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An American National Standard

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

This standard is issued under the fixed designation D 4425; 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

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

1.2 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

1.3 The values stated in SI units are to be regarded as the standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 217 Test Methods for Cone Penetration of Lubricating Grease[2]

3. Terminology

3.1 *Definitions:*

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

3.1.1.1 *Discussion*—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. **D 217**

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).
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.

The relative effect of centrifugal forces, when related to the gravitational standard acceleration (9.81 m/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, at 50°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

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

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[1] This test method is under the jurisdiction of Committee D02 on Petroleum Products and is the direct responsibility of Subcommittee D02.G on Lubricating Grease.

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[2] *Annual Book of ASTM Standards*, Vol 05.01.

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. 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:

6.1.1 *Fixed Angle Rotor*, multiple place, which can sustain a *G* value of 36 000.

6.1.2 *Thermometer*, preferably of a dial type, installed so that the temperature in the vicinity (5 to 15 mm) of the rotor can be measured. (For Sorvall centrifuge, see Fig. 1.)

6.1.3 *Air Choke*, installed at the air inlet of the centrifuge chamber (for Sorvall centrifuge, see Fig. 2), 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 for 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 with a minimum sensitivity of 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 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 and 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.

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

[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.

FIG. 1 Installation of Dial Thermometer (Sorvall Centrifuge)

FIG. 2 Choking Air Inlet of Chamber (Sorvall Centrifuge)

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 of grease for each cubic centimeter of tube capacity (example: 7 g of grease in a tube of 14 cm^3) taking care that the difference in mass of each does not exceed 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 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. 3, 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 to 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. 4.

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.

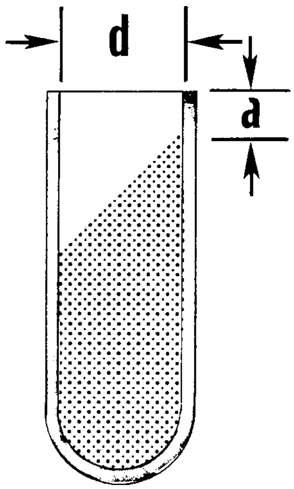

FIG. 3 Measuring Grease Volume

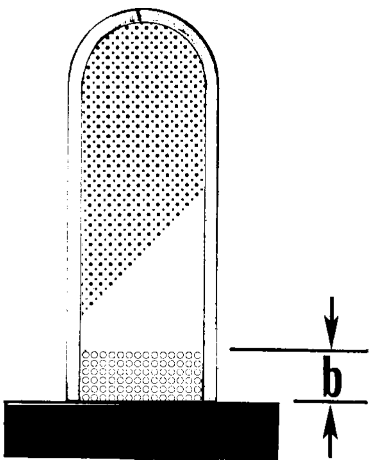

FIG. 4 Measuring Separated Oil

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. No more than 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 or 96 h if desired, but special reporting conditions prevail (see 11.4.2).

10. Calculation

10.1 Calculate the grease volume to the nearest cm ^{3} as follows:

$$V_g = V_t - \left[\left(a + \frac{d}{2 \tan A} \right) (0.785\, d^2) \times 10^{-3} \right] \quad (3)$$

where:
V_g = grease volume, cm^3,
V_t = tube volume, cm^3 (see 9.1.1),
a = distance to grease surface, mm (see 9.7),
d = tube inside diameter, mm (see 9.1.2), and
A = angle of the rotor, in degrees. This angle is specified by the rotor manufacturer.[4]

10.2 Calculate the oil volume to the nearest cm ^{3} as follows:

$$V_o = 0.785\, d^2\, b \times 10^{-3} \quad (4)$$

where:
V_o = separated oil volume, cm^3, and
b = height of the separated oil, mm (see 9.10).

10.3 Calculate the amount of oil separated from the grease to the nearest percent as follows:

$$V = \frac{V_o}{V_g} \times 100 \quad (5)$$

where
V = oil separation, in volume percent.

11. Report

11.1 The amount of oil separated from a lubricating grease subjected to centrifugal forces is a function of the *G* level, the amount of time under test,[2] and the test temperature. Thus, to evaluate the resistance of a grease to separate the oil, the following data are required:

11.1.1 The oil separation, in volume percent (see 10.3).

11.1.2 The level of centrifugal acceleration, in *G* (see 3.1).

11.1.3 The duration of the test, in hours.

11.1.4 The test temperature, in °Celsius.

11.2 In order to simplify the testing and reporting of the oil separation from lubricating greases by centrifuging, the level of centrifugal acceleration and the test temperature are maintained constant, at a *G* value of 36 000 and 50°C. Hence, the resistance of a grease to separate the oil under centrifugal forces is reported as the fraction *K36*.

$$K36 = V/H \quad (6)$$

where:
V = oil separation, in volume percent (see 10.3), and
H = accumulated time of testing in hours (see 9.13).

The fraction *K36* should not be reduced, and the values of both *V* and *H* should be reported.

11.3 The results of two tubes filled with the same grease (see 9.3) are averaged for reporting purposes.

11.4 The values for *V* and *H* in Eq 6 are those at the time the oil separation stabilizes. This is defined as the time at which the oil separation increases by less than 10 % between two consecutive readings.

11.4.1 If the oil separation does not stabilize before the 24 h test, the 24 h test results are used for reporting.

11.4.2 If the testing is extended beyond the 24 cumulative hours, the same conditions in 11.4 apply, except more than 24 h can be used. Examples of calculations can be found in Appendix X1.

12. Precision and Bias [4]

12.1 *Precision*—When run under the conditioins stated in the test method, the precision, as determined by statistical examination of interlaboratory results is as follows:

[4] Supporting data are available from ASTM International Headquarters: Request RR:D02-1185.

12.1.1 *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 long run, in the normal and correct operation of the test method, exceed 4.8 percentage points (of the value of *V*, 10.3) only in one case in twenty.

12.1.2 *Reproducibility*—The difference between two single and independent results, obtained by different laboratories on identical test material, would in the long run, in the normal and correct operation of test method, exceed 18.7 percentage points (of the value *V*, 10.3) only in one case in twenty.

12.2 *Bias*—The procedure in this test method has no bias because the value of K36 can be defined only in terms of the test method.

13. Keywords

13.1 centrifuge; grease; lubricating grease; oil separation

APPENDIX

(Nonmandatory Information)

X1. EXAMPLES OF CALCULATIONS

X1.1 *Examples for 24 h Tests:*

X1.1.1

Cumulative hours, *H*	6	12	24
Oil separation, *V*	30	32	32

K36 = 30/6, because *V* increased by less than 10 % between the 6 and 12 h tests.

X1.1.2

Cumulative hours, *H*	6	12	24
Oil separation, *V*	30	35	38

K36 = 35/12, because *V* increased by 17 % between the 6 h and 12 h test, but only 8.5 % between the 12 and 24 h tests.

X1.1.3

Cumulative hours, *H*	6	12	24
Oil separation, *V*	30	35	42

K36 = 42/24, because the oil separation did not stabilize before the 24 h test.

X1.2 *Examples for Tests Exceeding 24 h:*

X1.2.1

Cumulative hours, *H*	6	12	24	48	96
Oil separation, *V*	30	35	42	44	44

K36 = 42/24, because *V* increased by less than 10% between the 24 h and 48 h tests.

X1.2.2

Cumulative hours, *H*	6	12	24	48	96
Oil separation, *V*	30	35	42	47	48

K36 = 47/48, because *V* increased by less than 10% between the 48 h and 96 h tests.

X1.2.3

Cumulative hours, *H*	6	12	24	48	96
Oil separation, *V*	30	35	42	47	53

K36 = 53/96, because the oil separation did not stabilize before the 96 h test.