BS 2000 :
Part 50 : 1993

Methods of test for

Petroleum and its products

Part 50. Determination of cone penetration of lubricating grease

(Identical with IP 50/88)

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BSi
British Standards

Foreword

This British Standard, having been prepared under the direction of the Petroleum Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 28 February 1993.

This British Standard supersedes BS 2000 : Part 50 : 1985, which is withdrawn.

In accordance with BSI procedure this Standard was made available for public comment as 92/56097 DC.

BS 2000 comprises a series of test methods for petroleum and its products that are published by the Institute of Petroleum (IP) and have been accorded the status of a British Standard. Each method should be read in conjunction with the preliminary pages of `IP Standard methods for analysis and testing of petroleum and related products' which gives details of the BSI/IP agreement for publication of the series, provides general information on safety precautions, sampling and other matters, and lists the methods published as Parts of BS 2000.

The numbering of the Parts of BS 2000 follows that of the corresponding methods published in `IP Standard methods for analysis and testing of petroleum and related products'. Under the terms of the agreement between BSI and the Institute of Petroleum, the revised version of BS 2000 : Part 50 will be published by the IP (in `Standard methods for analysis and testing of petroleum and related products' and as a separate publication). BS 2000 : Part 50 : 1993 is thus identical with IP 50/88. Square brackets marked in the margin of this IP Standard indicate text that differs from the previous edition.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Committee reference PTC/13

IP 50/88 ASTM D217–88 BS 2000: Part 50: 1993

Determination of cone penetration of lubricating grease[1]

This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations.

1. SCOPE

1.1. This method covers four procedures for measuring the consistency of lubricating greases by penetration of a standard cone. This method includes procedures for the measurement of unworked, worked, prolonged worked, and block penetrations. Penetrations up to 475 may be measured. Undisturbed penetrations are described in the Appendix.

NOTE 1: The National Lubricating Grease Institute has classified greases according to their consistency as measured by the worked penetration. The classification is as follows:

NLGI grade	Worked penetration range
000	445 to 475
00	400 to 430
0	355 to 385
1	310 to 340
2	265 to 295
3	220 to 250
4	175 to 205
5	130 to 160
6	85 to 115

NOTE 2: The procedure for the measurement of penetration of petrolatum is described in Method ASTM D937–IP 179, Test for Penetration of Petrolatum.[2]

1.2. 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.

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

2. SUMMARY OF METHOD

2.1. The penetration is determined at 25°C by releasing the cone assembly from the penetrometer and allowing the cone to drop freely into the grease for 5 seconds.

3. SIGNIFICANCE

3.1. Worked penetrations may be used to establish the consistency of lubricating greases within the NLGI consistency grades shown in Note 1. The change in worked penetration results of a grease after prolonged working are believed to be a measure of its shear stability, under the conditions of the test. All four test procedures are widely used for specification purposes, however no correlation with field service has been established.

4. DEFINITIONS

4.1. *Penetration* of lubricating grease is the depth, in tenths of a millimeter, that a standard cone penetrates the sample under prescribed conditions of weight, time, and temperature.

4.2. *Working* is the subjection of a lubricating grease to the shearing action of the standard grease worker.

4.3. *Unworked Penetration* is the penetration at 25°C of a sample of lubricating grease which has received only minimum disturbance in transferring to a grease-worker cup or dimensionally equivalent container.

NOTE 3: The penetrations of soft greases are dependent upon the diameter of the container. Therefore, greases having undisturbed and unworked penetrations above 265 should be tested in containers having the same diameter limitations as those of the worker cup. The results on greases having penetrations below 265 are not significantly affected if the diameter of container exceeds that of the worker cup.

4.4. *Worked Penetration* is the penetration of a sample of lubricating grease after it has been brought to 25°C and then subjected to 60 double strokes in a standard grease worker, and penetrated without delay.

4.5. *Prolonged Worked Penetration* is the penetration of a sample of lubricating grease after being worked more than 60 double strokes in a standard grease worker at a temperature of 15 to 30°C. After the prescribed number of double strokes the worker and contents are brought to 25°C, worked an additional 60 double strokes, and penetrated without delay.

4.6. *Block Penetration* is the penetration at 25°C of a sample of lubricating grease that is sufficiently hard

[1]This method is under the jurisdiction of the ASTM Committee D-2 on Petroleum Products and Lubricants.
In the IP, this method is under the jurisdiction of the Standardization Committee.
[2]*Annual Book of ASTM Standards*, Vol. 05.01.
IP Methods for Analysis and Testing, Part 1, Volume 1.

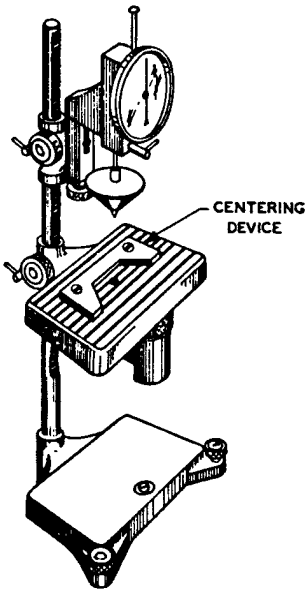


Fig. 1. Penetrometer.

to hold its shape, determined on the freshly prepared face of a cube cut from a block of the grease.

5. APPARATUS

5.1. *Penetrometer* – An instrument similar to that shown in Fig. 1, designed to measure in tenths of a millimetre the depth to which the standard cone penetrates the grease. 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 grease while maintaining a 'zero' reading on the indicator. When released, the cone should fall without appreciable friction [Note 4]. The tip of the cone should 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.

NOTE 4: Both the penetrometer shaft and the rack engaging the measuring dial should be of sufficient length to test greases with penetrations as great as 620. If only penetrations less than 400 are to be measured, the penetrometer may be designed such that when released, the cone falls for at least 40.0 mm.

5.2. *Standard Cone* – as specified in Annex A1.1. An optional cone, described in Annex A1.2, may be used for penetrations less than 400.

5.3. *Grease Worker* – as specified in Annex A1.3.

5.4. *Mechanical Grease Worker* – A machine designed to operate the plunger of the grease worker mechanically as specified in Annex A1.3. This apparatus is essential for the measurement of Prolonged Worked Penetration.

5.5. *Grease Cutter* – as specified in Annex A1.4.

5.6. *Water or Air Bath* – Bath capable of regulation to 25 ± 0.5°C and designed to bring the assembled worker to test temperature conveniently. If a water bath is to be used for samples for unworked penetration, means should be provided for protecting the grease surface from water and for maintaining the air above the sample at test temperature. An air bath is preferred for bringing block greases to test temperature, but a tightly sealed container placed in a water bath will suffice. A constant temperature test room may be used instead of the water or air bath.

Scraping Off.

Fig. 2. Preparing sample for penetration measurement.

5.7. *Spatula* – corrosion-resistant, having a stiff blade 32 mm wide and at least 150 mm long.

5.8. *Overflow Ring (Optional)* – as described in Annex A1.5. Both the grease removed in scraping and the grease forced by the penetrometer cone to overflow may be returned to provide a full cup for the next test. The overflow ring is convenient for returning overflowed grease to the worker cup.

6. PROCEDURE FOR UNWORKED PENETRATION

6.1. *Sample* – Sufficient sample (at least 0.4 kg) to overfill the cup of the standard grease worker shall be required. If sample size is insufficient and penetration ranges from NLGI grade 0 to 4, use Method ASTM D1403/IP 310 test for Cone Penetration of Lubrication Grease using One-quarter and One-half Scale Cone Equipment.[2] If the transferred penetration is above 200, at least three times the amount needed to fill the cup shall be required.

6.2. *Preparing Sample for Measurement* – Place the empty grease worker cup and an appropriate amount of the sample in a metal container in the water or air bath maintained at 25°C [Note 5] for sufficient time to bring the temperature of the sample and the worker cup to 25 ± 0.5°C. Transfer the sample, preferably in one lump, to overfill the cup of the grease worker [Note 3]. Make this transfer in such a manner that the grease will be worked as little as possible. Jar the cup to drive out trapped air and pack the grease with the spatula, 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 (Fig. 2). Do not perform any further levelling or smoothing of the surface throughout the determination of unworked penetration and determine the measurement immediately.

NOTE 5: 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 assure that the sample is at 25 ± 0.5°C before proceeding. Also, if the sample is larger than 0.4 kg, allow sufficient additional time to assure that the sample is at 25 ± 0.5°C. Testing may proceed if the sample is at a uniform temperature of 25 ± 0.5°C.

6.3. *Cleaning Cone and Shaft* – 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. Do not permit 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.

6.4. *Penetration Measurement* – Place the cup on the penetrometer table, making certain that it cannot teeter. Set the mechanisms 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 sample. Watching the shadow of the cone tip is an aid to accurate setting. For grease with penetrations over 400, the cup (Fig. A3) must be centered to within 0.3 mm of the tip of the cone. One way to centre the cup accurately is to use a centering device (Fig. 1). Release the cone shaft rapidly, and allow it to drop for 5.0 ± 0.1s. The release mechanism should not drag on the shaft. Gently depress the indicator shaft until stopped by the cone shaft and read the penetration from the indicator.

6.4.1. If the sample has a penetration over 200, center the cone carefully in the container; this sample can then be used for only one test. If the sample has a penetration of 200 or less, perform three tests in a single container, spacing these tests on three radii 120° apart and midway between the center 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.

6.5. *Additional Testing* – Make a total of three tests on the sample (either in three containers or in one, as described in 6.4), and report the average value, to the nearest unit, as the unworked penetration of the sample.

7. PROCEDURE FOR WORKED PENETRATION

7.1. *Sample* – Sufficient sample (at least 0.4 kg) to overfill the cup of the standard grease worker is required. If sample size is insufficient and penetration ranges from NLGI Grade 0 to 4, use Method ASTM D1403, IP 310.

7.2. *Working* – Transfer sufficient sample to the cup of the clean grease worker to fill it heaping full (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 air inadvertently entrapped. Assemble the worker and, with the vent cock open, depress the plunger to the bottom. Insert a thermometer through the vent cock so that its tip is in the centre of the grease. Place the assembled worker in the water or air bath maintained at 25°C [Notes 5 and 6] until the temperature of the worker and contents are 25 ± 0.5°C as indicated by the thermometer. Then remove the worker from the bath and wipe off the excessive water adhering to its surfaces. Remove the thermometer and close the vent cock. Subject the grease to 60 full (67 to 71.5 mm) double strokes of the plunger, completed in about 1 min, and return the plunger to its top position. Open the vent cock, 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 7.3, 7.4 and 7.5 immediately.

NOTE 6: If it is desired to immerse the portion of the worker above its closure, take care that the lid is watertight in order to prevent the entrance of water to the worker.

7.3. *Preparing Sample for Measurement* – Prepare the worked sample in the cup for testing so that a uniform and reproducible structure of the grease will be obtained. Jar the cup sharply on the bench or floor and pack the grease down with a spatula to fill the holes left by the plunger and to remove any air pockets [Note 7]. 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 (Fig. 2), retaining the portion removed [Note 8].

NOTE 7: The jarring should be as vigorous as required to remove the entrapped air without splashing the sample from the cup. In performing these operations, a minimum of manipulation should be used, as any agitation of the grease may have the effect of increasing the working beyond the specified 60 strokes.

NOTE 8: Particularly when testing soft greases, retain the grease removed from the cup in scraping 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 preparing the sample for the next test.

7.4. *Penetration Measurement* – Determine the penetration of the sample as described in 6.3 and 6.4. (6.4.1 applies to unworked penetration only).

7.5. *Additional Tests* – Immediately make two more tests in succession on the same sample, returning to the cup the portion previously removed with the spatula; repeat the operations described in 7.3 and 7.4. Report the average of the three tests, to the nearest 0.1 mm, as the worked penetration of the sample.

8. PROCEDURE FOR PROLONGED WORKED PENETRATION

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

Since ambient temperature may affect results, it should be noted precision data were obtained within a 21 to 29°C range.

8.2. *Working* – Fill a clean grease-worker cup and assemble the worker as described in 7.1. Subject the grease sample to the prescribed number of double strokes.

NOTE 9: In order to minimize leakage during working, special attention must be paid to the gland in the worker cover.

8.3. *Penetration Measurement* – Immediately after the working is concluded place the worker in a

constant-temperature water or air bath to bring the test sample 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 samples as described in 7.4 and 7.5, except for reporting the average of three tests as the prolonged worked penetration for the prescribed number of double strokes.

9. PROCEDURE FOR BLOCK PENETRATION

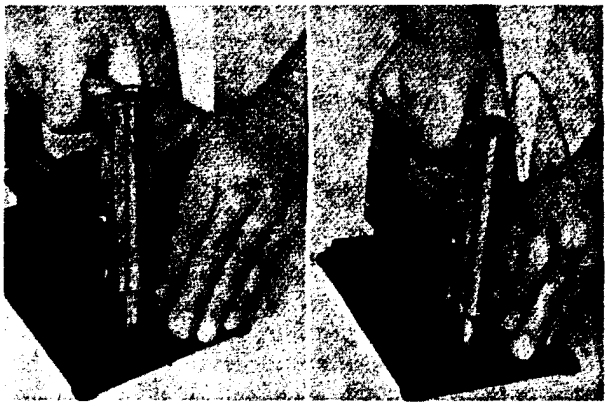
9.1. *Sample* – Obtain sufficient sample of the grease, which must be hard enough to hold its shape, to permit cutting from it a 50 mm cube as a test specimen.

9.2. *Preparing Sample for Measurement* – By means of the specified grease cutter, cut as a test specimen from the sample at room temperature a cube about 50 mm on the edge (Fig. 3(*a*)). While holding this specimen so that the unbevelled edge of the cutter is towards it (Fig. 3(*b*)), 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 (Fig. 3(*c*) and Note 10). 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 specimen to 25±0.5°C by placing it in a constant-temperature air bath maintained at 25°C for at least 1 h [Note 5].

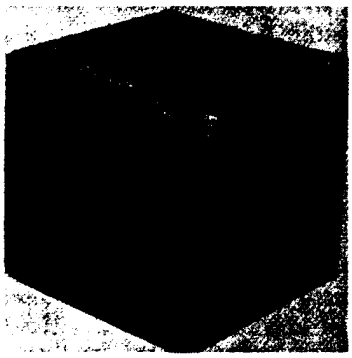
NOTE 10: The testing of three faces is intended to equalize in the final value the effect of fibre orientation in testing fibrous greases. Smooth-textured, non-fibrous greases may be tested on one face only, when agreed upon between interested parties.

9.3. *Penetration Measurement* – Place the test specimen on the penetrometer table 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 teeter 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 sample. Determine the penetration as described in 6.3 and 6.4. Make a total of three tests on the exposed face of the specimen, 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, make additional tests until three values agreeing within three units are obtained. Average these three values for the face being tested.

9.4. *Additional Tests* – Repeat the procedure described in 9.3 on each of the prepared faces of the specimen. Report one third of the sum of the averages for the three faces, to the nearest unit, as the block penetration of the grease under test.



(*a*) Cutting Test Specimen. (*b*) Slicing Off Layer.



(*c*) Truncated Corner for Identification.

Fig. 3. Preparing block sample for penetration measurement.

10. PRECISION

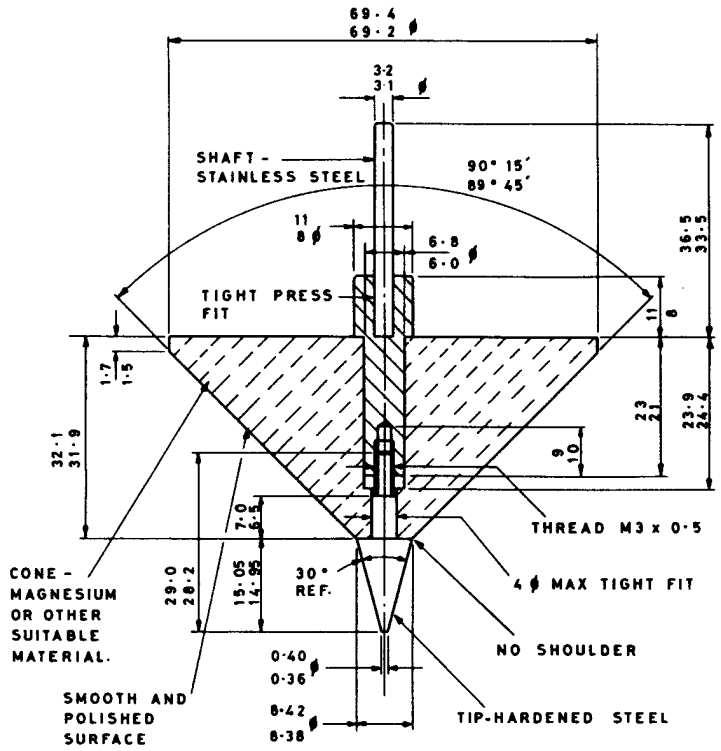
10.1. *Precision* – The precision of the method as determined by statistical examination of inter-laboratory results is as follows:

10.1.1. *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 in the table below in only 1 case in 20.

10.1.2. *Reproducibility* – The difference between two single and independent 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 in the table below in only 1 case in 20.

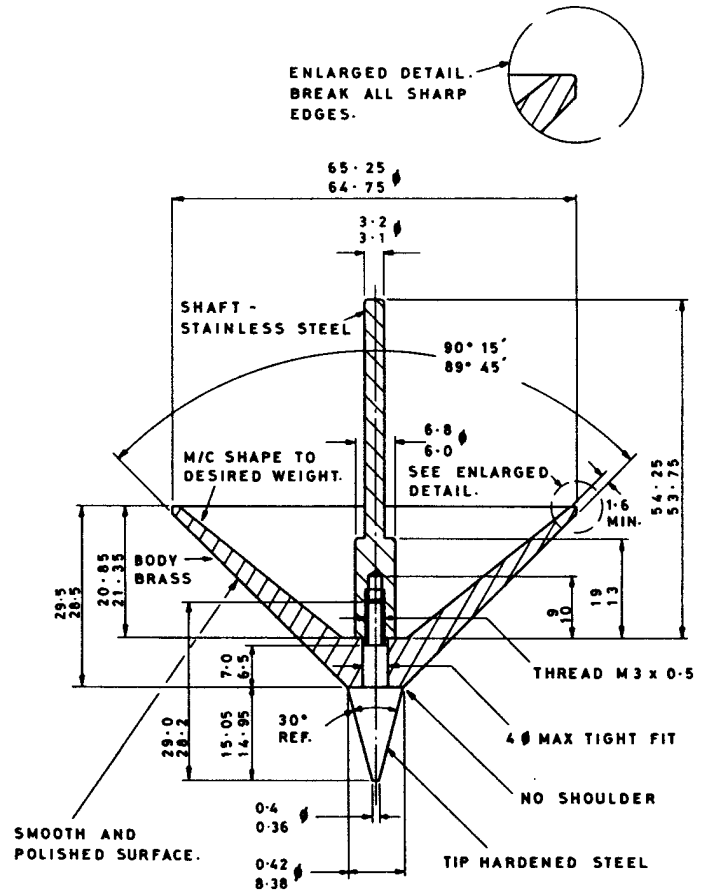
Penetration	Penetration range	Repeatability, one operator and apparatus	Reproducibility, different operators and apparatus
Unworked	85 to 475	8 units	19 units
Worked	130 to 475	7 units	20 units
Prolonged worked	130 to 475	15 units[a]	27 units[a]
Block	Under 85	7 units	11 units

[a]Determined at 100,000 double strokes within 15 to 30°C ambient temperature range.



NOTE: The total weight of the cone shall be 102.5 ± 0.05 g and the total weight of its movable attachments shall be 47.5 ± 0.05 g.

Fig. A1. Standard Penetrometer Cone.



NOTE: The total weight of the cone shall be 102.5 ± 0.05 g and the total weight of its movable attachments shall be 47.5 ± 0.05 g.

Fig. A2. Optional Penetrometer Cone.

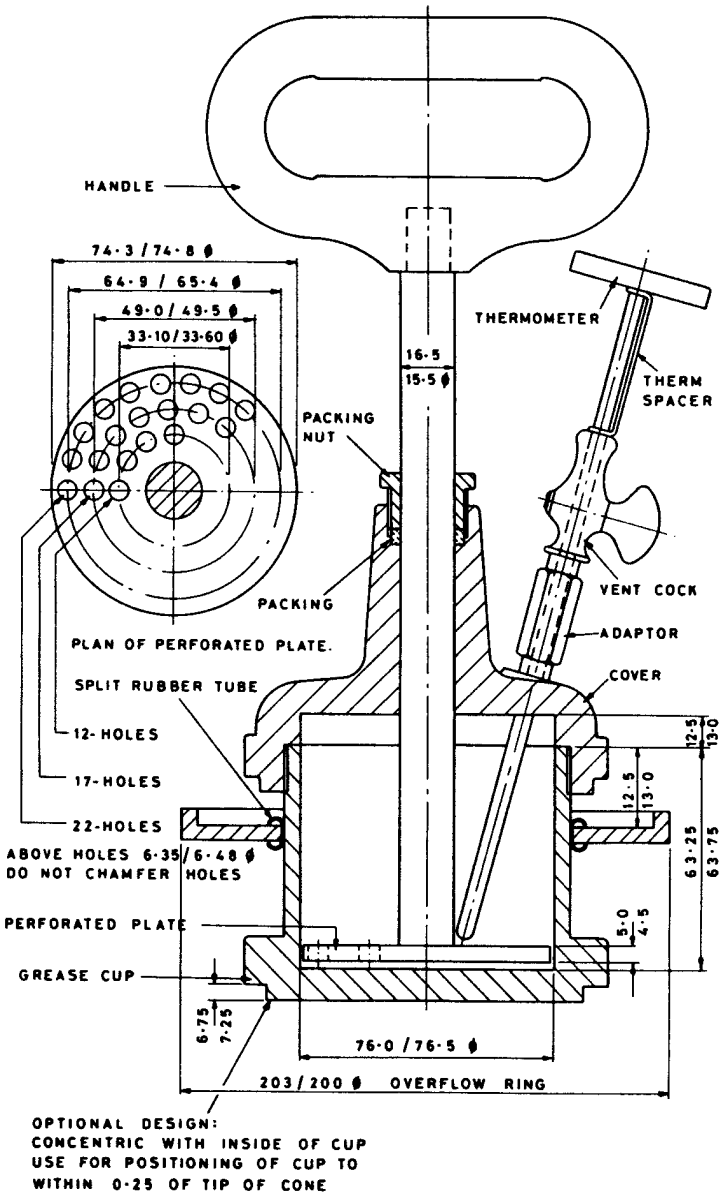


Fig. A3. Grease worker.

ANNEX

A1. APPARATUS

A1.1. *Standard Cone,* for measuring penetrations up to 475.

Cone, consisting of a conical body of magnesium or other suitable material with detachable, hardened steel tip, shall be constructed to conform to the tolerances shown in Fig. A1. The total weight 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 suitable means, at its lower end, for engaging the cone. The interior construction may be modified to achieve the specified weight, provided that the general contour and weight distribution are not altered. Polish the outer surface to a very smooth finish.

A1.2. *Optional Cone,* for measuring penetrations up to 400.

Cone, consisting of a conical body of brass or corrosion-resistant steel with detachable, hardened steel tip, shall be constructed to conform to the tolerances shown in Fig. A2. The total weight 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 suitable means, at its lower end, for engaging the cone. The interior construction may be modified to achieve the specified weight, provided that the general contour and weight distribution are not altered. Polish the outer surface to a very smooth finish.

A1.3. *Grease Worker* conforming to the dimensions given in Fig. A3. The dimensions not shown may be altered and other methods of fastening the lid and securing the worker may be used. The

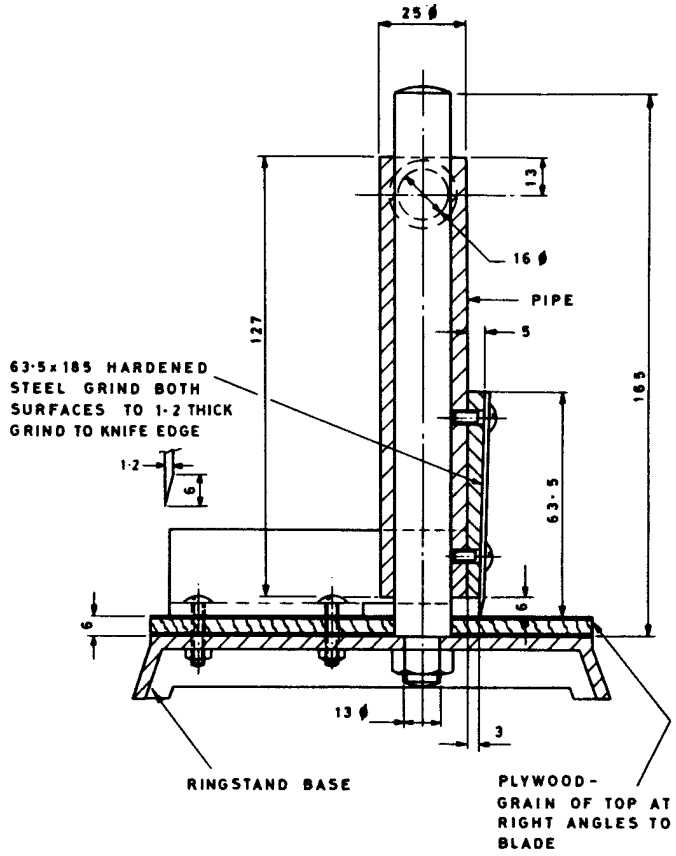


Fig. A4. Grease cutter.

worker may be constructed for either manual or mechanical operation. Design must be such that a rate of 60 ± 10 strokes per minute with a minimum length of 67 mm, can be maintained. A suitable temperature indicator, standardized at 25°C, shall be provided for insertion through a vent cock. If mechanically operated, the grease worker must be provided with a pre-setting counter to permit the apparatus to be automatically stopped after any required number of double strokes up to 99,999.

A1.4. *Grease Cutter,* having a sharp, rigidly mounted, bevelled blade, shall be essentially as shown in Fig. A4. It is necessary that the blade be straight and sharpened, as shown.

A1.5. *Overflow Ring,* conforming in principle to the illustration in Fig. A3 is a useful aid for returning displaced grease to the 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.

APPENDIX

UNDISTURBED PENETRATION

Undisturbed penetration is defined as the penetration at 25°C of a sample of grease in its container with no disturbance. Typical uses of the undisturbed penetration are: (1) to control the consistency of certain greases during manufacture, and (2) to assess the degree to which a grease develops a 'false body' or 'set' upon prolonged storage. The use of undisturbed penetration as a quality control test does not involve the transfer of grease samples from one container to another prior to testing. However, in evaluating the effect of prolonged storage upon a lubricating grease, samples may be repackaged prior to storage in an appropriately sized container. Parties interested in measuring the undisturbed penetration on grease samples should use the general procedure described in Sections 6.3, 6.4, and 6.5 and establish mutually agreeable conditions for storage and container size.