INCH-POUND
MIL-DTL-85548C (OS)
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SUPERCEDING
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DETAIL SPECIFICATION

PROPELLANT, CAST COMPOSITE, N-60

This specification is approved for use by the Naval Sea Systems Command, Department of the Navy, and is available for use by all departments and agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the manufacture and acceptance of one type of cast composite propellant, N-60, referred to herein as the propellant.

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3, 4, and 5 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections of this specification, whether or not they are listed.

2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

DEPARTMENT OF DEFENSE SPECIFICATIONS

MIL-A-192	Ammonium Perchlorate
MIL-A-85495	Antioxidant, T-Butylphenol Type 2, 2-Methylenebis (4-Methyl-6-T-Butylphenol)
MIL-I-85370	Iron Oxide, (Monohydrate) Yellow

Comments, suggestions, or questions on this document should be addressed to Commander, Indian Head Division, Naval Surface Warfare Center, Air Engineering Branch, 101 Strauss Avenue, Indian Head, MD 20640-5035, or e-mailed to amanda.penn@navy.mil. Since contact information can change, you may want to verify the currency of this information using the ASSIST Online database at https://assist.daps.dla.mil.

AMSC N/A FSC 1370

MIL-R-85545 Rocket, GTR-18A

DEPARTMENT OF DEFENSE STANDARDS

DOD-STD-2101 Classification of Characteristics

MIL-STD-286 Propellants, Solid: Sampling, Examination, and Testing

(Copies of these documents are available online at https://assist.daps.dla.mil/ or from the Standardization Documents Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.2.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

DRAWINGS

NAVAL AIR SYSTEMS COMMAND (NAVAIR) (CAGE Code 30003)

1335AS280 Rocket, GTR-18A

1335AS286 Zinc Powder

1335AS287 Dicyclohexylmethane 4, 4^1 – diisocyanate

1335AS288 Cure Catalyst; Triphenyl Bismuth

(Unless otherwise indicated, copies of Naval Air Systems Command drawings are available from the Naval Air Technical Services Facility (NATSF) (Code 312), 700 Robbins Avenue, Philadelphia, PA 19111-5096.)

PUBLICATIONS

NAVAL SEA SYSTEMS COMMAND

WS 19740 Material Specification for Dioctyl Adipate

WS 20700 Polybutadiene, Linear, Hydroxyl Terminated (R45 Type II)

(Unless otherwise indicated, copies of Naval Sea Systems Command drawings are available from the Naval Ordnance Station, Technical Documents (Code 802), Louisville, KY 40214-5001.)

2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

CHEMICAL PROPULSION INFORMATION AGENCY

CPIA, Publication No. 21 The Solid Propellant Mechanical Behavior: Method for Determining Uniaxial Tensile Test at Constant Strain Rate

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

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM B 822	Standard Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering
ASTM D 792	Specific Gravity and Density of Plastics by Displacement

(Copies of these documents are available online at http://www.astm.org or from the American Society for Testing and Materials Customer Service, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.4 Order of precedence. Unless otherwise specified herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 First article. When specified (see 6.2), a sample shall be subjected to first article inspection (see 6.4) in accordance with 4.3.

3.2 Composition. Composition of the propellant and requirements for each material shall be as specified in Table 1.

Table 1: Composition

Material[1]	Requirements	% by weight[2]
Hydroxyl terminated polybutadiene (HTPB)	WS 20700	
2, 2[1] – Methylenebis – (4-methyl-6 tertiary butylphenol) (DMMBP)	MIL-A-85495	11.970[3]
Dicyclohexylmethane 4, 4[1] – diisocyanate (H_{12}MDI)	1335AS287	
Di – (2 ethylhexyl) adipate (DOA)	WS 19740	3.000
Triphenyl bismuth	1335AS288[4]	0.030
Ferric oxide (yellow) ($Fe_2O_3H_2O$)	MIL-I-85370	1.000
Zinc Powder	1335AS286[5]	39.000 – 41.000[6]
Ammonium Perchlorate (AP), Grade C bimodal blend (see Table 3)	MIL-A-192	43.000 – 45.000[6]

[1] For possible sources see 6.5
[2] See Table 2
[3] The total for HTPB, DMMBP, and H_{12}MDI is 11.970%. See 6.6 and 6.61 for calculations.
[4] See 6.7
[5] See 3.3
[6] The total for Zinc Powder and AP shall be 84.000 - 86.000%

Table 2: Weighing Accuracies

Material	Tolerances (± %)[7]
HTPB and DMMBP mixture	
HTPB	2.5
DMMBP	2.5
Propellant Mixture	
HTPB-DMMBP	0.5
H_{12}MDI	0.5
DOA	0.5
Triphenyl bismuth	1.5
Ferric Oxide	0.5
Zinc Powder	0.5
AP	0.5

Table 3: Ammonium Perchlorate Composition

Component Nominal Size	Ratio In Blend[8]	MIL-A-192		Comments
		Grade	Class	
Ground12 ± 4 micron	61.37 – 75.00	C	N/A	See 4.5.1 and 4.5.3
200 Micron	25.00 – 38.63	C	4	Round type

3.3 Gassing of zinc powder. The zinc powder shall not exhibit gassing when tested according to 4.5.2.

3.4 Chemical, mechanical, and ballistic properties. The cured propellant samples used for the determination of the characteristics listed below shall be stored at 77 ± 10°F in a condition where moisture absorption is minimized. Excursions from these conditions for periods of up to 8 hours for handling, transportation, and machining are permissible. The propellant shall not exceed a stored life of 2 weeks between the end of cure and the start of analysis.

3.4.1 Density (M101). The density of the cured propellant shall be 2.23 ± 0.03 $\frac{g}{cm^3}$ at 77 ± 3°F (see 4.6.1).

3.4.2 Mechanical Properties. The mechanical properties of the cured propellant shall be as follows (see 4.6.2):

a. Tensile Strength: 100psi minimum
b. Elongation: Not less than 8% at maximum tensile load

[7] % of weight of material in the mixture
[8] May be varied within the range shown to adjust burning rates to the requirements of 3.4.3

3.4.3 Strand Burning Rate. The strand burning rate (r_b) in inches per second, shall be obtained as specified in 4.6.3 and shall fall within the band specified by the following equation:

$$R_b = AP^{0.4} \frac{in}{s}$$

Where:

$A = 0.030 \pm 0.006 \left(\left(\frac{in}{s}\right)(psi_a)^{-0.4}\right)$
P = average actual pressure (psi_a) (see 4.6.3)

The equation is valid for pressures between 15 and 50 psi absolute psi_a at 77 ± 3°F (see 4.6.3).

3.4.4 Heat of Explosion (M102). The heat of explosion shall be 650 ± 70 $\frac{cal}{g}$ (see 4.6.4).

3.5 Stability. During the time the tests specified herein are performed, the propellant shall not exhibit any physical, mechanical, or ballistic changes from start to completion of tests.

3.6 Identification and Markings. Each propellant batch shall be assigned an identification designation, which shall include the date of mix. Each chemical used in the batch shall be recorded by its name, production lot number, and quality assurance department certification. The marking of any propellant samples, containerized propellant, or other items which contain propellant shall have the propellant's batch designation clearly shown.

3.7 Workmanship. The propellant shall be free from any contamination or foreign material that would render it unsuitable for its intended use.

4. VERIFICATION

4.1 Classification of Inspections. The inspection requirements specified herein are classified as follows:

a. First article inspection (see 4.3)

b. Quality conformance inspection (see 4.4)

4.2 Inspection Conditions. Unless otherwise specified (see 6.2), inspection conditions shall be as follows:

Temperature: 65 - 90°F

Humidity: 95% maximum

4.3 First Article inspection. First article inspection shall be performed by the contractor after award of contract and prior to production, at a location acceptable to the Government. First article inspection shall be performed on samples which have been produced with equipment and procedures normally used in production (see 6.3).

4.3.1 Sampling. Unless otherwise specified in the contract (see 6.2), the first article sample shall consist of a block of propellant which has been vacuum cast and cured in accordance with note 3 of drawing 1335AS280. The propellant sample shall be large enough for all first article samples required in Table 4, but not larger than 700in^3.

4.3.2 Inspection Routine. The first article sample shall be subjected to the tests specified in Table 4.

4.3.3 Failure. Failure of the sample to meet any of the requirements shall be cause for first article rejection.

Table 4: Inspection Routine

Tests	Number of Inspection Units[9]		Acceptance Criteria	
	First Article	Quality Conformance	Accept	Reject
Density	10	2	0	1
Mechanical properties	10		0	1
Strand Burning Rate	9		0	1
Heat of Explosion	10	2	0	1

4.4 Quality Conformance Inspection. Quality conformance inspection shall be performed on each lot of propellant (see 6.3).

4.4.1 Sampling. Unless otherwise specified in the contract (see 6.2), the quality conformance sample shall consist of a block of propellant which has been vacuum cast and cured in accordance with note 3 of drawing 1335AS280. The propellant sample shall be large enough for all quality conformance samples required in Table 4, but not larger than 300in^3.

4.4.2 Lot. Unless otherwise specified in the contract, a lot shall consist of all the propellant manufactured in one batch from a single lot of each raw material.

4.4.3 Inspection Routine. The inspection routine shall be as specified in Table 4.

[9] See 6.9.2 for definition.

4.4.4 Classification of Characteristics. The characteristics verified by the tests and examinations herein are classified as critical, major, or minor in accordance with DOD-STD-2101. Requirements that are critical are identified by the symbol (C) and those that are major are identified by the symbol (M). The number following the classification symbol indicates the serial number of the requirement. Requirements not annotated with a classification code are minor requirements.

4.5 Test Methods for Material.

4.5.1 Particle Size of Ammonium Perchlorate. The 6 micron (nominal) ammonium perchlorate shall be tested for particle size in accordance with ASTM B 822.

4.5.2 Gassing of Zinc Powder. Each lot of zinc powder shall be tested for gassing as follows:

Mix 75g of HTPB (containing 0.99% DMMBP), 8.8g of H_{12}MDI, 0.2g triphenyl bismuth, and 280g of zinc powder. Apply a vacuum and place in an oven at 135° ± 5°F for 24 ± 4 hours. Remove the sample from the oven and section it. The zinc powder will have settled and the portion of the sample with the settled zinc will have a large number of small bubbles if the zinc causes gassing. If the zinc powder exhibits gassing, it may be degassed and retested.

4.5.3 Surface Moisture Requirement of Ammonium Perchlorate. The 6 micron (nominal) ammonium perchlorate shall not exhibit a surface moisture weight greater than 0.035 percent when tested in accordance with MIL-A-192.

4.6 Test Methods.

4.6.1 Density. The density shall be determined on test specimens in accordance with ASTM D 792, Method A-2. The test temperature shall be 77° ± 3°F. An optional method for determining the density of the immersion liquid (see 6.8) is to test a piece of glass of accurately known density in the same manner as the specimens.

$$\text{Density of immersion liquid} = \frac{D(W_A - W_L)}{W_A}$$

Where:

D = density of glass standard

W_A = weight of glass standard in air

W_L = apparent weight of glass standard while immersed in liquid

4.6.2 Mechanical Properties Test. Tensile strength and elongation at maximum tensile load shall be determined on class B or class C tensile test specimens in accordance with the procedures for preparation, storing , conditioning, and testing of tensile specimens given in section 4.3 of CPIA Publication No. 21, class B – "Quality Control Testing" or class C – "Development Screening Testing". These two methods shall give equivalent results for the material. The specimen shall be tested using a crosshead separation rate of 2 $\frac{in.}{min.}$ at a test temperature of 77° ± 2°F and 85% maximum relative humidity. An effective gauge length of 2.7in shall be used to calculate elongation.

4.6.3 Strand Burning Rate. Strand burning rate on cured specimens shall be determined according to MIL-STD-286, method 803.1. Strands shall be inhibited with epoxy inhibitor (Versamid 140/EPON 828 two part epoxyor equivalent). A minimum of three strands shall be tested at each of three pressures: 15 $^{+5}_{-0}$, 30 ± 5, and 45 ± 5 psi_a. Strands shall be burned at 77° ± 3°F. The chamber (starting) pressure and the maximum pressure attained shall be recorded and averaged. The pressure gage for this test shall have an accuracy of 1 psi_a and the timer shall have an accuracy of 0.5 seconds.

4.6.4 Heat of Explosion. This test shall be performed using an adiabatic calorimeter such as the Parr Instrument Company Series 1200. A Parr No. 1101 or 1108 double valve oxygen bomb (or equivalent) shall be used. The calorimeter shall be standardized with benzoic acid using oxygen according to the manufacturer's instructions. The heat of explosion test shall be conducted in the same manner as the standardization with the following limitations and exceptions:

a. Water shall not be added to the bomb.

b. The bomb shall be purged two times with 25 ± 1 atmospheres of helium or argon, then pressurize the bomb to 25 ± 1 atmospheres (using helium or argon) for testing.

Note: Impurities in the inert gas, such as oxygen or water can cause systematic high results. Each new bottle should be tested for suitability for this test by comparison with gas of known purity.

4.6.4.1 Calculation.

$$\text{Heat of Explosion } \left(\frac{cal}{g}\right) = \frac{W(t_2 - t_1)EE(\Delta T) - e_1 - e_2}{wt}$$

Where:

EE = energy equivalent of the bomb $\left(\frac{cal}{°C}\right)$

ΔT= temperature change (°C)

e_1 = fuze correction factor (cal)

e_2 = acid correction factor (cal)

wt = weight of the sample (g)

4.6.4.2 Specimens. A heat of explosion test specimen is defined as a single piece of solid propellant 2.5 ± 0.5g in weight, weighed to the nearest 0.0001g.

5. Packaging

This section is not applicable to this specification.

6. Notes

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended Use. The propellant covered by this specification is intended for use in the GTR-18A rocket. Since it was developed specifically for use in a military rocket, there is no commercial application.

6.2 Acquisition Requirements. Acquisition documents should specify the following:

a. Title, number, and date of this specification

b. If required, the specific issue of individual documents referenced (see 2.2.1 and 2.2.2).

c. If first article testing is required (see 3.1).

d. Inspection conditions if other than specified in 4.2.

e. Quality conformance sampling if other than specified in 4.4.1.

f. That the safety precaution requirements of the "Contractor's Safety Manual for Ammunition, Explosives, and Related Dangerous Material" (DoD 4145.26M) are applicable.

Note: When this specification 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" (NAVSEA OP 5) are applicable.

6.3 Data requirements. When this specification is used in an acquisition which incorporates a DD Form 1423, Contract Data Requirements List (CDRL), the data requirements identified below should be developed and delivered in accordance with the approved CDRL incorporated into the contract. When the provisions of DAR 7 104.9(n)(2) are invoked and the DD Form 1423 is not used, the data specified below should be delivered by the contractor in accordance with the contract or purchase order requirements. Deliverable data required by this specification is cited in the following paragraphs:

Reference Paragraph	Title
4.3 and 4.4	Test Plan
4.3 and 4.4	Report, Test -

6.4 First Article. When a first article inspection is required, the inspection should be performed on a first article sample as specified in 4.3.1. The contracting officer should include specific instructions in acquisition documents regarding arrangements for examinations, approval of first article test results, and disposition of first articles. Invitations for bids should state that the Government reserves the right to waive the requirement for samples for first article inspection for those bidders offering a product that has been previously acquired or tested by the Government. Bidders offering such products who wish to rely on previous production or test must furnish evidence with the bid that prior Government approval is presently appropriate for the pending contract. Bidders should not submit alternate bids unless specifically requested to do so in the solicitation.

6.5 Possible Sources of Supply. Products that have met the requirements of this specification in past procurement actions are listed below. This information is provided to assist with procurement and is in no way restrictive. It is provided to the procuring activity for informative purposes and is not to be construed as a waiver of any requirement of this specification, or as any limitation of additional supply.

Material	Possible Source
Hydroxyl-Terminated Polybutadiene (R-45HT)	Sartomer Company Inc. 502 Thomas Jones Way Exton, PA. 19341
Dicyclohexylmethane 4, 4^1 – diisocyanate (Desmodur W)	Bayer Polymers Coatings, Adhesives, and Sealants Business 100 Bayer Rd. Pittsburgh, PA. 15205
Di – (2-Ethylhexyl) Adipate (DOA)	Ashland Distribution Company 5200 Blazer Pkwy. Dublin, OH. 43017
2, 2^1 – Methylenebis – (4-methyl-6-tertiary-butylphenol) (CAO-5)	Sherwin Williams Co. Chemical Division Fords, NY. 03863
Triphenyl Bismuth	Boulder Scientific Co. P.O. Box 5480 Mead, CA. 80542
Ammonium Perchlorate (AP)	Kerr McGee Chemical Corp. Kerr McGee Blvd. Oklahoma City, OK. 73102
Zinc Powder	LDL Technology, Inc. 137 Pennsylvania Ave. Paterson, NJ. 07503
Ferric Oxide, Yellow ($Fe_2O_3H_2O$) (Yellow Iron Oxide)	Cities Service Co. 1440 N. Harbor Blvd. Fullerton, CA. 92635

6.6 Composition Calculation. The DMMBP is added to the HTPB and allowed to dissolve before mixing. One part of DMMBP is added to 100 parts of HTPB and this mixture is used in the propellant mix. Weight percentage of the HTPB and DMMBP mixture and H_{12}MDI are calculated as follows (see 6.6.1 for sample calculation):

$$\% \text{ HTPB and DMMBP mixture} = \frac{11.97}{\left(\frac{\left(NCO/_{OH}\right)\left(E_x + 0.00508\right)}{\left(1.01E_y\right)}\right) + 1}$$

% H_{12}MDI = 11.97 – (% HTPB and DMMBP mixture)

Where:

HTPB and DMMBP is a 100 part HTPB to 1 part DMMBP mixture

$NCO/_{OH}$ = Cure ratio. 0.90 to 1.10 will give near optimum tensile strength and hardness for the propellant. The cure ratio may be varied within 0.90 to 1.10 if needed so that the physical properties requirements are met.

E_x = OH equivalency per 100g of HTPB as determined by the laboratory for the lot of HTPB used

E_y = NCO equivalency per 100g of H_{12}MDI as determined by the laboratory for the lot of H_{12}MDI used

6.6.1 Sample Calculation. The percentages of H_{12}MDI and HTPB and PMMBP mixture used in each particular propellant mix are determined as follows:

a. The NCO (isocyanate) and OH (hydroxyl) equivalent weights are determined by laboratory analysis for the lots of H_{12}MDI and HTPB to be used in the propellant mix.

b. The following three equations are solved for percent IDPI and percent HTPB and DMMBP mixture:

(1) $$\frac{\left(NCO\ equivalents/_{100g}\right)(\%\ H_{12}MDI)}{\left(OH\ equivalents/_{100g}\right)(\%\ HTPB) + (0.568)(\%\ DMMBP)} = 0.90 \text{ to } 1.10$$

(2) (% HTPB and DMMBP mixture) = (1.01)(%HTPB)

(3) (% H_{12}MDI) + (% HTPB and DMMBP mixture) = 11.97%

Example:

Let

X = % HTPB and DMMBP mixture

X_1 = % HTPB

X_2 = % DMMBP

Y = % H_{12}MDI

Then

$X_1 + X_2 = X$

$X + Y = 11.97$

And

$$\frac{0.759(11.97 - X)}{0.081\left(X/_{1.01}\right) + 0.588\left(X - X/_{1.01}\right)} = 1.026$$

Or

$$X = \frac{11.97}{\frac{(1.026)(0.081 + 0.00588)}{(1.01)(0.759)} + 1.0} = 10.723\%$$

Therfore

$$X_1 = \frac{10.723}{1.01} = 10.617\%$$

X_2 = 10.723 – 10.617 = .106%

Y = 11.97 – 10.723 = 1.247%

6.7 Solvent for Triphenyl Bismuth. Grade A dichloromethane, in accordance with MIL-D-6998, may be used as a solvent for the triphenyl bismuth. This solvent shall be removed (evaporated) from the composition during mixing at low pressure.

6.8 Immersion Liquid. N-hexane has been found to be a satisfactory immersion liquid.

6.9 Definitions. For the purpose of this specification, the following definitions apply:

Batch – that quantity of material which at one time has been subjected to one or more chemical or physical processes (or combination thereof) intended to produce a product having uniform characteristics.

Sample Unit – a unit of propellant that is subjected to first article or quality conformance testing. The size of the unit depends upon the testing to be performed and is cited in the applicable test paragraph or a document reference within the paragraph.

Cure Ratio = $\frac{\left(NCO\ equivalents/_{100g}\right)(\%\ H_{12}MDI)}{\left(OH\ equivalents/_{100g}\right)(\%\ HTPB) + (0.588)(\%\ DMMBP)}$

6.10 Subject Term (key word) Listing.

Ammonium Perchlorate

HTPB (Hydroxyl terminated polybutadiene)

Heat of Explosion

6.11 Changes from Previous Issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extensive nature of the changes. Changes to this specification were authorized by ECP IH03002.

Custodian:
Navy - OS

Preparing activity:
Navy - OS
(Project No. 1370-2010-003)

Note: The activities listed above were interested in this document as of the date noted. Since organizations and responsibilities can change, you should verify the currency of the above information using the ASSIST online database at https://assist.daps.dla.mil/.