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Petroleum products and lubricants — Determination of low-temperature cone penetration of lubricating greases

Produits pétroliers et lubrifiants — Détermination de la pénétrabilité au cône à basse température des graisses lubrifiantes

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Contents

Page

Foreword

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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 13737 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Petroleum products and lubricants — Determination of low-temperature cone penetration of lubricating greases

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 determining the cone penetration of lubricating greases at low temperatures.

NOTE 1 Precision has only been determined in the temperature range −40 °C to −20 °C. This International Standard may be used at other temperatures, e.g. −20 °C to +20 °C, however precision has not been determined under these conditions.

NOTE 2 The cone penetration is expressed in units of 0,1 mm.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 2137:—[1)], *Petroleum products and lubricants — Determination of cone penetration of lubricating greases and petrolatum*

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

IEC 60751:1983, *Industrial platinum resistance thermometer sensors*

3 Principle

The penetration is determined using the penetrometer and the full-scale cone as defined in ISO 2137. The measurement is carried out on a sample cooled, under specified conditions, to the test temperature, with the cone also being cooled to the same temperature.

4 Apparatus

Usual laboratory apparatus and glassware, together with the following.

4.1 Equipment

1) To be published. (Revision of ISO 2137:1985)

All equipment needed to perform penetration measurements as specified in ISO 2137.

NOTE 1 A penetrometer with automatic triggering may be used.

NOTE 2 Only the full-scale cone (or the optional cone for penetrations up to 400 units) is required.

NOTE 3 Only the full-scale grease worker is required.

NOTE 4 Two identical cups are needed (one for the temperature control and one for the test).

4.2 Thermostatically controlled and ventilated chamber, making it possible to operate from −50 °C to 0 °C with an accuracy of ±1 °C. The cooling capacity of the chamber shall be such that the test temperature can be reached within 2 h with the chamber empty.

4.3 Temperature sensor, preferably a 100 Ω at 0 °C platinum thermistor complying with IEC 60751, or a total immersion thermometer conforming to Annex A.

4.4 Spatula, corrosion-resistant, square-ended, having a stiff blade approximately 32 mm wide and at least 150 mm long.

4.5 Timer, with an audible alarm capable of timing to 4 h ± 5 min.

5 Preparation and conditioning of the test portion

5.1 Preparation

5.1.1 General

Ensure that the chamber is set and stabilized at the test temperature. Prepare two test portions, one for the temperature control, the other for penetration determination.

5.1.2 Preparation of the temperature-control test portion

Carry out the procedure for preparing a test portion for unworked penetration (full-scale method) as specified in Clause 7 of ISO 2137, i.e. transfer from the container a test portion, preferably in one lump, to overfill the cup of the grease worker. 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, 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.

5.1.3 Preparation of the penetration-determination test portion

5.1.3.1 General

Carry out the procedure for preparing a test sample for worked penetration (full-scale method) as described in Clause 7 of ISO 2137, and as given in 5.1.3.2 and 5.1.3.3.

5.1.3.2 Preparation of test sample

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

5.1.3.2.2 For the working of the grease, transfer a sufficient quantity of the laboratory sample to the cup of the clean grease worker to overfill it (mounded up approximately 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 to 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 Notes 1 and 2 and the last two paragraphs of this subclause) until the temperature of the grease worker and contents is 25 °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 double stokes 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 may readily be removed. As the worked penetration of a lubricating grease may change significantly on standing, proceed in accordance with 5.1.3.3 without delay.

NOTE 1 A constant temperature test room or an air bath may be used instead of a water bath.

NOTE 2 The penetration of soft greases is dependent upon the diameter of the container. Therefore, greases having unworked penetration 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 ± 0,5 °C is used, sufficient additional time should be allowed to ensure that the test portion is at 25 °C ± 0,5 °C before proceeding. In addition, if the sample is larger than 0,5 kg, sufficient additional time should be allowed to ensure that the test sample is at 25 °C ± 0,5 °C. Testing may proceed if the test portion is at a uniform temperature of 25 °C ± 0,5 °C.

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

5.1.3.3 Preparation of test portion

5.1.3.3.1 Prepare the worked test sample (5.1.3.2.2) in the cup for testing so that a uniform and reproducible structure of grease will be obtained.

5.1.3.3.2 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.

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.

5.1.3.3.3 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 removed.

5.1.3.3.4 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 may be returned to the cup prior to preparing the sample for the next test.

5.2 Conditioning

Place the temperature sensor (4.3) in the temperature-control test portion (prepared according to 5.1.2), positioning its end at the centre and at a depth of approximately 30 mm.

Place the two test portions (5.1.2 and 5.1.3) in the thermostatically controlled chamber (4.2) previously set to the test temperature; the time between smoothing the test sample and placing it in the chamber shall be less than 30 min.

Also insert the cone of the penetrometer in the chamber. Allow the assembly to remain at the test temperature for 4 h ± 5 min. At this time, the difference between the temperature of the grease and the test temperature shall not differ by more than 1 °C. If this is not so, repeat the test.

6 Penetration measurement

6.1 Place the penetrometer stand near the chamber (4.2). Make sure that the cone shaft is absolutely plumb (vertical) and free for sliding in the release mechanism.

6.2 Record the test temperature of the temperature-control portion, containing the temperature sensor, to the nearest 0,5 °C.

6.3 Remove the cone from the chamber quickly and fit it on the vertical shaft of the penetrometer, ensuring that the release mechanism is perfectly free. Then transfer the test sample from the chamber to the penetrometer stand.

Without smoothing the grease, carry out the penetration measurement immediately, as follows:

a) Place the stem of the dial gauge and that of the cone in contact, then set the dial gauge to the “zero” position. Carefully adjust the penetrometer so that the tip of the cone is just in contact with the centre of the surface of the test portion.

b) Release the shaft of the cone quickly and allow it to take effect for 5 s ± 0,1 s. Lower the stem of the dial gauge gently until it is stopped by the shaft of the cone and read the penetration on the scale of the indicator.

The time between the measurement and the removal from the chamber shall be less than 1 min in order to avoid too great a variation in the temperature of the grease and of the cone.

7 Expression of results

Report the penetration of the grease as the result obtained from the measured sample, rounded off to the nearest unit (0,1 mm).

8 Precision

8.1 General

The precision, as determined by statistical examination of interlaboratory test results involving 9 laboratories, on NLGI class 2 greases (see ISO 6743-99) at temperatures in the range from −40 °C to −20 °C and for penetrations varying in the range from 60 units to 210 units, is given in 8.2 and 8.3.

NOTE The precision applies to any grease of any NLGI grade at any temperature between 25 °C and −40 °C.

8.2 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 9 units, in only one case in 20.

8.3 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 28 units, in only one case in 20.

9 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 7);

d) the test temperature selected;

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

f) the date of the test.