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AUTHORITY

usnwc ltr, 30 aug 1974

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SPECIFICATION

1 of 1

1. COMPONENT/PART NAME PER GENERIC CODE: PROPULSION PARTS & MATERIALS, SOLID FUEL ENGINES, PROPELLANTS	2. PROGRAM OR WEAPON SYSTEM: MULTIPLE	D0994			
	3. ORIGINATOR'S SPEC. NO.: WS 7682	3. DATE OF:	DAY	MO	YR
		ISSUE	11	1	68
4. ORIGINATOR'S SPECIFICATION TITLE: PURCHASE DESCRIPTION, COPOLYMER, VINYLIDENE DIFLUORIDE AND HEXAFLUOROPROPYLENE		REVISION			

6. SPECIFICATION IS: ☐ DRAFT ☐ PRELIMINARY ☒ FINAL

7. THIS SPECIFICATION COMPLEMENTS REPORT NO:

8. TYPE OF SPECIFICATION

- ☒ (A) GENERAL PRODUCT REQUIREMENTS FOR A FAMILY OF PARTS – PROCUREMENT DOCUMENT
- ☐ (B) INDIVIDUAL DETAIL PARTS DOCUMENT; STDS BOOK PAGES – FOR PROCUREMENT
- ☐ (C) DETAIL INSPECTION, PROCESS CONTROL, AND/OR TEST PROCEDURES FOR SPECIFIC PARTS
- ☐ (D) PROCESS (PAINTING, WELDING, FINISHING, HEAT TREATING ETC.) APPLICABLE TO MANY PARTS
- ☐ (E) SPEC. FOR PERFORMANCE, RELIABILITY, AND/OR ENVIRONMENT FOR ASSEMBLIES, EQUIPMENTS, SUBSYSTEMS AND SYSTEMS
- ☐ (F) PERFORMANCE AND APPLICATION DATA FOR DESIGN ENG. USE ON PARTS – NOT FOR PROCUREMENT
- ☐ (G) OTHER (DETAIL IN 10.)

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W.K. Sloan 12-5-68	NWC/CL	

13. SPEC. NO. 565.70.00.00-X7-17 S

NOTICES PAGE

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Code Ident
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WS 7682

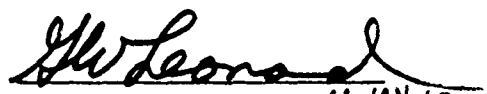
NAVAL ORDNANCE SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

COPOLYMER, VINYLIDENE DIFLUORIDE AND HEXAFLUOROPROPYLENE

Approved:



11 JAN 68

By direction

RECORD OF REVISIONS		
Revision Letter	Date	Changes

This document consists of pages i to ii and 1 to 10 inclusive.

FSC 6810

11ND-NOTS-4120/11(8-65)

3

Code Ident
10001

WS 7682

NAVAL ORDNANCE SYSTEMS COMMAND

DEPARTMENT OF THE NAVY

PURCHASE DESCRIPTION

COPOLYMER, /INYLIDENE DIFLUORIDE AND HEXAFLUOROPROPYLENE

1. SCOPE.

1.1 This purchase description covers one type of soluble fluoroelastomer (SFE) used as a high density binder material.

2. APPLICABLE DOCUMENTS.

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

STANDARDS

Military

MIL-STD-129	Marking for Shipment and Storage.
MIL-STD-414	Sampling Procedures and Tables for Inspection by Attributes.

(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.)

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3. REQUIREMENTS.

3.1 Product characteristics and performance. The copolymer is a product prepared by reacting vinylidene 1,1-difluoride and hexafluoropropylene. The presence of small amounts (less than 1 percent) of polymerization catalysts, coagulants, antioxidants or curing terminal groups may be acceptable. When tested in accordance with section 4, the binder material shall meet the following product characteristics and performance.

3.1.1 Chemical and physical properties. Chemical and physical properties of the binder material shall be in accordance with Table I.

Table I. Chemical and Physical Properties

Characteristic	Requirement Min	Max
Vinylidene difluoride, % by weight	55	63
Volatile material, %	---	0.30
Insolubles in acetone, %	---	0.50
Inherent viscosity at 30°C	0.65	1.15

3.2 Workmanship. The binder material shall be made using good workmanship, and shall be a product free from foreign materials. It shall be uniform in quality and shall be manufactured in accordance with standard manufacturing procedures of the industry.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in this document where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

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4.2 Lot. A lot shall consist of binder material produced by one manufacturer in one continuous operation employing not more than one lot of each ingredient with no change in formulation or process. If manufacture is by batch process, batches may be combined to form a lot provided that not more than one lot of each ingredient is used. A batch shall be as defined in 6.3.

4.3 Sampling. Sampling for quality conformance tests shall be in accordance with MIL-STD-414, Standard Deviation Method, Variability Unknown, Acceptable Quality Level of 1.0 percent defective.

4.4 Quality conformance tests. The following test procedures shall be used to determine that the requirements of this document have been met. Any proposed change in test methods or equipment shall necessitate, before adoption, prior approval of the procuring activity. In case of dispute between the results obtained using the methods and equipment specified herein and any proposed methods and equipment, the results obtained from what is specified herein shall prevail.

4.4.1 Vinylidene difluoride.

4.4.1.1 Apparatus. Infrared spectrophotometer, covering a minimum range of from 5 (2000 cm^{-1}) to 10 (1000 cm^{-1}) microns, and set for maximum efficiency for this analysis.

4.4.1.2 Sample preparation.

(a) Place a 0.7 ±0.1 gram (gm) sample into a small bottle (screw cap with aluminum foil liner).

(b) Add 10 milliliters (ml) of acetone to the clean sample bottle containing the sample and a small Teflon coated stirring bar.

(c) After screwing the cap in place, seal with masking tape.

(d) Place the bottle on a magnetic stirrer and allow to stir until completely dissolved.

4.4.1.3 Procedure.

(a) Let the dissolved sample settle (from 15 to 30 minutes) before the SFE solution is placed on the salt window (a sodium chloride window is satisfactory).

NOTE. Approximately two to three drops of the clear liquid from an eyedropper on an indented salt window will give a workable IR curve. After evaporation of the acetone, the film should give an absorbance between 0.6 and 0.9 at the 1400 cm^{-1} band to allow sufficiently accurate measurements to be made. If not, start again with a freshly prepared film.

(b) Scan the film on the salt plate from approximately 2000 cm^{-1} to 1000 cm^{-1}.

(c) Make certain that all the acetone has been removed from the film by checking the 1720 cm^{-1} region for an absorption band. If one is present, further drying is necessary.

(d) Determine the wave numbers of minimum absorbance at approximately 1425 cm^{-1} and 1335 cm^{-1} and draw tangent line "ac" (see Figure 1). Determine wave numbers of maximum absorbances at 1405 cm^{-1} and 1355 cm^{-1} and draw lines tangent at "d" and "e" and normal to the absorbance scale. Draw a line from tangent point "d" perpendicular to the wave number scale intersecting line "ac" at "x" and another line from tangent point "e" perpendicular to the wave number scale intersecting line "ac" at "y".

(e) Determine the absorbance differences: d - x and e - y.

(f) Calculate the absorbance ratio $\frac{d - x}{e - y}$.

Percent VF_2 is read from the calibration curve (see Figure 2).

4.7.1.4 Acceptance criteria. For the lot represented to pass the vinylidene difluoride test, the result obtained shall be within the range given in Table I.

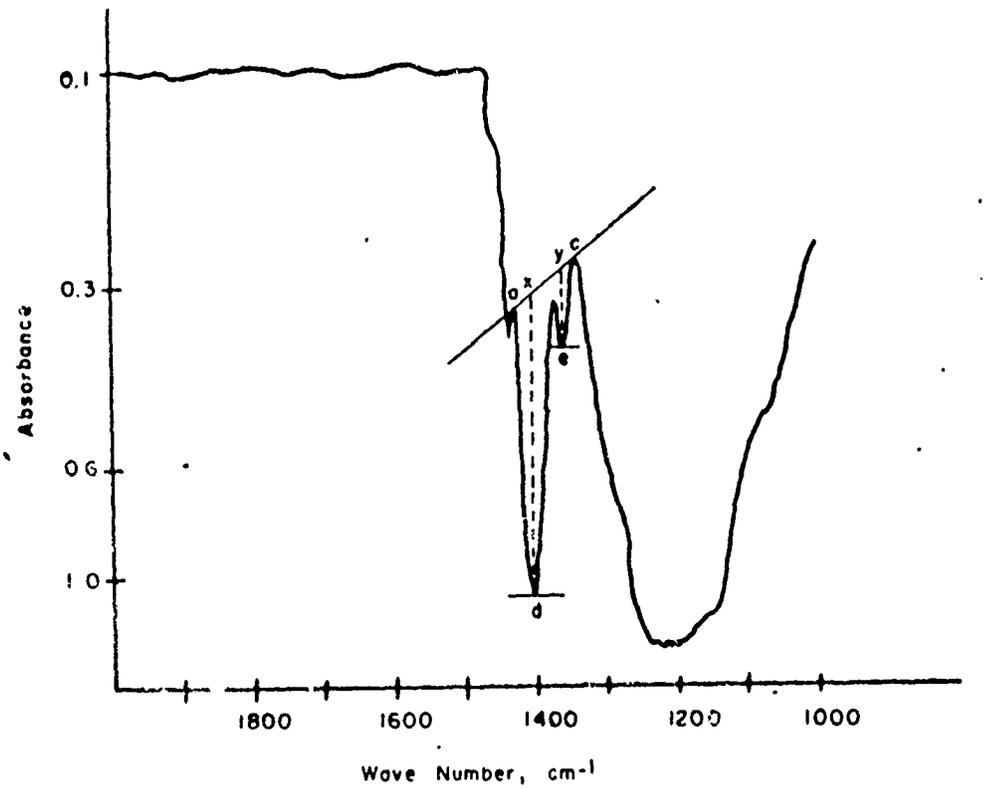

Figure 1

4.4.2 Volatile material. Volatile material shall be determined by oven drying at 204 ±4 degrees centigrade (°C) as follows: Prepare the sample by cutting small pieces 0.010 to 0.020 inch in thickness. Store in a glass stoppered bottle until ready for use. Clean and dry an aluminum pan at 204°C. Cool in a desiccator and weigh. Add approximately 15 gm of the sliced copolymer and reweigh. Place the pan and sample in the 204°C oven for 120 ±1 minute. Cool in a desiccator to room temperature, then reweigh. Calculate the content of volatiles as follows:

$$\text{Volatiles, percent} = \frac{W_o - W_d}{W_o - W} \times 100$$

Where: W_o = weight of pan and original sample, gm

W_d = weight of pan and dried sample, gm

W = weight of dried and cooled pan, gm

4.4.2.1 Acceptance criteria. For the lot represented to pass the volatile-material test, the value obtained shall be no more than that given in Table I.

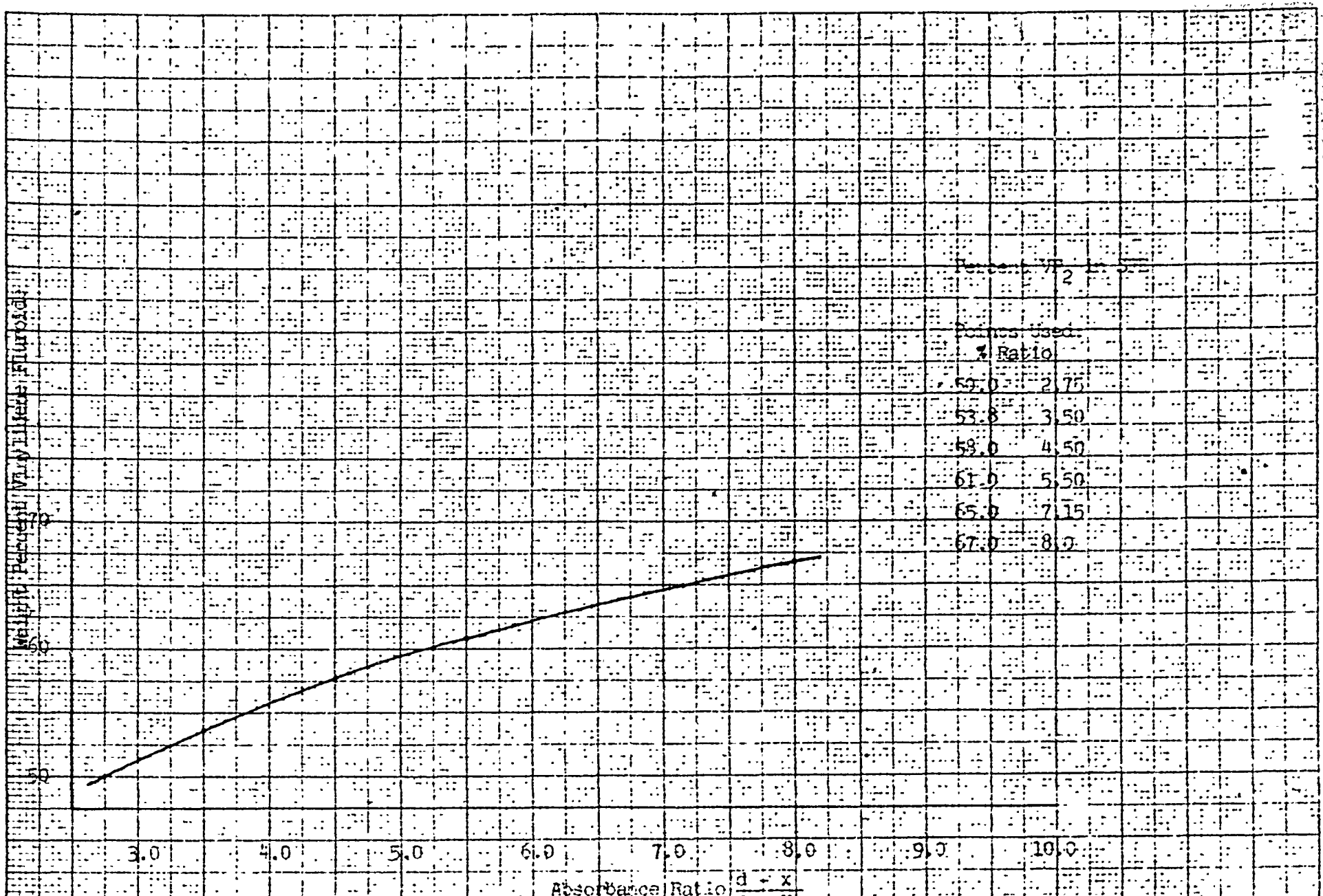

Figure 2. Calibration Curve for 4.4.1.3(f).

4.4.3 Insolubles in acetone. Solubility in acetone shall be determined by the following procedure. Place 10 gm of the SFE (weighed to the nearest 0.001 gm) into a clean, dry, 250 ml beaker and add 150 ml of reagent-grade acetone. Cover with aluminum foil and permit the SFE to soften for about 1 hour. Then stir until dissolved (about 1 hour). Let stand, covered, until insolubles settle (overnight) to aid filtration. Pack a GF/C glass filtering paper[1] wet with acetone over the fritted glass bottom of a 30 ml coarse filtering crucible.

Dry at 80°C, cool in desiccator, and weigh. Filter the SFE solution through the crucible. Police the beaker and transfer all residue to the crucible with a jet of acetone from a wash bottle. Wash all soluble material through the crucible with acetone. Use vacuum as needed, but do not let the frit become dry until the washing is complete. Dry in a well ventilated oven at 80°C for 30 minutes, cool in a desiccator, and weigh. Calculate the acetone insolubles as follows:

$$\text{Acetone insolubles, percent} = \frac{100\ (R - P)}{W}$$

Where: R = weight of prepared crucible and residue, gm

P = weight of prepared crucible, gm

W = weight of sample, gm

4.4.3.1 Acceptance criteria. For the lot represented to pass the insolubles-in-acetone test, the result obtained shall be no more than that given in Table I.

4.4.4 Viscosity, inherent. Inherent viscosity shall be determined on a dilute solution of the copolymer SFE as follows:

Weigh 0.100 ±0.001 gm of copolymer with an accuracy of ±0.0002 gm and place in a 250 ml glass stoppered Erlenmeyer flask. Add, by pipet, 100 ml of solvent prepared by mixing 20 volumes of tetrahydrofuran with 3 volumes of dimethylformamide. Dissolve by shaking mechanically.

CAUTION

Avoid contact and fumes from these flammable solvents. Use in well ventilated hood.

[1]Whatman GF/C glass fiber filters from Braun Corporation, 1363 South Bonnie Beach Place, Los Angeles, California 90023 proved satisfactory.

Filter the solution with the aid of a slight vacuum and centrifuge the filtrate in a Goetz tube for 30 minutes at 750 revolutions per minute. Transfer, by pipet, 10 ml of the gel-free and residue-free solution to a weighed 28 x 59 millimeter (mm) flat weighing bottle and evaporate the solvent gently in a warm vacuum oven. Weigh the bottle and residue to the nearest 0.0003 gm. Calculate the concentration of the solution as follows:

$$c = 10\left(W_2 - W_1\right)$$

Where: c = concentration of SFE, gm/100 ml

W_2 = weight of weighing bottle and dried copolymer, gm

W_1 = weight of empty weighing bottle, gm

Suspend a clean and dry Ostwald-Fenske viscometer tube, number 50 series, in a constant temperature water bath at 30 ±0.03°C. Pipette 10.0 ml of the mixed solvent to the large bulb and allow 10 minutes for temperature equalization. Calibrate the tube by drawing up the solvent, releasing, and determining the flow time for the meniscus to pass the two marks. Record the reading to the nearest 0.1 second. Drain and dry the tube. Determine the flow time for the copolymer SFE solution at 30 ±0.03°C in the same manner. Calculate the relative viscosity of the solution as follows:

$$r = t/t_o$$

Where: r = relative viscosity, dimensionless ratio

t = flow time for sample solution, seconds

t_o = flow time for mixed solvent, seconds

Calculate the inherent viscosity of the solution as follows:

$$inh = \frac{2.303 \log r}{c}$$

Where: inh = inherent viscosity number

r = relative viscosity

c = copolymer concentration, gm/100 ml

4.4.4.1 Acceptance criteria. For the lot represented to pass the inherent-viscosity test, the value obtained shall be within the range given in Table I.

4.5 Packaging, packing, and marking. Determine that packaging, packing, and marking conforms to section 5 of this document.

5. PREPARATION FOR DELIVERY.

5.1 Preservation and packaging.

5.1.1 Level A. Not applicable.

5.1.2 Level B. Not applicable.

5.1.3 Level C. The material shall be packaged in uniform quantities in sealed polyethylene bags or liners.

5.2 Packing.

5.2.1 Level A. Not applicable.

5.2.2 Level B. Not applicable.

5.2.3 Level C. Each individual package shall be packed in a fiberboard drum. The net weight of SFE shall be 50 or 100 pounds. The containers shall be acceptable to the carrier for safe delivery.

5.3 Marking.

5.3.1 Special marking. Each individual shipping container shall be marked to indicate the number of the batch or lot, whichever is the smaller, and the container number.

5.3.2 Normal marking. Shipping containers shall be marked in accordance with the requirements of MIL-STD-129.

6. NOTES.

6.1 Intended use. Lacquers made from this SFE are intended for use as binder material for pyrotechnics, propellants, and explosives.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number, and date of this document.

(b) Material form (crumb or slab) desired.

6.3 Batch. A batch is defined as that material which has been subjected to one or more chemical or physical processes (or combinations thereof) intended to produce a desired product having substantially uniform characteristics.

6.4 Chemical composition. The copolymer is the product prepared by reacting vinylidene 1,1-difluoride with hexafluoropropylene. This should not be interpreted as preventing the addition of less than 1 percent of suitable modifiers in the polymerization mix for the addition of terminal or intermediate reactive groups to the polymer chain in order to produce a curable product, or to promote adhesion to recommended materials so long as all other specified performance criteria are met by the final product during testing. The presence of small amounts of polymerization catalysts, coagulants, antioxidants or curing terminal groups may be acceptable.

Custodian:
NAVORD ORD9343

Preparing Activity:
NAVWPNSCEN/China Lake, California