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Petroleum products and lubricants - Determination of the dropping point of grease with an automatic apparatus

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Foreword

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This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The dropping point of lubricating grease is the temperature at which grease passes from a semi-solid state to a liquid state under test conditions.

This change of state is typical of greases that contain organic thickeners (soaps mainly) exhibiting phase changes and softening when the temperature is increased. Non-soap greases (like bentonite or silica greases) are described as “not fusible”, i.e. have no dropping point. Upon heating, they can separate oil.

Results of the dropping point test are indicative of the maximum temperature at which grease can be heated without complete liquefaction or oil separation. The dropping point is a useful indication of the grease type. For soap-based greases, it is related to the nature of the cation. At equivalent cation, complex soap-based greases have dropping points higher than that of simple soap-based greases. This characteristic can be used for manufacturing quality controls and for establishing product specifications.

The dropping point is not considered as having a direct relation with the service performance; dropping point is in no case the maximum temperature for use of grease.

Some cooperative testing has indicated that concordance exists between the results obtained using this document and the results obtained using ISO 2176[1] and ISO 6299[3].

This document is based on References [4] and [5].

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WARNING — Use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to application of the document, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document specifies a method for the determination of the dropping point of lubricating grease using an automatic apparatus.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
dropping point
temperature at which grease passes from a semi-solid state to a state sufficiently fluid to flow, under the effect of its own mass, through a calibrated orifice drilled at the bottom of a standardized cup, under the form of a drop

Note 1 to entry: When the dropping of the grease is not observed at temperatures less than 300 °C, it is considered as not having a dropping point.

4 Sampling

Unless otherwise specified in commodity specifications, samples of greases shall be drawn in accordance with ASTM D 4057.

5 Principle

A cup, containing the test grease, is inserted in a cup holder. The assembly is placed in an oven. The oven temperature is gradually increased, at a specified rate, until a drop of grease flows through the orifice present in the bottom of the cup. The temperature is recorded at the moment when the flow is detected by means of a photoelectrical cell.

6 Significance and use

The results of the present method are indicative of the maximum temperature at which grease can be heated without complete liquefaction or excessive oil separation.

7 Apparatus

7.1 Grease cup, shall have the dimensions specified in Annex A.

7.2 Cup plug gauge, shall have the dimensions specified in Annex A.

7.3 Calibrated rods, one with a diameter of 2,78 mm and one with a diameter of 2,82 mm.

7.4 Metal rod, 150 mm length, 1,2 mm to 1,6 mm diameter.

7.5 Cup holder, supplied with the equipment.

7.6 Measuring cell, capable of heating the cup/cup holder assembly in the range from 0 °C to 300 °C, with a precision of 0,5 °C on the whole temperature range, equipped with a cooling device and including a photoelectrical detector.

7.7 Control unit, capable of controlling the heating rate of the measuring cell in the range of 1 °C/min to 10 °C/min and showing the temperature of the cell with a precision of 0,1 °C throughout the heating duration up to the final dropping point.

8 Test procedure

8.1 Selection of the cup

Take a cup (7.1) conforming to the internal dimensions of the rounded edges of the cup plug gauge (7.2). Check the diameter of the bottom opening of the cup using, as gauges, the calibrated rods (7.3). The orifice shall allow the 2,78 mm rod to pass easily, but not the 2,82 mm rod. If the orifice is undersized, it shall be reamed to the correct size, or the cup shall be discarded. If the orifice is too large, the cup shall be discarded.

8.2 Filling of the cup

The cup selected in 8.1 is filled by pressing the larger cup opening into the grease container until the cup is filled. The excess of grease is removed with a spatula.

Gently press the cup, held in vertical position with the smaller opening at the bottom, down over the metal rod (7.4) until the latter protrudes approximately 25 mm.

Press the rod against the cup in such a manner that the rod makes contact at both the upper and the lower peripheries of the cup.

Maintain the contact, rotating the cup on the rod along the index finger to give a spiral-like motion down the rod to remove a conical section of the grease which adheres to the rod.

As the cup approaches the end of the rod, carefully slip the rod out of the cup so that a smooth film of grease, free of air bubbles and of reproducible thickness, remains inside the cup. The orifice of the cup shall not be obstructed with grease.

Place the filled cup in the cup holder (7.5).

8.3 Adjusting the apparatus

Adjust the apparatus following the instructions of the manufacturer.

8.4 Dropping point determination

8.4.1 When an approximate value of the dropping point is known, adjust the starting temperature 20 °C above the ambient temperature, while ensuring that this starting temperature is equal or below 50 °C. Place the cup/cup holder assembly in the measuring cell (7.6), conforming to the instructions of the equipment manufacturer.

When the temperature of the measuring cell has stabilised at the selected start temperature, commence heating at a rate of 10 °C/min until a temperature 20 °C below the expected drop point has been reached. At this point, reduce the heating rate to 1 °C/min until the drop point is detected.

8.4.2 When the dropping point of the grease is not known, an approximate result can be obtained using a temperature rise at a rate of 10 °C/min throughout the duration of the measure. Using the so obtained result, start the determination as indicated in 8.4.1.

8.4.3 When the dropping point is reached, (detection of the flow of a drop of grease by the photoelectrical cell), the apparatus stops automatically and indicates a temperature which is the dropping point.

NOTE For safety reasons, some equipment stops automatically when the temperature of 300 °C is reached, even if no drop flow has been detected.

9 Expression of the results

The results shall be expressed according to the temperature recorded in 8.4.3, in °C, rounded to the nearest °C.

10 Precision

10.1 General

The precision, as determined by statistical examination in accordance with ISO 4259-1[2] of interlaboratory test results, is given in 10.2 and 10.3.

10.2 Repeatability

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, within a short interval of time, under the same test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, can be calculated using Formula (1):

$$r = 1{,}214 \cdot 10^{-4} \cdot (x^2 + 7\,000) \quad (1)$$

where

x is the average of the two test results being compared.

10.3 Reproducibility

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, under different test conditions, that is