NOTE: The cover page of this standard has been changed for administrative reasons. There are no other changes to this document.

INCH-POUND

MIL-STD-2100(OS)
30 MARCH 1979
SUPERSEDING
(SEE 6.10)

DEPARTMENT OF DEFENSE
TEST METHOD STANDARD

PROPELLANT, SOLID, CHARACTERIZATION OF (EXCEPT GUN PROPELLANT)

AMSC N/A

FSC 1376

DEPARTMENT OF DEFENSE
Washington, DC 20301

Propellant, Solid, Characterization of (Except Gun Propellant)

MIL-STD-3100(OS)

1. This Military Standard is approved for use by the Naval Sea Systems Command (OS), Department of the Navy, and is available for use by all Departments and Agencies of The Department of Defense.

2. Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Ordnance Station, Standardization Documentation Division (501), Indian Head, MD 20640, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FOREWORD

1. This standard has been prepared to establish procedures for determining the chemical, physical and interface properties of solid propellants other than gun propellants. In the past, propellants have been incorporated into weapon systems without a sufficient data base available to assure proper safety and operational parameters; therefore, improvement of a propellant or incorporation of an existing propellant into a new system usually requires a large test program. Immediate characterization of new propellants, according to a standard procedure, is intended to establish the necessary data base to allow more efficient improvement and design programs while reducing the requirement for additional testing. This standard is intended to be cited in contracts to ensure that the necessary data are available to permit determination of optimum safety and performance.

CONTENTS

CONTENTS - Continued

CONTENTS - Continued

CONTENTS - Continued

1. SCOPE

1.1 Purpose. The purpose of this standard is to provide a compendium of standardized methods to determine the intrinsic physical, chemical and interface properties of a solid propellant or propellants proposed for use in tactical rockets, missiles, aircrew escape systems, or auxiliary devices. These methods are listed in TABLE I.

1.2 Intended use. This standard is intended for use in development programs for new propellants or in new applications research for which the existing data base is incomplete. The data generated shall be the basis for (a) design selection between several alternatives, and (b) generating specification requirements and quality assurance provisions for future procurement.

1.3 Application. This standard shall be applied to the extent necessary to establish the required data base for design selection and to determine any hazardous characteristics of the formulation and ingredients as required by FED-STD-313. It should be noted that all the tests in TABLE I may not be applicable to every propellant. This standard shall, therefore, be tailored by the procuring activity via the contract to require those tests and test conditions necessary to meet program objectives and to satisfy DoD directives (refer to MIL-HDBK-248 for additional tailoring guidance). Alternatively, additional tests or test conditions outside the environmental conditions foreseen may be necessary for any one specific program. Characterization shall not be considered a substitute for, or modification of, development, qualification, quality control, quality assurance, type life, or surveillance requirements.

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on the date of invitation for bids or request for proposals form a part of this standard to the extent specified herein.

SPECIFICATIONS

MILITARY

MIL-C-45662	Calibration System Requirements

STANDARDS

FEDERAL

FED-STD-313	Material Safety Data Sheets, Preparation and the Submission of

MILITARY

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129	Marking for Shipment and Storage
MIL-STD-286	Propellants, Solid; Sampling, Examination and Testing
MIL-STD-453	Inspection, Radiographic
MIL-STD-1218	ACS Chemicals

DRAWING

NAVAL ORDNANCE STATION, INDIAN HEAD, MD (CODE IDENT 14083)

22980	FPC Inhibited Grain

PUBLICATIONS

NAVAL SEA SYSTEMS COMMAND (CODE IDENT 10001)

NAVORDINST 8020.3	Explosive Hazard Classification Procedures
OD 9376	Standard Method and Process for the Strand Burning Rate Evaluation of Rocket Propellant Powder
OD 19554	Determination of Burning Rate Versus Pressure from Propellant Test Slabs
OD 44811	Safety and Performance Tests for Qualification of Explosives
OP 5	Ammunition and Explosives Ashore

NAVAL WEAPONS CENTER, CHINA LAKE, CA (CODE IDENT 12934)

Cruise, D.R., Notes on the Rapid Computation of Chemical Equilibrium

(Copies of specifications, standards, drawings, and publications required by suppliers in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this standard to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposals shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM C 493-70	Bulk Density and Porosity of Refractory Materials by Mercury Displacement
ASTM D 1193-74	Reagent Water
ASTM D 2240-75	Indentation Hardness of Rubber and Plastics by Means of a Durometer
Special Publication 234 (1958)	Reporting Particle Size Characteristics

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

AMERICAN NATIONAL STANDARDS INSTITUTE (ANSI)

ANSI Z129.1-1976	Precautionary Labeling of Hazardous Industrial Chemicals

(Application for copies should be addressed to the American National Standards Institute, 1430 Broadway, New York, NY 10018.)

CHEMICAL PROPULSION INFORMATION AGENCY (CPIA)

Publication 21	Solid Propellant Mechanical Behavior Manual
Publication 230	Solid Propellant Structural Integrity Handbook
Handbook Vol. II	Solid Rocket Propellant, Processing, Handling, Storage and Transportation

(Application for copies should be addressed to the Chemical Propulsion Information Agency, Johns Hopkins University, Applied Physics Laboratory, Johns Hopkins Road, Laurel, MD 20810).

CODE OF FEDERAL REGULATIONS

49 CFR 100-199 Transportation

(The Code of Federal Regulations is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Orders should specify "49 CFR 100-199 (latest revision)".)

DEPARTMENT OF DEFENSE

4145.26M DOD Contractor's Safety Manual for Ammunition Explosives and Related Dangerous Material

(Applications for copies of DoD Manual 4145.26M should be addressed to Superintendent of Documents, Government Printing Office, Washington, DC 20402.)

NATIONAL MOTOR FREIGHT TRAFFIC ASSOCIATION, INC., AGENT

National Motor Freight Classification

(Application for copies should be addressed to American Trucking Associations, Attn: Traffic Department, 1616 P Street, Washington, DC 20036.)

UNIFORM CLASSIFICATION COMMITTEE, AGENT

Uniform Freight Classification

(Application for copies should be addressed to the Uniform Classification Committee, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606.)

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are distributed among technical groups and using Federal agencies.)

3. DEFINITIONS

3.1 Characterization plan. A characterization plan is a schedule of tests that will result in the accumulation of the necessary data to determine the physical and chemical properties and the capabilities of a propellant.

3.2 Critical temperature. The critical temperature shall be defined as the transition point where the temperature becomes more important than time in the degradation reactions of a propellant.

3.3 Performing activity. The performing activity is the activity assigned the responsibility for conducting and managing the characterization plan. The performing activity may conduct the specified tests or may delegate part or all of the tests to other qualified activities.

3.4 Procuring activity. The procuring activity is the Government contracting office or agency procuring the equipment, system or subsystem for which this standard is being contractually applied.

4. REQUIREMENTS

4.1 Samples. Sample propellant intended for characterization shall conform to the following requirements:

4.1.1 Materials. Propellant shall be formulated from representative materials of the composition, grade, type, and class described in the applicable documentation and proposed for use during production. Lot numbers and test data shall be reported under the provisions of 4.2 as specified in the contract (see 6.1).

4.1.2 Composition. Variance for each individual ingredient shall not exceed that required in the applicable documentation. In general, variation shall not exceed ± 0.3 percent.

4.1.3 Processing. Equipment, procedures, and conditions shall be the same as, or equivalent to, those described in the applicable documentation and proposed for subsequent production (see CPIA Handbook, Volume II).

4.1.4 Inspection. Samples for characterization shall be inspected visually and radiographically and defective items removed before submission.

4.1.5 Workmanship. The propellant shall be uniform in quality and substantially free of impurities, foreign materials, porosity, voids, cracks, and other assignable defects (see 6.7). It shall be manufactured in accordance with the best practices of the industry.

4.1.6 Form and quantity. Unless otherwise specified in the contract, the form and quantity of a sample shall conform to the following requirements:

4.1.6.1 Bulk form. Sixteen 6x6x9-inch bulk samples shall be required. Alternatively, 20 cylinders 8 inches in diameter and 7 inches long may be acceptable (see 6.4).

4.1.6.2 Strain evaluation cylinders. Fifteen strain evaluation cylinders conforming to CPIA Publication 21 shall be required (see 6.4). These shall be furnished in three sets of five. Within a set, bores shall be varied to approximate strain levels of 10, 20, 30, 40 and 50 percent at -40° C or other levels tailored to the specific formulation and its intended use.

4.1.6.3 Subscale motors. Fifteen small-scale motors conforming to OD 19554 shall be required. Alternatively, an equal number of preferred test vehicles, not to exceed 40 pounds of propellant each, may be acceptable provided that prior written approval is obtained from the Government procuring activity (see 6.4). Forty-pound charges, if supplied, shall conform to NOS drawing 22980. Tooling and beakers for 40-pound charges may be obtained from: Commanding Officer, Naval Ordnance Station, Indian Head, MD 20640, Attn: NSWC R20.

4.1.6.4 Interface samples. A minimum of thirty (30) specimens shall be furnished for each interface in the proposed motor design.

a. In the case of propellant/liner/case and propellant/liner/insulator interface samples, the specimens shall be 2.0 inch poker chips and the liner thickness shall be that of the proposed motor design (see 5.4.22 and CPIA Publication 21 Section 4.7).

b. In the case of a propellant to propellant interface a sufficient number of bulk samples shall be furnished. Each propellant layer shall be a minimum of 3 inches (see 5.4.22.4).

4.1.7 Lot. Unless otherwise specified in the contract, the sample submitted for characterization shall be prepared from one propellant lot. The term "lot" shall be as defined in MIL-STD-105. A propellant lot shall contain individual ingredients from one ingredient lot only. In the event that more than one lot is represented, units of the sample shall be identified by lot number.

4.1.8 Packaging.

4.1.8.1 Preservation, packaging, and packing. Samples shall be preserved, packaged, and packed in a manner that will ensure protection against damage, loss of ingredients, and contamination by foreign material, including atmospheric moisture. Unless otherwise specified, containers shall be acceptable to commercial carriers for shipment at the lowest rate. Refer to the Uniform Freight Classification Rules or the National Motor Freight Rules as applicable. In all cases, the protection shall be not less than that required by 49 CFR 170-178 (see CPIA Handbook, Vol II).

4.1.8.2 Marking. In addition to any special marking required by the contract, each container shall be marked in accordance with MIL-STD-129 and 49 CFR 100-199. Marking shall include, but not be limited to, the following information:

a. Manufacturer's name and address
b. Contract or order number
c. Item identification
d. Lot number or other identification

e. Environmental limitations for storage
f. Explosive classification and warnings
g. Other applicable warnings (see ANSI Z 129.1 - 1976)

4.2 Documentation. The contractor shall furnish composition, processing information, results of tests performed (see 6.5), and assurance of compliance with requirements of 4.1.1 through 4.1.6 inclusive. This information shall be certified and submitted to the procuring activity as specified in the contract (see 6.1).

4.3 Submission of samples. Characterization samples shall be submitted to the performing activity as designated by the procuring activity (see 6.6). A copy of the documentation (4.2) shall accompany the samples.

4.4 Data submission. As specified in the contract, the performing activity shall report the results of all characterization tests, data, evaluations, and assessments to:

a. The procuring activity
b. The contractor
c. Other, as specified in the contract (see 4.10)

4.5 Retests. Retests required by the procuring activity shall be conducted as specified in the contract (see 6.8).

4.6 Safety precautions. The safety precaution requirements of the "Contractor's Safety Manual for Ammunition, Explosives, and Related Dangerous Material" (DOD 4145.26M) are applicable and shall be as specified in the contract.

NOTE: When this standard is used as part of the description of work to be accomplished by a Government activity, the safety precaution requirements of "Ammunition and Explosives Ashore" (OP 5) shall be made applicable.

4.7 Material safety data sheets. The contractor shall prepare and submit material safety data sheets in accordance with FED-STD-313 as specified in the contract (see 6.2).

4.8 Undue hazards. The manufacturer shall not use materials, ingredients, or practices that present any undue toxic or explosive hazard without expressed consent, in writing, from the procuring activity. Warnings of such hazards, together with a full description, shall be included in the documentation (see 4.2).

4.9 Proprietary and nondomestic technology. The manufacturer shall not use proprietary or nondomestic materials, ingredients, equipment, processes, or practices without written consent of the procuring activity. This

provision shall not apply if the Government receives, without cost, full disclosure rights of usage for proprietary materials at the time of sample submission.

4.10 Security classification. Security classification for all technical data including proposed usage shall be as specified by the procuring activity.

5. DETAILED REQUIREMENTS

5.1 Inspection by contractor.

5.1.1 Ingredients and processing. The ingredients and processing shall be inspected for conformance to the requirements of 4.1.1 through 4.1.5 and 4.1.7.

5.1.2 Visual inspection. The sample shall be visually inspected before and the containers after packing for conformance to the requirements of 4.1.5, 4.1.6 and 4.1.8.

5.1.3 Radiographic. The individual units of the sample shall be examined radiographically in accordance with MIL-STD-453 to ensure compliance with 4.1.4 and 4.1.5. When bonded units are required (see 4.1.6.3), examination details and conditions shall be sufficient to verify adequate bonding.

5.1.4 Records. The manufacturer shall maintain records of manufacturing inspections for a period of at least six months after acceptance of the final characterization report. These records shall be made available for inspection by the procuring activity upon request. Records of propellant tests performed by the contractor as specified in the contract shall be maintained and be available for review by the Government for a period of at least two years.

5.2 Inspection by the performing activity. The sample shall be inspected by the performing activity upon receipt in accordance with the following:

5.2.1 Form and quantity. The sample shall be visually inspected to verify conformance to the requirements of 4.1.6.

5.2.2 Dimensions. The dimensions of strain evaluation cylinders and subscale motors shall be measured to verify conformance to the requirements of the applicable reference documents.

5.2.3 Borescopic examination. The bore of each strain evaluation cylinder and subscale motor shall be examined with a borescope. The presence of surface defects such as cracks, voids, or discontinuities shall be cause for rejection of individual units (see 6.7).

5.2.4 Radiographic inspection. Each unit of the sample shall be inspected radiographically. The presence of cracks, voids, fuel rich or oxidizer rich areas, or unbonding if applicable, shall be cause for rejection of an individual unit (see 6.7).

5.2.4.1 Bulk samples. Each unit shall be inspected in two positions:

a. With the beam parallel to the vertical axis
b. With the beam parallel to the horizontal axis

5.2.4.2 Strain evaluation cylinders and subscale motors. Each unit shall be inspected with the beam parallel with the diameter at a zero point on the radius and with the unit or grain turned 90° from the initial point.

5.3 Characterization inspection. The performing activity shall conduct the characterization tests and examinations specified in the characterization plan as approved by the procuring activity (see 1.3).

5.3.1 Calibration. Instruments, tools, gages, and other measurement devices shall be maintained in accordance with MIL-C-45662.

5.3.2 Proprietary items. Proprietary and trade-marked devices or materials may be indicated in the test methods for definition or convenience. In such cases, equivalent items may be acceptable providing prior approval is obtained from the procuring activity.

5.3.3 Reagents. Unless otherwise specified, required chemicals and reagents shall meet the American Chemical Society (ACS) standards as specified in MIL-STD-1218. When water is required, it shall be a distilled or demineralized product in accordance with Type I or Type II of ASTM D 1193-74 containing less than 2 parts per million total solids and a pH in the range of 5.8 to 7.0.

5.3.4 Temperature tolerance. Where not specifically indicated, temperature tolerances shall be ± 1°C (± 2°F).

5.3.5 Inspection conditions. Unless otherwise specified, all inspections shall be performed under the following ambient conditions:

a. Temperature: Room ambient 18 to 35°C (65 to 95°F)
b. Altitude: Normal ground
c. Vibration: No requirement
d. Humidity: Room ambient to 95 percent relative, maximum

5.4. Test methods.

5.4.1 Theoretical performance calculations. Theoretical performance calculations shall be performed in accordance with NWC publication, "Notes on the Rapid Computation of Chemical Equilibrium."

TABLE I. Test list.

Test Description	Test Method Paragraph
Theoretical calculation	5.4.1
Heat of explosion	5.4.2
Strand burning rate	5.4.3
Environmental impact (of firings)	5.4.4
Card-gap	5.4.5
Cap	5.4.6
Impact	5.4.7
Friction sensitivity	5.4.8
Electrostatic discharge sensitivity	5.4.9
Ignition test	5.4.10
Unconfined burning	5.4.11
Velocity of detonation	5.4.12
Critical diameter	5.4.13
Vacuum stability (see 6.4)	5.4.14
Differential thermal analysis (DTA)	5.4.15
Thermogravimetric analysis (TGA)	5.4.16
Cube cracking (see 6.4)	5.4.17
Taliani (see 6.4)	5.4.18
Critical temperature (isothermal DTA)	5.4.19
Hardness	5.4.20
Density	5.4.21
Mechanical properties, uniaxial	5.4.22
Propellant/liner/case bond	5.4.22.1
Propellant/liner peel	5.4.22.2
Propellant/liner bond	5.4.22.3
Propellant/propellant bond	5.4.22.4
Boot/insulator	5.4.22.5
Mechanical properties, uniaxial, high rate	5.4.23
Mechanical properties, uniaxial, high pressure	5.4.24
Stress relaxation modulus	5.4.25
Mechanical properties, biaxial	5.4.26
Strain evaluation cylinders	5.4.27
Coefficient of thermal expansion	5.4.28
Heat capacity	5.4.29
Thermal conductivity	5.4.30
Transition point(s)	5.4.31
Hygroscopicity	5.4.32
Vapor pressure	5.4.33
Propellant/liner/insulator	5.4.34
Swelling ratio (see 6.4)	5.4.35
Sol/gel ratio (see 6.4)	5.4.36
Cross-link density (see 6.4)	5.4.37
Subscale motors	5.4.38
Other properties	5.4.39
Aging effects	5.4.40
Sealed units	5.4.40.1
Subscale motors	5.4.40.1a
Strain evaluation cylinders	5.4.40.1b
Bulk propellant	5.4.40.2
Storage conditions	5.4.40.2.1
Storage preparation	5.4.40.2.2
Under dry nitrogen	5.4.40.2.2a
Under nitrogen at the specified relative humidity, %	5.4.40.2.2b
Under dry air	5.4.40.2.2c
Under wet air	5.4.40.2.2d
Special cross storage	5.4.40.2.3
Storage	5.4.40.2.4
Testing of stored propellant	5.4.40.2.5
Exterior	5.4.40.2.5a
Hardness	5.4.40.2.5a.1
Impact test	5.4.40.2.5a.2
Mechanical properties, uniaxial	5.4.40.2.5a.3
Density	5.4.40.2.5a.4
Strand burning rate	5.4.40.2.5a.5
Interior	5.4.40.2.5b.
Mechanical properties, uniaxial	5.4.40.2.5b.1
Hardness	5.4.40.2.5b.2
Density	5.4.40.2.5b.3
Heat of explosion	5.4.40.2.5b.4
Strand burning rate	5.4.40.2.5b.5
Differential thermal analysis (DTA)	5.4.40.2.5b.6
Sol/gel, swelling ratio and cross-link density	5.4.40.2.5c
Microscopy	5.4.41

5.4.2 Heat of explosion. Heat of explosion shall be determined in accordance with method 802.1 of MIL-STD-286.

5.4.3 Strand burning rate. The strand burning rate shall be determined as specified in OD 9376 or MIL-STD-286 as appropriate. Nominally (see 1.3), the burning rate shall be measured at 500, 1000, 2000, 3000, 4000 and 5000 psi and at -65, 77 and 165°F. The report shall include a log-log plot of the data with calculation of pressure exponent and temperature dependence shown.

5.4.4 Environmental impact (of firings). An environmental impact assessment based on products in, and heat content of, the expanded after plume zone shall be prepared. Effects at sea level and intermediate or high altitudes, if applicable, shall be considered. Noise and electromagnetic (radar) attenuation shall not be factors in this assessment. Primary and secondary smoke and handling toxicity, if applicable, should be considered qualitatively.

5.4.5 Card-gap test. Testing shall be performed in accordance with NAVORDINST 8020.3.

5.4.6 Cap. The cap test shall be conducted in accordance with NAVORDINST 8020.3.

5.4.7 Impact. Impact sensitivity tests shall be performed in accordance with NAVORDINST 8020.3.

5.4.8 Friction sensitivity. The testing shall be performed in accordance with OD 44811.

5.4.9 Electrostatic discharge sensitivity. The test shall be performed in accordance with OD 44811.

5.4.10 Ignition test. The test shall be performed in accordance with 49 CFR 173.88.

5.4.11 Unconfined burning. The testing shall be performed in accordance with NAVORDINST 8020.3.

5.4.12 Velocity of detonation. NOTE: This paragraph is applicable only to Class 1, Division 1 compositions. The detonation velocity shall be determined in accordance with OD 44811.

5.4.13 Critical diameter. NOTE: This paragraph is applicable only to Class 1, Division 1 compositions. Critical diameter shall be determined as specified in OD 44811.

5.4.14 Vacuum stability. Testing shall be conducted in accordance with method 403.1 of MIL-STD-286. The sample size shall be selected to yield a volume of gas between 0.1 milliliter (mL) and 5.0 mL at standard temperature and pressure during a 40-hour test period (applicable only to high explosives/warhead materials and nitrate ester compositions, see 6.4).

5.4.15 Differential thermal analysis (DTA). The test shall be conducted as follows:

5.4.15.1 Equipment.

a. Aluminum heating block having three tube wells
b. Linear programmer with controlling sensor capable of holding a heating rate of 1°C/minute from ambient to 400°C
c. Differential thermocouple
d. XY Recorder
e. Glass Tubes

5.4.15.2 Materials.

a. Glass beads, 30-micrometer (μm) diameter

5.4.15.3 Specimen.

a. Cylinder, 0.5 x 0.5 inch, drilled to accept the thermocouple shield and to position the thermocouple at center of sample.
b. Teflon tape
c. Teflon positioning discs

5.4.15.4 Procedure. Place the specimen in a glass sample tube, the thermocouple shield in the drilled hole, and position the shield as necessary with one or two Teflon discs. Prepare a reference tube and a tube for the control probe, using a weight of glass beads, equal to the specimen weight, to replace the specimen. Insert one side of the differential thermocouple in the specimen shield, the second arm of the thermocouple in the reference tube, and the control probe in the remaining tube. Cover each tube with a piece of Teflon tape. Place the tubes in the heating block wells, and heat the assembly at a controlled linear rate of 1°C/minute to destruction of the specimen. Record the temperature and temperature differential on the XY recorder.

NOTE: The assembly must be barricaded during the actual testing.

5.4.15.5 Report. The report shall include a copy of the thermogram showing sample identification, sample weight, heating rate, temperature of reference at autoignition (T_i), if any, and an interpretation of other features of the plot, if any.

5.4.16 Thermogravimetric analysis (TGA). The test shall be conducted using a DuPont 900 Thermal Analyzer equipped with a 951 TGA unit, or equivalent. The equipment shall be operated in accordance with the manufacturer's recommended procedure. Nominally, the specimen shall be 10 milligrams (mg), the heating rate shall be 5°C/minute, and the atmosphere shall be nitrogen. These conditions may be varied to obtain the best fit for the particular sample. As a minimum, the report shall include a copy of the weight/loss temperature plot with sample identification, date, sample size, heating rate, atmosphere, and an interpretation of the features of the plot given.

5.4.17 Cube cracking test. Prepare three regular smooth faced cubes. These shall be 2 inches, 3 inches and 4 inches in size, respectively. Seal each in a lightweight aluminum container and radiograph in sufficient detail to detect voids, cracks, or fissures. Discard and replace any initially defective cubes. Place the cubes in their containers in an oven preheated and maintained at 80 ± 2°C. Reexamine radiographically after 72 hours and every 24 hours thereafter, but minimize temperature cycling. Report the number of days for each cube, by size, to crack. Tests shall be conducted at temperatures other than 80°C, if specified by the contract (usually for nitrate ester compositions, see 6.4).

5.4.18 Taliani test. The test shall be conducted in accordance with method 406.1 of MIL-STD-286. The test temperature shall be 110°C and the atmosphere nitrogen gas. (applicable only to nitrate ester containing compositions, see 6.4).

5.4.19 Critical temperature. The critical temperature test shall be performed in accordance with the following:

5.4.19.1 Equipment and specimen. The equipment and specimen shall conform to 5.4.15.

5.4.19.2 Procedure. Preheat the heating block, sample tube (empty), reference tube assembly, and controller probe assembly to a predetermined temperature. This temperature shall be determined from the DTA thermogram and shall be that reference temperature, T_T, where ΔT equals ±2°C. Insert the specimen and differential thermocouple assembly into the preheated sample tube and seal lightly with Teflon Tape. Record ΔT of the specimen until damage occurs as evidenced by the start of an exotherm. The time shall be measured from the point where the sample attains the preset temperature to the start of the exotherm. Conditions must be adjusted to minimize equilibration time. The procedure shall be repeated using fresh specimens at other selected temperatures to obtain a minimum of six points showing time to damage ranging from about 5 minutes to about 400 minutes.

5.4.19.3 Data treatment. Plot time to damage versus temperature for the several isotherms to obtain an L-shaped curve. Extrapolate to obtain the critical temperature. The report shall include a copy of the final plot showing the extrapolation, sample identification, and date.

5.4.20 Hardness. Hardness shall be determined in accordance with ASTM D 2240-75. Test temperature shall be 77 ± 2°F.

5.4.21 Density. The density shall be determined at 77 ± 2°F in accordance with method T 510.3 of MIL-STD-286.

5.4.21.1 Alternate method. Density may be determined at 77 ± 2°F in accordance with ASTM C 493-70.

5.4.22 Mechanical properties, uniaxial. Uniaxial mechanical properties shall be measured in accordance with CPIA Publication 21. As a minimum, five specimens shall be tested at each of three temperatures and strain rates. The test temperatures shall be -65, 77 and 165°F and the crosshead rates shall be 0.02, 0.20 and 2.0 in/min (see 1.3).

NOTE: Unless the intent is to show properties after glass transition, the lowest temperature taken should be at least 5°F above the transition point (see 5.4.31).

Conditioning of specimens at the highest temperatures shall not result in significant material loss by volatization or constitute an undue safety hazard. The report shall include values for maximum stress, stress at rupture, elongation at maximum stress, elongation at rupture, and initial (tangential) modulus, at each temperature and strain rate.

5.4.22.1 Propellant/liner/case bond test. Testing shall be in accordance with CPIA Publication 21, Section 4.7. A minimum of five specimens (see 4.1.6.4 for sample requirements) shall be tested at each of three temperatures and strain rates. The test temperatures shall be -65, 77 and 165°F and the crosshead rates shall be 0.02, 0.20 and 2.0 in/min (see 1.3). The report shall include values for maximum stress, stress at rupture, and modes of failure. The samples shall also be examined by microscopic methods (see 5.4.41).

5.4.22.2 Propellant/liner peel test. Testing shall be in accordance with CPIA Publication 21, Section 4.7. A minimum of five specimens shall be tested at each of three temperature and strain rates (see 4.1.6.4 for sample requirements). The test temperatures shall be -65, 77 and 165°F and the crosshead rates shall be 0.02, 0.20 and 2.0 in/min (see 1.3). The report shall include values for maximum stress, stress at rupture and modes of failure. The samples shall also be examined by microscopic methods (see 5.4.41).

5.4.22.3 Propellant/liner bond test. Testing shall be in accordance with CPIA Publication 21, Section 4.7.

5.4.22.4 Propellant/propellant bond test. Example: (Booster/sustainer propellant system). Testing shall be in accordance with CPIA Publication 21, Section 4.3. Standard JANNAF specimens containing equal lengths of each propellant in the gage length shall be obtained from a bulk sample of booster/sustainer loaded rocket motor. A minimum of five specimens shall be tested at each of three temperatures and strain rates (see 4.1.6.4 for sample requirements). Nominally (see 1.3), the temperatures shall be -65, 77 and 165°F and the crosshead rates shall be 0.02, 0.20 and 2.0 in/min. The report shall include values for maximum stress, stress at rupture, and modes of failure. The samples shall also be examined by microscopic methods (see 5.4.41).

5.4.22.5 Boot/insulator test. Prepare boot/insulator coupons; age at 120 to 165°F under a pressure which will simulate the load experienced by the boot/insulator in a fully loaded motor. Ten samples shall be tested for each withdrawal at 77°F and 2.0 in/min.

5.4.23 Mechanical properties, uniaxial, high rate. Testing shall be in accordance with CPIA Publication 230 using the specified materials test systems or equivalent. Five specimens shall be tested at each of three temperatures and strain rates. The test temperatures shall be -65, 77 and 165°F (see 1.3) and the crosshead rates shall be 500, 750 and 1000 inches per minute (see note in 5.4.22).

5.4.24 Mechanical properties, uniaxial, high pressure. Uniaxial properties of pressurized specimens shall be determined in accordance with CPIA Publication 230. Five specimens shall be tested at 77°F using a crosshead rate of 20 in/min at each of three pressures. Unless otherwise defined, the pressures shall be 400, 700 and 1000 psi.

5.4.25 Stress relaxation modulus. Stress relaxation modulus shall be measured in accordance with CPIA Publication 21 using tab-bonded JANNAF specimens. Nominally, specimens shall be strained 5 percent at a crosshead rate of 2.0 in/min. Five specimens shall be tested at each temperature. The test temperatures shall be -65, -40, -20, 0, 77 and 165°F. (see 1.3). Temperature considerations given under mechanical properties, uniaxial (5.4.22) shall apply.

5.4.26 Mechanical properties, biaxial. Testing shall be in accordance with CPIA Publication 21 using strip biaxial specimens. Five specimens shall be tested at each of three temperatures and rates. The test temperatures shall be -65, 77 and 165°F and the crosshead rates 0.02, 0.20 and 2.0 in/min (see 1.3). Temperature considerations discussed under mechanical properties, uniaxial (5.4.22) shall apply.

5.4.27 Strain evaluation cylinders. Cylinders shall be conditioned at 77 ± 2°F and examined radiographically and by borescope. The temperature of each shall be reduced at the rate of 10°F per day to 0°F and then at the rate of 5°F per day to failure. They shall be reexamined each day and cracked units removed (see CPIA Publication 21).

5.4.28 Coefficient of thermal expansion. The coefficient of thermal expansion shall be determined according to CPIA Publication 21.

5.4.29 Heat capacity. The determination of the mean specific heat of the propellant shall be made as described in CPIA Publication 21.

5.4.30 Thermal conductivity. The determination of the thermal conductivity shall be made in accordance with CPIA Publication 21.

5.4.31 Transition point(s). The transition point(s) shall be determined by one of the following methods. In case of dispute, the method of 5.4.31.1 shall be determinative.

5.4.31.1 Standard method. The transition point or points, over the temperature range of 180 to -70°F, shall be determined according to the method described in CPIA Publication 21.

5.4.31.2 Alternate method. Transition point(s) may be determined using a DuPont 942 Thermal Mechanical Analyzer or equivalent. Operation of the instrument shall be according to the manufacturer's recommendations.

5.4.32 Hygroscopicity. Twelve freshly cut specimens approximately 0.12 inches (3.1mm) thick and weighing approximately 10 g shall be prepared, placed in individual tared weighing dishes, and each reweighed. The sample shall be desiccated over anhydrous magnesium perchlorate at ambient temperature and pressure until constant weight is attained. These desiccated specimens shall be exposed at 30°C as follows:

Number of specimens	% Relative humidity
3	90
3	75
3	52
3	20

These humidities may be obtained using constant humidity cabinets or over the following solutions:

Relative humidity	Solution
90	18.6 weight % (wt %) H_2SO_4
75	Saturated NaCl
52	Saturated $Mg(NO_3)2 \cdot 6H_2O$
20	59.2 wt % H_2SO_4

The specimens shall be exposed until weight equilibrium is attained. The average percent water absorbed at each humidity shall be reported.

NOTE 1: When propellant is exposed over sulfuric acid solution, contact of the propellant with the acid must be avoided.

NOTE 2: If data at other temperatures and humidities are required, solutions may be obtained from the following:

a. International Critical Tables 1,67
b. Stokes and Robinson, Ind. Eng. Chem., 41, 2013 (1949)
c. Carr and Harris, ibid, 41, 2014 (1949)

5.4.33 Vapor pressure. The partial pressures of constituents over the propellant shall be measured by a dynamic (transpiration) method. Nominally, measurements shall be made at 100, 120, and 140°F. (Not applicable to propellants containing appreciable amounts of occluded volatile solvents such as ethyl ether, acetone, etc).

5.4.33.1 Sample. The sample is ground to pass a U.S. sieve screen number 35 (500µm). Alternatively, lathe turnings approximately 0.5 mm thick may be used. The prepared sample shall be dried over anhydrous molecular sieves in a desiccator at ambient temperature and pressure for 48 to 72 hours.

5.4.33.2 Apparatus. A glass apparatus conforming in general detail to descriptions given in advanced physical chemistry texts shall be used. The sample container, approximately 9 mm inside diameter x 190 mm long, shall be connected with glass to a cold trap (cooled with a mixture of dry ice acetone). The cold trap outlet shall be connected with glass to a constant pressure gasometer having a capacity of about 3 liters(L). The sample and most of the connection to the cold trap shall be maintained at the indicated temperature(s) ± 0.005°C (± 0.01°F). Dry prepurified nitrogen shall be used as the effluent.

5.4.33.3 Procedure. Adjust the constant temperature bath to the proper temperature. Pack the dried sample loosely in the sample tube, assemble into the apparatus and immerse in the bath. Vent the top of the sample tube to the air, start the nitrogen flow and adjust the rate to approximately 2 liters in 3 hours. Connect the sample tube to the cold trap and gasometer, pass about 2 liters of nitrogen through the system, and measure the actual amount used with the gasometer. Remove the cold trap and analyze the contents by infrared absorbance or other analytical technique. Calculate the apparent partial pressure for each constituent from the expression $PV = nRT$. Repeat the measurement using fresh samples and flow rates of about 2 liters in 4 and 5 hours, respectively. To obtain the true partial pressure(s), extrapolate to zero flow rate.

5.4.34. Propellant/liner/insulator. Remove the insulator from the propellant/liner interface and cut in half, depth wise. Chop a portion of each section and extract approximately 0.3 g of sample with di-ethyl ether for 10 - 15 min. The extraction is repeated until a total volume of 40 mL of ether is used. Evaporate the ether and dissolve the residue in a 20 volume % 1, 2-dimethoxyethane/heptane solution; dilute to 25 mL in a volumetric flask. The solution is then checked for plasticizers, stablizers, etc. by high performance liquid chromatograph (HPLC, analytical scale).

NOTE: If stabilizers present in the formulation are nitroso compounds, extraction equipment should be wrapped in foil to retard possible photolytic effects. If the analysis is delayed for more than one hour, the samples and standards should be stored in an area free from all sources of ultraviolet light.

Analysis of Propellant Extract:

a. High Performance Liquid Chromatograph (HPLC, Analytical Scale): Instrumentation:

1. HPLC, modular construction, consisting of:

A. Pump, M-6000A Solvent Delivery System, Waters Associates, Milford, MA

B. Injector, Model U6K Universal Injector, Waters Associates, Milford, MA

C. Column, μ-Porasil, 10 μm particle silica gel, Waters Associates, Milford, MA

D. Spectrophotometer, Beckman Model 25 UV-Visible, adapted to HPLC using a Waters Associates Microcell Assembly, Beckman Instruments Inc., Irvine, CA

E. Photometer, Model 440 UV-Visible, Waters Associates, Milford, MA

F. Refractometer, Model R-401, Waters Associates, Milford, MA

G. Recorder, Model 282 Integrating, Linear Instruments Corporations, Irvine, CA, and Servo Riter II, dual channel, Texas Instruments, Inc., Houston, TX

2. Syringes, Series LC-210 and Series C, Precision Sampling Corporation, Baton Rouge, LA

3. Glass wool, Corning Glass Works, Corning, NY

4. Nalgene thistle tops, Fisher Scientific Co.

5. Solvent dispenser, "REPIPET", Lab Industries, Berkeley, CA

6. Micrometer buret, Ultra-Precision, Roger Gilmont Instruments, Inc., Great Neck, NY

b. Reagents:

1. Diethyl ether, "Distilled in Glass", UV cutoff 218 nm, Burdick and Jackson Laboratories, Inc., Muskegon, MI

2. 1,2-Dimethoxyethane (Glyme), "Distilled in Glass", UV cutoff 220 nm, above supplier.

3. n-Heptane, "Distilled in Glass", UV cutoff 200 nm, above supplier.

4. Silica gel, 40-140 mesh, "Baker Analyzed", J. T. Baker Chemical Co., Phillipsburg, NJ

c. Standards:

100 mg of stabilizers, plasticizers, etc., are weighed into respective 100-mL volumetric flasks, dissolved and diluted to volume with glyme. Using a pipette or a precision buret (recommended), the standard stock solution is transferred into a 25-ml volumetric flask containing 20 ml of n-heptane, diluted to volume with glyme and stored, tightly closed, along with the stock solutions in an area free of light.

d. Instrument Conditions:

1. Mobile phase - 10 to 35 volume % glyme in heptane depending on column efficiency, achievable separation, and desired separation parameters.

2. Flow rate - 1.0 to 2.0 mL/min depending on solvent polar strength, group (Solvent Use Index I, Waters Associates) and desired separation.

3. Detector settings - 280 nm for 2NDPA, 340 nm for MNA and NNOMNA. Response settings must be determined for each detector; e.g., .25 AUFS on the spectrophotometer, .05 AUFS onthe Model 440, 2X-4X on the R-401.

e. Analysis:

With the injector in the "load" position, insert 20 µL of the standard solution into the sample loop. Close the loop, and inject the standard into the system. When the standard has been eluted from the system, in the order of 2NDPA, NNOMNA, MNA, measure the peak heights or peak areas for each compound.

Inject the sample solution in like manner and calculate the percentage of each component as follows:

$$\text{wt\% Component} = \frac{A}{B} \times \frac{C}{D} \times 100\%$$

Where A = weight in mg of the standard component in the 25-mL volumetric (mg/25 mL)

B = peak height (peak area) of the standard component in relative units

C = peak height (peak area) of the component in the sample in relative units

D = weight in mg of the sample in the 25-mL volumetric (mg/25 mL)

f. Limitations:

1. The concentration of the substances to be measured should approximate the concentration of standard. Follow the guidelines in the Methods and Procedures section.

2. Depending on separation techniques via HPLC, other species eluting along with the species of interest could interfere with quantitation.

3. Samples containing nitroso compounds should be prepared with minimum exposure to any source of ultraviolet (UV) light, as decomposition of the nitroso group will occur.

5.4.35 Swelling ratio. The swelling ratio shall be determined in accordance with CPIA Publication 21 (Applicable only to crosslinked formulations (see 6.4)).

5.4.36 Sol/gel ratio. Samples conforming to CPIA Publication 21 shall be extracted with a suitable solvent in a continuous extractor. (Applicable only to crosslinked formulations (see 6.4)). Criteria for selection of a solvent shall be as follows:

a. Solvent shall swell the gel phase to a maximum extent
b. Boiling point shall be low enough to permit extraction on a hot water bath
c. Solvent shall not dissolve or react with fillers

The extraction shall be continued until the soluble phase has been completely removed. The solvent shall be removed from the solution portion of the extract to constant weight in a stream of dry nitrogen. Calculations shall be as shown in CPIA Publication 21.

5.4.37 Cross-link density. The cross-link density shall be calculated as specified in CPIA Publication 21 (Applicable only to cross-linked formulations (see 6.4)).

5.4.38 Subscale motors. Nine subscale motors shall be tested at three pressures and three temperatures. Nominally (see 1.3), the temperatures shall be -65, 77 and 165°F, and the pressures shall be approximately 1000, 2000, and 4000 psi. The motors shall be fitted with nozzles sized to operate at the designated average pressure(s), conditioned at the required temperature(s), examined radiographically, and static fired.

NOTE: In selecting motor operating pressure, consideration of the strength of the case and propellant burning rate exponent must be the controlling factors. Maximum pressure must not exceed 85 percent of the capability of the case and the rate exponent at the maximum pressure must not exceed 1.0.

5.4.39 Other properties. Determination of properties other than those described herein may be necessary to characterize a specific solid propellant or to describe suitability for a given application (see 1.2 and 6.8). Additional tests may be required by the procuring activity when specified in the contract. These may include but shall not be limited to the following:

a. Mechanical properties and burning rates in the direction of major and minor axes of extruded or compressed forms

b. Stress relaxation modulus at additional strain levels and for various time intervals

c. Composition of evolved gas as a function of time, temperature, and loading density

d. Solubility of evolved gas species in the propellant

e. Diffusivity of evolved gas species

f. Constant strain capability

g. High rate, high pressure, cold, mechanical properties

5.4.40 Aging effects. Propellant shall be aged at ambient temperature and under accelerated conditions and tested as indicated in TABLE II and the following paragraphs:

5.4.40.1 Sealed units. Subscale motors and strain evaluation cylinders shall be flushed with dry nitrogen, sealed, and subjected to accelerated aging at a specified temperature.

a. Subscale motors. Six subscale motors shall be aged at T_3 (see TABLE II). Three shall be removed after 6 weeks and examined radiographically at ambient temperature. Motors exhibiting cracks, slump, or other defects shall not be fired. Acceptable motors shall be fitted with nozzles to yield operating pressures of 1000, 2000 and 4000 psi, respectively, and static-fired (see 4.1.6.3 and 5.4.38). The remaining three motors shall be withdrawn after 12 weeks conditioning, examined, defective units rejected. acceptable units fitted with nozzles and fired.

b. Strain evaluation cylinders. Two sets of strain evaluation cylinders (see 4.1.6.2) shall be aged at T_3. One set shall be withdrawn after 6 weeks and the remaining set after 12 weeks. Each set of cylinders shall be examined and tested as soon as possible after withdrawal, as specified in 5.4.27,

TABLE II. Typical Aging Effects Matrix.*

Tests	Atmosphere																											
	Dry Nitrogen								Nitrogen at a relative humidity of H_1%								Dry Air						Wet Air Air at H_1%					
	Temperature																											
	T_1			T_2				T_3				T_2				T_3			T_2				T_3				T_2				T_3		
	Typical Storage Times, Weeks																											
	8	16	26	4	8	12	16	4	6	8	12	4	8	12	16	4	8	12	4	8	12	16	4	8	12	4	8	12	16	4	8	12
Subscale motors	-	-	-	-	-	-	-	-	X	-	X	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Strain evaluation cylinders	-	-	-	-	-	-	-	-	X	-	X	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Bulk, exterior surface, hardness	X	X	X	X	X	X	X	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Exterior 1/16 inch, impact	-	-	X	-	X	-	X	X	-	X	X	-	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Exterior 1/4 inch, mechanicals	X	X	X	-	X	-	X	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Exterior 1/4 inch, density	-	-	X	-	X	-	X	-	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Exterior 1/4 inch, burning rate	-	-	X	-	-	-	X	-	-	-	X	-	-	-	X	-	-	X	-	-	-	X	-	-	X	-	-	-	X	-	-	X
Bulk, interior, mechanicals	X	X	X	X	X	X	X	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Bulk, interior, hardness	X	X	X	X	X	X	X	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Bulk, interior, density	-	-	X	-	-	-	X	-	-	-	X	X	X	X	X	X	X	X	-	X	-	X	X	X	X	-	X	-	X	X	X	X
Bulk, interior, heat of explosion	-	-	X	-	-	-	X	-	-	X	X	-	X	-	X	X	X	X	-	X	-	X	X	X	X	-	X	-	X	X	X	X
Bulk, interior, burning rate	-	X	X	-	X	-	X	-	-	-	X	-	X	-	X	-	X	X	-	X	-	X	-	X	X	-	X	-	X	-	X	X
Bulk, interior, DTA	-	X	X	-	X	-	X	-	-	X	X	-	X	-	X	-	X	X	-	X	-	X	-	X	X	-	X	-	X	-	X	X
Bulk, interior, sol/gel	X	X	X	X	X	X	X	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Bulk, interior, swelling ratio	X	X	X	X	X	X	X	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
Bulk, interior, crosslink density	X	X	X	X	X	X	X	X	-	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X

*This matrix shall be tailored by the procuring activity to require those tests and test conditions necessary to meet program objectives. The procuring activity shall also determine the aging temperatures and humidity (T_1, T_2, T_3 and H_1), storage conditions and times (see 1.3).

$T_1 < T_2 < T_3$

5.4.40.2 Bulk propellant.

5.4.40.2.1 Storage conditions. Twenty-four slabs of propellant approximately 6 X 6 X 3 inches shall be aged for various periods and under various conditions as specified in TABLE II and as shown below:

a. Under dry nitrogen at T_1, T_2 and T_3 to show the effect of thermal aging

b. Under wet nitrogen at T_2 and T_3 to show the effects of thermal and hydrolytic attack

c. Under dry air at T_2 and T_3 to show thermal and oxidative effects

d. Under air at T_2 in the presence of the specified relative humidity, H_1%

e. Under air at T_3 in the presence of the specified relative humidity, H_1%

The slabs in a. b. and c. above shall be stored in a 10-inch Pyrex vacuum desiccator fitted with a glazed porcelain support disc. Flanges and cocks shall be lubricated with a thin film of high vacuum silicone grease. The slabs in d. and e. above shall be stored in a controlled temperature and relative humidity conditioning oven in such manner that the slabs are exposed to the desired environment.

5.4.40.2.2 Storage preparation.

a. Under dry nitrogen. Place anhydrous molecular sieves in the bottom compartment of a desiccator, insert the support plate, place the slab on the plate and seat the top. Ten such assemblies shall be required. Evacuate the desiccator to about 30 mm of mercury (Hg) and fill with dry nitrogen. Repeat evacuation and filling two additional times. Pressure at ambient after final filling with nitrogen shall be:

1. Three containers, T_1 storage = atmospheric minus 10 mm of Hg
2. Four containers, T_2 storage = atmospheric minus 70 mm of Hg
3. Three containers, T_3 storage = atmospheric minus 90 mm of Hg

b. Under nitrogen at the specified relative humidity, H_1%. Four assemblies for T_2 storage and three for T_3 storage shall be prepared. Preparation shall be as specified in 5.4.40.2.2a with the exception that the bottom compartment of the desiccator shall be filled with a solution to yield the specified relative humidity, H_1%, at the respective storage temperatures.

c. Under dry air. The lower compartment of the desiccator shall be filled with anhydrous molecular sieve and the propellant slab supported above the desiccant on a support plate. Prepare four assemblies for T_2 storage and three for T_3 storage. While in elevated temperature storage the cock or valve shall be open. A Teflon tube shall be connected to the

cock and extended through the side or top of the heated chamber. The external end of the Teflon tube shall be connected to a vented drying tower filled with molecular sieves. The drying tower shall be at room (ambient) temperature.

d. Under wet air. The bulk propellant slabs shall be stored in a controlled temperature and relative humidity oven in such a manner that the slabs are exposed to the environment (see 5.4.40.2.1 and TABLE II for instructions and conditions).

5.4.40.2.3 Special ozone storage. Test set-up and equipment. The ozone accelerated aging equipment setup is shown in Figure 1. Filtered regulated air with a pressure of 69 kiloPascals (kPa) (10 psig) is supplied by an air pump. The air passes through an ozone generator, a distribution manifold, and flow meters prior to reaching the sample containers.. UV radiation converts oxygen in the air to ozone in the generators where the level of ozone is controlled by the amount of exposure of the UV source to the air flow. The ozone level is monitored by a Bendix Model 8002 Ozone Analyzer. This analyzer, currently state-of-the-art in ozone measurement, uses a photometric method for detecting the chemiluminescense reaction of ozone and ethylene. The analyzer is connected so as to measure both input and exhaust levels of ozone from any of eight desiccators. These desiccators are standard 7-liter glass chambers housed in temperature controlled ovens. The ozone/air mixture is introduced at the bottom of the desiccators since it has a higher specific gravity than air. The mixture then exits through the top of the desiccator.

To maintain strain on the propellant samples during testing, a special strain controlling fixture was designed and fabricated. This fixture can be adjusted to control the amount of strain on the propellant dogbones. The test procedure is presented and the special fixture is illustrated in the 1978 publication of "JANNAF Structures and Mechanical Behavior Working Group" (Service Life Sub-committee) 15th Meeting.

5.4.40.2.4 Storage. The propellant-desiccator assemblies shall be stored at the temperatues and for the times indicated in TABLE II.

5.4.40.2.5 Testing of stored propellant. After the specified storage time(s), the propellant shall be examined radiographically. Slabs that are cracked, fissured, or otherwise defective shall be removed from the program. The remaining propellant shall be tested to the extent necessary to describe properties after each aging period and condition. Tests may include but shall not be limited to those given in TABLE II and the following paragraphs:

5. Strand burning rate. The burning rate of intermediate withdrawals shall be determined at 77 ± 2°F and 1000 psi. The burning rate of the terminal withdrawals at each storage condition shall be measured at the conditions of the initial assessment (see 5.4.3).

6. Differential thermal analysis (DTA). DTA tests, as indicated in TABLE II, shall be conducted in accordance with 5.4. 15. In cases where appreciable degradation is indicated, as compared to the original assessment, vacuum stability tests as specified in 5.4.14 shall be required.

c. Sol/gel, swelling ratio and cross-link density. Duplicate tests shall be conducted in accordance with 5.4.36, 5.4.35, and 5.4. 37, respectively. Individual and average values shall be reported.

5.4.41 Microscopy.

a. Particle size analysis. The particle size analysis of solid ingredients shall be determined in accordance with the ASTM recommended practice for reporting particle size characteristics, ASTM Special Publication #234, (1958).

b. Interface samples (all types). Characteristics such as unbonding, migration phenomena, cracks, voids and porosity at a propellant/insulator interface, propellant/propellant interface, etc., shall be microscopically determined utilizing scanning electron microscopy (SEM) and/or stereomicroscopy. 1.0 x 1.0 x 0.25cm sections of the interface shall be shadowed with a conductive coating before analysis by SEM. The working distance of the stereomicroscope is the determining factor for sample size for stereo microscopical analysis.

c. Bulk propellant morphology. Propellant morphology shall be characterized utilizing polarized light microscopy for determining the presence of agglomerates, dewetting phenomena, crystalline desolvation, straining, particle growth and fragmentation, homogeneity and phase changes of oxydizer ingredients. The crystalline ingredients shall be removed from the propellant, placed on a microscope slide and an immersion medium such as a Cargille liquid added to the slide. After the slide has been prepared, a polarizing research microscope shall be utilized for characterizing the crystalline material. SEM analysis shall be utilized for determining bonding characteristics between the crystalline ingredients and the propellant matrix. Refer to 5.4.41b for sample preparation for analysis by SEM.

6. NOTES

6.1 Contract data requirements. Any data required for delivery in connection with this document shall be specified on a DD Form 1423

incorporated into the contract. Such data will be delivered as identified on completed (numbered) DIDs (Data Item Descriptions/DD 1664) which will be documented in the Applicable ADL (Authorized Data List) (see 4.2, 4.4 and 6.5).

6.2 Material safety data. Material safety data sheet requirements are applicable and should be specified in the contract as required by the Armed Services Procurement Regulations (ASPR) 1-323.2 (see 4.7).

6.3 Safety precautions. Safety precaution requirements are applicable and should be specified in the contract as required by ASPR 1-323 (see 4.6).

6.4 Deviations. Propellant properties, processing methods, and type of applications are quite diverse. Consequently, it may not be feasible to furnish the forms and quantities specified in 4.1.6 or perform all of the tests indicated in TABLE I. Such situations, if not clarified in the contract, must be resolved before the start of the program. Requests for deviations shall be addressed to the procuring activity and prior approval for deviations obtained in writing (see 5.4.14, 5.4.17, 5.4.18, 5.4.35, 5.4.36 and 5.4.37).

6.5 Documentation. When results of chemical analysis or other test data are supplied by the manufacturer, a detailed description of the procedure, or a nonproprietary reference, shall be required. In many cases, interaction among ingredients occurs and the final composition differs significantly from statement of nominal composition. In such cases, the best available information calculated from the amounts of ingredients actually added shall be acceptable (see 4.2 and 6.1).

6.6 Submission of samples. Unless otherwise specified in the contract, the characterization sample shall be sent to: Receiving Officer, Naval Ordnance Station, Indian Head, MD 20640, Attn: NSWC R20 (see 3.3). A copy of the documentation specified in 4.2 shall accompany the sample as specified in the contract.

6.7 Defects. Surface defects, cracks, voids, fissures, fuel rich areas, oxidizer rich areas, unbonding foreign material, or other defects of sufficient magnitude to constitute a safety problem or to compromise test results, shall be cause for rejection of individual units of a sample. In cases of disagreement, the judgement of the contracting officer shall be final.

6.8 Retests and additional tests. Provisions for retests and/or additional tests should be specified in the contract (see 4.5 and 5.4. 39).

6.9 Conversions. The following conversions shall apply for all temperatures specified in this document:

6.9.1 Temperatures.

°F	°C	°F	°C
-70	-57	77	25
-65	-54	86	30
-40	-40	100	38
-20	-29	120	49
0	-18	140	60
		165	74
		176	80
		180	82
		230	110

6.9.2 Rates.

°F/minute	°C/minute
1.8	1.0
9	5

~~6.9.3~~ Tolerances.

°F	°C
±1	±0.5
±2	±1
±5	±3
±10	±5.5

6.10 Supersession information. This standard includes the requirements of and, for Naval Sea Systems Command procurements, shall be used in lieu of Naval Air Systems Command (30003) purchase description AS 4177, "Solid Propellant Characterization: Sample Requirements and Test Plan".

Custodian:
Navy - OS

Preparing Activity:
Navy - OS

Project No. 1376-N135

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

OMB Approval No. 22-R255

INSTRUCTIONS: The purpose of this form is to solicit beneficial comments which will help achieve procurement of suitable products at reasonable cost and minimum delay, or will otherwise enhance use of the document. DoD contractors, government activities, or manufacturers/vendors who are prospective suppliers of the product are invited to submit comments to the government. Fold on lines on reverse side, staple in corner, and send to preparing activity. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements. Attach any pertinent data which may be of use in improving this document. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity.

DOCUMENT IDENTIFIER AND TITLE: MIL-STD-2100(OS) PROPELLANT, SOLID, CHARACTERIZATION OF (EXCEPT GUN PROPELLANT)

NAME OF ORGANIZATION AND ADDRESS

CONTRACT NUMBER

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE DOCUMENT CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?

   A. GIVE PARAGRAPH NUMBER AND WORDING.

   B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES

2. COMMENTS ON ANY DOCUMENT REQUIREMENT CONSIDERED TOO RIGID

3. IS THE DOCUMENT RESTRICTIVE?

   ☐ YES ☐ NO *(If "Yes", in what way?)*

4. REMARKS

SUBMITTED BY *(Printed or typed name and address - Optional)*

TELEPHONE NO

DATE

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