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Designation: ~~D4425 – 09 (Reapproved 2014)~~ D4425 – 19

Standard Test Method for Oil Separation from Lubricating Grease by Centrifuging (Koppers Method)[1]

This standard is issued under the fixed designation D4425; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. ~~Scope~~ Scope*

1.1 This test method covers a procedure for determining the tendency of lubricating grease to separate oil when subjected to high centrifugal forces.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D217 Test Methods for Cone Penetration of Lubricating Grease

iTeh Standards (https://standards.iteh.ai) Document Preview

3. Terminology

3.1 *Definitions:*

3.1.1 *lubricating grease, n*—a semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant.

3.1.1.1 *Discussion*—

ASTM D4425-19

The dispersion of the thickener forms a two-phase system and immobilizes the liquid lubricant by surface tension and other physical forces. Other ingredients are commonly included to impart special properties. **D217**

3.1.2 *thickener, n—in lubricating grease*, a substance composed of finely-divided particles dispersed in a liquid to form the products's structure.

3.1.2.1 *Discussion*—

Thickeners can be fibers (such as various metallic soaps) or plates or spheres (such as certain non-soap thickeners), which are insoluble or, at most, only very slightly soluble in the liquid lubricant. The general requirements are that the solid particles be extremely small, uniformly dispersed, and capable of forming a relatively stable, gel-like structure with the liquid lubricant.

3.2 *Symbols:*

a = distance from top of grease surface to tube mouth (mm).
b = height of liquid column in an inverted test tube (mm).

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[1] This test method is under the jurisdiction of Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.G0.03 on Physical Tests.

Current edition approved ~~Oct. 1, 2014~~June 1, 2019. Published ~~November 2014~~July 2019. Originally approved in 1984. Last previous edition approved in ~~2009~~2014 as ~~D4425 – 09~~.D4425 – 09 (2014). DOI: ~~10.1520/D4425-09R14.~~10.1520/D4425-19.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

***A Summary of Changes section appears at the end of this standard**

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d = test tube inside diameter (mm).
H = accumulated test time at a given reading (h).
$K36$ = resistance to centrifugal separation (V/H).
r = measured at the maximum radius of rotation (mm).
rpm = rotational speed (r/min).
V = volume of separated oil, as a percentage of the original grease volume (%).
V_g = grease volume in a test tube (cm^3).
V_o = volume of separated oil (cm^3).
V_t = test tube total volume (cm^3).
A = angle of rotor, between the test tube axis and axis of rotation (degrees).
ω = rotational speed (rad/s).
G = relative centrifugal acceleration.

3.3 The relative effect of centrifugal forces, when related to the gravitational standard acceleration ~~(9.81~~ (9.81 m ~~m/s~~/s^2), is noted with the symbol *G*. It can be calculated as follows:

$$G = 1.02 \times 10^{-4} \times r \times \omega^2 \quad (1)$$

or

$$G = 1.12 \times 10^{-6} \times r \times \overline{\text{rpm}}^2 \quad (2)$$

4. Summary of Test Method

4.1 Pairs of centrifuge tubes are charged with grease samples and are placed in the centrifuge. The grease samples are subjected to a centrifugal force equivalent to a *G* value of ~~36 000,~~36 000, at ~~50°C~~50 °C ± ~~1°C,~~1 °C, for specific periods of time. The resistance of the grease to separate the oil is then defined as a ratio of the percent of oil separated to the total number of hours of testing.

5. Significance and Use

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5.1 This test method is useful in evaluating the degree to which a grease would separate into fluid and solid components when subjected to high centrifugal forces. Flexible shaft couplings, universal joints, and rolling element thrust bearings are examples of machinery which subject lubricating greases to large and prolonged centrifugal forces. This test method has been found to give results that correlate well with results from actual service. The test method may be run at other conditions with agreement between parties but the precision noted in this test method will no longer apply.[3]

6. Apparatus

6.1 *High-Speed Centrifuge,* capable of developing a *G* value of ~~36 000.~~36 000. Mount the unit on a flat level surface to allow unrestricted air flow to the motor. This is essential for long motor life. The centrifuge should be equipped with:

ASTM D4425-19
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6.1.1 *Fixed Angle Rotor,* multiple place, which can sustain a *G* value of ~~36 000.~~36 000.

6.1.2 *Thermometer,* preferably of a dial type, installed so that the temperature in the vicinity ~~(5~~(5 mm to ~~15 mm)~~ 15 mm) of the rotor can be measured.

6.1.3 *Air Choke,* installed at the air inlet of the centrifuge chamber, and used to control the temperature if the unit lacks an automatic temperature control. Some designs require outlet choking as well.

6.1.4 *Centrifuge Tubes,* made of transparent material, capable of withstanding a *G* value of ~~36 000~~36 000 for ~~100 h~~ 100 h minimum (Note 1).

NOTE 1—Polypropylene tubes were found to be the most durable.

6.2 *Balance,* having a capacity of about ~~100 g~~ 100 g with a minimum sensitivity of ~~0.1 g.~~ 0.1 g.

7. Sampling

7.1 The sample presented for analysis should be large enough to make possible the selection of a representative portion for testing. Each run will require approximately ~~0.5 g~~ 0.5 g for each cubic centimetre of tube capacity . Examine for any indication of non-homogeneity such as oil separation, phase changes, or gross contamination. If any abnormal conditions are found, obtain a new sample.

7.2 The sample temperature at time of loading is to be between ~~15°C~~15 °C and ~~35°C.~~35 °C.

8. Preparation of Apparatus

8.1 Inspect the centrifuge unit paying particular attention to the cleanliness of the rotor which will be unbalanced by any surface deposits.

[3] Detailed discussion is found in Calistrat, M. M., Grease Separation under Centrifugal Forces, ASME Paper 75-PTG-3. Presented at the Joint ASLE-ASME Lubrication Conference, Oct. 21–23, 1975.

8.2 Examine the required number of tubes to be used for the test, rejecting any with surface scratches or imperfections.

9. Procedure

9.1 For each grease, two centrifuge tubes are required. New tubes must be used for each test and they must be handled with care to avoid scratches.

9.1.1 Determine the total volume, *V*, in cubic centimetres, of each tube by filling with water and then pouring into a graduated cylinder and measuring.

9.1.2 Measure the inside diameter, *d*, in millimetres with a vernier caliper.

9.2 Take grease samples from the container without including any free oil found on the grease surface.

9.3 Charge each tube with approximately ~~0.5 g~~ 0.5 g of grease for each cubic centimeter of tube capacity (example: ~~7 g~~ 7 g of grease in a tube of ~~14 cm~~14 cm^3) taking care that the difference in mass of each does not exceed ~~0.3 g~~ 0.3 g to minimize centrifuge imbalance.

9.4 Place the tubes in diametrically opposite compartments if all rotor compartments are not used. Always use even numbers of tubes.

9.5 The centrifuge lid must always be closed when the rotor is turning. The rotor should never be touched while rotating.

9.6 Operate the centrifuge at a *G* value of 1000 for ~~3 min~~ 3 min to eliminate any trapped air bubbles in the grease charge.

9.7 Measure the distance, *a*, in millimetres from the top of the test tube to the closest point on the grease surface as shown in Fig. 1, and calculate the grease volume as in 10.1.

9.8 Replace the tubes in the rotor head, close the lid, and bring the speed up to a relative acceleration, *G*, of 36 000. Consult manufacturer's instructions for proper speed.

9.9 At the end of the test interval, the rotor must come to a complete stop before opening the lid.

9.10 Measure the amount of oil separated as follows:

9.10.1 Cover the mouth of the tube with a piece of hard rubber or plastic material and invert for ~~1~~1 min to ~~2 min~~ 2 min or until all the oil flows to the top of the tube.

9.10.2 Measure the height, *b*, of the separated oil in millimetres, as shown in Fig. 2.

9.11 Calculate the oil volume as indicated in 10.2.

9.12 Calculate the percent of oil separated from the grease as indicated in 10.3.

9.13 If the test is to be continued, return the tubes to their respective compartments in the centrifuge rotor. When the covering over the tube mouth is removed all adhering oil must be returned to the tubes.

9.14 Measure the amount of oil separation after one or more of the following time periods: 6, 12, 24, 48, or 96 cumulative hours of testing at a *G* value of ~~36 000.~~36 000. No more than ~~72 h~~ 72 h of interruption is acceptable between two test periods.

9.15 The normal duration of a test is 24 cumulative hours.

9.15.1 The test can be extended to ~~48~~48 h or ~~96 h~~ 96 h if desired, but special reporting conditions prevail (see 11.4.2).

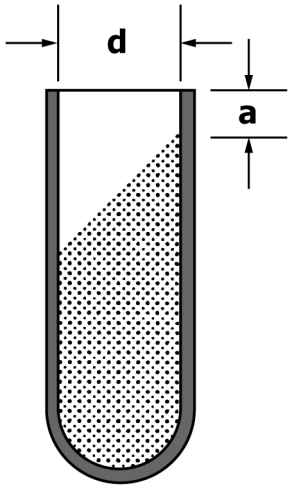

FIG. 1 Measuring Grease Volume