

Designation: D7342 - 07

Standard Test Method for Shear Stability of Lubricating Grease in Presence of Water (Water Stability Test)¹

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1. Scope

- 1.1 This test method covers two procedures for determining the shear stability of lubricating grease in the presence of water (wet shear stability) by a full scale grease worker or a roll stability test apparatus. Both procedures can be used to determine the relative wet shear stability of greases, but the results between procedures are not directly comparable. This test method is also known as the water stability test.
- 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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 ASTM Standards:²

D217 Test Methods for Cone Penetration of Lubricating Grease

D1403 Test Methods for Cone Penetration of Lubricating Grease Using One-Quarter and One-Half Scale Cone Equipment

D1831 Test Method for Roll Stability of Lubricating Grease D1193 Specification for Reagent Water

3. Terminology

- 3.1 Definitions:
- 3.1.1 *consistency*, *n*—*of lubricating grease*, degree of resistance to movement under stress.
- 3.1.1.1 *Discussion*—The term *consistency* is used somewhat synonymously with *penetration*. Generally, consistency

refers to the worked penetration of a grease. **D217**

- 3.1.2 *lubricating grease*, *n*—semi-fluid to solid product of a dispersion of a thickener in a liquid lubricant.
- 3.1.2.1 *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.

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- 3.1.3 *penetration*, *n*—*of lubricating grease*, depth that the standard cone, when released to fall under its own weight for 5 s, enters the sample.

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- 3.1.4 *thickener*, *n*—*in lubricating grease*, substance composed of finely divided particles dispersed in a liquid lubricant to form the product's structure.
- 3.1.4.1 *Discussion*—The thickener can be fibers (such as various metallic soaps) or plates or spheres (such as certain non-soap thickener) which are insoluble or, at most, only very slightly soluble in liquid lubricant. The general requirements are that the solid particles to be relatively stable, gel-like structure with the liquid lubricant.

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- 3.1.5 wet shear stability, n—of lubricating grease, change in consistency of a mixture of sample and small amount of water after a specified amount of working in a grease worker or a roll stability test apparatus.
- 3.1.6 worked penetration, n—of lubricating grease, the penetration at 25°C, without delay, of a sample after 60 double strokes in a standard grease worker.

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- 3.1.7 working, n—of lubricating grease, subjection of a sample to the shearing action of the standard grease worker.

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4. Summary of Test Method

4.1 A grease sample mixed with a small amount of water is subjected to low shear at 20 to 35°C for a specified time or strokes in a standard grease worker (Procedure A) or a roll stability apparatus (Procedure B). The difference between the cone penetration before working and the cone penetration after is used as a measure of the wet shear stability of the grease.

5. Significance and Use

5.1 It is known that contamination by water can affect the shear stability of some greases in service. Both test procedures specified in this method are widely used to determine the wet shear stability of greases in service. Many grease specifications

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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.

require these procedures as a wet shear stability test. No accurate correlation is established between the test results and wet shear stability of grease in actual service.

6. Apparatus

- 6.1 *Motorized Grease Worker*, as specified in Test Methods D217.
- 6.2 Roll Stability Test Apparatus, as specified in Test Method D1831.
 - 6.3 Penetrometer, as specified in Test Methods D217.
- 6.4 One-Quarter or One-Half Scale Cone and Shaft with Worker, as described in Test Methods D1403.
 - 6.5 Electric Kitchen-type mixer, Low speed of 500 rpm.
 - 6.6 Suitable Mixing Bowl.

7. Reagents and Materials

- 7.1 Appropriate Volatile Gum-free Solvent, for example, mineral spirit solvent.
- 7.2 *Cloth or Paper Wiper*, for wiping grease from the penetrometer cone. The wiper should be soft, so as not to scratch the cone.
- 7.3 *Distilled Water*, Specification D1193, Type II minimum purity.

8. Procedure

Procedure A-Wet Prolonged Worked Test

- 8.1 Measure the worked penetration for the grease to be tested according to Test Methods D217.
- 8.2 For each determination, prepare a homogeneous mixture of grease and water by placing 450 ± 1 g of grease and 50 ± 1 g of distilled water in a suitable mixing bowl using an electric kitchen-type mixer or mixing by hand in a suitable container
- 8.2.1 Gradually add the calculated amount of distilled water to the grease in the bowl.
- 8.2.2 Mix the contents for 4 ± 1 min. A spatula may be used to channel the constituents to the mixer.
- 8.2.3 The objective of foregoing sections is to produce a mixture of nine parts of grease and one part of water that transfer to a worker cup.

Note 1—If free water is observed after mixing, then additional mixing is required.

- 8.3 Fill a standard Test Methods D217 grease worker with the mixture and work the sample on a motorized grease worker for 100 000 double strokes (approximately 28 h). After working is completed, bring the grease worker and the sample to 25 \pm 0.5°C in accordance with the procedure described in Test Methods D217 for prolonged worked penetration.
- 8.4 Immediately after the grease sample reaches 25 \pm 0.5°C, rework the sample an additional 60 double strokes. Determine the worked penetration.

Procedure B—Wet Shell Roll Test

- 8.5 Determine the worked penetration of the grease to be tested in accordance with Test Methods D1403.
- 8.6 Transfer 63.0 ± 0.2 g of unworked grease to test cylinder. Distribute the grease uniformly on the inside wall of the cylinder with a spatula.

- 8.7 Place the weighed roll in the cylinder.
- 8.8 Add 7.0 \pm 0.2 g distilled water to the cylinder, and tighten the cap.
- 8.9 Mount the cylinder in position, start the machine, and record the time and room temperature which should be limited to 20 to 35°C. If the cylinder is enclosed within a cabinet, the temperature around cylinder shall be maintained at 20 to 35°C.
- 8.10 After rolling the cylinder for $2 \text{ h} \pm 5 \text{ min}$, remove the grease from the cylinder promptly and proceed with the requirements of worked penetration in Test Methods D1403. Record the worked penetration. After transferring the grease to the worker, clean the test apparatus by wiping with clean cloth or tissue.
- 8.11 Convert the fractional scale penetration values determined by Test Methods D1403 (before and after test) into the equivalent full scale cone penetration values using the appropriate equations described in Test Methods D1403.

9. Calculation

9.1 Calculate the change in consistency of the sample as follows:

Penetration change =
$$P_2 - P_1$$
 (1)

where:

 P_2 = final full-scale penetration reading, and

 P_1 = initial full-scale penetration reading.

Note 2—Penetration reading is measured in tenths of a millimetre. A negative penetration change indicates hardening of grease while a positive penetration change indicates softening.

10. Report

10.1 The value calculated in 9.1 is reported with the test procedure as the wet shear stability of the grease. For Procedure B, a notation if free water is present is useful information.

11. Precision and Bias

- 11.1 *Precision*—The precision of this test method as determined by a statistical examination of interlaboratory test results shown in Tables 1 and 2. Their round robin samples are listed in Table 3.
- 11.1.1 Repeatability—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in normal and correct operation of the test method exceed the following values only in one case in twenty:

Procedure A 20 units Procedure B 18 units

11.1.2 *Reproducibility*—The difference between two single and independent 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 the following values only in one case in twenty:

Procedure A 26 units Procedure B 31 units

TABLE 1 Round Robin Test Results for Procedure A

Lab	Sample					
	S1	S2	S3	S4	S5	
1	+25	+122	+100	+55	+29	
'	+24	+126	+103	+60	+32	
2	+11	+117	+88	+72	+25	
2	0	+112	+90	+71	+28	
0	+154	+107	+97	+62	+24	
3	+159	+118	+99	+53	+7	
4	+1	+120	+97	+53	+27	
4	+6	+123	+96	+80	+27	
	+29	+115	+86	+54	+28	
5	+12	+114	+109	+63	+28	
6	+25	+95	+99	+49	+25	
б	+20	+88	+104	+44	+31	
7	+11	+117	+79	+48	+25	
7	-2	+89	+92	+61	+23	
0	0	+117	+96	+62	+36	
8	+3	+122	+88	+68	+33	
0	+11	+118	+100	+59	+18	
9	+17	+113	+87	+64	+20	
10	+11	+108		+70	+32	
· -	+10	+91		+67	+37	

TABLE 2 Round Robin Test Results for Procedure B

Sample					
S1	S2	S3	S4	S5	
-3	+32	+64	+40	+24	
+2	+16	+68	+39	+23	
+4	+38	+54	+52	+18	
+3	+48	+60	+37	+26	
+26	-12	+52	+22	+3	
+12	0	+50	+38	-2	
-18	+26	+38	+37	+2	
-21	+29	+39	+42	+27	
+8	+64	+58	+40	+24	
+4	+26	+60	+36	+20	
-6	+44	+55	+36	+20	
-2	+37	+58	+34	+24	
-12	+60	+30	+32	+12	
-4	+60	+52	+26	+10	
+2	+42	+58	+40	+20	
0	+50	+62	+44	+22	
+30	+79	+41	+26	+19	
+19	+90	+33	+22	+22	
+2	+50		+20	+10 +12	
	-3 +2 +4 +3 +26 +12 -18 -21 +8 +4 -6 -2 -12 -4 +2 0 +30 +19	-3 +32 +16 +4 +16 +4 +38 +3 +48 +26	S1 S2 S3 -3 +32 +64 +2 +16 +68 +4 +38 +54 +3 +48 +60 +26 -12 +52 +12 0 +50 -18 +26 +38 -21 +29 +39 +8 +64 +58 +4 +26 +60 -6 +44 +55 -2 +37 +58 -12 +60 +30 -4 +60 +52 +2 +42 +58 0 +50 +62 +30 +79 +41 +19 +90 +33 +2 +50	S1 S2 S3 S4 -3 +32 +64 +40 +2 +16 +68 +39 +4 +38 +54 +52 +3 +48 +60 +37 +26 -12 +52 +22 +12 0 +50 +38 -18 +26 +38 +37 -21 +29 +39 +42 +8 +64 +58 +40 +4 +26 +60 +36 -6 +44 +55 +36 -2 +37 +58 +34 -12 +60 +30 +32 -4 +60 +52 +26 +2 +42 +58 +40 0 +50 +62 +44 +30 +79 +41 +26 +19 +90 +33 +22 +2 +50 +20	

11.2 *Bias*—The interlaboratory tests confirm that there is a significant bias between Procedure A and Procedure B in this test method. Both procedures can be used to determine the

relative wet shear stability of greases, but the results between procedures are not directly comparable.



TABLE 3 Round Robin Samples

Sample Code	NLGI Grade	Thickener Type
S1	2	Al-complex
S2	3	Lithium
S3	2	Polyurea
S4	1	Lithium
S5	1	Li-complex

12. Keywords

12.1 cone penetration; consistency; grease worker; lubricating grease; penetration; roll stability; water stability test; wet shear stability

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