INTERNATIONAL STANDARD

ISO 2137

Third edition
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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

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Foreword

SO (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.

Introduction

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. Unfortunately, most of the cones available do not conform to ISO 2137:1985 and users were obliged to establish waivers, which was difficult to justify to quality auditors. In the present edition of ISO 2137, 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.

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

WARNING — The use of this International Standard can 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. See 8.1 for the limitations on the use of one-quarter-scale cones due to the poor precision.

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 can 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.

ASTM D 4057, *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.

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 Adapted from ISO 1998-2:1998, 2.80.001.

3.2
working
subjecting a lubricating grease to the shearing action of a grease worker

3.3
unworked penetration
cone penetration of a test portion that 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.

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, capable of measuring, 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 is performed using either mechanical devices (mechanical indicator) or electronic devices (digital indicator).

5.2 Cones

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 g ± 0,05 g and that of its movable attachments shall be 47,50 g ± 0,05 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 as shown in Figure 3 can 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 37,50 g ± 0,05 g. The mass of the cone shall be 22,500 g ± 0,025 g. The mass of the movable attachments shall be 15,000 g ± 0,025 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 g ± 0,025 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 minimum length of 63 mm, can be maintained. A suitable thermometer, standardized at 25 °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 °C ± 0,5 °C and holding the assembled grease worker.

If the bath is to be used for samples for unworked penetrations, a 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 °C.

An air bath, maintained at 25,0 °C ± 0,5 °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 can be used instead of a water bath.

5.6 Thermometer, calibrated at 25 °C, for the water bath or air bath.

5.7 Oven, capable of maintaining a temperature of 85 °C ± 2 °C, for melting the petrolatum samples.

5.8 Spatula, corrosion-resistant, square-ended, with 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, having a flat bottom 100 mm ± 5 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 9.1.3).

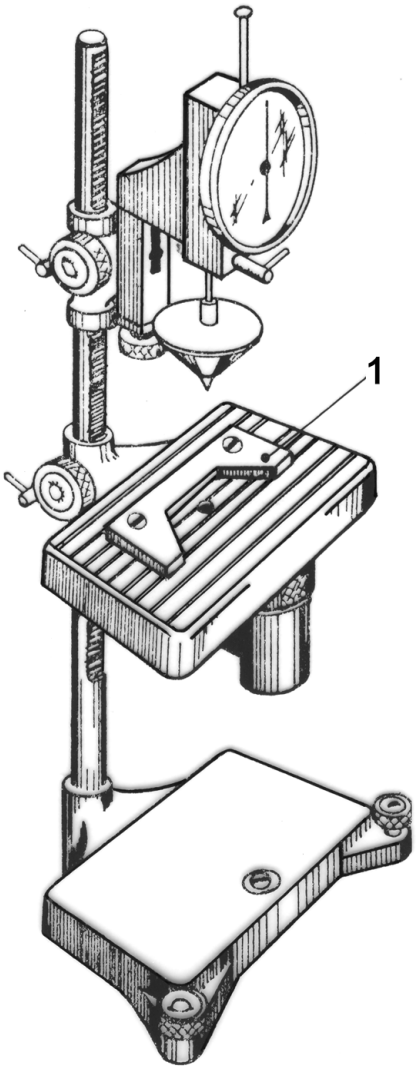
Containers of the “ointment box” type having somewhat flexible sides should not be used, for these permit slight working of the petrolatum, due to flexing of the sides in handling.

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.



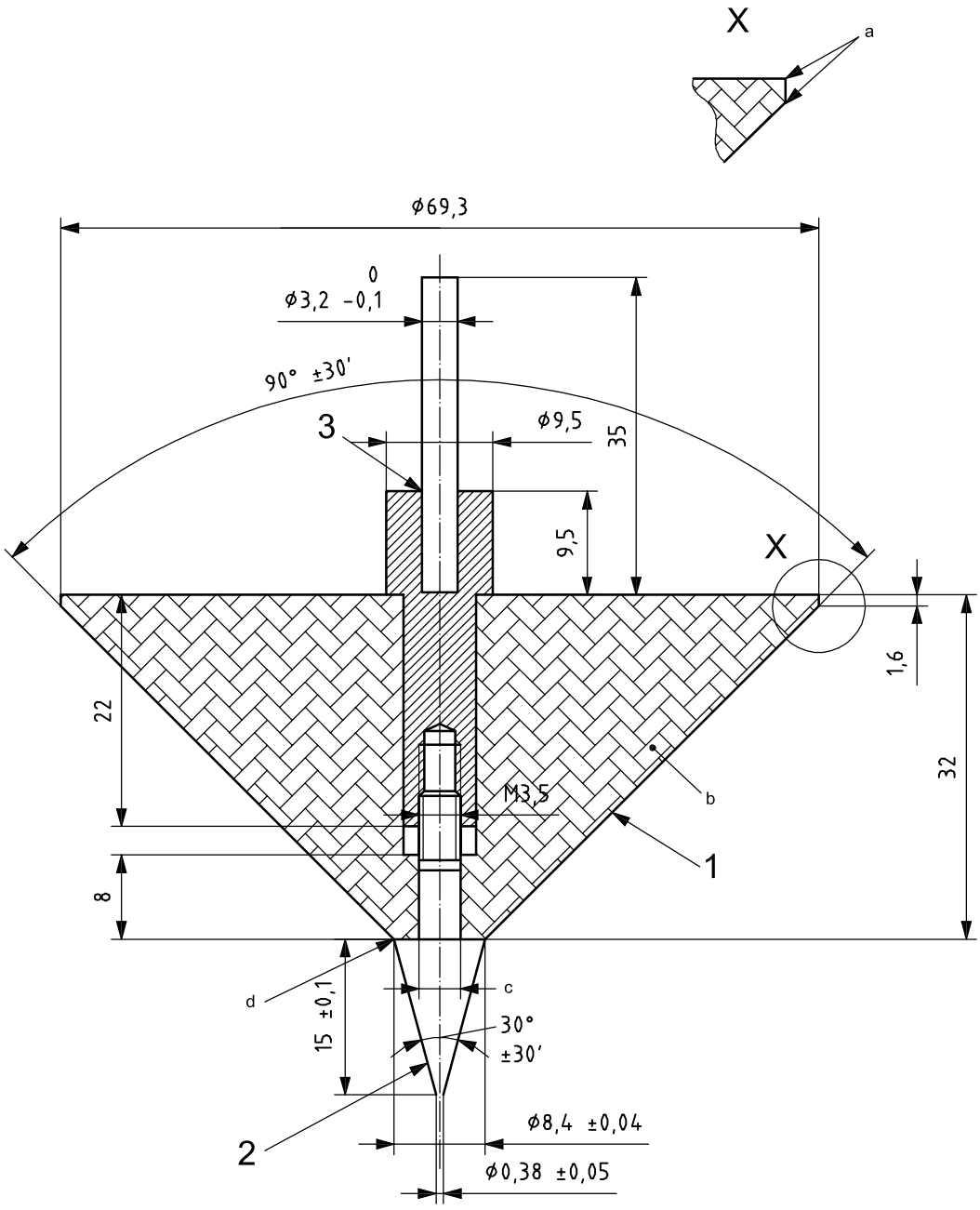
Key

1 centring device

NOTE This figure shows a combined assembly; generally, it is possible to displace vertically either the cone assembly or the plate.

Figure 1 — Penetrometer

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 1 mm



Key

1 smooth and polished surface
2 hardened steel tip
3 shaft stainless steel — tight press fit

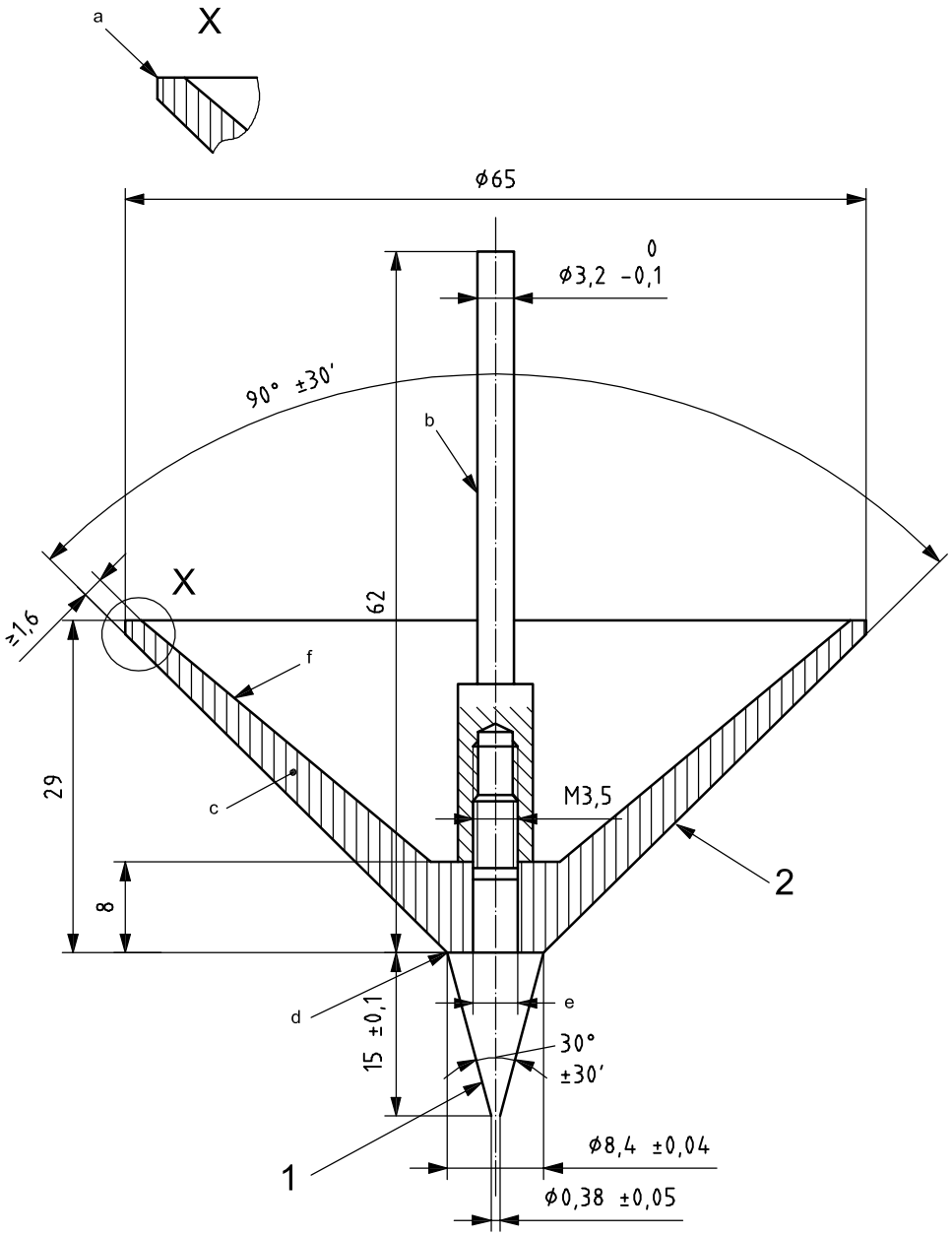
a Do not round the edges.
b Magnesium or any other suitable material.
c ∅ 4 max., tight fit.
d No shoulder.

Total mass of cone: 102,50 g ± 0,05 g

Total mass of movable attachments: 47,50 g ± 0,05 g

Figure 2 — Cone of the penetrometer — Full-scale cone

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 1 mm



Key

1 hardened steel tip
2 smooth and polished surface

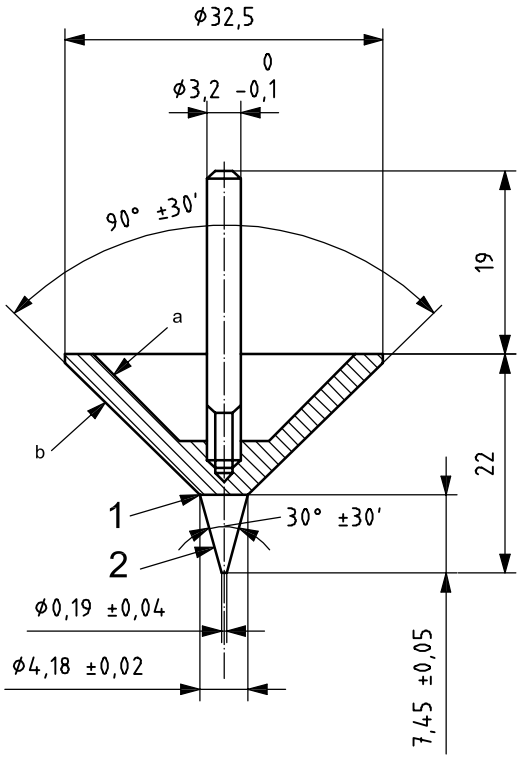
a Break all sharp edges.
b Stainless steel.
c Brass or corrosion resistant steel.
d No shoulder.
e ∅ 4 max., tight fit.
f Machining to the required mass.

Total mass of cone: 102,50 g ± 0,05 g

Total mass of movable attachments: 47,50 g ± 0,05 g

Figure 3 — Cone of the penetrometer — Optional cone

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 0,5 mm



Key

1 sharp corner
2 hardened steel tip

a Machine this surface to give specified mass.

b Polish all outside surfaces.

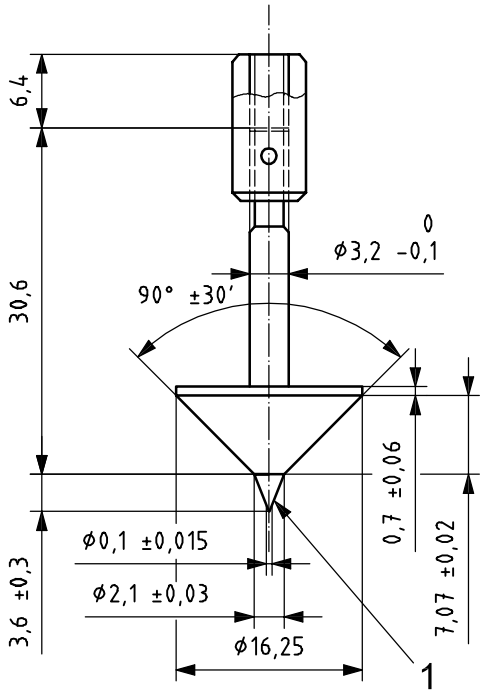
Mass of cone: 22,500 g ± 0,025 g

Mass of movable attachments: 15,000 g ± 0,025 g

Total mass of cone and its movable attachments: 37,50 g ± 0,05 g

Figure 4 — One-half-scale cone

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 0,25 mm



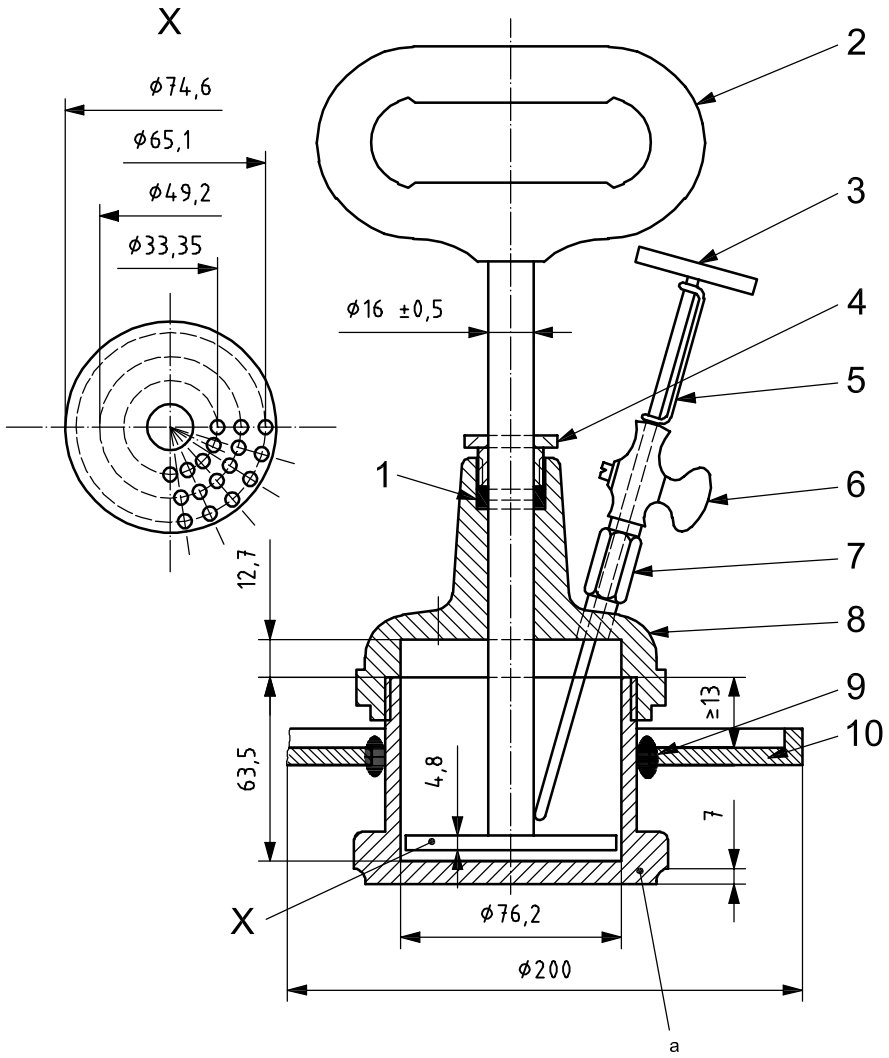
Key

1 hardened steel tip — Angle: 30° ± 30'

Total mass of cone and its movable attachments: 9,380 g ± 0,025 g

Figure 5 — One-quarter-scale cone

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 0,25 mm



Key

1	packing	6	vent valve
2	handle	7	adapter
3	thermometer	8	cover
4	packing nut	9	split rubber tube
5	thermometer gauge	10	overflow ring (optional)

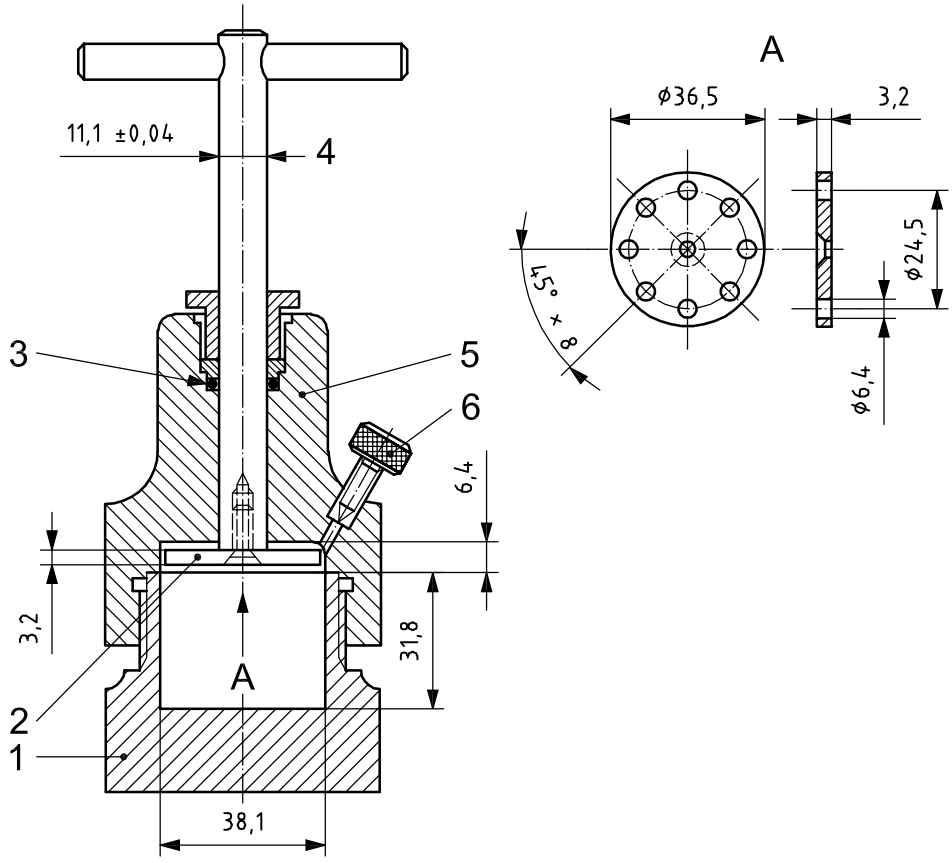
[a] optional, concentric to the interior of the cup, for use with the centring device.

Plan of perforated plate (detail X) — circle 1 (∅ 33,35): 12 holes circle 3 (∅ 65,10): 22 holes

circle 2 (∅ 49,20): 17 holes all holes: $6{,}4 < \varnothing < 6{,}53$

Figure 6 — Full-scale grease worker

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 0,25 mm



Key

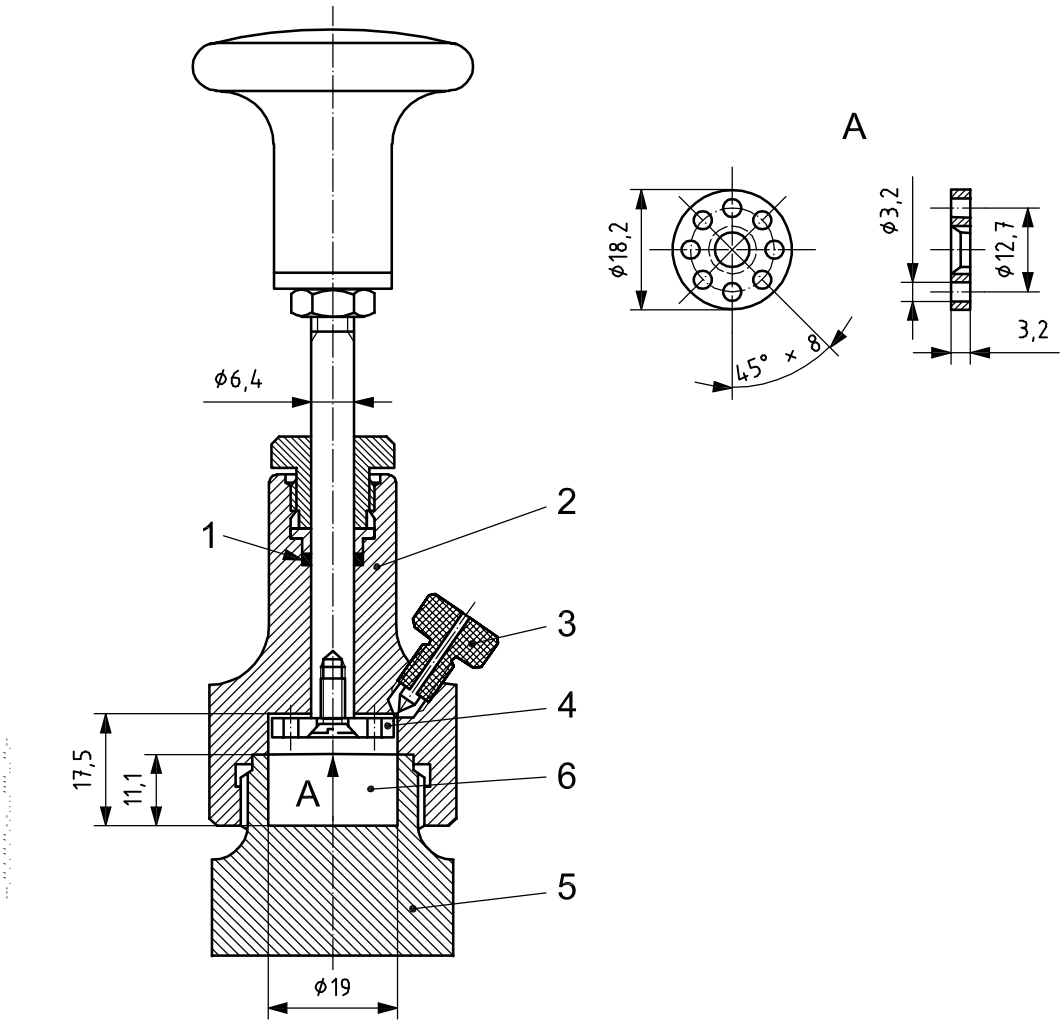
1 base
2 grease plate
3 ring
4 shaft
5 cover
6 valve

Material: stainless steel

Capacity: 50 ml

Figure 7 — One-half-scale grease worker

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 0,25 mm



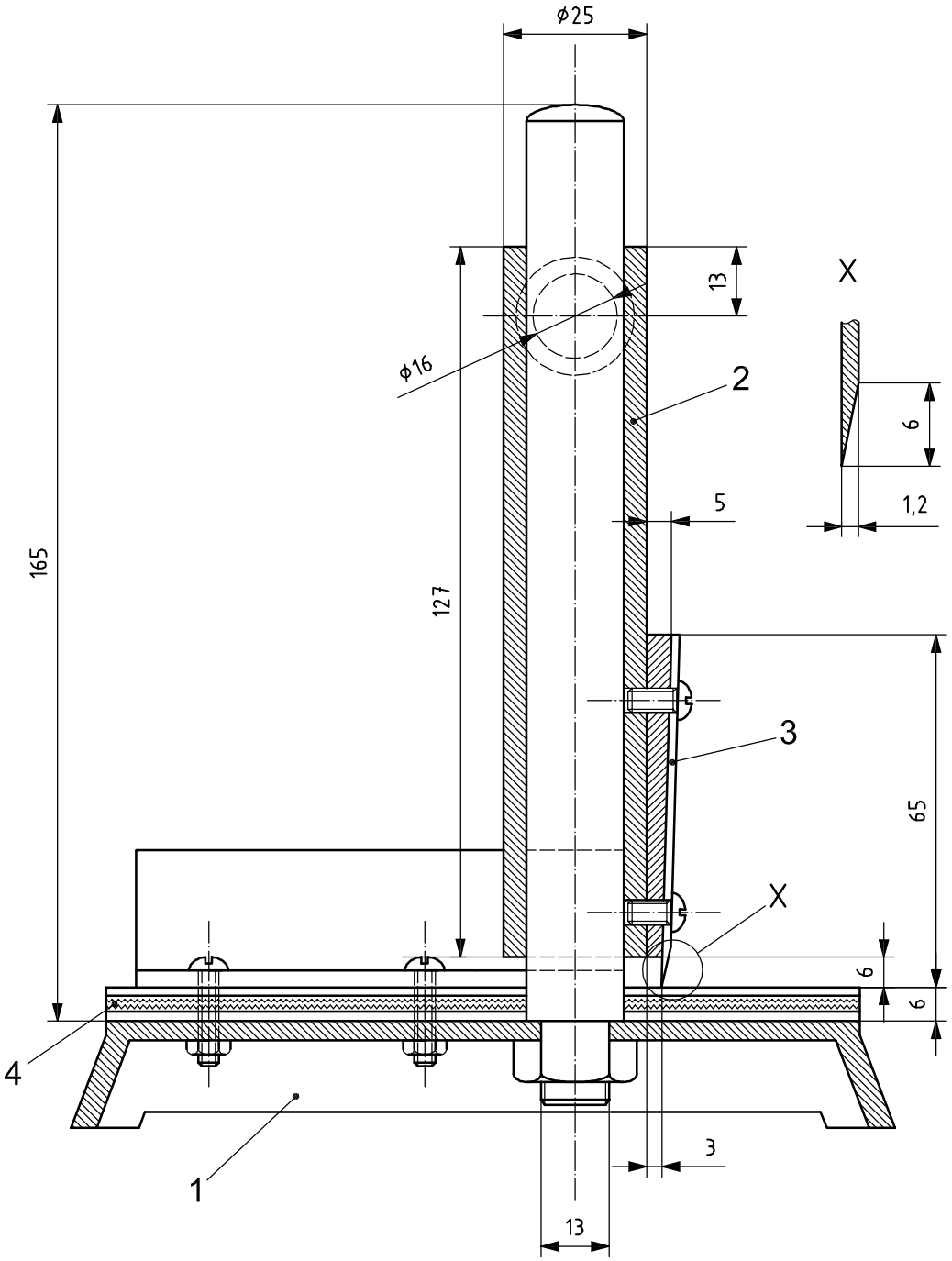
Key

1 ring
2 cover
3 valve
4 grease plate
5 base
6 chamber: ∅ 19,0 × 17,5 height — Capacity with the plate, as shown: 4 ml

Material: stainless steel

Figure 8 — One-quarter-scale grease worker

Dimensions in millimetres
Tolerances on dimensions except where otherwise stated: ± 0,25 mm



Key

1 base
2 tube
3 blade 65 × 185 — Hardened steel. Grind both surfaces to 1,2 mm thick. Grind lower edge
4 plywood or hard wood, 6,4 mm thick. Grain of top perpendicular to blade

Figure 9 — Grease cutter

7 Procedures for determining the cone penetration of lubricating grease — Full-scale methods

7.1 Procedure for unworked penetration

7.1.1 Preparation of test portion

7.1.1.1 Take sufficient sample (at least 0,5 kg) to overfill the cup of the grease worker (5.3.1). If the penetration is above 200 units, at least three times the amount needed to fill the cup is required.

7.1.1.2 Place the empty assembled grease worker, or metal container of equal inside dimensions, and an appropriate amount of the test sample in a metal container in the water bath (5.5) maintained at 25 °C (see the note to 5.5, and the last two paragraphs of this subclause) for sufficient time to bring the temperature of the sample to 25 °C ± 0,5 °C. Transfer from the container a test portion, preferably in one lump, to overfill the cup of the grease worker or metal container of equal inside dimensions (see below). Make this transfer in such a manner that the grease is worked as little as possible. Jar the container to drive out trapped air and pack the grease with the spatula (5.8), with as little manipulation as possible, to obtain a cupful without air pockets. Scrape off the excess grease extending above the rim, creating a flat surface, by moving the blade of the spatula, held inclined toward the direction of motion at an angle of approximately 45°, across the rim of the cup. Do not perform any further levelling or smoothing of the surface throughout the determination of unworked penetration, and carry out the measurement immediately.

The penetration of soft greases is dependent upon the diameter of the container. Therefore, greases having unworked penetrations above 265 units should be tested in containers having the same diameter limitations as those of the grease worker cup. The results on greases having penetrations below 265 units are not significantly affected if the diameter of the container exceeds that of the grease worker cup.

If the initial sample temperature differs from 25 °C by more than approximately 8 °C, or if an alternative method of adjusting the sample to 25 °C is used, sufficient additional time should be allowed to ensure that the test portion is at 25,0 °C ± 0,5 °C before proceeding. Also, if the sample is larger than 0,5 kg, sufficient additional time should be allowed to ensure that the test sample is at 25,0 °C ± 0,5 °C. Testing may proceed if the test portion is at a uniform temperature of 25,0 °C ± 0,5 °C.

7.1.2 Cleaning the cone and movable attachments

Clean the penetrometer cone carefully before each test. Bending of the cone shaft can be avoided by holding it securely in its raised position whilst cleaning. Remove all grease or oil on the penetrometer shaft, as they can cause drag on the shaft assembly. Do not rotate the cone, as this can cause wear on the release mechanism.

NOTE For penetrations up to 400 units, the optional cone as shown in Figure 3 can be used.

7.1.3 Penetration measurement

7.1.3.1 Place the cup on the penetrometer table, which shall be adjusted to a perfectly horizontal position, making certain that it cannot rock. Set the mechanism to hold the cone in the “zero” position, and adjust the apparatus carefully so that the tip of the cone just touches the surface of the test portion at the point specified in 7.1.3.2 or 7.1.3.3. Watching the shadow of the cone tip is an aid to accurate setting. For greases with penetrations over 400 units, centre the cup to within 0,3 mm of the tip of the cone. One way to centre the cup accurately is to use a centring device (see Figure 1). Release the cone shaft rapidly, allow it to drop for 5,0 s ± 0,1 s and re-clamp it in this position. The release mechanism shall not drag on the shaft. Gently depress the indicator shaft until it is stopped by the cone shaft and read the penetration from the indicator scale.

7.1.3.2 If the test portion has a penetration over 200 units, centre the cone carefully in the container. This test portion shall be used for only one test.

7.1.3.3 If the test portion has a penetration of 200 units or less, perform three tests in a single container, spacing the penetrations on three radii approximately 120° apart, and approximately midway between the centre and the side of the container, so that the cone neither strikes the side of the container nor impinges on the disturbed area made in a previous test.

7.1.3.4 Make a total of three determinations on the test portion [either in three containers (see 7.1.3.2) or in one container (see 7.1.3.3)] and record the values obtained.

7.2 Procedure for worked penetration

7.2.1 Preparation of test sample

7.2.1.1 Sample

Take sufficient sample (at least 0,5 kg) to overfill the cup of the grease worker (5.3.1).

7.2.1.2 Working

Transfer a sufficient quantity of the laboratory sample to the cup of the clean grease worker (5.3.1) to overfill it (mounded up approximately 13 mm at the centre), avoiding the inclusion of air by packing with the spatula (5.8). Jar the cup from time to time as it is being packed to remove any trapped air.

Assemble the grease worker with the plunger raised and, with the vent valve open, depress the plunger to the bottom. Insert a thermometer (5.6) through the vent valve so that its tip is in the centre of the grease. Place the assembled grease worker in the water bath maintained at 25 °C (see the note to 5.5, the second and third paragraphs of 7.1.1.2) until the temperature of the grease worker and contents is 25,0 °C ± 0,5 °C as indicated by the thermometer. Then remove the grease worker from the bath and wipe off excess water adhering to its surfaces. Remove the thermometer and close the vent cock. Subject the grease to 60 full (63 mm to 71,5 mm) double strokes of the plunger, completed in approximately 1 min, and return the plunger to its top position. Open the vent valve, remove the top and plunger, and return to the cup as much of the grease clinging to the plunger as can readily be removed. As the worked penetration of a lubricating grease can change significantly on standing, proceed in accordance with 7.2.2 and 7.2.3 without delay.

If it is desired to immerse the part of the grease worker above its closure, care should be taken that the lid is watertight in order to prevent the entrance of water to the grease worker.

7.2.2 Preparation of test portion

7.2.2.1 Prepare the worked test sample (7.2.1.2) in the cup for testing so that a uniform and reproducible structure of grease is obtained.

7.2.2.2 Jar the cup sharply on the bench or floor and pack the grease down with the spatula (5.8) to fill the holes left by the plunger and to remove any air pockets.

The jarring should be as vigorous as is required to remove the entrapped air without splashing the sample from the cup. In performing these operations, a minimum of manipulation shall be used, as continued agitation of the grease can have the effect of increasing the working beyond the specified 60 strokes.

7.2.2.3 Scrape off the excess grease extending above the rim of the cup, creating a flat surface, by moving the blade of the spatula, held inclined toward the direction of motion at an angle of approximately 45°, across the rim of the cup. Retain the portion of grease that is removed.

7.2.2.4 Particularly when testing soft greases, retain the grease removed when scraping the cup to provide a full cup for subsequent tests. Keep the outside of the rim of the cup clean so that the grease forced by the penetrometer cone to overflow the cup can be returned to the cup prior to preparing the sample for the next test.

7.2.3 Penetration measurement

7.2.3.1 Determine the penetration of the sample as described in 7.1.2 and 7.1.3.

7.2.3.2 Immediately make two more determinations in succession on the same test portion. First, return to the cup the portion of grease previously removed with the spatula in 7.2.2.3. Then repeat the operation specified in 7.2.2 through 7.2.3.1. Record the three values obtained.

7.3 Procedure for prolonged worked penetration

7.3.1 Preparation of apparatus and test portion

7.3.1.1 Temperature

Maintain the temperature of the room used for the test within the range of 15 °C to 30 °C. No further control of the grease worker temperature is necessary, but, before starting the test, the grease shall have been in the room for sufficient time to bring its temperature within the range of 15 °C to 30 °C.

7.3.1.2 Working

Fill a clean grease worker cup and assemble the worker as described in 7.2.1.2. Subject the grease sample to the prescribed or agreed number of double strokes.

In order to minimize leakage during working, special attention should be paid to the gland in the grease worker cover.

7.3.2 Penetration measurement

Immediately after the working has been completed, place the grease worker in a constant-temperature water bath or air bath (5.5) to bring the test portion temperature to 25,0 °C ± 0,5 °C within 1,5 h. Remove the worker from the bath and subject the grease to a further 60 (63 mm to 71,5 mm) double strokes. Prepare and penetrate the test portions as described in 7.2.2 and 7.2.3.

7.4 Procedure for block penetration

7.4.1 Preparation of test portion

7.4.1.1 Take sufficient sample of the grease, which shall be hard enough to hold its shape to permit cutting from it a test sample comprised of a cube with sides approximately 50 mm in length.

7.4.1.2 By means of the grease cutter (5.4), cut as a test sample from the laboratory sample at room temperature, a cube with sides approximately 50 mm in length. While holding this test sample so that the unbevelled edge of the cutter is toward it, slice off a layer approximately 1,5 mm in thickness from each of the three faces adjacent to a single corner, which may be truncated for identification. Take care not to touch those portions of the newly exposed faces that are used for testing or to set a prepared face against the base plate or guide of the cutter. Bring the temperature of the finished test portion to 25,0 °C ± 0,5 °C by placing it in a constant-temperature air bath maintained at 25 °C for at least 1 h (see the last paragraph of 7.1.1.2).

7.4.2 Penetration measurement

Place the test portion on the table of the penetrometer, which shall be adjusted to a perfectly horizontal position, with one of the prepared faces upward, and press it down by the corners to make it rest level and firmly on the table so that it cannot rock during the test. Set the mechanism to hold the cone in the “zero” position and adjust the apparatus carefully so that the tip of the cone just touches the surface at the centre of the test portion. Determine the penetration as described in 7.1.2 and 7.1.3. Carry out a total of three tests on the exposed face of the test portion, locating the tests at least 6 mm from the edge and as far apart as possible without impinging on any touched portion, air hole or other apparent flaw in the surface. If the result

of any of these tests differs from the others by more than three units, carry out additional tests until three values agreeing within three units are obtained. Average these three values for the face being tested.

7.4.3 Additional determinations

In order to equalize, in the final value, the effect of the fibre orientation in testing fibrous grease, repeat the procedure described in 7.4.2 on each of the other prepared faces of the test portion and record the average values obtained.

Smooth-textured, non-fibrous greases may be tested on one face only, when agreed upon between the interested parties.

8 Procedures for determining the cone penetration of lubricating grease — Half- and quarter-scale cone method

8.1 General

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.

8.2 Procedure for unworked penetration

8.2.1 Preparation of test portion

Take sufficient sample to overfill the cup of the grease worker (5.3.2 or 5.3.3). If the penetration by the quarter-scale cone is greater than 47 units or by the half-scale cone greater than 97 units, at least three times the amount needed to fill the cup shall be taken, as only one test shall be made in one grease cup.

Proceed as described in 7.1.1.2.

8.2.2 Cleaning cone and movable attachments

Clean the penetrometer cone (5.2.2 or 5.2.3) carefully before each test. Bending of the cone shaft can be avoided by holding it securely in its raised position while cleaning. Remove all grease or oil from the penetrometer shaft, as they can cause drag on the shaft assembly. Do not rotate the cone, as this can cause wear of the release mechanism.

8.2.3 Penetration measurement

8.2.3.1 Carry out a preliminary determination of the penetration as specified below with the cone at the centre of the test portion surface; if the approximate value of the penetration is already known, this step may be omitted.

8.2.3.2 If the test portion has a penetration over 47 units by the quarter-scale cone (5.2.3) or over 97 units by the half-scale cone (5.2.2), centre the cone carefully in the container; this test portion can then be used for only one test.

8.2.3.3 If the test portion has a penetration of 47 units or less by the quarter-scale cone or 97 units or less by the half-scale cone, perform three tests in a single container, spacing these tests on three radii approximately 120° apart, and approximately midway between the centre and side of the container so that the cone neither strikes the side of the container nor impinges on the disturbed area made in a previous test.

8.2.3.4 Proceed as described in 7.1.3.1 and 7.1.3.4.

8.3 Procedure for worked penetration

8.3.1 Preparation of test sample

8.3.1.1 Sample

Take sufficient sample to overfill the cup of the appropriate grease worker (5.3.2 or 5.3.3).

8.3.1.2 Working

Proceed in accordance with 7.2.1.2, but mounding up to approximately 6 mm and without using a thermometer in the grease worker.

8.3.2 Preparation of test portion

Proceed in accordance with 7.2.2.

8.3.3 Penetration measurement

8.3.3.1 Immediately determine the penetration of the test portion as specified in 8.2.2 and in 8.2.3.1 to 8.2.3.3.

8.3.3.2 Proceed as specified in 7.1.3.1. Immediately carry out two more determinations in succession on the same test portion. First return to the cup the portion of grease previously removed with the spatula as described in 7.2.2.3. Then repeat the operations specified in 7.2.2, 8.2.2, 8.2.3.1 to 8.2.3.3 and 7.1.3.1. Record the three values obtained.

9 Procedure for determining the cone penetration of petrolatum

9.1 Preparation of the test portion

9.1.1 Take a laboratory sample of approximately 1 kg for petrolatum having a penetration over 200 units, and of approximately 700 g for petrolatum having penetration equal to or less than 200 units.

9.1.2 If the penetration of the petrolatum is over 200 units, prepare three separate test portions. If the penetration is equal to or less than 200 units, prepare one test portion as specified in 9.1.3.

9.1.3 Melt the test sample in the oven (5.7) maintained at 85 °C ± 2 °C. Place the required number of test portion containers (5.10) in the oven along with the test sample to bring them up to 85 °C. When the test sample has melted and comes to within 3 °C of that temperature, remove the test sample and the heated test portion containers and fill the required number of containers to within 6 mm of their rims. Allow the filled containers to cool for 16 h to 18 h in a location free from draughts and at a temperature controlled to 25 °C ± 2 °C. Then place the filled test portion containers in the water bath (5.5) for 2 h to bring the temperature to 25,0 °C ± 0,5 °C before testing. Do not cut level the surface of the test portions or work them in any other way. Remove the filled test portion container from the water bath and with minimum delay proceed with the determination.

Some compounded petrolatums are affected by contact with water. The test portion of such a petrolatum should be covered with a sealed lid as required for greases (see Clause 7). Uncompounded petrolatum is not affected by water and it is not necessary that it be covered.

Some petrolatums containing higher-melting-point waxes can require higher pouring temperatures; in that case, the precision data given in Table 3 might not apply to the results.

If the room temperature varies from 25 °C by 2 °C or more, adjust the cone temperature to 25,0°C ± 0,5 °C immediately before testing the test portion by immersing it in the water bath (5.5) and subsequently drying it

with a lint-free cloth or tissue. Frequent adjustment of the cone temperature can be necessary if the room temperature varies appreciably from 25 °C.

9.2 Penetration measurement

Proceed in accordance with 7.1.2 and 7.1.3.

Some harder petrolatums tend to form a marked depression in the centre on solidifying. Such test portions should not be tested in this depression, as the values obtained can be different from those obtained in off-centre positions on the level surface.

10 Calculation

10.1 Calculation of full-scale penetration

Calculate the average of the values recorded in the determination.

10.2 Conversion to full-scale penetration for the half- and quarter-scale cones

10.2.1 General

When required, the penetration values obtained from the quarter-scale and half-scale cone equipment may be converted to full-scale penetrations determined in accordance with Clause 7 using the equations given in 10.2.2 or 10.2.3.

10.2.2 Quarter-scale cone

The cone penetration, P, as determined using full-scale equipment, can be approximated as given in Equation (1):

$$P = 3{,}75p + 24 \tag{1}$$

where p is the cone penetration determined using quarter-scale equipment.

10.2.3 Half-scale cone

The cone penetration, P, as determined using full-scale equipment, can be approximated as given in Equation (2):

$$P = 2p + 5 \tag{2}$$

where p is the cone penetration determined using half-scale equipment.

11 Expression of results

Round the result to the nearest unit (0,1 mm).

12 Precision

12.1 General

The precision, as determined by statistical examination in accordance with ISO 4259 of interlaboratory test results, is given in 12.2 and 12.3.

12.2 Repeatability

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 values given in Tables 1 to 3 in only one case in twenty.

12.3 Reproducibility

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 values given in Tables 1 to 3 in only one case in twenty.

Table 1 — Precision — Lubricating grease — Full scale

Dimensions in units of 0,1 millimetres

Grease	Penetration range	Repeatability	Reproducibility
Unworked	85 to 475	8	19
Worked	130 to 475	7	20
Prolonged working	130 to 475	15 [a]	27 [a]
Block grease	Below 85	7	11
[a] Determined for 100 000 cycles and for an ambient temperature between 15 °C and 30 °C.			

Table 2 — Precision – Lubricating grease — Half scale and quarter scale

Dimensions in units of 0,1 millimetres

Penetration	Scale	Repeatability	Reproducibility
Unworked	1/2	5 (10)	13 (26)
Worked	1/2	3 (6)	10 (20)
Unworked	1/4	3 (11)	10 (38)
Worked	1/4	3 (11)	7 (26)
NOTE The values given in parentheses represent the repeatability and reproducibility resulting from the conversion to full-scale penetration.			

Table 3 — Precision – Petrolatum

Repeatability	$2 + 0{,}05P$
Reproducibility	$9 + 0{,}12P$
NOTE P is the penetration in 0,1 mm units.	

13 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard, the procedure used (as specified in Clause 7, 8 or 9), the method used (unworked, worked, prolonged worked, or block) and the cone used (full-scale, optional, half-scale or quarter-scale);

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

c) the results of the test (see Clause 11);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) date of the test.

Bibliography

[1] ISO 1998-2:1998, *Petroleum industry — Terminology — Part 2: Properties and tests*

[2] ISO 4259:2006, *Petroleum products — Determination and application of precision data in relation to methods of test*

[3] ISO 6743-99:2002, *Lubricants, industrial oils and related products (class L) — Classification — Part 99: General*

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