



Standard Test Method for Lithium and Sodium in Lubricating Greases by Flame Photometer¹

This standard is issued under the fixed designation D 3340; 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 covers determination of the lithium and sodium content of lubricating greases by means of a flame photometer.

1.2 A multi-element analysis method for greases, which includes the measurement of lithium and sodium concentrations using inductively couple plasma-atomic emission spectroscopy (ICP-AES), is available in Test Method **D 7303**.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only. The preferred units are mass %.

1.4 *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:*

D 1193 Specification for Reagent Water²

D 6299 Practice for Applying Statistical Quality Assurance Techniques to Evaluate Analytical Measurement System Performance

D 6792 Practice for Quality System in Petroleum Products and Lubricants Testing Laboratories

D 7303 Test Method for Determination of Metals in Lubricating Greases by Inductively Coupled Plasma Atomic Emission Spectrometry

3. Summary of Test Method

3.1 The sulfated ash of the grease is extracted with water and the lithium and sodium content of the resulting solution is determined by means of a suitable flame photometer.

¹ This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

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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.

4. Significance and Use

4.1 Lubricating greases can contain the lithium soap of hydroxy stearic acid or the sodium soap of various fatty acids as thickeners. The determination of total lithium or total sodium is a measure of the amount of thickener in the grease.

5. Apparatus

5.1 *Flame Photometer*, suitably equipped to determine lithium and sodium over a range from 0 to 15 mg/L lithium and from 0 to 5 mg/L sodium.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification **D 1193**.

6.3 *Lithium Sulfate, Stock Solution*—1.188 g of lithium sulfate dried to constant weight at 180°C (356°F), is dissolved in water and made up to 1 L. This will contain 150 mg/L lithium.

6.4 *Sodium Sulfate, Stock Solution*—0.1544 g of sodium sulfate, dried to constant weight at 105°C (221°F), is dissolved in water and made up to 1 L. This will contain 50 mg/L sodium.

6.5 *Sulfuric Acid (rel. den. 1.84)*—Concentrated sulfuric acid (H₂SO₄).

7. Sampling

7.1 A homogeneous sample shall be obtained to ensure quantitative analytical results.

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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

8. Procedure

8.1 Weigh to the nearest 1 mg approximately 1 g of the grease into a platinum dish or crucible. Heat the dish until the sample can be ignited with a flame. Maintain at such a temperature that the sample burns at a uniform and moderate rate, leaving only ash and carbon when the burning ceases.

8.2 Cool the dish and completely moisten the residue by the dropwise addition of concentrated sulfuric acid. Heat the dish at a low temperature, taking care to avoid spattering of the contents, and continue heating until fumes are no longer evolved. Place the dish in a furnace at $550 \pm 25^\circ\text{C}$ ($1022 \pm 45^\circ\text{F}$) and continue heating until the oxidation of the carbon is practically complete.

8.3 Cool and repeat 8.2 to complete the sulfation.

8.4 Extract the sulfated ash with hot distilled water and transfer the aqueous extract, filtering if necessary, to a 100-mL volumetric flask. Allow to cool and make up to the mark with distilled water.

8.5 Prepare the following fresh calibration standards by appropriate dilution of the stock solutions:

8.5.1 *Lithium Sulfate*—Solutions containing 0, 3, 6, 9, 12 and 15 mg/L lithium.

8.5.2 *Sodium Sulfate*—Solutions containing 0, 1, 2, 3, 4, and 5 mg/L sodium.

8.6 Obtain the flame photometer readings for each calibration standard and prepare curves relating concentration to photometer reading.

8.7 Obtain readings for the extract derived from 8.4 and determine the lithium and sodium contents from the calibration curves.

NOTE 1—Further dilution may be necessary.

9. Quality Control

9.1 Confirm the performance of the test procedure by analyzing a quality control (QC) sample that is stable and representative of the sample of interest.

9.1.1 When the quality control/quality assurance (QA) protocols are already established in the testing facility, these may be used to confirm the reliability of the test results.

TABLE 1 Precision Values for Lithium and Sodium (Mass %)

Lithium, <i>x</i>	Repeatability $0.014(x)^{2/5}$	Reproducibility $0.075(x)^{2/5}$
0.01	0.002	0.011
0.05	0.004	0.022
0.1	0.005	0.029
0.2	0.007	0.039
0.3	0.008	0.046
0.4	0.009	0.052
Sodium, <i>x</i>	Repeatability $0.029(x)^{2/5}$	Reproducibility $0.166(x)^{2/5}$
0.01	0.004	0.026
0.05	0.008	0.050
0.1	0.011	0.066
0.2	0.015	0.087
0.3	0.017	0.102
0.4	0.020	0.115
0.5	0.022	0.125
0.6	0.023	0.135
0.7	0.025	0.143
0.8	0.026	0.151

9.1.2 When there is a quality control/quality assurance protocol established in the testing facility, Appendix X1 can be used to perform this function.

10. Calculation and Report

10.1 Calculate the lithium content of the grease as follows:

$$\text{Lithium, mass \%} = X/100 W \quad (1)$$

where:

X = lithium content of the water extract, mg/L lithium (see 8.7), and

W = mass of the grease taken, g.

Modify the formula if further dilution of the water extract was necessary, for example, if further dilution of 50 mL to 200 mL was made, then:

$$\text{Lithium, mass \%} = (X)(200)/(100 W)(50) \quad (2)$$

10.2 Report the result as lithium content.

10.3 Calculate the sodium content of the grease as follows:

$$\text{Sodium, mass \%} = Y/100 W \quad (3)$$

where:

Y = sodium content of the water extract in mg/L sodium (see 8.7), and

W = mass of the grease taken, g.

Modify the formula if further dilution of the water extract was necessary as in 10.1.

10.4 Report the result as sodium content.

11. Precision and Bias

NOTE 2—The precision data were derived within the IP. The method has not as yet been cooperatively tested by ASTM.

11.1 The precision of this test method as determined by statistical examination of interlaboratory results is as follows:

11.1.1 *Lithium*:

11.1.1.1 *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 the following values only in one case in twenty:

11.1.1.2 Lithium $0.014(x)^{2/5}$.

11.1.1.3 Sodium $0.029(x)^{2/5}$.

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:

11.1.2.1 Lithium $0.075(x)^{2/5}$.

11.1.2.2 Sodium $0.166(x)^{2/5}$, where *x* is the lower of two values.

NOTE 3—The applicable range of concentrations are 0 to 0.4 % mass/mass lithium and 0 to 8 % mass/mass sodium.

NOTE 4—These precision values have been obtained by statistical examination of interlaboratory test results by the Institute of Petroleum and were first published in 1966. They were obtained from the test results from nine laboratories using the following instruments:

No. of
Laborato-
ries
6
2
1

“EEL” Flame Photometer.
SP 900 Unicam (modified).
H 700 Uvispek spectrophotometer
(modified)

11.2 *Bias*—The bias of this test method cannot be determined because no suitable reference material is available.

NOTE 5—The values of these precision estimates for selected values of x are set out in the [Table 1](#).

12. Keywords

12.1 flame photometer; lithium content; lubricating grease; sodium content; thickener

APPENDIX

(Nonmandatory Information)

X1. QUALITY CONTROL MONITORING

X1.1 Confirm the performance of the instrument or the test procedure by analyzing quality control (QC) sample(s).

X1.2 Prior to monitoring the measurement process, the user of this test method needs to determine the average value and control limits of the QC sample (see Practices [D 6299](#) and [D 6792](#) and ASTM MNL 7⁴).

X1.3 Record the QC results and analyze by control charts or other statistically equivalent techniques to ascertain the statistical control status of the total testing process (see Practices [D 6299](#) and [D 6792](#) and ASTM MNL 7⁴). Investigate any out-of-control data for root cause(s). The results of this investigation may, but not necessarily, result in instrument recalibration.

X1.4 The frequency (see [Note X1.1](#)) of QC testing is dependent on the criticality of the quality being measured, the

demonstrated stability of the testing process, and customer requirements. Generally, a QC sample should be analyzed each testing day with routine samples. The QC frequency should be increased if a large number of samples are routinely analyzed. However, when it is demonstrated that the testing is under statistical control, the QC testing frequency may be reduced. The QC sample testing precision should be periodically checked against the ASTM method precision to ensure data quality (see Practices [D 6299](#) and [D 6792](#) and ASTM MNL 7⁴).

NOTE X1.1—In the absence of explicit requirements given in the test method, this clause provides guidance on QC testing frequency.

X1.5 It is recommended that, if possible, the type of QC sample that is regularly tested be representative of the material routinely analyzed. An ample supply of QC sample material should be available for the intended period of use, and it must be homogenous and stable under the anticipated storage conditions.

X1.6 See Practices [D 6299](#) and [D 6792](#) and ASTM MNL 7⁴ for further guidance on QC and Control Charting techniques.

⁴ ASTM MNL 7, *Manual on Presentation of Data Control Chart Analysis*, 6th ed., ASTM International, W. Conshohocken, PA.

SUMMARY OF CHANGES

Subcommittee D02.03 has identified the location of selected changes to this standard since the last issue (D 3340–98(2003)) that may impact the use of this standard.

(1) Added Test Method [D 7303](#) and Practices [D 6299](#) and [D 6792](#) to the standard text and the Referenced Documents.

(2) Added quality control information in new Section [9](#) and [Appendix X1](#).

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