

MIL-T-18833(NOrd)

30 June 1955

SUPERSEDING
NAVORD OS 5038B
6 APRIL 1954

MILITARY SPECIFICATION

TAPE INHIBITING, ETHYL CELLULOSE

1. SCOPE

1.1 Scope. - This specification covers inhibiting tape manufactured for use as the peripheral inhibiting wrap for Rocket Propellant Grains (Ballistite Type).

1.2 Classification. - The inhibiting tape covered by this specification shall be ethyl cellulose as specified (see 3.1).

2. APPLICABLE DOCUMENTS

2.1 The following specifications, standards, drawings, and publications, of the issue in effect on date of invitation for bids, form a part of this specification:

SPECIFICATIONS:

FEDERAL:

L-P-406 - Plastics, Organic: General Specifications, Test Methods.
VV-L-791 - Lubricants, Liquid Fuels, and Related Products; Methods of Inspection, Sampling, and Testing.

MILITARY:

JAN-P-125 - Packaging and Packing for Overseas
Shipment; - Barrier-Materials, Waterproof,
Flexible.

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NAVY DEPARTMENT:

General Specifications for Inspection of Material.

STANDARDS:

MILITARY:

MIL-STD-105

Sampling Procedures and Tables for
Inspection by Attributes.

MIL-STD-129

Marking for Shipment and Storage.

DRAWINGS:

BUREAU OF ORDNANCE:

BuOrd Drawings 652148 Inhibiting Tape (Ethyl Cellulose).

PUBLICATIONS:

BUREAU OF ORDNANCE:

OD 7904

Taliani Test for Determination of
Stability of Solid Propellants

OD 8480

Tensile Strength Determination of
Propellant Powder (Peripheral)
Inhibitor Bond

(Copies of specifications, standards, drawings and publications required by contractors 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 publications form a part of this specification:

AMERICAN SOCIETY FOR TESTING MATERIALS PUBLICATIONS

A.S.T.M. D914-50

Standard Methods of Testing Ethyl
Cellulose.

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(A.S.T.M. Publications are available from the American Society for Testing Materials, 1916 Race Street, Philadelphia 3, Pa.)

3. REQUIREMENTS

3.1 Material. - The material shall consist of suitable ethyl cellulose and plasticizer(s) so compounded as to be suitable for processing into tape. Suitable dyes or other colorants may be added where required to obtain colors specified by the procuring agency (see 3.3).

3.1.1 Plasticizer. - The contractor shall certify the chemical name of any plasticizer(s) used in the tape, and sample(s) shall be submitted to the procuring agency when requested.

3.1.2 Cellulose Modification. - The manufacturer shall state the average percent of ethylation and the average viscosity (as measured by viscosity of a five percent solution of the flake in 60:40 toluene-Formula 2B Alcohol solvent mixture) of the ethyl cellulose used in manufacturing the tape and shall specify the methods of analysis used in making the determinations. Both determinations are to be run in triplicate and the average values reported.

3.2 Method of Manufacture. - The tape shall be made by the continuous extrusion method or equivalent as approved by the procuring agency.

3.3 Color. - Unless otherwise specified by the procuring agency, the tape shall be furnished uncolored. When colored tapes are specified, the color shall match the sample furnished by the procuring agency (see 6.2 and 6.3).

3.4 Formulation. - The tape shall be compounded from the following materials:

3.4.1 Ethyl cellulose flake
Ethoxyl content 45.5% to 46.5% (A.S.T.M. D914-50)
Viscosity of flake 65 to 85 cps.
(When measured at 25°C on a 5% solution of the flake in a solvent consisting of 60 parts toluene and 40 parts Formula 2B Alcohol by Method 305.2 of Specification VV-L-791

NOTE: Two acceptable sources of this material are:

Dow Chemical Company (Ethocel, Standard Grade, 70cp), and Hercules Powder Company (Ethyl cellulose, K70A Grade, completely de-ashed).

3.4.2 Plasticizer, substituted Phenyl Ether - with bis (p-(1, 1, 3, 3, - tetramethyl butyl) phenyl) ether as the principal constituent.*

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(1) Color	Light Amber
(2) Minimum % Transmission through 1 cm. of liquid (450 millimicron wave length)	45
(3) Viscosity at 25°C, Centistokes	4000 - 8000
(4) % Volatile (148.5°C and 4 mm. mercury for one hour) maximum	9
(5) Acid number maximum	0.5
(6) Specific Gravity	0.92 - 0.95

*Dow P-1099 plasticizer is one commercial form of this material.

3.4.3 Dye or Colorants. - No dye or other colorant is permissible unless otherwise specified by the procuring agency.

3.4.4 Stabilizer. - Diamyl Phenol 110 (manufactured by Sharples Chemical Company) and Stabilizer A-5 (manufactured by Carbide and Carbon Company). The use of these two stabilizers is permitted with Hercules ethylcellulose (see 3.4.5). Alternate stabilizers may be used only with the permission of the procuring agency.

3.4.5 Proportions. - The ingredients of the tape shall be compounded in the following proportions:

Ethyl Cellulose Flake	100 Parts
Stabilizers (See Section 3.6D)	0-2.2 Parts *
Plasticizer	17.7-25.0 Parts

The variation in formulation given above is allowed so that the proportions of flake and plasticizer can be varied to bring the compounded material within that required to give the proper extrudability and desired physical properties. (See 3.7).

*The stabilizers are included for use with Hercules Flake only. When stabilizers are used, a corresponding reduction in the limits of plasticizer is required.

3.4.6 Rework. - Rework or scrap may be included in the plastic if the use of reworked material does not affect the tape properties listed in this specification. It is recommended that rework content of the extruded tape be held to 10 percent or less. Scrap or rework incorporated in the

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tape shall be of the same source and basic formulation as the virgin material, but need not be from the same factory run or batch as the virgin material.

3.5 Dimensional Requirements. - All dimensions and limits shall be as selected and requested by the procuring activity from the schedule of sizes shown on BuOrd Drawing No. 652148. All dimensional requirements hold only when the material is conditioned and measured at 77 \pm 2°F and 50 \pm 5% Relative Humidity.

3.5.1 Roll Characteristics

3.5.1.1 Core. - The Inhibiting Tape shall be tightly wound on a multilayer paper core with dimensions as shown on Drawing 652148. The core shall not be collapsed during or after the winding operation.

3.5.1.2 Tightness of Roll. - Tape rolls shall be tightly wound so as to prevent telescoping of the roll during handling and shipping operations (see 4.4.1.4.1).

3.5.1.3 Splices. - One splice shall be permissible within the 3000 foot length rolls. No splices shall be permissible within the 1500 foot length rolls (see 4.4.1.4.4).

3.5.1.4 Telescoped or Crimped Edges. - The rolls of tape shall be smoothly wound so that no tape edges project or become crimped during winding or subsequent handling and packaging (see 4.4.1.4.6).

3.5.1.5 Time Limits and Storage Conditions. - Because of the nature of this material and the tendency for dimensions to change upon storage, the manufacturer shall be held accountable for the dimensions of his product only during the period of two days to 120 days after final slitting and winding of the tape rolls. Any other properties directly affected by dimensional changes are also subject to this time limitation. In order to minimize dimensional changes of the samples chosen for testing, the samples shall be stored and shipped under conditions which do not exceed the following limits: Temperature, 50 to 100°F - Relative Humidity, 30 to 80%. The manufacturer will not be held accountable for the dimensions of his product under the 120 days limit imposed above after receipt of the product by the procuring agency unless the procuring agency stores the product under the conditions outlined above.

3.6 Visual Appearance

3.6.1 Extrusion Defects

3.6.1.1 Fisheyes and Gels. - The tape shall be relatively free from fisheyes or gels when inspected as indicated in section 4.4.1.5.1.1.

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For purposes of this specification, a fisheye or gel shall be considered as any solid particle imbedded in the tape that measures more than 0.0005 inch thicker than the surrounding tape.

3.6.1.2 Bubbles. - No bubble or void in the body of the plastic into which a 1/16 inch diameter circle can be fitted shall be permissible. No elongated bubble or void with a length of 1 inch or more and a width of 0.010 inch or more shall be permissible. No group or cluster of pinpoint bubbles or bubble streaks with a cluster grouping into which a circle of 1/4 inch diameter can be fitted shall be permissible (see 4.4.1.5.1.2).

3.6.1.3 Striations. - No striations shall be permissible that permit the tape thickness to vary more than 0.0003 inch from the thickness of the tape surrounding the striation, as determined by gage measurements at the point of striation and on the surrounding tape (see 4.4.1.5.1.3).

3.6.1.4 Laminations. - The tape shall not contain laminations that can be detected by the inspection method described in section 4.4.1.5.1.4.

3.6.2 Color. - The natural color of the tape should be a light clear yellow or amber transparent. A dark yellow or brown color is indicative of undesirable excessive degradation of the plastic during processing. The color of a single layer of tape viewed against a white background under a daylight lighting shall be no darker than Kodak C. C. 15 color compensating filter viewed under the same conditions as the tape (see 4.4.1.5.2).

3.6.3 Clarity. - The major requirement of this section is sufficient clarity of the tape so that inhibited grains may be inspected visually for inhibiting defects. (see 4.4.1.5.3).

3.7 Physical Properties. - The tape covered by this specification shall conform in physical properties of the values listed in Table 1:

TABLE 1-PHYSICAL REQUIREMENTS

<u>Property</u>	<u>Specification</u>
Tensile strength, min psi	4,950
Elongation, min, percent	28
Specific gravity	1.06 to 1.17
Water absorption, max percent	2.8
Soluble matter lost, max percent	0.2
Weight loss on heating, mg/sq cm, max percent	0.2

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Flow temperature, °C	127 to 137°C
Heat distortion temperature, min °F	130°F
Coefficient of linear expansion, 10 ⁻⁵ in/in/°F	4.3 to 6.4
(see 4.4.1.6).	

3.8 Migration Characteristics. - When four samples of tape (taken from separate tape rolls) are each tested in duplicate by the procedure described in section 4.4.1.7, the migration properties of the tape shall fall within the following limits: (Duplicate tests are to be averaged for each roll and reported as a single value.)

Average weight gain of four samples	20 to 24%
Range of weight gain (difference between highest and lowest of the four average sample results), max.	2.5%
Average nitroglycerin gain of four samples	14 to 18%
Range of nitroglycerin gain (difference between highest and lowest of the four sample results), maximum	4%
Average inert gain of four samples (weight gain minus nitroglycerin gain)	4 to 8%

3.9 Wrappability. - Inhibiting tape shall wrap satisfactorily when used to inhibit propellant grain (Ballistite Type) as described in section 4.4.1.8. The presence of stretched or warped tape sections within a roll is likely to cause the tape to wrap unsatisfactorily.

3.10 Adhesion. - Inhibiting tape shall adhere to the propellant grain with a bond strength of 850 psi or greater, when tested as specified in section 4.4.1.9.

3.11 Taliani Stability. - When tested simultaneously by the same procedure, in triplicate, carefully blended samples (each consisting of 1/2 gram of finely chopped ethyl cellulose tape and 1/2 gram of the same lot of comminuted N-5 propellant) shall not exhibit an average Taliani slope in mm/minute at the 100 mm level in excess of the average value obtained with the one gram sample of N-5 propellant alone. The individual value of slope shall be determined as that of the tangent to a plot of pressure versus time on linear coordinate paper.

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3.12 Test Limitations. - The requirements of paragraph 3.8, 3.9, 3.10 and 3.11 shall be deemed to have been met by the manufacturer without testing by the local Government Inspector so long as the quality of the material in all other respects continues to equal the quality of the pilot lot. However, if within forty-five days of delivery, it is determined by a test facility designated by the procuring agency (see 4.3.3) that the tape fails to meet the requirements of paragraphs 3.8, 3.9, 3.10 or 3.11, the procuring agency may, at its discretion, return the defective lot (or lots) to the manufacturer for replacement.

4. QUALITY ASSURANCE PROVISIONS AND TEST REQUIREMENTS

4.1 Lot^s

4.1.1 Preproduction Lot. - Prior to production under contract the manufacturer shall produce an acceptable preproduction lot of inhibiting tape consisting of the first 50 rolls of inhibiting tape made at the plant in which he is to fill his contract, from the same materials, on the same type of equipment, and by the same process(es) that he proposes to use in executing the contract.

4.1.2 Production Lot. - Unless otherwise specified, a production lot shall consist of inhibiting tape produced in a single continuous run or extrusion of film from which the tape rolls are cut or slit according to one set of drawings and prepared as an individual shipment. Maximum size of a production lot shall be limited to 60,000 pounds of material. When material lots or major operating conditions are changed, a new production lot shall be designated in accordance with the practice outlined in MIL-STD-105.

4.1.3 Inspection Lot. - As applied to Government inspection of units of product, the term "lot" shall mean "inspection lot", i.e., a collection of units of product used as a basis for Government inspection. Unless otherwise specified, the number of units of product in "inspection lots" shall be determined by the Government Inspector and may differ from the quantity designated in the contract or order as a lot for production, shipment or other purpose.

4.2 Inspection Unit. - For purpose of acceptance or rejection in accordance with this specification and MIL-STD-105, an inspection unit shall be a finished roll of tape.

4.3 Sampling

4.3.1 Sampling Preproduction Lot and Periodic Production Acceptance Tests. - Fifty rolls shall be submitted as a preproduction lot and fifty rolls shall be selected at random during production of each 50,000 roll increment of a manufacturer's contract production to be sent to a testing activity designated by the procuring agency for tests in accordance with Table II. Acceptance or rejection criteria of the lot tested shall be in accordance with the rejection requirements of Table II.

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TABLE II PREPRODUCTION LOT AND PERIODIC PRODUCTION

SAMPLING PLAN

<u>Test</u>	<u>Section</u>	<u>Sample Size</u>	<u>Rejection Level</u>	
Migration Characteristics	3.8	4 rolls	Any deviation from specification	
Taliani Stability	3.11	3 rolls		
Thickness	4.4.1.1	10 rolls	One or more defective ratings	
Width	4.4.1.2	10 rolls		
Core Dimensions	3.7.1.1	10 rolls		
Roll Diameter	4.4.1.4.3	10 rolls		
Width of Roll	4.4.1.4.5	10 rolls		
Telescoped or Crimped Edges	3.7.1.4	10 rolls		
Wrappability	3.9	5 rolls		
Adhesion	3.10	5 rolls		
Tightness of Roll	4.4.1.4.1	10 rolls		Two or more defective ratings
Physical Properties	3.7	3 rolls		
Extrusion Defects	3.6.1	10 rolls		
Color of Tape	3.6.2	10 rolls		
Clarity of Tape	3.6.3	10 rolls		

The following properties are considered as special requirements and shall be certified by the manufacturer as meeting the requirements of the specification:

Material	(3.1)
Method of Manufacture	(3.2)
Color	(3.3)
Formulation	(3.4)
Splices within Roll	(3.5.1.3)
Packaging	(5.1)
Label on Roll	(5.2.1)
Marking of Container	(5.2.2)
Length of Tape on Roll	(4.4.1.4.2)

Copies of this certification shall be furnished to the Government Inspector concerned, the activity designated to test the preproduction lot, and the procuring agency.

Sample rolls may be tested for more than one property: Failure of the test rolls to conform with any requirement previously evaluated does not prevent testing of that roll for additional defects.

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4.3.2 Sampling for Production Lot Acceptance Tests. - The Government Inspector shall select tape rolls for testing in accordance with MIL-STD-105 unless otherwise specified in the appropriate detail description of the tests involved. Unless otherwise approved, no tape shall be accepted as a production lot until the pilot lot has been tested and found to conform to this specification.

4.3.3 Inspection for Production Lots. - Inspection for production lots may be made at the plant of the manufacturer by the Government Inspector, or at such other place as the procuring agency may specify.

4.4 Tests4.4.1 Preproduction Lot and Periodic Production Lot Tests

4.4.1.1 Thickness. - Unroll a section of tape (100 feet or more), measure and record the thickness of the unrolled section at twenty points selected at random along the tape strip. Determine the average of the twenty measurements. Select five places along the tape strip and measure the thickness of the center and both edges at these five points across the face of the tape. Grade any roll defective that does not meet the requirements of Drawing 652148.

4.4.1.2 Width. - Unroll a section of tape (100 feet or more), measure and record the width of the unrolled section at twenty (or more) points selected at random along the tape strip. Grade any roll defective that does not meet the requirements of Drawing 652148.

4.4.1.3 Core. - Measure the inside diameter, width, and wall thickness of the tape roll cores. Grade any roll defective that does not meet the requirements of Drawing 652148.

4.4.1.4 Roll Dimensions and Characteristics

4.4.1.4.1 Tightness of Roll. - Secure a cardboard core or pipe with the dimensions as follows: Outside diameter - not less than $3/32$ inch smaller than the minimum inside diameter of the roll core, Wall Thickness $3/16$ inches minimum, Length 12 inches minimum. Fasten this core in an upright position. Insert on this core a sponge rubber cushion pad with the following dimensions: Thickness 1 inch \pm $1/16$ inch, inside diameter $1/32$ inch interference to $1/16$ inch clearance with the outside diameter of the cardboard pipe, outside diameter $3\ 3/4$ inches \pm $1/16$ inch. Then insert in place over the core above, a paper core the same size as that used for the tape roll to be tested and let it rest over the rubber cushion pad at the base of the vertical inner core.

Insert the roll on the upper end of the vertical core and let the roll drop a distance of eight inches onto the outer core at the bottom of the twelve-inch vertical core. Any rolls that do not still meet the width of roll requirements of Drawing 652148 shall be graded defective.

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4.4.1.4.2 Tape Length on Roll. - Check the label on the inside of the roll core for the marked length of tape on the roll. This statement by manufacturer may be verified by unrolling the tape and measuring the linear feet of tape found on the roll. (Since it is very difficult to rewind tape satisfactorily after unrolling, any tape roll that is checked for length will probably have to be scrapped if the tape is not used as it is unreeled.) The manufacturer should be notified if any roll length is found to deviate by more than 50 feet from the length marked on the roll label. If the inspector samples a tape roll, he should correct the roll label to allow for the amount of tape he has removed. Check the roll labels and measured footage readings against the requirements of Drawing 652148 and notify the Government Inspector and procuring agency if more than 1% of any shipment is found to be incorrectly labeled as to tape length on roll, or if the tape rolls are found to contain less than the limits specified.

4.4.1.4.3 Roll Diameter. - Measure the outside diameter of the finished rolls and grade any roll defective that exceeds the requirements of Drawing 652148.

4.4.1.4.4 Splices. - Hold the tape roll with the flat side of the roll facing a light source and visually inspect the roll by transmitted light. Any splice will usually show up as a break or discontinuity in the tape layers on the roll and by a spot where the light enters between the edges of the splice. Any splices encountered during the use of the inhibiting tape shall be reported to the procuring agency and the inspector concerned. Any rolls found to contain splices in excess of the requirements of Drawing 652148, shall be graded defective.

4.4.1.4.5 Width of Roll. - Place the tape roll between two parallel plates and adjust the plates to fit against both faces of the roll. Measure the distance between the parallel plates and grade as defective any roll that does not meet the requirements of Drawing 653148 for width.

4.4.1.4.6 Telescoped or Crimped Edges. - Visually inspect the tape roll for presence of crimped edges or edges that protrude and are liable to become crimped during handling, packaging or shipping operations. Any roll showing crimped or telescoped edges shall be graded defective as required by section 3.5.1.4.

4.4.1.5 Visual Appearance.

4.4.1.5.1 Extrusion Defects.

4.4.1.5.1.1 Fisheyes and Gels. - Unroll a section of tape (100 feet or more) and cut five sections three feet long at random from the unrolled tape. Mark the fisheyes and gels visible in the three feet sections.

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Measure their thickness versus the thickness of the surrounding tape by means of a micrometer or other suitable thickness gage that is capable of accurate measurement of 0.0001 inch. Count and record the number of fisheyes or gels in each strip with a thickness differential of 0.0005 to 0.0025 inch. Count and record the number of fisheyes or gels with a thickness differential of 0.0025 inch or greater. If any roll contains a particle with a thickness differential of 0.0025 inch or more, grade the roll defective. If any three foot section contains fifty or more particles with a thickness differential in the range 0.0005 to 0.0025 inch, grade the roll from which the strip was taken as defective (section 3.6.1.1).

4.4.1.5.1.2 Bubbles. - Unroll a section of tape (100 feet or more), and cut five sections three feet long at random from the unrolled tape. Examine the cut sections visually for presence of bubbles. If any bubbles or bubble clusters are observed, measure either the diameters or lengths and widths of these bubbles or clusters and grade any roll defective that exceeds the requirements of section 3.6.1.2.

4.4.1.5.1.3 Striations. - Unroll a section of tape (100 feet or more) and cut five sections three feet long at random from the unrolled tape. Examine the cut sections visually for presence of striations (continuous or semi-continuous ridges or valleys running lengthwise or across the face of the tape). If striations are observed, measure the thickness of the tape at the point of striation and on the surrounding tape. Grade any roll defective that exceeds the requirements of section 3.6.1.3.

4.4.1.5.1.4 Laminations. - Unroll a section of tape (100 feet or more), and cut five sections three feet long at random from the unrolled tape. Take each three foot strip and tear the strip lengthwise in a series of "Z" or sawtooth rips extending to within 1/4 inch of each edge of the tape. Examine the torn edges for thin sections or feather edges that peeled away from the body of the tape during the tearing operation. These sections or edges that peel away from the tape body are indications of laminations. Grade any roll defective that shows laminations as specified in section 3.6.1.4.

4.4.1.5.2 Color. - Cut a small section of tape (about one foot) from the end of the tape roll. Place this section against a white background. Place a Kodak C. C. 15 color compensating filter beside the tape specimen and visually compare the color of the sample and the filter under daylight lighting. A roll showing color darker than the standard shall be graded defective as specified in section 3.6.2.

4.4.1.5.3 Clarity. - Cut a strip (about three feet) from the end of the tape roll. Cut this strip into six pieces. Stack the six pieces and place the stack in contact with a sheet printed with Century Roman 8 point type (such as a sheet from Federal Specification L-P-406). Press the stack of

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tape firmly against the printed sheet and try to read the printing through the six layers of plastic. If the printing is not distinct and legible when viewed through the plastic, the roll shall be graded defective in accordance with section 3.6.3.

4.4.1.6 Physical Test. - Physical test shall be tested in accordance with the procedures on samples prepared as indicated below:

Cut the tape roll into strips and condition these strips as specified in specification L-P-406, Testing conditions. Use these conditioned strips in the following tests:

	Spec L-P-406	Method No:
a. Tensile Strength		1013
b. Elongation		1013
c. Specified Specific Gravity		5011
d. Water absorption		7031
e. Soluble matter loss		7031
f. Weight loss on heating		7041

4.4.1.6.1 Flow Temperature Test. - Punch out the tape into a preformed pellet. Use this preformed pellet in testing the tape for flow temperature, in accordance with specification L-P-406, method 2041.

4.4.1.6.2 Heat Distortion Test. - Mold a 1/2 x 1/2 x 5 inch test bar in an A.S.T.M. compression type mold, such as shown in BuOrd Sketch 337131 (see Fig. 1), from randomly oriented chopped (1/4 inch or smaller) tape segments. Charge weight to the mold is 22.5 grams. Molding conditions are as follows:

Using a laboratory platen press with heating and cooling elements, heat the charged mold to 320°F (applying contact pressure only). Cool the mold to 300°F (contact pressure only). When the mold has cooled to 300°F, apply a load of 20,000 to 25,000 lb. to the mold (continue cooling). When the mold temperature reaches 280°F, let the press load fall off to contact pressure only, and continue cooling. When the mold temperature falls to 120°F, remove the mold from the press and take out the molded bar. Mold cycle should normally be 45 to 60 minutes, depending upon the heating and cooling capacity of the press used. After molding, trim the flash from the test bar and check the dimensions of the bar against the dimensional requirements of specification L-P-406 method 2011.

After conditioning the test bar, use the molded bar in the test for heat distortion temperature.

4.4.1.6.3 Coefficient of Thermal Expansion. - Using a 1/2 x 1/2 x 5 inch molded bar prepared as outlined in section 4.4.1.6.2, machine a rod from the test bar to the dimensions 3/8 inch diameter by 3 3/4 inches long with flat

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square ends so as to conform to the requirements of specification L-P-406 method 2031. Condition the machined rod and use the conditioned rod in the test for coefficient of linear expansion. In this test, it is not necessary to cement the steel plates onto the specimen. The ultimate contraction and expansion of the specimen is determined by no further movement or change of the dial indicator. No temperature readings are made of the inside of the specimen (see 3.7).

4.4.1.7 Migration Characteristics.

4.4.1.7.1 Procedure for Standard Plasticizer-Migration Screening Test.

4.4.1.7.1.1 Materials. - The inhibitor plastic to be tested should be in the form of a film, not more than 0.010 inch thick. The propellant (N-5, MIL-P-16401) should be prepared as lathe turnings, 0.0015 inch thick.

4.4.1.7.1.2 Procedure. - A 30 to 40 mg piece of the plastic is weighed, then placed in vacuum dessicator and pumped continuously with a good mechanical pump for one hour. The loss in weight is a measure of the volatile materials in the film. The same piece is then placed in a 20 by 150 mm test tube containing a weight of N-5 propellant turnings equal to twice the weight of the film (Fig. 2A). The test tube is then necked down (Fig. 2B) and a piece of 7 mm tubing provided with two constrictions sealed on Fig. 2C. This unit is evacuated for one hour, either alone or in manifold with others, by a pump capable of reducing the pressure to one micron or below, then sealed off at the upper constriction (Fig. 2D). After three days of storage completely immersed in an oil or water bath thermostat controlled at $40 \pm 1^\circ\text{C}$, the tube is removed, and the vacuum released at once by breaking the 7 mm tube at the second constriction. The tube is then opened, the propellant and inhibitor weighed, and the inhibitor film analyzed polarographically for nitroglycerin content. Each film sample is run in duplicate, and results reported as "Weight Gain" and "Nitroglycerin Gain," both in percent of the original film weight, after all volatile matter has been removed.

4.4.1.7.2 Nitroglycerin Determination

4.4.1.7.2.1 Inhibitor samples are dissolved in warm 95 percent ethanol. An aliquot of the diluted ethanol solution is polarographed in a 0.025 molar tetramethyl ammonium chloride solution (75 percent ethanol). The concentration of nitroglycerin is read from a standard graph.

4.4.1.7.2.2 Special Apparatus.

Sargent Model XXI Polarograph, or equivalent.
25 ml volumetric flasks.
30 ml beakers.
Hot Plate.
Constant temperature bath.

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Dropping mercury electrode assembly. Note 1.
Cylinder of dry nitrogen gas (containing little or no oxygen). Note 2.

4.4.1.7.2.3 Reagents.

Pure nitroglycerin (distilled from Kieselguhr dynamite).
Ethyl cellulose of the same source as the unknown samples.
95 percent ethanol.
Redistilled mercury.
0.1 molar tetramethyl ammonium chloride $(\text{CH}_3)_4\text{NCl}$, practical grade,
containing 10 ml of 0.1 percent alcoholic solution of methyl red per
liter of final solution.

4.4.1.7.2.4 Preparation of Sample.

Weigh the duplicate inhibitor samples and place into clean 25 ml
volumetric flasks.

Add 15 ml of 95 percent ethanol, warm slightly and shake until samples
are dissolved. Allow solutions to cool and dilute to volume with 95
percent ethanol. Samples should be in solution, but if a slight turbidity
of the solution is observed it will not interfere with the analysis.

4.4.1.7.2.5 Standard Graph.

a. Use an ethyl cellulose sample which is the same type as the unknown
sample Note 3 (this sample contains no nitroglycerin, but shall be the
same in every other respect).

NOTE 1. The dropping mercury electrode assembly consists of a platinum
contact electrode, a sintered-glass flushing tube, and a dropping mercury
electrode (should have a drop rate of about 6 to 8 seconds per drop at
-1.00 volts in the 0.025 molar $(\text{CH}_3)_4\text{NCl}$ solution). These are held in
place by a rubber stopper and the assembly is raised and lowered on a
metal rod.

NOTE 2. The nitrogen gas should contain less than 0.1 percent oxygen.
Excess may be removed by passing the nitrogen gas over absorbents such
as hot chromous chloride.

NOTE 3. From experiment it has been shown that any effects of ethyl
cellulose on the nitroglycerin polarographic wave are compensated for
by using the same type and amount of ethyl cellulose in preparing the
Standard Graph as one expects in the unknown samples.

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b. Weigh a 30 to 40 milligram sample and place in a clean 25 ml volumetric flask. Add 95 percent ethanol, warm slightly and shake until sample is in solution. Allow to cool and dilute to volume with 95 percent ethanol.

c. Weigh 50 milligrams of pure nitroglycerin into a clean 50 ml volumetric flask that has been previously calibrated. Dilute to volume with 95 percent ethanol.

d. Add 10 ml of ethyl cellulose solution (paragraph b) to a clean 30 ml beaker. Add 5 ml of the pure nitroglycerin solution (paragraph c) to the beaker. Add 5 ml of the 0.1 molar tetramethyl ammonium chloride solution (4.4.1.7.2.3) and stir the contents of the beaker well.

e. Decant about one-fourth of the beaker contents (this will allow no overflow of solution when flushing the sample with nitrogen). Add enough redistilled mercury to the beaker to cover the tip of the platinum contact electrode in the dropping mercury electrode assembly. Place the beaker into the constant temperature bath and lower the stopper containing the dropping mercury electrode assembly until it fits well into the beaker. Lower the glass tube for introduction of dry nitrogen into the solution and flush nitrogen through the solution for 5 to 10 minutes. Raise the glass tube and allow nitrogen to flow over the solution during the polarographic analysis. Polarograph the sample from 0.0 volts to -1.60 volts at a sensitivity of 0.060 $\mu\text{a}/\text{mm}$ (using the Sargent Model XXI Polarograph). Note 1.

f. Three additional points for the Standard Graph are obtained in the same manner (paragraph b) by substituting 4 ml and 2 ml aliquots respectively for the 5 ml aliquot of the pure nitroglycerin solution and adjusting the final volume.

g. The data should show a linear relationship when a plot of wave height (current) in millimeters versus milligrams (concentration) of nitroglycerin is made.

4.4.1.7.2.6 Polarographic Analysis of Sample.

Add 10 ml of the prepared sample (4.4.1.7.2.5, paragraph d) to a clean 30 ml beaker.

Add 5 ml of 95 percent ethanol and 5 ml of 0.1 molar tetramethyl ammonium chloride solution.

Stir the contents of the beaker well and treat as given in 4.4.1.7.2.5, paragraph e.

NOTE 1. The sensitivity used will depend upon the type of polarograph. This should be adjusted to give about 100 millimeter wave heights.

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Obtain duplicate polarograms (current-voltage curves) and refer the average height in millimeters to the Standard Graph. Note 1.

4.4.1.7.2.7 Calculation

The average wave height in millimeters for each inhibitor analyzed, referred to the Standard Graph, will determine the concentration of nitroglycerin present in the solution.

Percent Nitroglycerin equals

$$\frac{\text{Concentration} \times \text{total solution volume} \times 100}{\text{Weight of sample (mg)}}$$

(See 3.8)

4.4.1.8 Wrappability. - Select five rolls of tape at random and use these rolls for evaluation as to the wrappability of the plastic tape in inhibiting propellant grains (Ballistite Type). Wrap a minimum of 30 grains with the sample tape and 30 grains with a previously approved standard tape. Wrap the grains in accordance with standard practices under similar operating conditions. Wrapping operations should be observed and inhibited grains should be inspected for quality of inhibiting in accordance with applicable drawings and Ordnance Classification of Defects. If the sample tape is found to be significantly more difficult to process satisfactorily than the standard tape, the pilot lot shall be graded unsatisfactory for wrappability. Since this is a subjective test, depending upon the judgment of the operators and observers, the inspection results on the inhibited grains shall be used to assist in judging the performance of the sample tape for conformance to the requirements of section 3.9.

4.4.1.9 Adhesion. - Select fifteen propellant grains inhibited with the sample tape and let them cure in open storage at 70° to 80°F for two weeks. After curing, test the grains for bond strength in accordance with the procedure given in NAVORD OD 8480 (see 3.10).

4.4.1.10 Taliani Stability. - Taliani stability test shall be performed on a mixture of 1/2 gram sample tape and 1/2 gram acceptable N-5 propellant in accordance with OD 7904. (See 3.11).

4.4.2 Production Lot Tests. - Normal production lot tests may be conducted at the manufacturer's plant by or under the direction of the

NOTE 1. If nitrocompounds such as 2-nitrodiphenylamine are present in the inhibitors, a correction for its wave height must be determined and the value subtracted from the total wave height observed polarographically. The remaining wave height is then referred to the Standard Graph for nitroglycerin. Phthalate esters may be present in the inhibitor; however, these will not interfere in the nitroglycerin analysis.

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Government Inspector. The Government Inspector shall check each production lot for the properties as specified herein. In addition, the inspector shall satisfy himself that the manufacturer is meeting the requirements of Packaging (5.1) and Marking of Container (5.2.2).

The procuring agency may, at its discretion submit tape samples from any production lot received for evaluation of any or all of the properties specified in the preproduction lot and periodic production tests (see 4.4.1), and may reject the production lot on the basis of these tests if the sample tape fails to meet any of the test requirements.

4.5 Resubmission. - A lot that has been rejected under the provisions of this specification may be resubmitted for inspection and testing provided the contractor (after being informed of the reason(s) for rejection) submits a signed statement that he has corrected the deficiency(ies) noted and has screened the rejected lot to remove the defective material. When any production lot is tested and fails to conform to the following requirements and before acceptance of any production lots, resubmission of samples for pilot lot tests in accordance with section 4.4.1 shall be required:

Migration Characteristics (see 3.8).

Taliani Stability (see 3.11).

Wrappability (see 3.9).

Adhesion (see 3.10).

Physical Properties (see 3.7).

5. PREPARATION FOR DELIVERY.

5.1 Packaging. - Unless otherwise specified, the tape rolls shall be packed in cardboard boxes or drums with a minimum bursting strength of 250 psi. The rolls shall be inserted on a core having an outside diameter not less than 1/16 inch under the minimum roll core diameter specified (wall thickness of core 3/16 inch or more) and protected from contact with each other and the ends of the container by interlayers of 0.10 inch thick solid fiberboard. The interlayers shall support the rolls and inner core so that the tape rolls do not contact each other or the sides of the container. A clearance of one-half inch shall be maintained between the face of the tape surface on the outside diameter of the rolls and the inside surface at the sides of the box or packaging container. The weight of a single complete package, including the rolls of inhibiting tape, core, interlayers and the lined container shall not exceed 75 pounds. Containers should be lined with a waterproof barrier material conforming to Specification JAN-P-125.

5.2 Marking.

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5.2.1 Marking of Tape Rolls. - Unless otherwise specified, each roll of tape should be identified by a label cemented to the inside of the tape roll core. This label should contain the following information:

Name of Manufacturer.
 Nominal Thickness and Width of Tape.
 Contract Number or Order Number.
 Production Lot Number.
 Drawing or Specification Number.
 Linear Feet of Tape in Roll.
 Date of Manufacture.

5.2.2 Marking of Containers. - Unless otherwise specified, each container shall be marked with the following information:

Name of Manufacturer.
 Drawing or Specification Number.
 Nominal Thickness, Width and Length of Tape Rolls.
 Contract Number or Order Number.
 Number of Rolls in Container.

In addition, shipments shall be marked in accordance with MIL-STD-129, Marking for Shipment and Storage. The fifty roll pilot lot shipments should be marked with the following information in addition to the standard marking outlined above:

Samples for pilot lot acceptance tests in accordance with MIL-T-18833 Authorized by (reference letter of contract number):

6. NOTES.

6.1 Intended Use. - The ethyl cellulose tape covered by this specification is for use in inhibiting Rocket Propellant Grain (Ballistite Type).

6.2 Ordering Data. - Requests, requisitions and contracts or orders should specify the following:

The title, number, and date of this specification.

The laboratory to which samples specified in 4.1.1 should be sent for testing.

Color of tape required (see 3.1 and 6.3).

6.3 Color. - When colored tape is required, color samples will be furnished with requests for bids.

Patent Notice. - When Government drawings, specifications or other data are used for any purpose other than in connection with a definitely

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related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications or other data is not to be regarded by implication or otherwise as in any manner licencing the holder or any other person or corporation or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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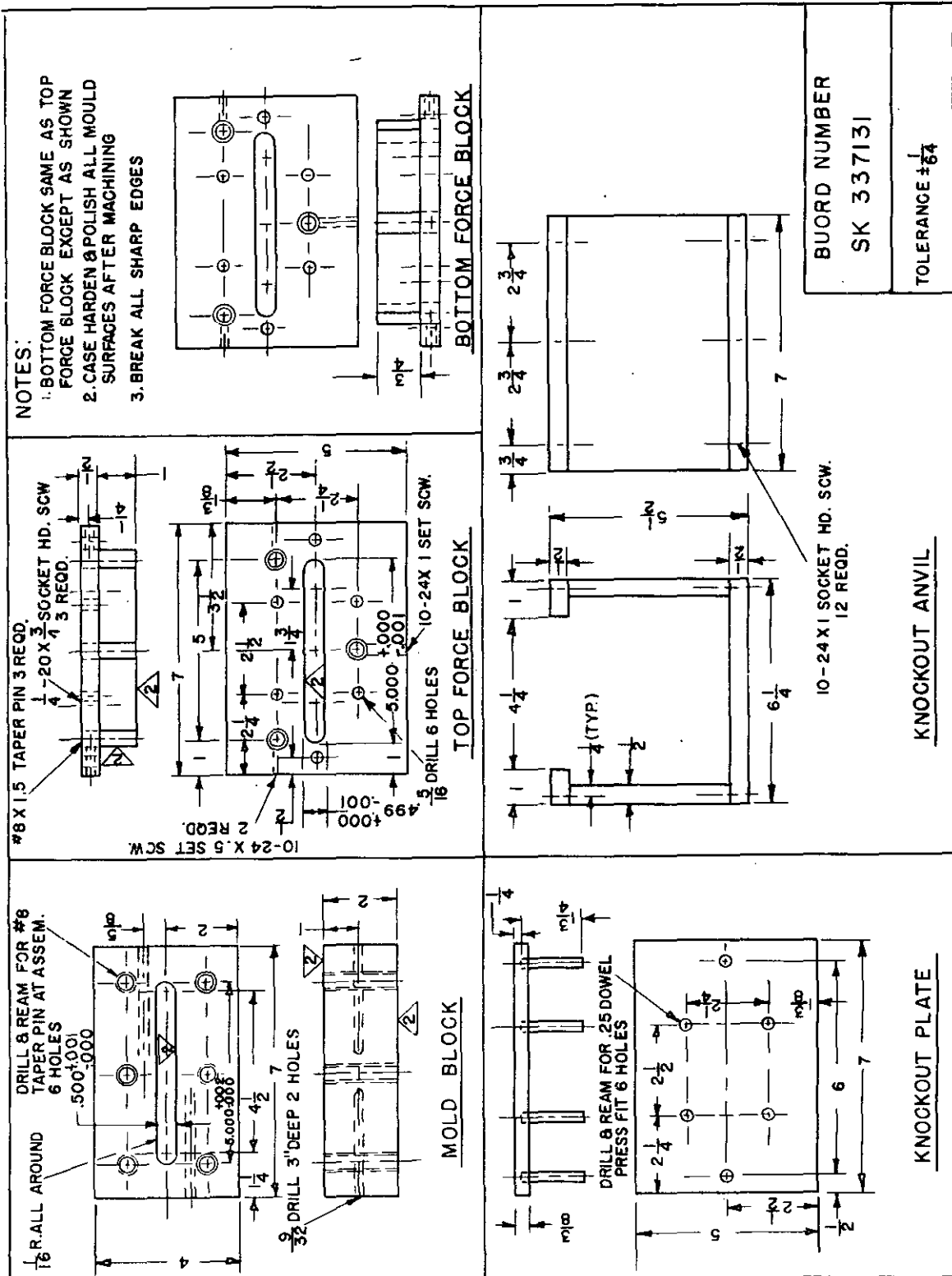


FIGURE 1, MOLD DETAILS

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FIGURE 2

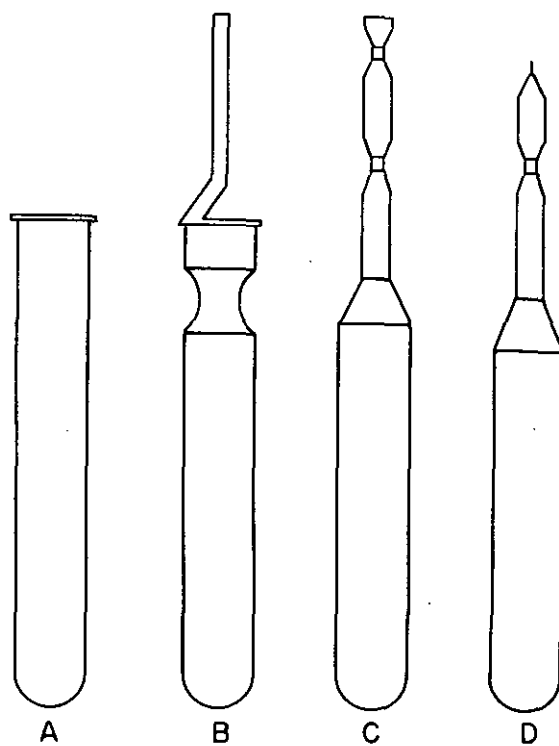


FIGURE 2

APPARATUS FOR MIGRATION TEST

FOLD

DEPARTMENT OF THE NAVY

POSTAGE AND FEES PAID
NAVY DEPARTMENT

OFFICIAL BUSINESS

CHIEF, BUREAU OF NAVAL WEAPONS
ENGINEERING DIVISION
ATTN: CODE RREN-5
DEPARTMENT OF THE NAVY
WASHINGTON, D. C. 20360

FOLD

SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 119-R004
<u>INSTRUCTIONS</u>		
This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity (as indicated on reverse hereof).		
SPECIFICATION		
ORGANIZATION (Of submitter)		CITY AND STATE
CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT \$
MATERIAL PROCURED UNDER A		
<input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?		
A. GIVE PARAGRAPH NUMBER AND WORDING.		
B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3. IS THE SPECIFICATION RESTRICTIVE?		
<input type="checkbox"/> YES <input type="checkbox"/> NO IF "YES", IN WHAT WAY?		
4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Printed or typed name and activity)		DATE