

Designation: F 359 – 82 (Reapproved 1994)^{ε1}

Standard Practice for Static Immersion Testing of Unstressed Materials in Nitrogen Tetroxide (N₂O₄)¹

This standard is issued under the fixed designation F 359; 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.

^{ε1} NOTE—Section 15 was added editorially in September 1994.

1. Scope

1.1 This practice covers the evaluation of the compatibility of nitrogen tetroxide (N₂O₄) rocket engine propellant with materials which will be used in aerospace systems. The practice includes immersion procedures in the vapor phase, liquid phase, critical vapor, and vapor-liquid interface for metals, elastomers, plastics, lubricants, and other materials as applicable.

1.2 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.*

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:

- D 380 Test Methods for Rubber Hose²
- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers-Tension³
- D 471 Test Method for Rubber Property—Effect of Liquids³
- D 543 Practice for Evaluating Resistance of Plastics to Chemical Reagents⁴
- D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing⁴

¹ This practice is under the jurisdiction of ASTM Committee F-7 on Aerospace and Aircraft and is the direct responsibility of Subcommittee F07.02 on Propellant Technology.

Current edition approved Aug. 27, 1982. Published October 1982. Originally published as F359 – 72. Last previous edition F359 – 72 (1977).

² Annual Book of ASTM Standards, Vol 09.02.

³ Annual Book of ASTM Standards, Vol 09.01.

⁴ Annual Book of ASTM Standards, Vol 08.01.

D 638 Test Method for Tensile Properties of Plastics⁴

D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials⁴

D 883 Terminology Relating to Plastics⁴

D 1414 Test Methods for Rubber O-Rings²

D 1460 Test Method for Rubber Property—Change in Length During Liquid Immersion³

D 2240 Test Method for Rubber Property—Durometer Hardness³

D 2510 Test Method for Adhesion of Solid Film Lubricants⁵

D 3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products³

E 8 Test Methods of Tension Testing of Metallic Materials⁶

G 1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens⁷

2.2 ANSI Standards:

ANSI MC 96.1 Temperature Measurement Thermocouples⁸

2.3 Military Standards:⁹

MIL-C-81302 C Cleaning Compound, Solvent, Trichlorotrifluoroethane

MIL-C-27401 C Propellant Pressurizing Agent, Nitrogen

MIL-P-26539 Propellant, Nitrogen Tetroxide.

3. Summary of Practice

3.1 The practice is essentially an examination to detect and measure changes in physical, chemical, and mechanical properties of the specimens and the propellant. The changes are observed visually or measured with precise tools before and after static, unstressed immersion of the material of construction in N₂O₄ under predetermined standard conditions of time, temperature, pressure, and other selected parameters. Liquid phase, vapor phase, critical, and interfacial tests are included.

3.2 Because N₂O₄ is a very reactive substance, compatibility tests are generally conducted in two successive steps as follows:

⁵ Annual Book of ASTM Standards, Vol 05.01.

⁶ Annual Book of ASTM Standards, Vol 03.01.

⁷ Annual Book of ASTM Standards, Vol 03.02.

⁸ Available from the American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036.

⁹ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

F 359

3.2.1 First, a carefully executed exposure of the specimen to determine if any unpredictable, spontaneous, or gross chemical reaction or physical deterioration will occur.

3.2.2 Second, corrosion and compatibility measurements in accordance with this practice.

4. Significance and Use

4.1 Use limits are based on end-use requirements of the N_2O_4 . The quality status, revealed by tests, compared to use limits affords a basis for accepting N_2O_4 into a subsequent storage or handling system. Use limits must not be the criteria for procurement acceptance where the military specification is the applicable standard.

5. Apparatus

5.1 *General*—Apparatus that comes in contact with N_2O_4 should be made of borosilicate glass, 304 stainless steel, 6061 aluminum, polytetrafluoroethylene plastic, or other materials known to be compatible, and be free of contaminants, lubricants, sealants, and extractables that might react with the N_2O_4 or otherwise interfere with the practice.

5.2 Containers:

5.2.1 Vials.

5.2.2 Corrosion Tubes.

5.2.3 Pressure Glass Jars, graduated in millilitres.

5.2.4 Stainless Steel Pressure Containers.

5.2.5 Staco Cells (optional).

5.3 Temperature-Monitoring Instruments and Controls:

5.3.1 Constant-Temperature Bath.

5.3.2 Constant-Temperature Oven.

5.3.3 Thermocouples (see 2.2).

5.4 Pressure-Monitoring Instruments and Controls.

5.5 Measuring Devices:

5.5.1 Balance, accurate to ± 0.0001 g.

5.5.2 *Microscope* with photographic attachment (30 power minimum).

5.5.3 *Color Comparison Indicators* for N_2O_4 and specimens.

5.6 Transfer Equipment:

5.6.1 Tubing.

5.6.2 Valves.

5.6.3 Disposal Vessel.

5.6.4 Cylinder Sand.

5.6.5 Flowmeters.

5.6.6 Sight Glasses.

5.7 Safety Equipment:

5.7.1 Protective Clothing.

5.7.2 Fans.

5.7.3 Shields.

5.7.4 Controlled Environment.

5.7.5 Fire-Fighting Equipment.

5.8 *Dry Box* for preparation of sample, test fluid, and container.

5.9 First Aid Equipment.

6. Reagents and Materials

6.1 The N_2O_4 used in this practice must be of the same quality and type (NTO or MON-1) as used in the final application. Nitrogen tetroxide conforming to the use limits

established for the operating aerospace application must be used since the added impurities can drastically affect material compatibility. As a minimum the N_2O_4 must be analyzed in accordance with the specification prior to immersion.

6.2 Cleaning Solvents:¹⁰

6.2.1 Ethyl Alcohol, ACS reagent grade.

6.2.2 *Trichlorotrifluoroethane*, conforming to specification MIL-C-81302.

6.3 *Gases for Purging and Pressurizing*—Gaseous nitrogen conforming to specification MIL-P-27401.

6.4 *Liquid*—Nitrogen tetroxide conforming to specification MIL-P-26539.

7. Properties of Nitrogen Tetroxide

7.1 Boiling Point, 21.1°C (70°F),

7.2 Freezing Point, -11.2°C (11.8°F),

7.3 Density at 20°C (68°F), 1447 kg/m³ (1.447 g/cm³), and

7.4 Critical Temperature, 158.2°C (316.8°F).

8. Safety Precautions

8.1 *Toxicity*—Nitrogen tetroxide is a dangerous substance. It is classified as a Class-A poison (1).¹¹ The threshold limit value for nitrogen dioxide is 5 ppm (2). Breathing N_2O_4 vapors or spilling liquid N_2O_4 onto the body must be avoided. It is recommended that the user familiarize himself with the hazards of N_2O_4 and arrange for the necessary safety precautions prior to running any tests (3,4).

8.2 *Flammability and Explosion Hazards*—When properly stored, pure N_2O_4 is a stable, nonflammable, shock-insensitive liquid. It is during handling that explosion or fire hazards might develop. For example, accidental contact with paper, cloth, various organic substances, solvent residues, certain chlorocarbons, alcohols, and other chemicals can result in spontaneous ignition or form a shock-sensitive mixture (3,5,6,7,8,9).

9. Test Specimens

9.1 *General Requirements*—The test materials shall be the same chemically, physically, or metallurgically or both as the material of construction to be used in the operating N_2O_4 system. Organic materials, such as plastics, greases, etc., vary considerably from batch to batch. Specification on critical materials require performing this evaluation on every batch or lot. The cleaning and preparation steps to be used must be equivalent to those used in the system.

9.2 Types of Specimens:

9.2.1 *Pure Specimens*—Materials as received from vendors or prepared are cleaned and polished (metals) prior to test.

9.2.2 *Processed Specimens*—Materials as received from vendors or prepared are processed in the same fashion as those materials used in operating systems. In effect, these samples

¹⁰ *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.

¹¹ The boldface numbers in parentheses refer to the list of reference at the end of this practice.