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# Petroleum products and lubricants — Determination of oil separation from grease — Pressure filtration method



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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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 www.iso.org/directives).

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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 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*.

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

Storage stability is an important parameter in the quality of lubricating greases.

Oil separation, also called “bleeding”, is an indication of an inadequate structure of the thickening agent, e.g. a too low concentration of a large network of fibres, or insufficient interaction between the base oil and the thickening agent, or a too high interaction inside the crystalline network of the gel leading to a contraction of the latter and expulsion of oil.

Oil separation at storage renders the grease improper for use. An oil separation which is too high leads to a hardening that can be significant or not, depending on the quantity of oil separated. This hardening can lead, when a bearing is running, to the blocking of the rolling elements and of the retainer, and to damage. Hardening can also lead to the clogging of the grease ducts, to the bearing and to difficulties for the functioning of the grease relief valves.

The evaluation of the oil separation is therefore of prime importance to verify the good structure of the gel.

Many methods exist to evaluate the tendency of greases to separate oil. These methods include depositing the grease on a metallic grid, in prescribed temperature conditions and evaluating, after a certain time, the quantity of oil separated. In some cases, additional pressure is applied on the top of the grease surface.

This document is derived from IP 121[5], DIN 51817[2] and NF T 60-191[3]. This is a method where a pressure of 0,66 kPa is applied on the grease.

This document differs from ASTM D 1742[4] which uses a different test device and different conditions of applied pressure, temperature and duration.

# Petroleum products and lubricants — Determination of oil separation from grease — Pressure filtration method

**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 application of the document, and to determine the applicability of any other restrictions for this purpose.**



## 1 Scope

This document specifies a method for measuring the quantity of oil separated from lubricating grease in the test conditions.

It can be used to predict the behaviour of greases on storage in pails and drums.

This method is not applicable to very soft greases that, during the test, flow unchanged through the sieve.

## 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, *Petroleum products and lubricants — Determination of cone penetration of lubricating greases and petrolatum*

ISO 2194, *Industrial screens — Woven wire cloth, perforated plate and electroformed sheet — Designation and nominal sizes of openings*

ISO 12924, *Lubricants, industrial oils and related products (Class L) — Family X (Greases) — Specification*

ASTM D4057, *Standard Practice for Manual Sampling of Petroleum and Petroleum Products*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions 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/

**3.1**
**oil separation**
oil quantity that, in the test conditions, separates from the test grease and is collected, after the test duration, in the receiver

## 4 Sampling

Unless otherwise specified in commodity specifications, samples of greases shall be drawn in accordance with ASTM D4057.

## 5 Principle

A sample of grease is placed in a metallic cylinder closed by a cone made from a metallic gauze.

For greases of NLGI grade 2 and higher, a pressure is applied on the levelled surface by means of weight.

For greases of NLGI grades 0 and 1, the test can be performed without applying pressure.

For very soft greases (NLGI grades 00 and 000), the test is performed without applying pressure.

The test temperature applied can be chosen in the range between 40 °C and 120 °C. The test temperature shall be maintained constant at ±1 °C throughout the test.

The normal duration of the test is 168 h; the test can be shortened to 18 h.

For the tests performed without applying the pressure, the test duration to be chosen is 18 h.

The oil quantity separated through the metallic gauze after the test duration is the measure of the stability of the grease concerning oil separation during storage.

## 6 Significance and use

The results of the present method are representative of the oil separation phenomena in grease containers at storage.

The present method does not take into account the tendency of the grease to separate oil in the conditions of service.

The present method does not apply to grease softer than the NLGI 1 grade, because of the risks of overflow of the grease outside the cup when applying the weight.

The NLGI grades shall be as defined in ISO 12924 by the determination of the penetrability worked 60 strokes at 25 °C, according to ISO 2137.

## 7 Apparatus and reagents

**7.1 Separation apparatus,** shall have the dimensions specified in Figure A.1, consisting of the following.

**7.1.1 Separation cup,** (as in Figure A.2) consisting of a metallic cylinder closed by a stainless steel gauze cone, with an aperture of 63 µm and a wire diameter of 43 µm (240 mesh) shall be in accordance with ISO 2194. The cone, with a tip angle of 140° can be obtained either by forming or by welding, ensuring that the width of the welding is minimal.

**7.1.2 Metal weight,** of (100 ± 0,1) g, as in Figure A.3, to provide the additional mass.

**7.1.3 Receiver,** made from metal or glass, with a diameter and a depth allowing support of the separation cup (7.1.1) so that the tip of the gauze cone is at all times above the oil collected. A cup with a diameter of 55 mm and a depth of 35 mm as in Figure A.4 and according to EN 1426, may be used as a receiver.

**7.2 Oven,** capable of maintaining a temperature of (40 ± 1) °C, throughout the duration of the test.

**7.3 Precision balance,** capable of weighing to the milligram.

**7.4 Spatula,** stainless steel.

**7.5 Heptane or white-spirit.**

# 8 Test procedure

## 8.1 Cleaning of the separation cup

After each test, the separation cup (7.1.1) shall be cleaned using hot heptane or white-spirit, which may be brought to boiling in order to facilitate the elimination of the grease. It is possible to improve the efficiency of the cleaning using an ultrasonic bath. After solvent cleaning, the separation cup is air dried.

Before using any separation cup, it is recommended to check that the meshes of the gauze are not obstructed.

The separation cups shall be handled with care so as to avoid any change of the aperture of the gauze. Particularly avoid air drying with pressure that is too high.

## 8.2 Operating procedure

**8.2.1** Make the determination in triplicate.

For commodity and simplification reasons, the test may be run duplicate instead of triplicate. In such a case, the test will be considered as a "simplified test".

**8.2.2** Weigh the empty receiver (7.1.3) to the nearest milligram, using the balance (7.3), yields $m_1$.

**8.2.3** Weigh the separation cup (7.1.1) to the nearest milligram, using the balance (7.3), yields $M_1$.

**8.2.4** Work the grease as little as possible fill the cup (7.1.1) avoiding the formation of air pockets. If possible, let a small quantity of grease pass through the gauze. Arase the surface with the spatula (7.4) and wipe off the quantity of grease passed through the gauze with the finger. Then weigh the grease filled separation cup to the nearest milligram, using the balance (7.3), yields $M_2$.

**8.2.5** Place the separation cup filled in 8.2.4 on the receiver (7.3.1) tared in 8.2.2.

For greases of NLGI grades 2 and higher, position the 100 g weight (7.1.2) centring it on the surface of the grease.

For greases of NLGI grades 0 and 1, the test can be performed with or without applying the weight. This shall be agreed with the test requester.

For very soft greases of the NLGI grades 00 and 000 that can flow unchanged through the metallic gauze, the test is performed without applying the weight.

**8.2.6** Place the assembly prepared in 8.2.5 in the oven (7.2) maintained at the chosen temperature, making sure of the horizontalness and maintain it during the duration chosen (18 h ± 0,25 h; 168 h ± 2 h). Other test durations can be applied. This shall be documented.

The test temperature applied can be chosen in the range between 40 °C and 120 °C. The test temperature shall be maintained constant at ±1 °C throughout the test.

**8.2.7** At the end of the chosen duration, withdraw the assembly from the oven; take out the separation cup. If an oil drop is suspended on the tip of the cone, recover it in the receiver (7.1.3). Weigh the receiver containing the separated oil to the nearest milligram, using the balance (7.3), yields $m_2$.

The oil separation of greases that flow unchanged through the metallic gauze without application of the weight cannot be determined using this document's method. To check that, fill the separation cup

carefully with the grease, place it on the receiver for 2 h. If after 2 h no grease has flown through the metallic gauze, it is possible to start the test. Otherwise, the oil separation of this grease cannot be determined by means of this document's test.

NOTE Some greases containing solid lubricants, as well as some thickener systems can affect the result because they can pass through the metallic gauze.

**8.2.8** Calculate the quantity of oil separated in mass fraction (%) of the grease, by means of Formula (1):

$$S = \frac{m_2 - m_1}{M_2 - M_1} \cdot 100 \quad (1)$$

where

$S$ is the mass fraction of oils separated after a given time;

$m_1$ is the mass of the empty receiver (in mg);

$M_1$ is the mass of the separation cup (in mg);

$m_2$ is the mass of the receiver containing the separated oil (in mg);

$M_2$ is the mass of the grease filled separation cup (in mg).

Calculate the arithmetical mean of the three determinations and round to the nearest 0,1 % (see 9).

NOTE In the case of application of the simplified procedure, the result will be expressed under the form of the arithmetical mean of the two determinations, round to the nearest 0,5 %.

## 9 Expression of the results

The results, symbolized by the letter *S*, are expressed in terms of mass fraction (%) of oil separated after a given time at a given temperature, applying or not the weight. *S* is the arithmetical mean of the three determinations rounded to the nearest 0,1 % as shown in Formula (2).

$$S \text{ (mass fraction)} = \frac{1}{3}\sum_{i=1}^{3} S_i \quad (2)$$

NOTE For the expression of the result of the simplified procedure, the result is in Formula (3):

$$S_{\text{simplfied}} \text{ (mass fraction)} = \frac{1}{2}\sum_{i=1}^{2} S_i \quad (3)$$

EXAMPLE 1 Grease with oil separation: mass fraction of 3,5 % tested at 80 °C with the normal duration of 168 h (N)

Oil separation ISO 22285: mass fraction of 3,5 % — 80 °C — N

EXAMPLE 2 Grease with oil separation: mass fraction of 1,5 % tested at 40 °C with the short duration of 18 h (S)

Oil separation ISO 22285: mass fraction of 3,5 % – 40 °C – *S*

EXAMPLE 3 Grease with oil separation: mass fraction of 4,5 % tested at 40 °C with the short duration of 18 h, without applying the weight (w/o W, i.e without pressure)

Oil separation ISO 22285: mass fraction of 4,5 % — 40 °C — *S* (w/o W)



# 10 Precision

## 10.1 General

The precision is taken from DIN 51817[2] The precision, as determined by statistical examination in accordance with ISO 4259-1[1] of interlaboratory test results for the temperatures of 40 °C, 80 °C and 120 °C is given in Table 1.

NOTE The precision has not been established for the simplified procedure, nor for other test durations.

## 10.2 Repeatability

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, within a short interval of time, under the same test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, can be calculated using the functions in Table 1.

## 10.3 Reproducibility

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, under different test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, can be calculated as described in Table 1.

**Table 1 — Precision**

| **Test duration**<br>h | **Repeatability,** $r$ | **Reproducibility,** $R$ |
|---|---|---|
| 18 | $0{,}06\,X + 0{,}20$ | $0{,}3\,X + 0{,}28$ |
| 168 | $0{,}07\,X + 0{,}27$ | $0{,}22\,X + 0{,}42$ |
| NOTE X is the average of the two test results being compared. | | |

# 11 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. ISO 22285:2018;

b) the complete identification of the test product;

c) whether this is the simplified (see 8.2.1) procedure which has been applied;

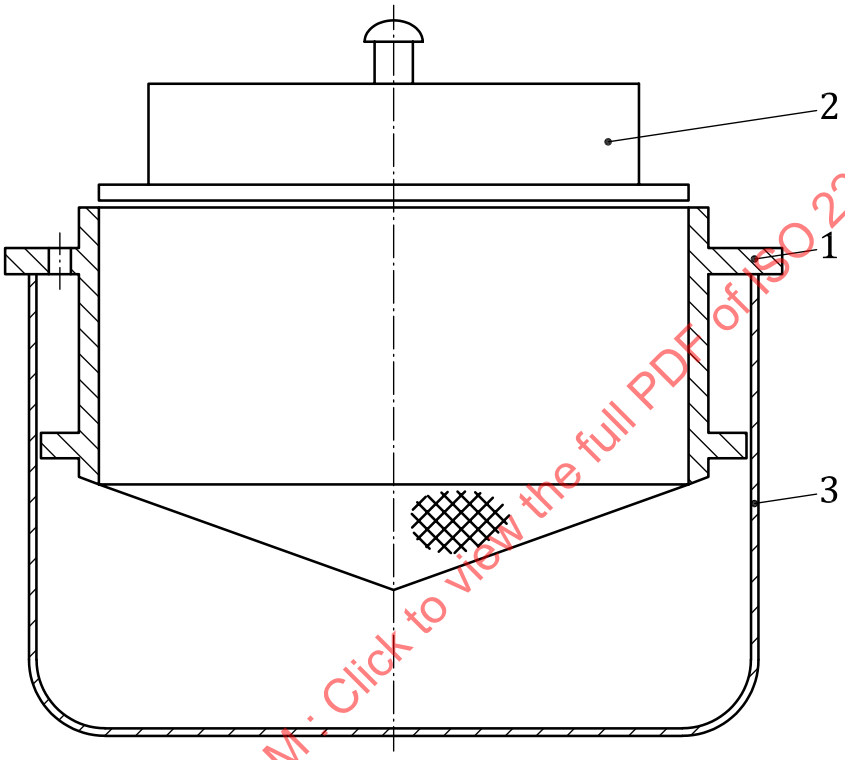
d) the result of the test (see Clause 9);

e) the date of the test.

# Annex A
(normative)

# Separation apparatus constituents

This annex presents the overall apparatus in Figure A.1 and the dimension of the other parts.




**Key**

1 separation cup
2 100 g weight
3 receiver

**Figure A.1 — Separation apparatus**