007-1.

ASTM D6138[12] and IP 220[11] are similar to ISO 11007-1.

ASTM D1743[10] covers the corrosion preventive properties of lubricating grease using grease lubricating tapered roller bearings stored under wet conditions. This test is, contrarily to ISO 11007-1 and ISO/TS 11007-2, a static test.

This document (similar to ASTM D7342[13], NF T60-626[14] and ASTM D8022[15]) includes tests which evaluate the behaviour of greases when submitted to working in presence of water. The action of water on greases can also be shown by a more or less important absorption of water, by more or less important hardening or softening, or by a loss of adhesiveness on metallic surfaces.

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