Designation: D7596 – 14

Standard Test Method for Automatic Particle Counting and Particle Shape Classification of Oils Using a Direct Imaging Integrated Tester[1]

This standard is issued under the fixed designation D7596; 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 the determination of particle concentration, particle size distribution, particle shape, and soot content for new and in-service oils used for lubrication and hydraulic systems by a direct imaging integrated tester.

1.1.1 The test method is applicable to petroleum and synthetic based fluids. Samples from 2 to 150 mm^2/s at 40°C may be processed directly. Samples of greater viscosity may be processed after solvent dilution.

1.1.2 Particles measured are in the range from 4 µm to ≥ 70 µm with the upper limit dependent upon passing through a 100 µm mesh inlet screen.

1.1.3 Particle concentration measured may be as high as 5 000 000 particles per mL without significant coincidence error.

1.1.4 Particle shape is determined for particles greater than approximately 20 µm in length. Particles are categorized into the following categories: sliding, cutting, fatigue, nonmetallic, fibers, water droplets, and air bubbles.

1.1.5 Soot is determined up to approximately 1.5 % by weight.

1.1.6 This test method uses objects of known linear dimension for calibration.

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]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D2896 Test Method for Base Number of Petroleum Products by Potentiometric Perchloric Acid Titration
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4175 Terminology Relating to Petroleum, Petroleum Products, and Lubricants
D4177 Practice for Automatic Sampling of Petroleum and Petroleum Products
D5185 Test Method for Multielement Determination of Used and Unused Lubricating Oils and Base Oils by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
D5967 Test Method for Evaluation of Diesel Engine Oils in T-8 Diesel Engine
D6304 Test Method for Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration
D6595 Test Method for Determination of Wear Metals and Contaminants in Used Lubricating Oils or Used Hydraulic Fluids by Rotating Disc Electrode Atomic Emission Spectrometry
D7279 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids by Automated Houillon Viscometer
E2412 Practice for Condition Monitoring of In-Service Lubricants by Trend Analysis Using Fourier Transform

---

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.96 on In-Service Lubricant Testing and Condition Monitoring Services.

Current edition approved June 1, 2014. Published July 2014. Originally approved in 2010. Last previous edition approved in 2010 as D7596 – 10. DOI: 10.1520/D7596-14.

[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**

Infrared (FT-IR) Spectrometry

G40 Terminology Relating to Wear and Erosion

2.2 *ISO Standards:*[3]

ISO 12103-1 1997 Road Vehicles—Test Dust for Filter Evaluation—Part 1: Arizona Test Dust

ISO 4406 Hydraulic Fluid Power—Fluids—Method for Coding Level of Contamination by Solid Particles

2.3 *SAE Standards:*[4]

SAE AS 4059 Aerospace Fluid Power – Cleanliness Classification for Hydraulic Fluids

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *contaminant particles, n*—particles introduced from an extraneous source into the lubricant of a machine or engine.

3.1.2 *direct imaging integrated tester, n*—an instrument for counting particles as they flow through a cell by means of imaging; instrument may also determine particle shape and fluid viscosity.

3.1.3 *ISO Codes, n*—standard method for coding the level of contamination by solid particles. This code simplifies the reporting of particle count data by converting the number of particles per mL into three classes covering ≥4 µm, ≥6 µm, and ≥14 µm. ISO 4406 classifications are used as an option to report results for this test method.

3.1.4 *new oil, n*—oil taken from the original manufacturer's packaging, prior to being added to the machinery. **E2412**

3.1.5 *particle size, circular diameter, µm, n*—diameter of a circle with an area equivalent to the projected area of a particle passing through the direct imaging integrated tester flow cell.

3.1.6 *soft particles, n*—particles present in the sample that are related to undissolved oil additives or additive by-products. Without dilution, at room temperature these particles are likely to be counted by an optical particle counter in a similar manner to dirt and wear metal particles, air bubbles, and free water droplets. They are not considered contaminants as they are either purposefully left undissolved, or are not harmful to the fluid system, or both.

3.1.7 *soot, n—in internal combustion engines*, sub-micron size particles, primarily carbon, created in the combustion chamber as products of incomplete combustion. **D4175**

3.1.8 *wear, n*—damage to a solid surface, usually involving progressive loss or displacement of material, due to relative motion between that surface and a contacting substance or substances. **D4175, G40**

3.1.9 *wear particles, n*—particles generated from wearing surfaces of a machine or engine.

4. Summary of Test Method

4.1 Lubricant samples are acquired periodically from a machine or engine being monitored. Samples are taken using clean receptacles in order to avoid altering the sample by method or container.

4.2 Particles are counted and sized by processing a sample through an appropriate particle sizing instrument. Sample size is instrument dependent. The instrument determines the size and shape of each particle detected in the sample as described in Section 1 of this test method. Adjustable cell gap instruments are set at a fixed gap width that allows for comprehensive analysis. Gap of 100 to 300 µm is a common distance, however instruments may vary and other gap distances may be employed as long as there is no restriction of particle flow into the measurement zone. See Fig. 1.

4.3 The instrument calculates the shape of all particles ≥20 µm in size. The instrument software sorts particles into the following categories: cutting, fatigue, severe sliding, nonmetallic, fibers. Air bubbles and water droplets ≥20 µm must be eliminated from the particle counting results by analysis or treatment. Further information regarding wear particle shape recognition may be found in Anderson' report.[5].

4.4 Nonmetallic particles are recognized by their partial transparency. Nonmetallic particles, in thin sections, do not block light, as do metallic particles. Therefore, particles displaying transparent interior pixels are classified as nonmetallic. Nontransparent particles are sorted into one of three metallic categories, namely, cutting, sliding, and fatigue.

4.5 Cutting wear particles are recognized by their elongated, curved, or curly shape.

4.6 Sliding wear particles are recognized by being longer than wide, often with straight edges.

4.7 Fatigue particles are recognized by being more or less as long as they are wide and often with jagged, irregular edges.

4.8 Fibers are recognized by their elongated shape and by partial transparency indicating nonmetallic composition.

4.9 Air bubbles are dark round circles, either completely dark or with small bright centers.

4.10 Water droplets are dark round circles with large bright centers. The difference in appearance between air bubbles and water droplets is due to the much different refractive index of each. When present in oil, air bubbles refract much of the light passing through them away from the direction of transmission, whereas water droplets, having a refractive index more nearly equal to that of oil, allow much of the light incident upon them to transmit through them to the CCD video chip.

4.11 Soot is measured by performing an optical extinction measurement with reference to new oil. Absorbance of light is measured and calibration is made to diesel engine oil samples

---

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

[4] Available from SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://aerospace.sae.org.

[5] Anderson, D., *Wear Particle Atlas (Revised)*, Prepared for Advanced Technology Office, Support Equipment Engineering Department, Naval Air Engineering Center, Lakehurst, NJ, 08733, 28 June 1982, Report NAEC – 92 – 163, approved for public release, distribution unlimited.

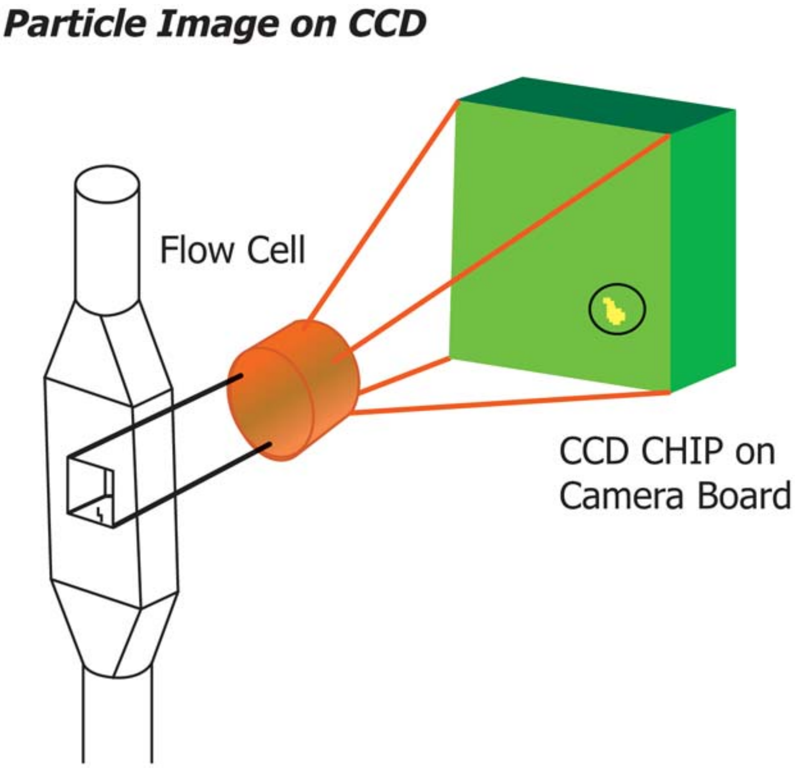

FIG. 1 Schematic of Direct Imaging Integrated Tester

with known percentage of soot as determined by thermal gravimetric analysis in accordance with Test Method D5967, Annex A4.

4.12 Condition alerts and alarms, based on trend and level, can be issued for the system being monitored according to particle count, size distribution, types of particles recognized and soot content.

5. Significance and Use

5.1 This test method is intended for use in analytical laboratories including on-site in-service oil analysis laboratories. Periodic sampling and analysis of lubricants have long been used as a means to determine overall machinery health. Atomic emission spectroscopy (AES) is often employed for wear metal analysis (Test Methods D5185 and D6595). A number of physical property tests complement wear metal analysis and are used to provide information on lubricant condition (Test Methods D445, D2896, D6304, and D7279). Molecular spectroscopy (Practice E2412) provides direct information on molecular species of interest including additives, lubricant degradation products and contaminating fluids such as water, fuel and glycol. Direct imaging integrated testers provide complementary information on particle count, particle size, particle type, and soot content.

5.2 Particles in lubricating and hydraulic oils are detrimental because they increase wear, clog filters and accelerate oil degradation.

5.3 Particle count may aid in assessing the capability of a filtration system to clean the fluid, determine if off-line recirculating filtration is needed to clean the fluid, or aid in the decision whether or not to change the fluid.

5.4 An increase in the concentration and size of wear particles is indicative of incipient failure or component change out. Predictive maintenance by oil analysis monitors the concentration and size of wear particles on a periodic basis to predict failure.

5.5 High soot levels in diesel engine lubricating oil may indicate abnormal engine operation.

6. Interferences

6.1 Dirty environmental conditions and poor handling techniques can easily contaminate the sample. Care must be taken to ensure test results are not biased by introduced particles.

6.2 Air bubbles < 20 µm may be counted as particles giving false positive readings. Air bubbles ≥ 20 µm are recognized and automatically eliminated from the count. Mixing or agitating the sample introduces air bubbles into the oil, but these readily dissipate with ultra-sonication or vacuum degassing.

6.3 Water droplets < 20 µm may be counted as particles giving false positive readings. If water droplets ≥ 20 µm are detected in a sample by the direct imaging integrated tester, there is reason to suspect water droplets < 20 µm are present and have spuriously increased particle count. Small amounts of water in the sample may be negated by the use of water masking solvent. See Appendix X1.

6.4 Certain additives or additive by-products that are not fully dissolved in the oil, most notably polydimethylsiloxane defoamant additive, are known to be present as soft particles that are not contaminants in the fluid system, but are counted as particles by the direct imaging integrated tester. These may be negated by use of a diluting solvent. See Appendix X1.

6.5 Samples with viscosity greater than approximately 150 mm^2/s at 40°C when processed by the direct imaging integrated tester at room temperature (approximately 20°C) may flow through the tester too slowly causing the same particle to be imaged twice. This effect may be negated by diluting the sample with clean solvent to lower viscosity. The tester software makes provision for input of the dilution factor so that particle counts are adjusted and reported for undiluted sample.

6.6 Soot levels above approximately 1.5 % by weight cause insufficient laser light to reach the CCD video detector. The software provides an error message and the sample may be diluted with clear, particle free oil and reprocessed. The tester software makes provision for input of the dilution factor so that particle counts are adjusted and reported for undiluted sample.

6.7 No correction for oil sample density or for soot density is made for the soot calculation.

6.8 High particle concentrations, in excess of approximately 5,000,000/mL, may cause reporting errors. The software provides an error message and the sample may be diluted with clear, particle free oil and reprocessed. The tester software makes provision for input of the dilution factor so that particle counts are adjusted and reported for undiluted sample.

6.9 The software categorizes particles into one of three possible metallic types. These are cutting, sliding or fatigue. The software was trained using particles generated by controlled wear modes under simulated conditions in a laboratory using wear testing equipment. Particles from actual in-service oil samples that appear similar in shape are classified into one of the direct imaging integrated tester's categories even though the nature of the particles may be different. For example, nonmetallic particles may be classified into one of the three metallic classes if the silhouette image captured by the CCD video camera contains no transparent interior pixels. The software assumes particles are nonmetallic if the image allows light to pass through interior pixels. Small, nonmetallic particles may deflect light so that they appear to be metallic. Fatigue platelets, generated by rolling contact fatigue, produce metallic platelets that are more or less as long as they are wide. There are, however, several other particle types with similar shape characteristics. These might be metallic sliding wear particles that are not long enough to be classified as sliding wear particles, or they may be something else entirely such as molybdenum disulfide flakes, coal dust particles, carbon seal material or dark metallo-oxides from sliding wear. Further examination of particles by other means may be necessary to determine their exact type.

6.10 Instruments may have different methodologies to count and size particles. It is more important that the handling and operating methods used do not introduce error into the result, and that the instrument itself is repeatable. Multiple test runs shall be made for each sample and repeatability values included in the report of results.

7. Apparatus

7.1 *Direct Imaging Integrated Tester,* a particle counter and shape recognition instrument consisting of a flow through cell, illumination source, imaging sensor, software, and readout system.

7.2 *Ultrasonic Bath,* an ultrasonic bath having a minimum power density of 4000 W/m^2 . The bath is used to coalescence and remove air bubbles that were introduced during sample shaking to homogeneously suspend the particles therein.

7.3 *Vacuum Degassing Apparatus,* capable of pulling a full vacuum on the sample container in a vacuum chamber. The vacuum degassing apparatus may be used as an alternative to the ultrasonic bath (in accordance with 7.2).

7.4 *Solvent Fltration Apparatus,* to facilitate filtering of rinse solvent or diluent oil through an 0.45 µm (or finer) membrane filter.

7.5 *Mechanical Shaker,* may be used optionally, rather than manual shaking, to redisperse particles before processing.

7.6 *Sample Container,* new container used for collecting the in-service oil sample. Recommended containers are cylindrical bottles (or jars) typically made of polypropylene, polystyrene, PET or glass with nominally flat bottoms, fitted with suitable non-shedding threaded caps. After performing any cleaning procedures, the bottles shall meet the cleanliness criterion of contributing less than 1 % of the total particles expected in the cleanest sample. Bottle must be compatible with fluid and able to withstand the temperature of the fluid when collecting the sample. Sample containers with certified cleanliness levels (for example, "ultra clean") may be used to collect samples for particle counting.

7.7 *Rinse Solvent Container,* a clean bottle or container of approximately 500 mL volume is used for rinse solvent. Recommended containers are cylindrical bottles with flat bottoms made of polypropylene, polystyrene, PET or glass with flat bottoms, fitted with suitable non-shedding threaded caps.

8. Reagents and Materials

8.1 *Check Fluid,* a check fluid or quality control fluid to ensure proper instrument operation. This is a suspension of ISO Medium Test Dust in oil or hydraulic fluid, either a primary sample obtained directly from the NIST (SRM 2806)[6] or a secondary standard purchased commercially or prepared by mixing ISO Medium Test Dust in clean oil or clean hydraulic fluid at a concentration of 2.8 mg/L.

8.2 *Rinse Solvent and Diluent,* kerosene is recommended although other mid-range boiling point fluids such as clean diesel fuel are acceptable. The main criterion for rinse solvent selection is that it is capable of dissolving and rinsing oil samples from the direct imaging integrated tester flow path. Consideration may be given to odor, flashpoint (high flashpoint is desired for reasons of safety), and cost.

---

[6] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

9. Sample Collection and Handling

9.1 Unless otherwise specified, take a representative sample in accordance with Practices D4057, D4177, or other comparable sampling practices using a clean and appropriate sample container. Containers previously holding a sample or any other type of fluid are not considered appropriate containers.

9.2 Ensure that enough sample is taken to perform all analysis methods of interest. The container must not be filled beyond 80% of its total capacity to allow volume for sufficient agitation.

9.3 Label the sample according to the expectations of the analyzing laboratory, including oil type at a minimum.

9.4 Upon receipt at the analyzing laboratory, the sample should be inspected and any non-standard conditions noted. This includes inappropriate container, overfilled container and free water. Recommend a re-sample if inappropriate container or overfilled container is noted.

9.5 Take appropriate safety precautions when collecting samples.

10. Preparation of Apparatus

10.1 Following manufacturer's instructions, prepare the instrument for operation. Check to make sure the instrument is clean and ready for operation by rinsing with solvent and analyzing particulate level in the rinse solvent. For new oils a maximum of 320 particles per mL is recommended. This cleanliness level corresponds to an ISO class of ≤ 16 for particles ≥4 µm. For demanding applications where exceptional cleanliness is required, the maximum allowable cleanliness level of the rinse solvent may be lowered. For routine in-service lubricant samples, the maximum allowable cleanliness level may be raised. A maximum level of 1000 particles/mL is recommended.

11. Calibration and Verification

11.1 Calibration of the direct imaging integrated tester is performed according to the manufacturer's instructions and must encompass the stated range of measurement, which is 4 µm to 99 µm.

11.2 Verification of performance may be performed periodically by using check fluid (8.1) and following manufacturer's operating manual. Software verifies proper analytical operation and records results for future reference. It is recommended that performance verification be done at least once a week.

11.3 If verification of performance fails, consult manufacturer's operating manual. The number counts and size distribution of the reported results of the check fluid may indicate certain faults such as a clogged inlet screen, a stuck particle in the flow cell or an improperly installed flow cell that usually can be corrected by the operator. If necessary, contact the manufacturer's service department.

12. Procedure

12.1 Agitate the sample bottle vigorously for 60 s to redistribute particles. Use hand shaking or a mechanical shaker. Do not use a magnetic stir bar.

12.2 For samples that have been dormant for more than a few days since they were collected, it is recommended that they be agitated in an ultrasonic bath (7.2), for a minimum of 10 min or until any visible deposits on the bottom or side walls of the container are gone. Then agitate the sample vigorously (12.1).

12.3 Place the sample bottle into the ultrasonic bath (7.2), for at least one minute or until any visible air bubbles have risen to the top of the sample surface and escaped.

12.4 Load the sample into the prepared instrument and, following manufacturer's instructions, initiate the measurement cycle.

12.5 After the sample has been processed and results have been displayed on the computer monitor screen, initiate the cleaning cycle following manufacturer's instructions.

12.6 After a sample is processed and the cleaning cycle is initiated, the software continuously monitors the cleanliness level of the rinse solvent until the rinse solvent contains less than the target level of ≤320 particles/mL ≥4 µm (or other value specified for the oil of interest). Once the target cleanliness value is reached, the cleaning cycle is ended and the tester will be ready for the next sample.

12.7 If the target cleanliness level of the rinse solvent is not reached, re-rinse and check particulate level. If the particle count is still too high, replace the rinse solvent with new and re-rinse

13. Report

13.1 Report the following information:

13.1.1 A reference to this test method.

13.1.2 The sample identification.

13.1.3 The date of the test.

13.1.4 Particle size cumulative counts reported by the instrument (for example, ≥4 µm per mL, ≥6 µm per mL, ≥14 µm per mL, ≥21 µm per mL, ≥38 µm per mL, and ≥70 µm per mL.)

13.1.5 ISO code result, according to ISO 4406 for ≥4 µm per mL, ≥6 µm per mL, ≥14 µm per mL. (Other classification systems may also be used, for example, SAE AS 4059.)

13.1.6 The number per mL of sliding, cutting, fatigue, nonmetallic and fiber particles.

13.1.7 The percent by weight of soot.

13.1.8 Any deviation, by agreement or otherwise, from the specified procedures.

14. Precision and Bias[7]

14.1 A temporary precision statement including repeatability is reported in Table 1 based on two samples tested thirty times each in the same laboratory. Full Precision and bias statements based on interlaboratory round robin testing will be determined within five years of adoption of this test method.

---

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1697.

TABLE 1 Temporary Precision Statement

Category	Typical Repeatability, percent	Lower Range, (Particles/mL)	Upper Range, (Particles/mL)
≥ 4 µm	24	250	2,000,000
≥ 6 µm	28	100	850,000
≥ 10 µm	41	40	250,000
≥ 14 µm	54	20	70,000
≥ 21 µm	69	5	18,000
≥ 38 µm	37	100	1500
≥ 70 µm	152	3	50
Cutting	34	50	11,5000
Sliding	31	100	4000
Fatigue	34	50	5000
Nonmetallic	25	200	45,000
Fibers	91	15	250
Soot	7	0.27%	0.27%

15. Keywords

15.1 condition monitoring; contaminant particles; contamination; direct imaging integrated tester; ISO codes; particle count; soot; wear; wear particles

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR NEGATING WATER DROPLET AND SOFT PARTICLES

X1.1 The effects of small amounts of free water and undissolved soft particles may be negated by mixing the sample to be analyzed in the ratio of 50:50 by volume with either of the following two solvents: 1) a volumetric mixture of toluene and 2-propanol (also called isopropanol or isopropyl alcohol), in a 75:25 ratio or 2) dipropylene glycol npropyl ether (also called DOWANOL DPnB[8]). The water and soft particle masking solvent must be filtered to minimize the contribution of particles from the masking solvent.

X1.2 After diluting the sample with water and soft particle masking solvent, record the dilution ratio, and proceed to 12.1. The software will prompt for input of the dilution ratio. Results will be reported for undiluted sample.

---

[8] A registered trademark of Dow Chemical Company.

SUMMARY OF CHANGES

Subcommittee D02.96 has identified the location of selected changes to this standard since the last issue (D7596 – 10) that may impact the use of this standard. (Approved June 1, 2014.)

(1) Added new term, direct imaging integrated tester, to Terminology.

(2) Revised Sections 4, 6, 10, 11, and 12, and revised subsection 7.1.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/