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

Produits pétroliers et lubrifiants — Détermination de la pénétrabilité au cône des graisses lubrifiantes et des pétrolatums

[Revision of second edition (ISO 2137:1985)]

ICS 75.100; 75.140

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 2137 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This third edition cancels and replaces the second edition (ISO 2137:1985), which has been technically revised (see the introduction).

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Introduction

International Standard ISO 2137 was first published in 1972. A second edition was issued in 1985. This third edition cancels and replaces the first two editions, of which it constitutes a technical revision. The revision mainly concerns the dimensional tolerances of the various cones, to better fit with what is available from laboratory equipment suppliers. This revision is necessary because of all the quality plans and data integrity programmes established by numerous laboratories to comply with the various ISO quality standards. These quality standards require the total compliance of the cones with the dimensions indicated in ISO 2137:1985. Unfortunately, this is not the case for most of the cones available and users were obliged to establish waivers, which was difficult to justify to quality auditors. The dimensional tolerances have been enlarged to allow most of the cones to fulfil the new requirements. A round robin test has been performed with cones conforming to these new requirements and has demonstrated that the precision of the method is not altered by this change in the tolerances. Tolerances have been retained only on the characteristics where it has been established that they have a direct impact on the penetration determination, i.e. tip angle, tip height, tip top thickness, tip base diameter, cone angle, total mass of cone plus movable attachments.

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

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 several methods for the empirical estimation of the consistency of lubricating greases and petrolatum by measuring the penetration of a standardized cone.

The National Lubricating Grease Institute (NLGI) classifies greases according to their consistency, as measured by the 60 strokes worked penetration. The NLGI classification includes nine consistency numbers or grades, each grade corresponding to a given range of worked penetration. The NLGI classification is given in ISO 6743-99.

Clause 7 of this International Standard specifies four procedures for determining the consistency of lubricating greases by measuring the penetration of a full-scale cone. These procedures cover the measurement of unworked, worked, prolonged worked, and block penetrations. Penetrations up to 500 units can be measured.

Clause 8 of this International Standard specifies methods for determining the consistency of lubricating greases when only small samples are available, by the use of cones a half-scale or quarter-scale of that used in Clause 7. The methods are applicable to greases having penetrations of 175 units to 385 units with the full-scale cone and are intended for use only if the size of the test sample prevents the use of cones described in Clause 7. They are not intended to replace the full-scale penetration as described in Clause 7, although a conversion to full-scale penetration is given in 10.2.

The use of the one-quarter-scale cone should be limited as much as possible, due to the poor precision, to cases where the quantity of test grease is not sufficient for use of the one-half-scale cone. It is not recommended to use the one-quarter-scale cone for the determination of the penetration change of greases after mechanical shear tests in bearings or in specific test rigs.

NOTE 1 Unworked penetrations do not generally represent the consistency of greases in use as effectively as do worked penetrations. The latter are usually preferred for inspecting lubricating greases.

NOTE 2 Penetration of block greases can be obtained on those products that are sufficiently hard to hold their shape. These greases generally have penetrations below 85 units.

Clause 9 of this International Standard specifies a method for the determination of the consistency of petrolatum by measurement of the penetration of a full-scale cone, having penetrations up to 300 units. This method may also be used to estimate the consistency of slack waxes.

2 Normative references

The following referenced documents are indispensable for the application 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 6743-99:2002, *Lubricants, industrial oils and related products (class L) — Classification — Part 99: General*

ASTM D 4057, *Standard practice for manual sampling of petroleum and petroleum products*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1 cone penetration

distance that a standardized cone penetrates into a test portion under standardized conditions of load, time, and temperature

NOTE 1 The cone penetration is expressed in units of 0,1 mm.

NOTE 2 This definition is adapted from definition 2.80.001 given in ISO 1998-2¹⁾.

3.2 working

subjection of a lubricating grease to the shearing action of a grease worker

3.3 unworked penetration

cone penetration of a test portion which has received only minimum disturbance in transfer from the sample container to the cup of the grease worker

3.4 worked penetration

cone penetration of a test portion after it has been subjected to a defined number of strokes in a grease worker

3.5 prolonged worked penetration

cone penetration of a test portion which has been worked more than the defined number of strokes in worked penetration (see 3.4)

3.6 block penetration

cone penetration determined on a test portion which is sufficiently hard to hold its shape without a container

4 Principle

The cone penetration of lubricating grease is determined at 25 °C by releasing the cone assembly from the penetrometer and allowing the cone to drop for 5 s, and measuring the extent of the penetration.

1) ISO 1998-2:1998, *Petroleum industry – Terminology – Part 2: Properties and tests*.

Unworked penetrations are determined on test portions transferred with a minimum of disturbance to a container suitable for test purposes.

Worked penetrations are determined immediately after working the test portion for 60 double strokes in a standard grease worker.

Prolonged worked penetrations are determined on test portions worked more than 60 double strokes.

Block penetrations are determined on a freshly prepared face of a cube cut from a block of grease with a standard cutter.

The cone penetration of petrolatum is determined by first melting and cooling a test sample under specified conditions, and then measuring the penetration as for lubricating grease.

5 Apparatus

5.1 Penetrometer

Similar to that shown in Figure 1, designed to measure, in tenths of a millimetre, the penetration of a cone in a material. The cone assembly or the table of the penetrometer shall be adjustable to enable accurate placement of the tip of the cone on the level surface of the material while maintaining a "zero" reading on the indicator. The cone shall fall, when released, without appreciable friction for at least 62 mm. The tip of the cone shall not hit the bottom of the sample container. The instrument shall be provided with level jig screws and a spirit level to maintain the cone shaft in a vertical position.

NOTE The measurement of the penetration depth can be performed using either mechanical devices (mechanical indicator) or electronic devices (digital indicator).

5.2 Cones

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5.2.1 Full-scale cone

Consisting of a conical body of magnesium or other suitable material with a detachable, hardened steel tip. Dimensions and tolerances shall be as shown in Figure 2. The total mass of the cone shall be $102,50 \text{ g} \pm 0,05 \text{ g}$ and that of its movable attachments shall be $47,50 \text{ g} \pm 0,05 \text{ g}$. The attachments consist of a rigid shaft having a stop at its upper end and a suitable means at its lower end for engaging the cone. The interior construction may be modified to achieve the specified mass, provided that the general contour and mass distribution are not altered. The outer surface shall be polished to a very smooth finish.

NOTE For penetrations up to 400 units, the optional cone (see Figure 3) may be used.

5.2.2 One-half-scale cone and shaft

Made of steel, stainless steel, or brass with a hardened steel tip of 45 Rockwell C hardness to 50 Rockwell C hardness, and constructed to conform to the dimensions and tolerances shown in Figure 4. The shaft may be made of stainless steel. The total mass of the cone and its movable attachments shall be $37,500 \text{ g} \pm 0,050 \text{ g}$. The mass of the cone shall be $22,500 \text{ g} \pm 0,025 \text{ g}$. The mass of the movable attachments shall be $15,000 \text{ g} \pm 0,025 \text{ g}$.

5.2.3 One-quarter-scale cone and shaft

Consisting of a conical body of plastics or other low density material with a hardened steel tip of 45 Rockwell C hardness to 50 Rockwell C hardness, and constructed to conform to the dimensions and tolerances shown in Figure 5. The shaft may be constructed of magnesium alloy. The total mass of the cone and its movable

attachments shall be $9,380 \text{ g} \pm 0,025 \text{ g}$. The total mass of the cone and its movable attachments may be adjusted by adding small shot to the cavity of the shaft.

5.3 Grease workers

5.3.1 Full-scale grease worker

Conforming to the dimensions shown in Figure 6. The sizes of non-dimensioned parts are not critical and may be varied according to individual requirements. Other methods of fastening the cover and securing the worker may be used. The grease worker may be constructed for either manual or mechanical operation. The design shall be such that a rate of 60 strokes per minute ± 10 strokes per minute, with a length of 67 mm to 71 mm, can be maintained. A suitable thermometer, standardized at $25 \text{ }^\circ\text{C}$, shall be provided for insertion through the vent valve.

5.3.2 One-half-scale grease worker

Conforming to the dimensions given in Figure 7. Other methods of fastening the cover and securing the worker may be used. The worker may be constructed for either manual or mechanical operation. The design shall be such that a rate of 60 strokes per minute ± 10 strokes per minute, with a minimum length of 35 mm, can be maintained.

5.3.3 One-quarter-scale grease worker

Conforming to the dimensions given in Figure 8. Other methods of fastening the cover and securing the worker may be used. The worker may be constructed for either manual or mechanical operation. The design shall be such that a rate of 60 strokes per minute ± 10 strokes per minute, with a minimum length of 14 mm, can be maintained.

5.3.4 Overflow-ring (optional)

Conforming in principle to the illustration shown in Figure 6. This is a useful aid for returning displaced grease to the grease worker cup. The overflow ring shall be positioned at least 13 mm below the rim of the cup while making a penetration measurement. A rim 13 mm high is helpful.

5.4 Grease cutter

Having a sharp, rigidly mounted, bevelled blade, essentially as shown in Figure 9. It is necessary that the blade be straight and sharpened as shown.

5.5 Water bath

Capable of being maintained at $25,0 \text{ }^\circ\text{C} \pm 0,5 \text{ }^\circ\text{C}$ and holding the assembled grease worker. If the bath is to be used for samples for unworked penetrations, means shall be provided for protecting the grease surface from water. A cover shall also be provided to maintain the air temperature above the sample at $25 \text{ }^\circ\text{C}$.

An air bath, maintained at $25,0 \text{ }^\circ\text{C} \pm 0,5 \text{ }^\circ\text{C}$, is required for determining block penetration; a tightly sealed container placed in the water bath will suffice.

NOTE A constant temperature test room or an air bath may be used instead of a water bath.

5.6 Thermometer

Calibrated at $25 \text{ }^\circ\text{C}$, for the water bath or air bath.

5.7 Oven

Capable of maintaining a temperature of $85\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$, for melting the petrolatum samples.

5.8 Spatula

Corrosion-resistant, square-ended, having a stiff blade approximately 32 mm wide and at least 150 mm long; for tests with half- and quarter-scale cones the width should be approximately 13 mm.

5.9 Timer

Graduated in 0,1 s.

5.10 Test portion containers

For petrolatum, cylindrical containers having a flat bottom $100\text{ mm} \pm 5\text{ mm}$ in diameter and 65 mm or more in depth, constructed of metal at least 1,6 mm thick and, if necessary, each provided with a well-fitting watertight cover (see note 1 to 9.1.3).

NOTE Containers of the "ointment box" type having somewhat flexible sides should not be used, for these permit slight working of the petrolatum, due to flexing of the sides in handling.

6 Sampling

Unless otherwise specified in a commodity specification, samples shall be drawn in accordance with ASTM D 4057.

The samples shall be examined for any sign of non-homogeneity such as oil separation, phase changes or gross contamination. If any abnormal conditions are found, a new sample shall be drawn.

The size of the sample shall be large enough to fill the requested number of cups.