INTERNATIONAL STANDARD

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Petroleum products — Determination of dropping point of lubricating greases (wide temperature range)

Produits pétroliers — Détermination du point de goutte des graisses lubrifiantes (domaine de température étendu)

Reference number
ISO 6299:1998(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 6299 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Annex A forms and integral part of this International Standard. Annex B is for information only.

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Petroleum products — Determination of dropping point of lubricating greases (wide temperature range)

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the dropping point of lubricating grease over a wide temperature range.

NOTES

1 The dropping point is useful as an aid to identification of lubricating grease as to type, and as a bench mark for quality control. The results should be considered to have only limited significance with respect to service performance because dropping point is a static test.

2 Cooperative testing indicates that, in general, dropping points determined according to this International Standard and to ISO 2176 are in agreement up to 260 °C. In cases where results differ, there is no known significance. However, agreement between the manufacturer and purchaser as to the test method used is advisable.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test.*

3 Definitions

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

3.1 dropping point: Numerical value assigned to a grease composition representing the corrected temperature at which the first drop of material falls from the test cup and reaches the bottom of the test tube

3.2 lubricating grease: Semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant

3.3 observed dropping point: Value noted on the thermometer monitoring the internal temperature of the test cup when the first drop of material falls from the test cup and reaches the bottom of the test tube

3.4 thickener: In lubricating grease, a substance composed of finely-divided particles dispersed in a liquid lubricant to form the structure of the product

4 Principle

A grease sample in a test cup is supported in a test tube placed in an aluminium block oven at a pre-set constant temperature. A sample thermometer is placed in the tube and positioned so that it measures the temperature in the test cup without coming into contact with the grease. The reading on the thermometer is recorded to the nearest 1 °C as the observed dropping point when the temperature is increased sufficiently for a drop of material to fall from the test cup to the bottom of the test tube. At the same time, the temperature of the aluminium block oven is also recorded to the nearest 1 °C. One-third of the difference between the two values is the correction factor which is added to the observed value and recorded as the dropping point of the grease.

5 Apparatus and accessories

5.1 Dropping point assembly, illustrated in figure 1 and consisting of the following parts:

5.1.1 Test cup, of chromium-plated brass, conforming to the dimensions given in A, of figure 1.

5.1.2 Test tube, of thin-walled soft glass with rim, having the dimensions given in B of figure 1.

5.1.3 Cup support, of glass tubing as shown in C of figure 1.

5.1.4 Thermometer, conforming to the specified requirements of annex A, and shown in D of figure 1.

5.2 Thermometer clamp, as shown in E-1 of figure 1.

5.3 Bushings, as shown in E-2 and E-3 of figure 1.

5.4 Bushing support ring, as shown in E-4 of figure 1.

5.5 Thermometer depth gauge, as shown in E-5 of figure 1.

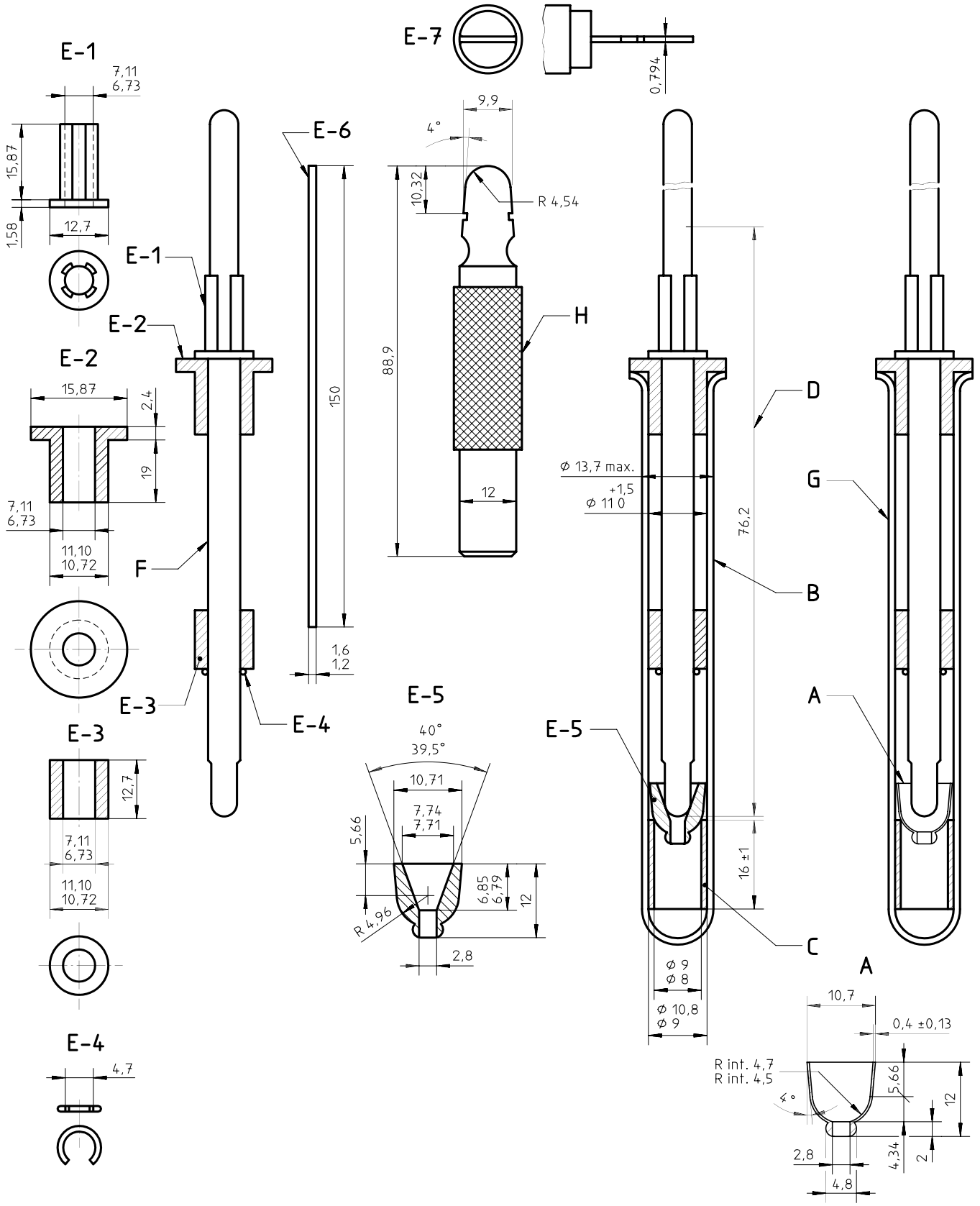
5.6 Metal rod, as shown in E-6 of figure 1.

5.7 Test cup plug gauge, as shown in E-7 of figure 1.

5.8 Aluminium block oven, of 700 W output and of the design and dimensions shown in figure 2. The block shall be equipped with an integral cartridge-type heater. Control of the heater shall be used to obtain and maintain the desired oven temperature.

5.9 Thermometer for aluminium block oven, as shown in F of figure 1.

Dimensions in millimetres



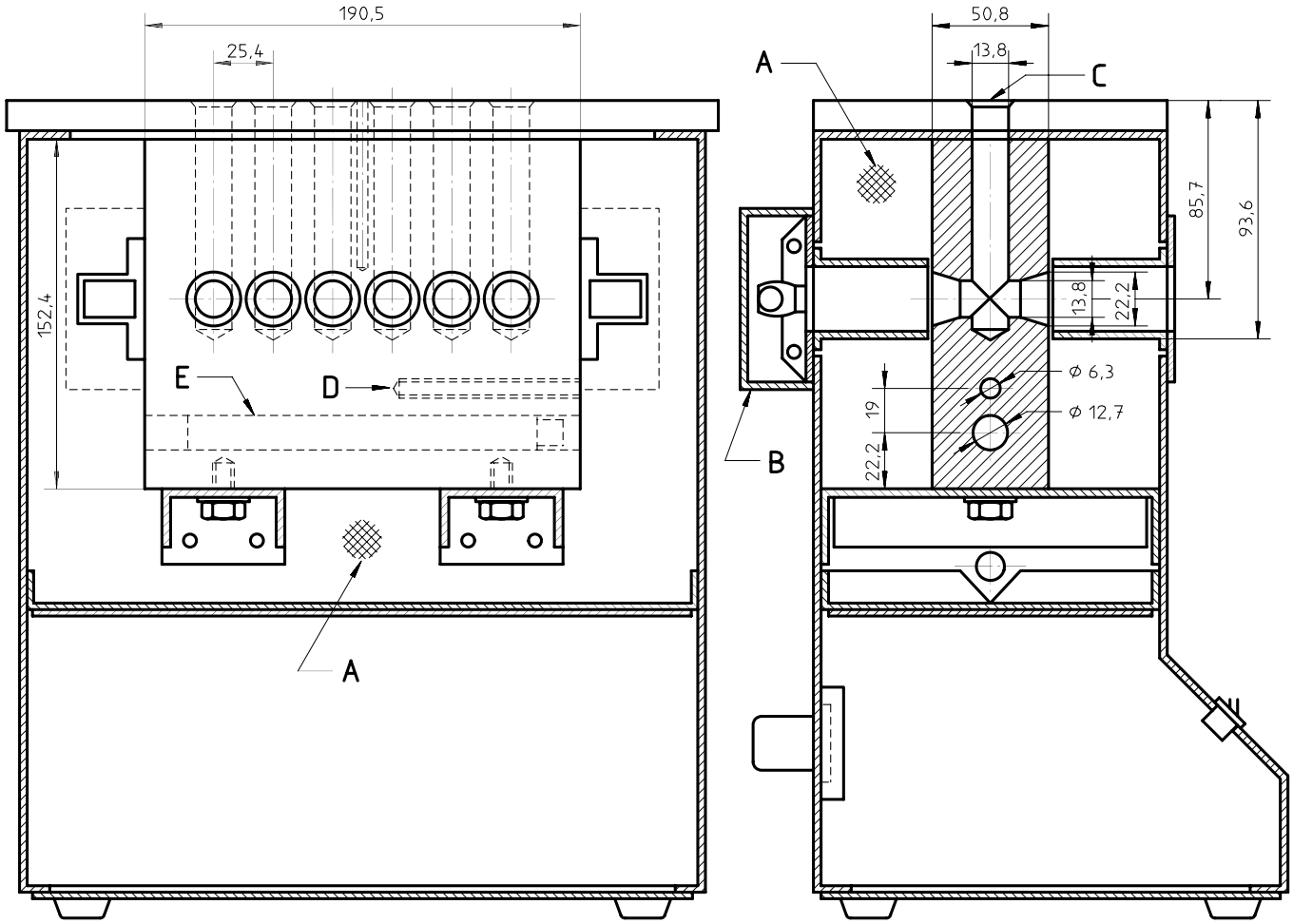
See over for key.

Figure 1 — Dropping point apparatus

Key

A Grease cup of chromium-plated brass
B Thin-wall test tube, length 100 mm ± 1 mm
C Cup support
D Thermometer immersion
E-1 Thermometer clamp
E-2 Aluminium or transite bushing
E-3 Aluminium or transite bushing
E-4 Bushing support ring. Material: 1,0 mm stainless steel wire
E-5 Thermometer depth gauge of brass
E-6 Rod
E-7 Cup plug gauge. Material: AISi-01. Harden to HRC 50-52
F Thermometer assembly
G Tube assembly
H Knurl

Dimensions in millimetres



Key

A Insulation
B Lamp assembly
C Thermometer well
D Thermistor probe
E 700 W Heater

Figure 2 — Aluminium block oven

6 Procedure

6.1 Asssembly of apparatus

6.1.1 Thoroughly clean the test cup, cup support, and test tube. Use only test cups that are clean and free of any residue. When the interior plating of the test cup shows indications of wear, discard the test cup.

When new test cups are used, check their dimensions by using the test cup plug gauge (5.7). A 2,78 mm rod shall fit easily through the bottom opening of the test cup, while a 2,82 mm rod shall not. If the hole is undersize, ream to the correct size. If the hole is too large, discard the test cup.

NOTE — Test cups of the correct size do not require rechecking prior to each test run.

6.1.2 Test tubes shall be clean and free of residues and conform to the dimensions shown in B of figure 1. Inspect the test tubes for chips or cracks and replace when necessary.

6.1.3 Sample thermometer bulbs shall be clean and free of residues. Inspect bushings (5.3) for cleanliness and ensure that the thermometer clamp (5.2) is sufficiently tight to hold the thermometer in position.

6.1.4 The glass sleeve used to support the test cup shall be free of any chips or cracks, residue or stain, and conform to the dimensions shown in C of figure 1. Replace when necessary.

6.1.5 Insert an empty test tube in each test-tube well, and a sample thermometer (5.1.4) in the thermometer well of the aluminium block oven. Select the lowest oven temperature setting, from those given in table 1, that will result in an observed dropping point at or below the figure given in table 1 for the corresponding oven temperature.

Table 1 — Maximum dropping points for oven-temperature settings

Oven temperature °C	Maximum dropping point °C
121 ± 3	116
232 ± 3	221
288 ± 3	277
316 ± 3	304

6.1.6 Select and use test tubes and accessories (E-1, E-2 and E-3 in figure 1) to minimize wobble of the thermometer. All components shall be at room temperature prior to the test. Place the components E-1 through E-4 on the thermometer in the order shown by the thermometer assembly F of figure 1. Adjust the bushing (5.3) and the bushing support ring (5.4) so that the latter is approximately 25 mm from the tip of the thermometer. Place the test cup support (5.1.3) in the test tube (5.1.2). Insert the thermometer depth gauge (5.5) and the thermometer assembly in the tube. Position the thermometer so that the tip bottoms in the gauge. Adjust the bushing and the thermometer clamp (5.2) so that the bushing shoulders on the top of the test tube.

6.2 Addition of the test sample

6.2.1 Fill the test cup (5.1.1) by pressing its larger opening into the grease to be tested until the cup is filled. Remove excess grease from the top edge of the cup with a spatula. Gently press the cup, held in a vertical position with the smaller opening at the bottom, down over the metal rod (5.6) until the rod protrudes approximately 25 mm. Press the rod against the cup in such a manner that the rod makes contact at both the upper and lower peripheries of the cup. Maintain this contact, rotating the cup on the rod along the index finger so as to give it a spiral-like motion down the rod to remove a conical section of the grease which adheres to the rod. A smooth film of reproducible thickness is left inside the cup.

6.2.2 Remove the thermometer assembly and depth gauge from the test tube. Position the grease cup and replace the thermometer assembly. Make no further adjustment as the thermometer bulb is now positioned so as to provide adequate clearance between the tip of the bulb and the grease sample in the cup.

6.2.3 Remove an empty test tube from the oven and gently insert the tube assembly (G of figure 1), ensuring that the cup remains upright in the cup support.

NOTE — If the cup is tilted, the thermometer bulb can come into contact with the film of grease and result in an erroneous value.

6.2.4 When the first drop of material falls free of the cup orifice and reaches the bottom of the test tube, record both the temperature of the cup and the oven to the nearest 1 °C. If certain greases, such as those with simple soap compositions or those containing some particular types of polymer, form a drop with a trailing thread, record the temperature in the cup as the observed dropping point when the drop reaches the bottom of the test tube.

NOTE — The aluminium block oven can accommodate up to six samples of the same or different greases in order that multiple determinations may be made simultaneously.

7 Calculation

Calculate the dropping point, D, in degrees Celsius, from the following equation:

$$D = t_0 + \frac{t_1 - t_0}{3}$$

where

t_0 is the thermometer reading when the first drop reaches the bottom of the test tube, in degrees Celsius;

t_1 is the block temperature when the drop falls, in degrees Celsius.

8 Expression of results

Report the result calculated in clause 7 to the nearest 1 °C.

9 Precision

The precision of the method, as produced by statistical examination of interlaboratory results according to ISO 4259, is given in 9.1 and 9.2.

NOTE — The precision values for greases with dropping points below 221 °C were obtained using older heavy-walled heat-resistant test tubes. Values for greases with dropping points above 221 °C were obtained using the thin-walled test tubes (5.1.2) of soft glass.

9.1 Repeatability

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the normal and correct operation of the test method, exceed the values given in table 2 only in one case in 20.

9.2 Reproducibility

The difference between two single and independent results, obtained by different operators working in different laboratories on nominally identical test material would, in the normal and correct operation of the test method, exceed the values given in table 2 only in one case in 20.

Table 2 — Precision data

Dropping point range °C	Repeatability limit °C	Reproducibility limit °C
Below 116	6	9
116 to 221	8	12
222 to 277	6	16
278 to 316	7	12

10 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard;
b) the type and complete identification of the product tested;
c) the result of the test (see clause 8);
d) any deviation, by agreement or otherwise, from the standard procedures specified;
e) the date of the test.

Annex A
(normative)

Thermometer specification

The thermometer specified in 5.1.4 shall meet the specification given in table A.1.

Table A.1 — Thermometer specification

Range, °C	– 5 to 400
Graduation, °C	1
Immersion depth, mm	76
Overall length, mm	408 to 418
Bulb length, mm	11 to 15
Bulb diameter, mm	5
Distance of bottom of bulb to:	
0 °C line, mm	100
400 °C line, mm	354
Longer line at each, °C	5
Figured at each, °C	10
Scale error, max., °C	1 up to 300 1,5 over 300

NOTE — An ASTM 3C/IP 73C thermometer conforms to the above specification.

Annex B
(informative)

Bibliography

[1] ISO 2176:1995, *Petroleum products — Lubricating grease — Determination of dropping point.*

ICS 75.100

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