International Standard 2137

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Petroleum products — Lubricating grease and petrolatum — Determination of cone penetration

Produits pétroliers — Graisse lubrifiante et pétrolatum — Détermination de la pénétrabilité au cône

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

International Standard ISO 2137 was first published in 1972. This second edition cancels and replaces the first edition, of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Petroleum products — Lubricating grease and petrolatum — Determination of cone penetration

0 Introduction

This International Standard describes several methods for the empirical estimation of the consistency of lubricating greases and petrolatum by measuring the penetration of a standard cone.

1 Scope and field of application

1.1 Section one of this International Standard specifies four procedures for determination of the consistency of lubricating greases by measurement of the penetration of a standard cone. These procedures cover the measurement of unworked, worked, prolonged worked, and block penetrations. Penetrations up to 620 units may be measured.

1.2 Section two of this International Standard specifies methods for the determination of the consistency of lubricating greases, when only small samples are available, by the use of cones a half or quarter scale of that used in Section one.

The methods are applicable to greases having penetrations of 175 to 385 units with the standard cone and are intended for use only if the size of the test sample prevents the use of Section one.

They are not intended to replace the full-scale penetration as described in Section one, although a conversion to full-scale penetrations is given in 13.2.

NOTES

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.

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

1.3 Section three of this International Standard specifies a method for the determination of the consistency of petrolatums by measurement of the penetration of a standard cone, having penetrations up to 300 units.

This method may also be used to estimate the consistency of slack waxes.

1.4 Section four of this International Standard describes the calculation of results, precision data and the test report.

2 Definitions

2.1 cone penetration: The distance that a standard cone penetrates into a test portion under standardized conditions of load, time, and temperature.

NOTE — It is expressed in units of 0,1 mm.

2.2 working: The subjection of a lubricating grease to the shearing action of a grease worker.

2.3 unworked penetration: The 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.

2.4 worked penetration: The cone penetration of a test portion after it has been subjected to a defined number of strokes in a grease worker.

2.5 prolonged worked penetration: The cone penetration of a test portion which has been worked more than the defined number of strokes in 2.4.

2.6 block penetration: The cone penetration determined on a test portion which is sufficiently hard to hold its shape without container.

3 Principles

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 prescribed conditions, and then measuring the penetration as for lubricating grease.

4 Apparatus

4.1 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 levelling screws and a spirit level to maintain the cone shaft in a vertical position.

4.2 Cones

4.2.1 Standard 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 102,5 ± 0,05 g and that of its movable attachments shall be 47,5 ± 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 (figure 3) may be used.

4.2.2 One-half scale cone and shaft, of steel, stainless steel, or brass with a hardened steel tip of 45 to 50 Rockwell hardness C 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 37,5 ± 0,050 g. The mass of the cone shall be 22,5 ± 0,025 g. The mass of the movable attachments shall be 15 ± 0,025 g.

4.2.3 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 to 50 Rockwell hardness C, 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 9,38 ± 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.

4.3 Grease workers

4.3.1 Grease worker, full-scale, 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 ± 10 strokes per minute, with a length of 67 to 71 mm, can be maintained. A suitable thermometer, standardized at 25 °C, shall be provided for insertion through the vent valve.

4.3.2 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 ± 10 strokes per minute, with a maximum length of 35 mm, can be maintained.

4.3.3 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 ± 10 strokes per minute, with a maximum length of 14 mm, can be maintained.

4.3.4 Overflow ring (optional), conforming in principle to the illustration 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.

4.4 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.

4.5 Water bath, capable of being maintained at 25 ± 0,5 °C and capable of 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 ± 0,5 °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 may be used instead of a water bath.

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

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

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

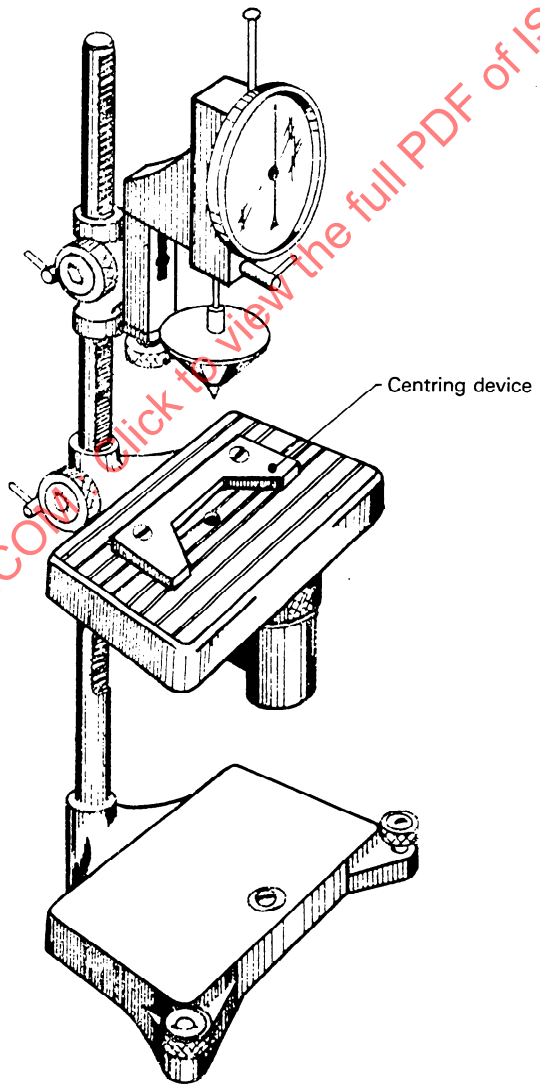
4.9 Timer, graduated in 0,1 s.

4.10 Test portion containers for petrolatum, cylindrical, having a flat bottom 100 ± 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 the note 1 to 12.1.3).

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.

5 Sampling

Take a representative sample of the product to be tested.



NOTE — This is a combined figure; generally either the cone assembly or the table can be moved vertically.

Figure 1 — Penetrometer

Dimensions in millimetres

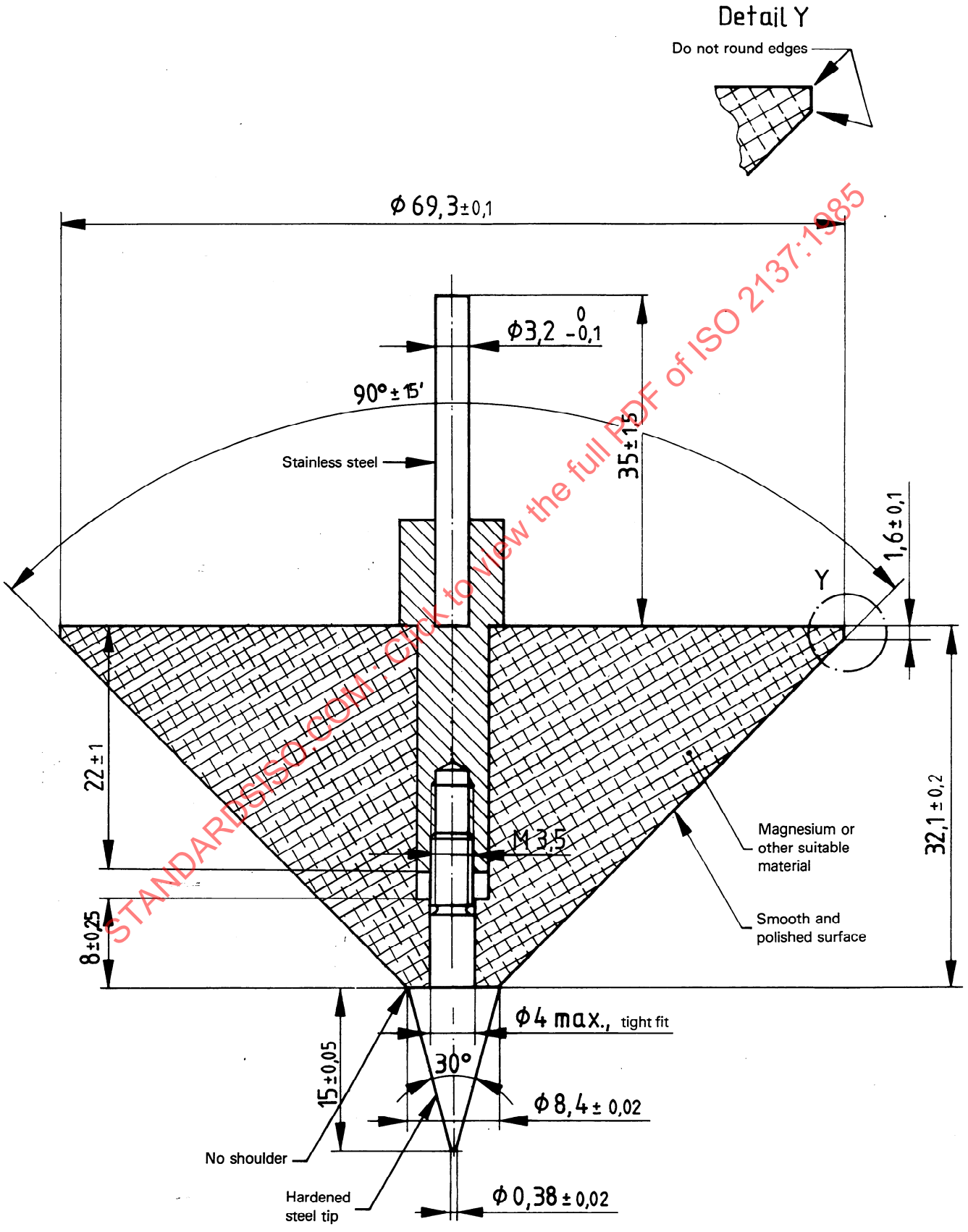
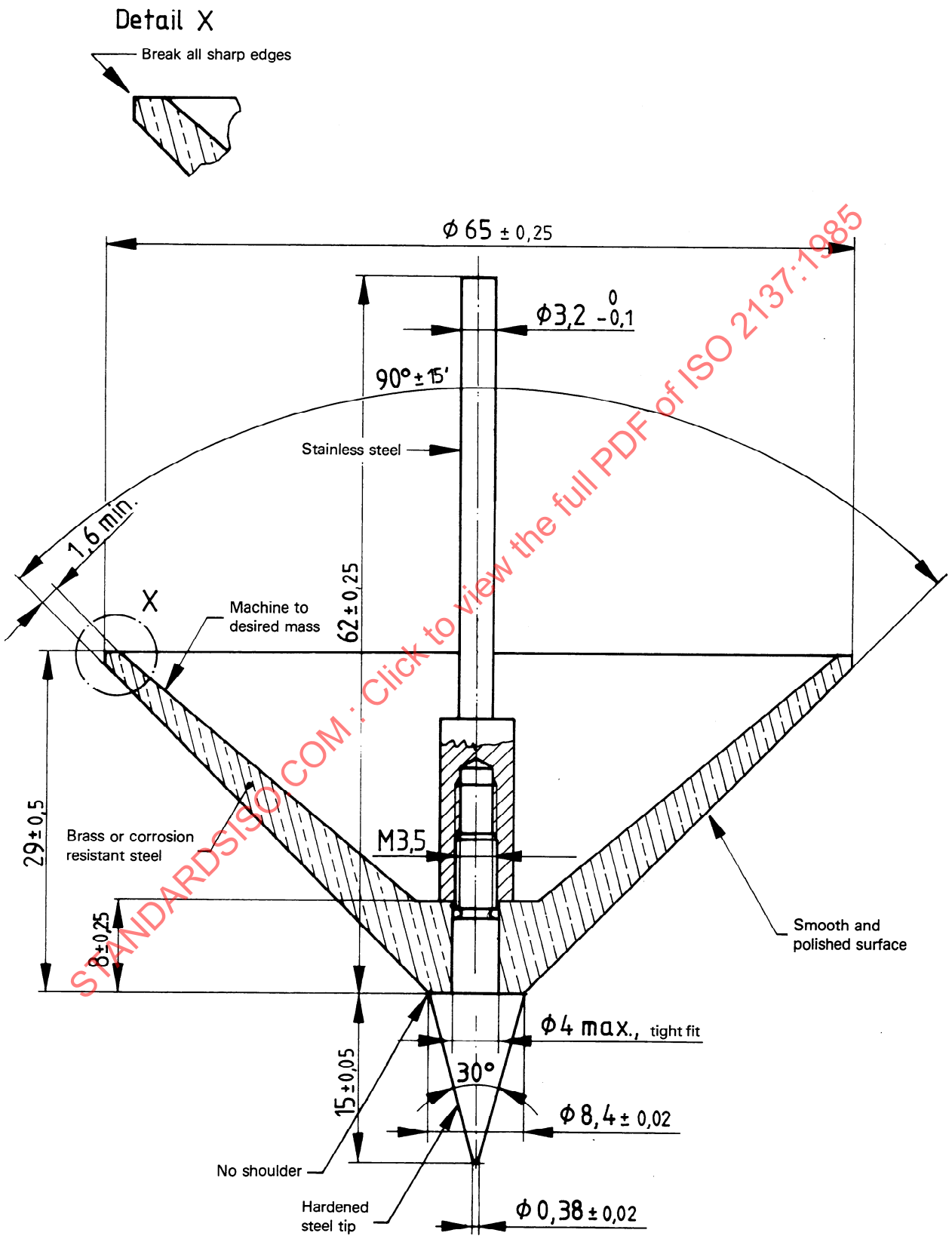


Figure 2 — Penetrometer cone, standard

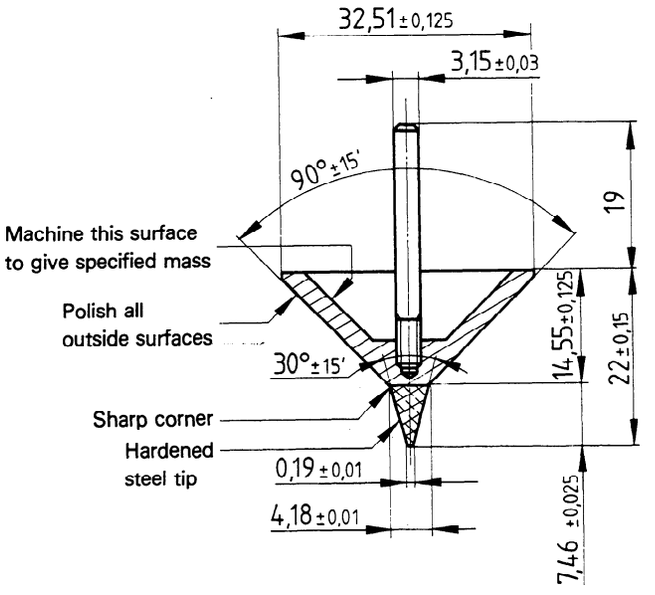
Dimensions in millimetres



Total mass of cone: 102,5 ± 0,05 g; total mass of movable attachments: 47,5 ± 0,05 g

Figure 3 — Penetrometer cone, optional

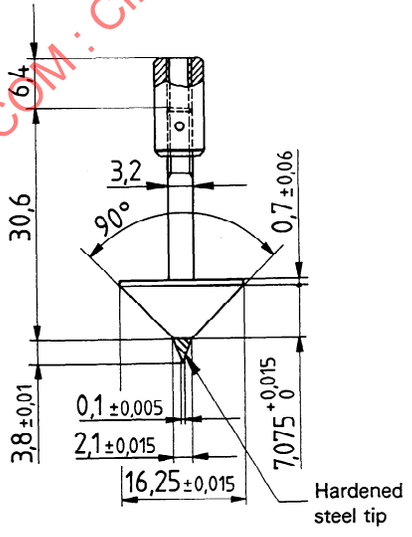
Dimensions in millimetres



Mass of cone: 22,5 ± 0,025 g
Mass of movable attachments: 15 ± 0,025 g
Total mass of cone and its movable attachments: 37,5 ± 0,050 g

Figure 4 — One-half scale cone

Dimensions in millimetres



Total mass of cone and movable attachments:
9,38 ± 0,025 g

Figure 5 — One-quarter scale cone

Dimensions in millimetres

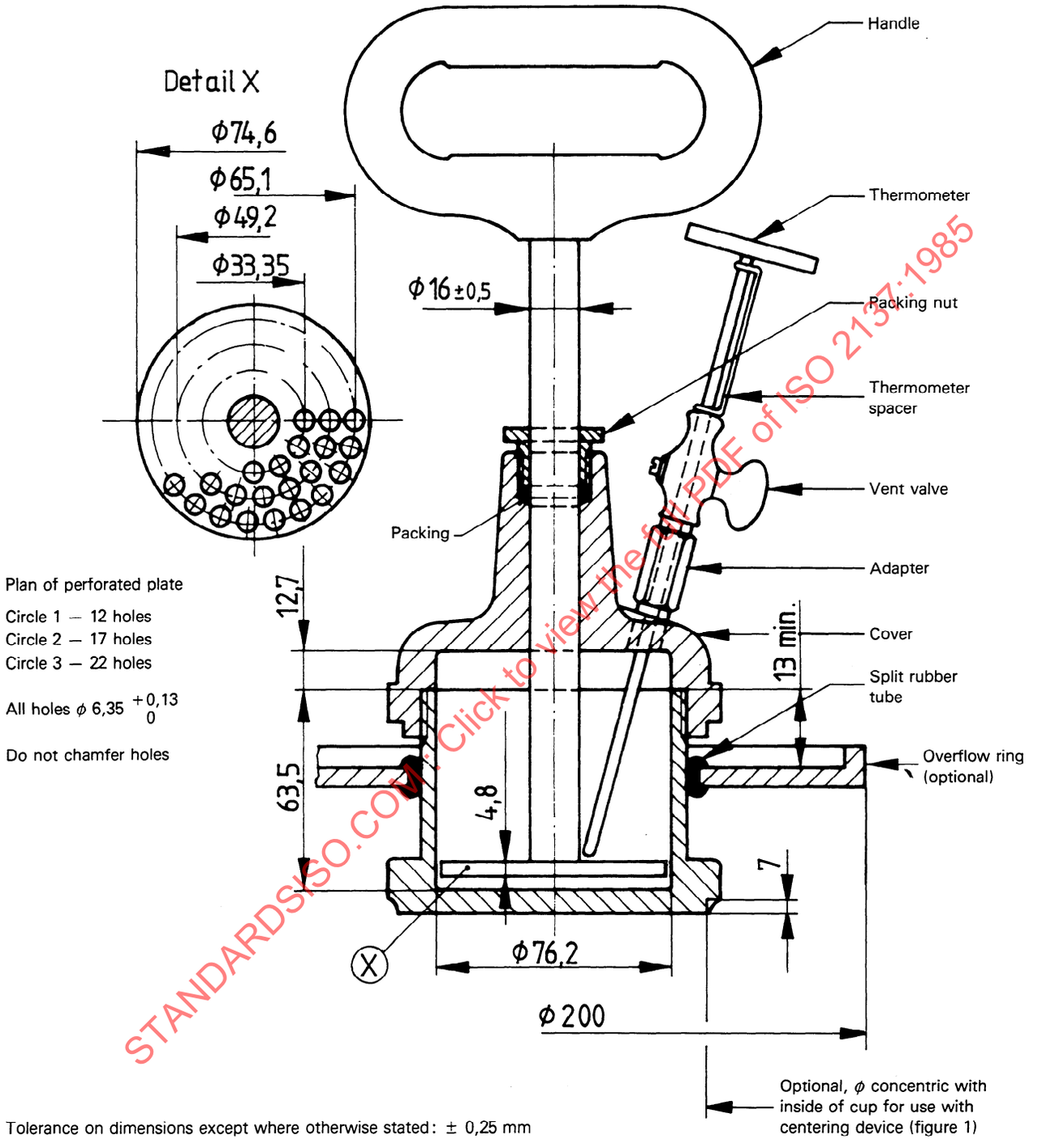
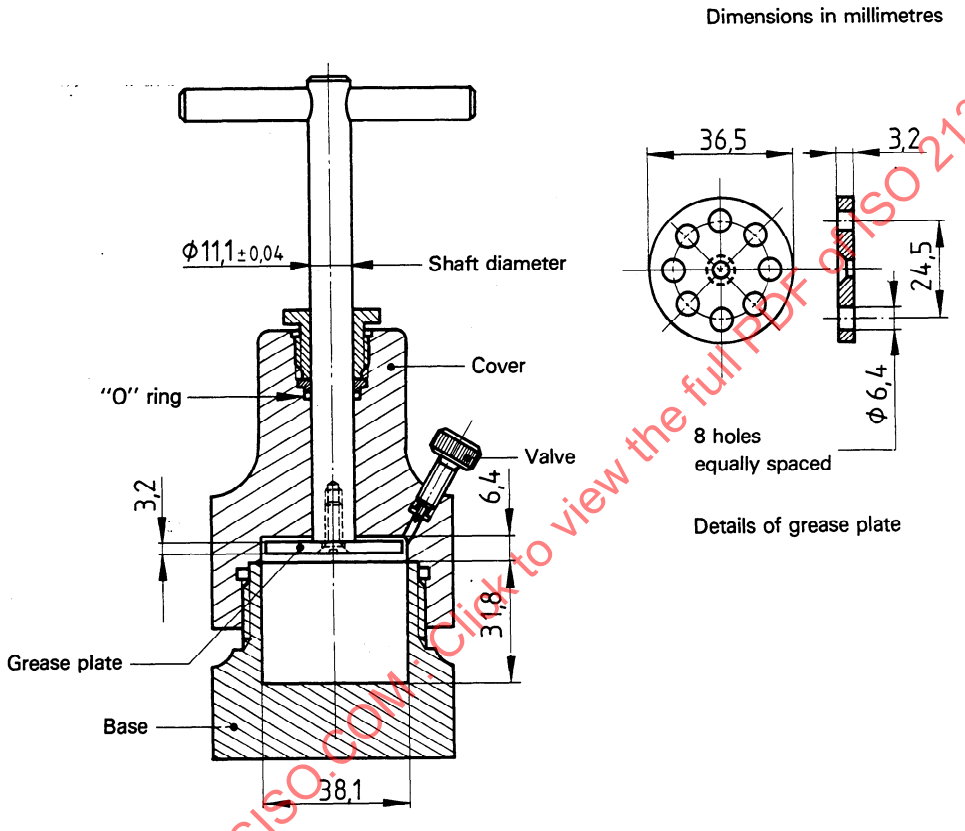
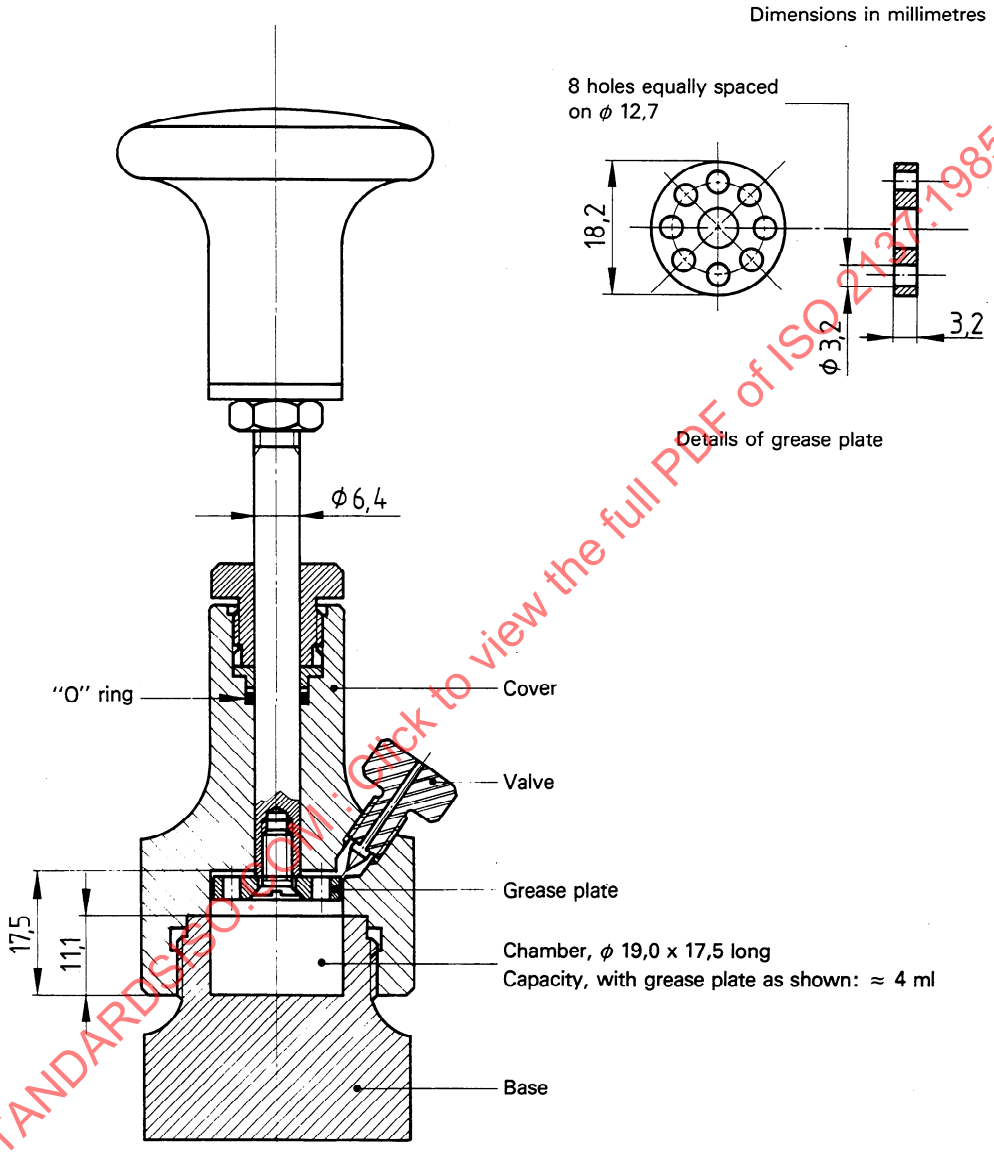


Figure 6 — Grease worker, full-scale



Tolerance on dimensions, except where otherwise stated : ± 0,25 mm

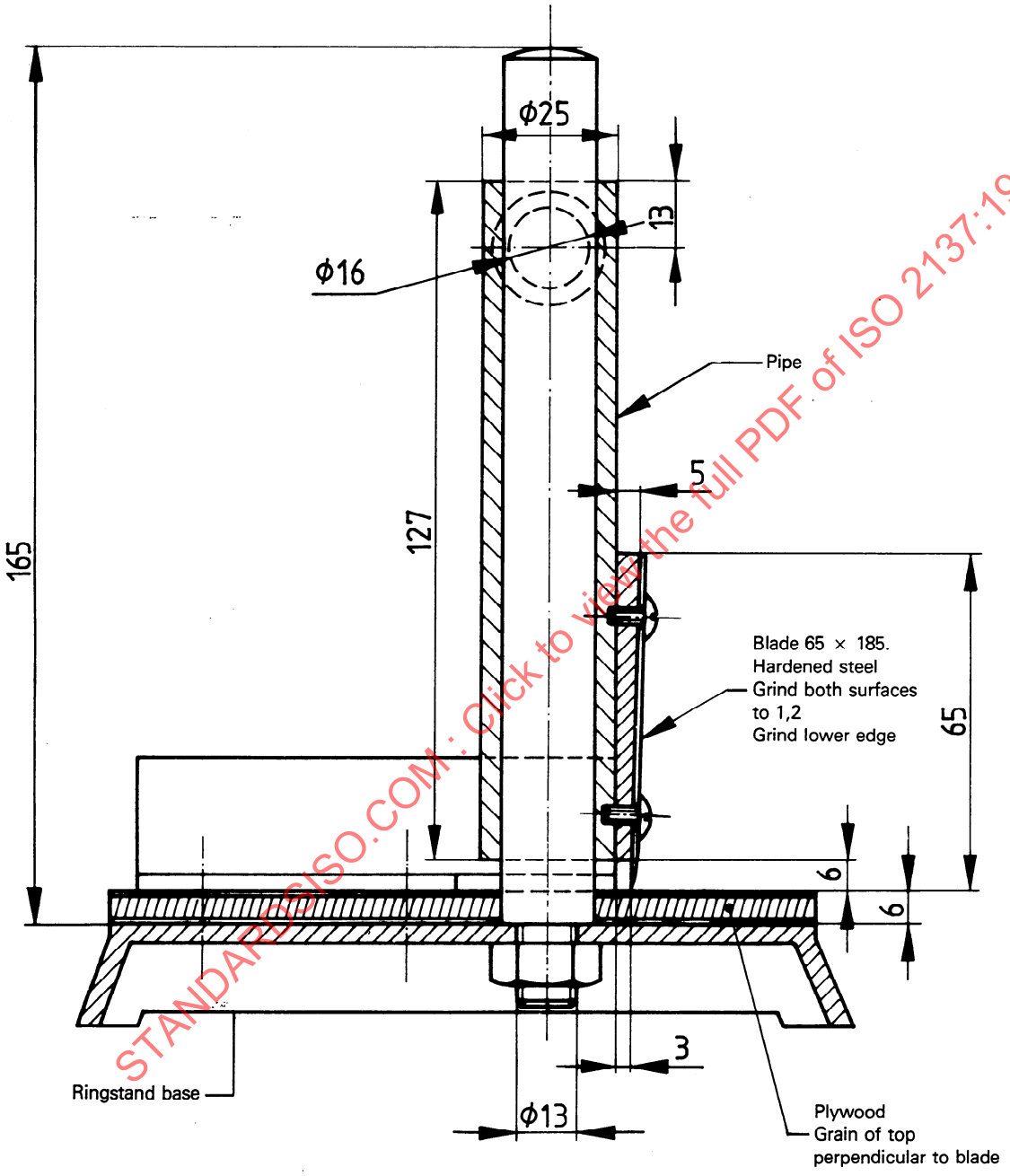
Figure 7 — One-half scale grease worker



Tolerance on dimensions, except where otherwise stated : ± 0,25 mm.

Figure 8 — One-quarter scale grease worker

Dimensions in millimetres



Tolerance on dimensions, except where otherwise stated : ± 0,25 mm.

Figure 9 — Grease cutter

Section one: Lubricating grease — Full-scale methods

6 Procedure for unworked penetration

6.1 Preparation of test portion

6.1.1 Take sufficient sample (at least 0,5 kg) to overfill the cup of the grease worker (4.3.1). If the penetration is above 200, at least three times the amount needed to fill the cup will be required.

6.1.2 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 (4.5), maintained at 25 °C (see note to 4.5 and the notes to this clause), for sufficient time to bring the temperature of the sample to 25 ± 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 note 1). Make this transfer in such a manner that the grease will be worked as little as possible. Jar the container to drive out trapped air and pack the grease with the spatula (4.8), with as little manipulation as possible, to obtain a cupful without air pockets. Scrape off the excess grease extending above the rim by moving the blade of the spatula, held inclined toward the direction of motion at an angle of 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.

NOTES

1 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.

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

6.2 Cleaning 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 while 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 may cause wear on the release mechanism.

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

6.3 Penetration measurement

6.3.1 Place the cup on the penetrometer table, which should 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 6.3.2 or 6.3.3. Watching the shadow of the cone tip is an aid to accurate setting. For greases with penetrations over 400, 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 ± 0,1 s, and reclamp 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.

6.3.2 If the test portion has a penetration over 200 units, centre the cone carefully in the container; this test portion can be used for only one test.

6.3.3 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 120 ° apart, and midway between the centre and the side of the container, so that the cone will neither strike the side of the container nor impinge on the disturbed area made in a previous test.

6.3.4 Make a total of three determinations on the test portion [either in three containers (see 6.3.2) or in one container (see 6.3.3)] and record the values obtained.

7 Procedure for worked penetration

7.1 Preparation of test sample

7.1.1 Take sufficient sample (at least 0,5 kg) to overfill the cup of the grease worker (4.3).

7.1.2 Working

Transfer a sufficient quantity of the laboratory sample to the cup of the clean grease worker (4.3) to overfill it (mounded up about 13 mm at the centre), avoiding the inclusion of air by packing with the spatula. 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 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 note to 4.5, note 1 to 6.1.2, and note to this clause) until the temperature of the grease worker and contents is 25 ± 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 double strokes of the plunger, completed in about 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 may readily be removed. As the worked penetration of a lubricating grease may change significantly on standing, proceed in accordance with 7.2 and 7.3 without delay.

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

7.2 Preparation of test portion

7.2.1 Prepare the worked test sample (7.1.2) in the cup for testing so that a uniform and reproducible structure of grease will be obtained.

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

NOTE — 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 may have the effect of increasing the working beyond the specified 60 strokes.

7.2.3 Scrape off the excess grease extending above the rim of the cup by moving the blade of the spatula, held inclined toward the direction of motion at an angle of 45 °, across the rim of the cup, retaining the portion removed.

NOTE — 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 may be returned to the cup prior to turning the sample for the next test.

7.3 Penetration measurement

7.3.1 Determine the penetration of the sample as described in 6.2 and 6.3.1.

7.3.2 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 7.2.3. Then repeat the operation specified in 7.2 through 7.3.1. Record the three values obtained.

8 Procedure for prolonged worked penetration

8.1 Preparation of apparatus and test portion

8.1.1 Temperature

Maintain the temperature of the room used for the test within the range of 15 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 to 30 °C.

8.1.2 Working

Fill a clean grease worker cup and assemble the worker as described in 7.1.2. Subject the grease sample to the prescribed or agreed number of double strokes.

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

8.2 Penetration measurement

Immediately after the working has been completed, place the grease worker in a constant temperature water or air bath to bring the test portion temperature to 25 ± 0,5 °C within 1,5 h. Remove the worker from the bath and subject the grease to a further 60 double strokes. Prepare and penetrate the test portions as described in 7.2 and 7.3.

9 Procedure for block penetration

9.1 Preparation of test portion

9.1.1 Take sufficient sample of the grease, which must be hard enough to hold its shape to permit cutting from it a 50 mm cube as a test sample.

9.1.2 By means of the grease cutter (4.4), cut as a test sample from the laboratory sample at room temperature, a cube about 50 mm on the edge. While holding this test sample so that the unbevelled edge of the cutter is toward it, slice off a layer about 1,5 mm in thickness from each of the three faces adjacent to a single corner, which may be truncated for identification (see note below). Take care not to touch those portions of the newly exposed faces which are to be 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 ± 0,5 °C by placing it in a constant temperature air bath maintained at 25 °C for at least 1 h (see note 2 to 6.1.2).

NOTE — The testing of three faces is intended to equalize in the final value the effect of the fibre orientation in testing fibrous greases. Smooth-textured, non-fibrous greases may be tested on one face only, when agreed upon between the interested parties.

9.2 Penetration measurement

Place the test portion on the table of the penetrometer, which should 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 6.2 and 6.3.1. Make 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 3 units, make additional tests until three values agreeing within 3 units are obtained. Average these three values for the face being tested.

9.3 Additional determinations

Repeat the procedure described in 9.2 on each of the other prepared faces of the test portion and record the average values obtained.

Section two: Lubricating grease — Half-and quarter-scale cone method

10 Procedure for unworked penetration

10.1 Preparation of test portion

10.1.1 Take sufficient sample to overfill the cup of the grease worker (4.3.2 or 4.3.3). If the penetration by the quarter-scale cone is greater than 47 or by the half-scale cone greater than 97, 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 in 6.1.2.

10.2 Cleaning cone and movable attachments

Clean the penetrometer cone (4.2.2 or 4.2.3) 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 may cause wear of the release mechanism.

10.3 Penetration measurement

10.3.1 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.

10.3.2 If the test portion has a penetration over 47 by the quarter-scale cone (4.2.3) or over 97 by the half-scale cone (4.2.2), centre the cone carefully in the container; this test portion can then be used for only one test.

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

10.3.4 Proceed as in 6.3.1 and 6.3.4.

11 Procedure for worked penetration

11.1 Preparation of test sample

11.1.1 Take sufficient sample to overfill the cup of the appropriate grease worker (4.3.2 or 4.3.3).

11.1.2 Working

Proceed according to 7.1.2, but mounding up to approximately 6 mm, and without using a thermometer in the grease worker.

11.2 Preparation of test portion

Proceed according to 7.2.

11.3 Penetration measurement

11.3.1 Immediately determine the penetration of the test portion as specified in 10.2 and 10.3.1 to 10.3.3.

11.3.2 Proceed as specified in 6.3.1. 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 7.2.3. Then repeat the operations specified in 7.2, 10.2, 10.3.1 to 10.3.3 and 6.3.1. Record the three values obtained.

Section three: Petrolatum

12 Procedure

12.1 Preparation of test portion

12.1.1 Take a laboratory sample of approximately 1 kg for petrolatums having a penetration over 200 and of approximately 700 g for petrolatums having penetration equal to or less than 200.

12.1.2 If the penetration of the petrolatums is over 200, prepare three separate test portions. If the penetration is equal to or less than 200, prepare one test portion as specified in 12.1.3.

12.1.3 Melt the test sample in the oven (4.7) maintained at 85 ± 2 °C. Place the required number of test portion containers (4.10) 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 to 18 h in a location free from draughts and at a temperature controlled to 25 ± 2 °C. Then place the filled test portion containers in the water bath (4.5) for 2 h to bring the temperature to 25 ± 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.

NOTES

1 Some compounded petrolatums are affected by contact with water. The test portion of such a petrolatum must be covered with a sealed lid as required for greases (see Section one). Uncompounded petrolatums are not affected by water and need not be covered.

2 Some petrolatums containing higher melting point waxes may require higher pouring temperatures; in that case, the precision data given in table 3 may not apply to the results.

If the room temperature varies from 25 °C by 2 °C or more, adjust the cone temperature to 25 ± 0,5 °C immediately before testing the test portion by immersing it in the water bath (4.5) and subsequently drying it with a lint-free cloth or tissue. Frequent adjustment of the cone temperature may be necessary if the room temperature varies appreciably from 25 °C.

12.2 Penetration measurement

Proceed according to 6.2 and 6.3.

NOTE — 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 may be different from those obtained in off-centre positions on the level surface.