

NOT  
MEASUREMENT  
SENSITIVE

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SUPERSEDING  
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## PERFORMANCE SPECIFICATION

### POLYBUTADIENE, LINEAR, CARBOXYL TERMINATED

This specification is approved for use by the Naval Air 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 establishes the requirements for two types of carboxyl terminated linear polybutadiene used in the manufacture of solid propellant grains.

1.2 Classification. The carboxyl terminated linear polybutadiene is of the following types:

Type I - Low acid content

Type II - High acid content

#### 2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this specification. This section does not include documents 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 documents cited in sections 3 and 4 of this specification, whether or not they are listed.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Air Warfare Center Aircraft Division, Code 414100B120-3, Highway 547, Lakehurst, NJ 08733-5100, by using the self addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

FSC 1336

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2.2 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

## AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- |            |   |  |
|------------|---|--|
| ASTM-D240  | - | Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter Standard Test Method for (DoD adopted).   |
| ASTM-D1159 | - | Bromine Number of Petroleum Distillates and Commercial Aliphatic Olefins by Electrometric Titration, Test Method for (DoD adopted).                                |
| ASTM-D1218 | - | Refractive Index and Refractive Dispersion of Hydrocarbon Liquids Test Method for (DoD adopted).   |
| ASTM-D1298 | - | Density, Relative Density (Specific Gravity), or API Gravity of Petroleum and Liquid Petroleum Products by Hydrometer Method, Standard Practice for (DoD adopted). |
| ASTM-E275  | - | Describing and Measuring Performance of Ultraviolet, Visible, and Near-Infrared Spectrophotometers, Standard Practice for  |

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.3 Order of precedence. 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 in accordance with 4.3.

3.2 Chemical and physical requirements. The chemical and physical requirements of the carboxyl-terminated linear polybutadiene, Types I and II, shall be in accordance with table I.

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3.3 Workmanship. The material furnished under this specification shall be a uniform product free from foreign contaminants. It shall be manufactured in accordance with standard manufacturing procedures of the industry.

TABLE I. Chemical and physical requirements.

Characteristic	Requirement			
	Type I		Type II	
	Minimum	Maximum	Minimum	Maximum
Viscosity, poise at 77°F	200	300	230	350
Viscosity ratio, 77°F/100 °F	1.5	3.0	1.5	3.0
Specific gravity, 60 /60°F	0.890	0.925	0.890	0.925
Index of refraction, 25/D	1.514	1.519	1.514	1.519
Carboxyl content, weight (wt.) percent	1.10	1.40	1.60	1.90
Unsaturation type,	25	45	25	45
Cis (percent of total)	35	55	35	55
Trans (percent of total)	15	25	20	30
Vinyl (percent of total)	-	1.0	-	1.0
Volatiles, wt. percent	-	0.05	-	0.05
Moisture, wt. percent	1.0	1.5	1.0	1.5
Antioxidant, wt. percent	-	0.2	-	0.2
Insoluble in trichloroethylene, wt percent	-	0.05	-	0.05
Ash, ignited, wt. percent	17,900	18,400	17,900	18,400
Heat of combustion, British Thermal Units (BTU) per pound mass, net				

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### 4. VERIFICATION

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

- a. First article inspection (see 4.3)
- b. Conformance inspection (see 4.4)

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

4.3 First article inspection. When required (see 6.2), the first article inspection shall consist of all the tests required for conformance inspection.

4.4 Conformance inspection. Conformance inspection shall consist of all the performance tests specified in 4.5.

4.4.1 Conformance sampling. Each lot shall be sampled as specified in the contract or order (see 6.2). All samples shall be selected at random from each lot. There shall be no defects.

4.5 Performance tests. All tests shall be performed to determine conformance with table I.

4.5.1 Viscosity at 77°F and 100°F. The apparatus and procedures for the determination of viscosity at 77°F and 100°F shall be as follows:

4.5.1.1 Apparatus.

a. Brookfield viscometer, Model RVF, Brookfield Engineering Laboratories, or equivalent.

b. Constant temperature water baths, maintained at 77°F and 100°F.

4.5.1.2 Procedure.

a. Remove the handle and mount the viscometer on a ring stand. Carefully attach the Brookfield No. 7 spindle. If a different spindle or speed is used, consult the chart accompanying the instrument for the multiplying factor.

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b. Pour the sample into a 600-milliliter (mL) beaker, place in the water bath at 77°F or 100°F, and stir with a thermometer until the sample comes to the test temperature. Do not stir air bubbles into the sample.

c. Lower the viscometer on the ring stand until the surface of the sample is in the narrow section of the spindle. Level the viscometer and operate at 20 revolutions per minute (rpm) for determinations in the 100 to 2,000 poise range. During operation maintain the spindle depth in the sample so that the polymer level climbs midway up the narrowest section of the spindle.

d. Obtain a series of readings until the value is constant.

4.5.2 Calculations. Using the No. 7 spindle at 20 rpm, and reading the 0 to 100 scale, calculate the viscosities as follows:

$$\text{Viscosity at } 77^{\circ}\text{F, poises} = (20)(\text{scale reading})$$

$$\text{Viscosity at } 100^{\circ}\text{F, poises} = (20)(\text{scale reading})$$

If a different spindle or speed is used, consult the chart accompanying the instrument for the multiplying factor. Convert the resulting centipoises to poises by dividing by 100.

Calculate the ratio of the viscosities at 77°F and 100°F as follows:

$$\text{Viscosity ratio, } 77^{\circ}\text{F} / 100^{\circ}\text{F} = \frac{\text{Viscosity at } 77^{\circ}\text{F, poises}}{\text{Viscosity at } 100^{\circ}\text{F, poises}}$$

4.5.3 Specific gravity. The specific gravity shall be determined in accordance with ASTM-D1298.

4.5.4 Index of refraction. The index of refraction shall be determined in accordance with ASTM-D1218.

4.5.5 Carboxyl content. The procedures for the determination of carboxyl content shall be as follows:

4.5.5.1 Apparatus.

a. Buret.

b. 250-mL Erlenmeyer flask or equivalent.

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c. Magnetic stirrer.

#### 4.5.5.2 Reagents and materials.

a. Foil.

b. Nitrogen.

c. Pyridine, reagent grade.

d. Sodium methylate, standard, 0.2 normal (N). Dissolve 4.6 grams (g) of clean metallic sodium in reagent grade methanol and dilute to one liter with the same solvent. Standardize against standard 0.1N HCl using thymol blue indicator.

e. Sodium methylate, 0.02N standard. Dilute 100.0 mL of the 0.2N standardized sodium methylate to exactly one liter with methanol.

f. Thymol blue indicator. Dissolve 0.3 g thymol blue in 100 mL of methanol.

#### 4.5.5.3 Procedure.

4.5.5.3.1 Test specimen preparation. Weigh, in duplicate, 0.4 to 0.6 g of polymer sample into a previously tared 250 mL Erlenmeyer flask (see 6.4.1). Add 50 mL of pyridine and a magnetic stirring bar. Cover the flask with foil to help exclude atmospheric carbon dioxide and dissolve the sample by stirring magnetically.

4.5.5.3.2 Titration. Insert the sodium methylate buret tip and the nitrogen flush tube into the flask by puncturing the foil. Flush the flask with nitrogen, and maintain a slow flow of nitrogen into the flask during the titration. Add ten drops of thymol blue solution and titrate to a blue end point with 0.02N sodium methylate solution prepared above. Repeat the titration procedure using 50 mL of pyridine as a blank.

4.5.5.4 Calculations. Carboxyl content shall be calculated as follows:

$$\text{Carboxyl content, wt. percent} = \frac{(A-B)(N)(45.02)}{(1,000)W} \times 100$$

Where:            A = mL of sodium methylate required for sample.  
                       B = mL of sodium methylate required for blank.  
                       N = Normality of sodium methylate.

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W = Weight of sample in grams.

45.02 = molecular weight of the carboxyl group, COOH.

Duplicate determinations shall not differ from the mean by more than  $\pm 0.05$  percent carboxyl in the range of 0.1 to 5.0 weight percent.

4.5.6 Unsaturation determination. The determination of unsaturation shall be as follows:

4.5.6.1 Apparatus.

a. Infrared spectrophotometer. Use a double beam instrument. Although a Perkin-Elmer Model 21 or its equivalent is preferred, one of the less expensive instruments such as Infracord or the IR-5 may be used with some sacrifice in reproducibility. ASTM-E275 may be used to compare the performance of the equivalent spectrophotometer unit to the Perkin-Elmer Model 21.

b. Sample cells. Use two matched pairs of cells with sample cell thickness of 1,500 and 500 micrometers ( $\mu\text{m}$ ). Build these cells in a laboratory if no commercial product is available. Directions for preparation of the cells are given in 4.5.6.3a.

c. Accessory equipment.

- (1) Vacuum desiccator
- (2) Vacuum pump
- (3) 25-mL volumetric flasks

4.5.6.2 Reagents and materials. Unless otherwise specified below, all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society (ACS).

a. Carbon disulfide.

b. Antioxidant. Phenyl-B-naphthylamine (PBNA).

4.5.6.3 Procedure.

a. Preparation of sample cells. Use pairs of infrared cells. One cell, of known thickness, serves as the sample cell, while the other should be of the thickness to compensate for the carbon disulfide solvent in the sample cell. If built in a laboratory, first build a sample cell of the latter thickness, and then build several cells 2.5 percent thinner. Obtain differential spectra between a

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2.5 percent polybutadiene solution in the sample cell and pure carbon disulfide in the reference cells. Examine the carbon disulfide absorption bands at 4.35, 4.65 and 11.65  $\mu\text{m}$  (see 6.4.2).

4.5.6.3.1 Preparation of solutions.

4.5.6.3.1.1 Regular procedure.

a. Prepare a stock solution of solvent by dissolving 0.1 g of PBNA or other antioxidant in a 2-kg (5-pound) bottle of carbon disulfide.

b. Tare a 25-mL volumetric flask to the nearest 0.1 mg. Add 0.625 g of polymer to the flask and reweigh the flask to  $\pm 0.1$  mg. Care should be taken to prevent the polymer from sticking to the lip or neck of the flask where solvent will not reach it. Add stock carbon disulfide to 2/3 final volume. Allow to stand, with occasional shaking until the polymer is dissolved.

c. Check the solution thoroughly for gel. Gel occurs in two forms: "tight gel" which is visible as definite particles and a "loose gel" which may be detectable only by the slight difference in refractive index from the surrounding solution. When gel is present discard the solution and use the alternate procedure of 4.5.6.3.1.2. When no gel is present, make the solution up to final volume and shake well before sampling for scanning (see 4.5.6.3.2).

4.5.6.3.1.2 Alternate procedure. This procedure shall be used instead of that in 4.5.6.3.1.1 for those samples which are found in the regular procedure to contain gel.

a. Weigh 1.3 g of polymer into a small bottle and add 50 mL of stock carbon disulfide. Allow to stand with occasional shaking until the polymer is dissolved. Examine the solution carefully for gel. If any is present, remove it by filtration.

b. Tare a small (25 mL) vial to the nearest 0.1 mg. At the time the infrared cells are filled, withdraw 10.0 mL of the solution with a pipet and place in the tared vial. Place the vial in a hood and allow the solvent to evaporate. Remove the last traces of solvent overnight in a vacuum desiccator. Weigh the vial to the nearest 0.1 mg and subtract the tare weight to obtain the weight of the solids. The concentration of the solution in g/L is 100 times the weight of the residue in grams.

4.5.6.3.2 Scanning of samples. Calibrate the instrument for electrical balance and the 100 percent T line. Set the zero percent T line to coincide with that of the chart paper. When these checks are completed, the instrument is ready for scanning.



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a. Make the polymer solution up to the final volume and shake thoroughly. Fill the 1,500- $\mu\text{m}$  sample cell. Fill the reference cell with the same stock carbon disulfide used to make the solution. Make a complete spectrum of the 1,500- $\mu\text{m}$  sample cell with the reference cell in the reference beam. Normal operating conditions may be used, although a slower scanning speed may be used if desirable. If the transmission at the band maximum of either the 10.35 or 11.0  $\mu\text{m}$  band is less than 20 percent, scan the sample in the 500- $\mu\text{m}$  cell.

b. Measurement of spectra. Measure all absorbencies for the trans and vinyl bands at the band maxima (see 6.4.3). Draw the baselines as shown on figure 1. Calculate the absorbance,  $A$ , as follows:

$$A = \log I_0 - \log I$$

Where  $I_0$  and  $I$  are measured as shown on figure 1. Make this measurement accurately.

4.5.6.4 Calculations.

a. Calculate the trans and vinyl concentrations as follows:

$$\text{Percent unsaturation} = \frac{100 A}{a b c}$$

Where:

$A$  = measured absorbance  
 $a$  = absorptivity as determined under calibration  
 $b$  = cell thickness in centimeters  
 $c$  = polymer concentration, g/L

b. Calculate the cis concentration from the relation:

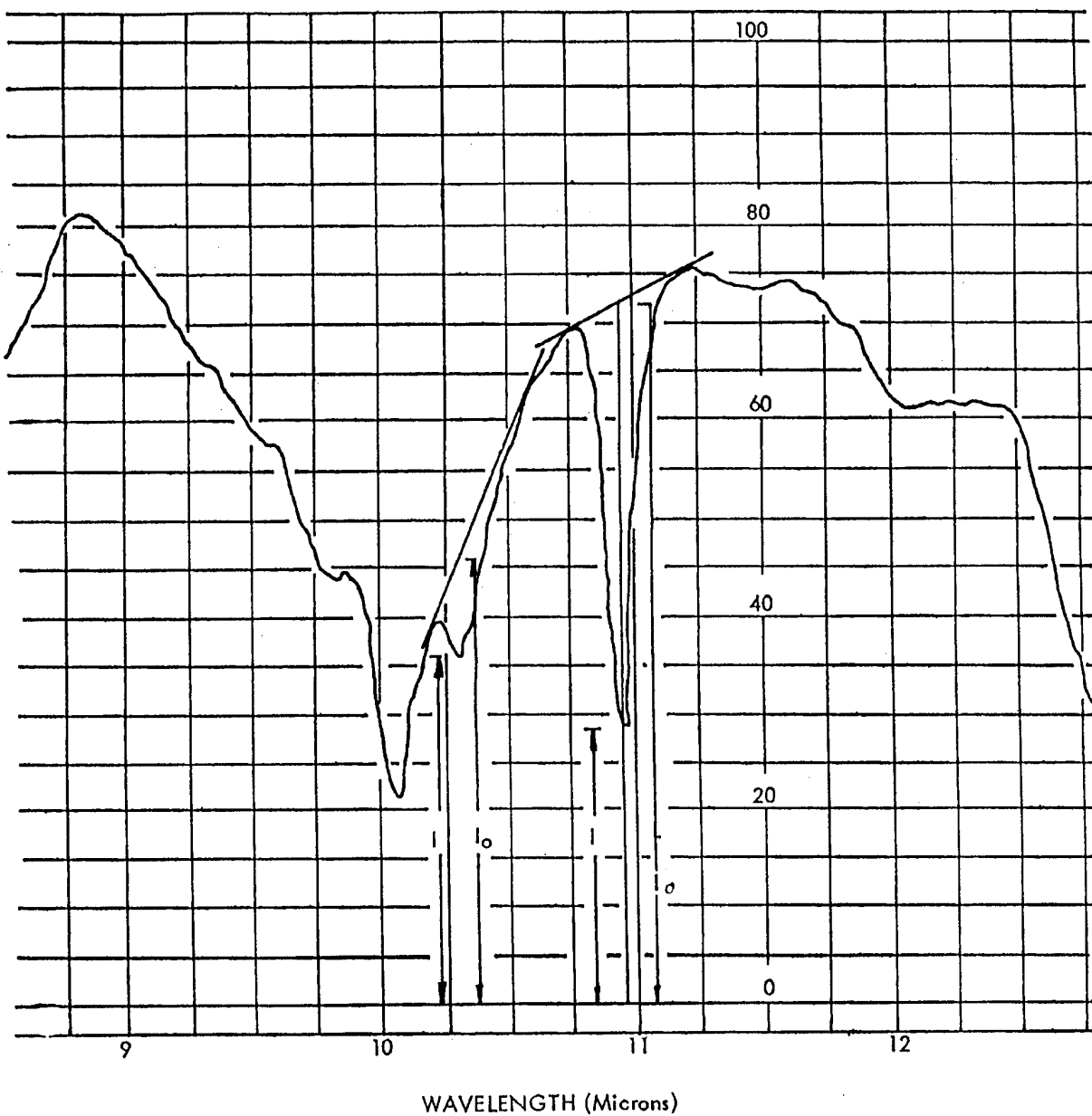
$$\text{percent cis} = 100 - (T + V)$$

Where:

$T$  = percent trans  
 $V$  = percent vinyl

4.5.6.5 Calibration. If the short method is to be used with the same instrument on which the complete method (see 6.4.4) has been calibrated, no additional spectra are required. If a different instrument is to be used, measure the samples selected as calibration standards on it also. Preferably, measure near the same time as the samples for the complete method and in the same cells. If this is not possible, purify the samples and prepare new solutions. Measure baseline

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Absorbances at 10.35 and 11.0  $\mu\text{m}$  per 4.5.6.5.

FIGURE 1. 95% CIS-1, 4 addition polybutadiene.

absorbances at 10.35 and 11.0  $\mu\text{m}$  on the spectra of the secondary standards, as described in 6.4.3. Calculate the absorptivities for trans and vinyl as follows:

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$$a = \frac{100 A}{b c d}$$

Where:

- a = absorptivity to be determined
- A = appropriate baseline absorbance
- b = cell thickness in cm
- c = polymer concentration, g/L
- d = percent concentration of the unsaturation type being measured as determined by the complete method

Several polymers should be used as secondary standards. The average absorptivity for each type of unsaturation is then substituted in the equation given under calculations (see 6.4.4).

4.5.7 Volatile materials determination. Volatile materials shall be determined by vacuum evaporation at 200°F and 1 millimeter (mm) of mercury. Twenty grams of sample ( $W_1$ ) shall be weighed to the nearest 0.1 mg in a previously tared 250-mL flask. The flask shall be attached to an appropriate evaporation system capable of maintaining a vacuum of 1 mm of mercury. The flask shall be submerged to the neck in an oil bath maintained at 200°F. The evaporation process shall continue for 15 minutes after the vacuum reaches 1 mm of mercury. The flask shall be cooled, then reweighed ( $W_2$ ) (gram of volatile material lost). Percent volatile material shall be determined as follows:

$$\text{Volatile, wt \%} = \frac{W_2 \text{ (in grams)}}{W_1 \text{ (in grams)}} \times 100$$

#### 4.5.8 Moisture content.

##### 4.5.8.1 Apparatus.

- a. Electrometric titration apparatus described in ASTM-D1159.
- b. A three necked, flat bottomed, 500-mL capacity titration flask may be used. The three necks are used for insertion of the sealed buret tip, electrode assembly, and sample and solvent introduction. If other titration vessels are used, provision for exclusion of atmospheric moisture shall be provided.
- c. Stirrer. A Teflon (TFE), or equivalent, covered magnetic stirring bar.
- d. Stopwatch or timer.

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- e. Buret, 10 mL.
- f. Platinum electrodes, furnished with a commercial titrimeter.
- g. Hypodermic syringe. The metal end of a 5-mL hypodermic syringe is removed and the glass opening is enlarged to 3 mm. This syringe is used for transferring weighed samples of the polymer.

4.5.8.2 Reagents and materials.

- a. Karl Fischer reagent, or equivalent, available from Mallinckrodt or Fischer Scientific Co.
- b. Karl Fischer reagent diluted 50 volume percent with Karl Fischer grade methanol.
- c. Titration solvent consisting of a mixture of 10 mL methanol, 20 mL pyridine, 10 mL of glacial acetic acid and 160 mL of chloroform. All solvents shall be ACS reagent grade except Karl Fischer grade methanol.

4.5.8.3 Standardization of reagent. Place 200 mL of dry methanol in the titration flask and titrate with Karl Fischer reagent to the end point. Add 0.06 g of water from a weighing pipet or a weighed amount of sodium tartrate equivalent to 0.06 g of water. Titrate again to the end point and record the volume of Karl Fischer reagent used. Repeat by adding successive weighed amounts of water or sodium tartrate and titrating until satisfactory checks are obtained. Duplicate titer values shall be within 0.00002 g water per mL of Karl Fischer reagent.

$$\text{Titer} = \frac{\text{grams water}}{\text{mL Karl Fischer reagent}}$$

4.5.8.4 Procedure. Add 200 mL of the titration solvent to the titration flask and titrate to a dead-stop end point with the Fischer's reagent. Slightly before the end point is reached (detectable by a change of color of the titration mixture), adjust the electric eye so that it is barely closed. At the end point, the electric eye should remain open for at least 60 seconds with one drop of reagent. If extremely dry titration solvents are used, the electric eye may not be able to be adjusted. This can be corrected by adding a drop of water and then titrating with the Karl Fischer reagent, or equivalent, to the end point (see 6.4.5). After the end point is reached, add a weighed 5-g sample from the modified hypodermic syringe. Titrate slowly near the end point with the Karl Fischer reagent to the same 60 second end point used in pretitrating the titration solvent. Calculate percent water as follows:

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$$\text{Water, percent by weight} = \frac{(\text{mL Karl Fischer reagent})(\text{titer})(100)}{\text{grams sample}}$$

Duplicate results by the same operator shall not differ by more than 5 ppm.

#### 4.5.9 Antioxidant determination.

##### 4.5.9.1 Apparatus.

- a. A mixing device capable of rotating the sample vials end over end at 50 rpm.
- b. Ultraviolet spectrophotometer, the Beckman Model DU or an equivalent instrument.
- c. 40 mL vials with plastic screw caps equipped with TFE liners or equivalent.
- d. Volumetric glassware.

4.5.9.2 Reagents and materials. All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society.

- a. Antioxidant.
- b. Cyclohexane, Spectro grade.
- c. Potassium hydroxide, alcoholic solution prepared by dissolving 28.05 g of potassium hydroxide in 500 mL of methanol, then filter the solution through glass fiber filter paper to remove the insoluble carbonates.

##### 4.5.9.3 Procedures.

4.5.9.3.1 Calibration curve. Weigh 1.0 g accurate to  $\pm 1$  mg of the same brand and grade of antioxidant used in the plant process into a weighing bottle. Dissolve the antioxidant in cyclohexane and quantitatively transfer to a 1-L volumetric flask, rinsing the weighing bottle three times with cyclohexane. Dilute to volume with cyclohexane and mix well. Pipet 1, 2, 3, and 4 mL of the standard antioxidant solution into separate 50-mL volumetric flasks and dilute to volume with alcoholic potassium hydroxide. The resulting solutions contain 20, 40, 60, and 80  $\mu\text{g/mL}$  of antioxidant. Measure the absorbance of each of the standards in a 1 cm cell versus alcoholic potassium hydroxide at wave lengths of 308, 330, and 292 nanometers (nm). Compute the differential absorbance as follows:

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$$\Delta A = A_{308} - A_{330} + \frac{22}{38} (A_{292} - A_{330}) \quad (\text{Equation 1})$$

Where:  $\Delta A$  = Differential absorbance and:  $A_{308}$ ,  $A_{330}$ , and  $A_{292}$  are the absorbance at 308, 330, and 292 nm, respectively

Prepare a calibration curve by plotting values for  $\Delta A$  versus corresponding concentrations of antioxidant in  $\mu\text{g/mL}$ .

4.5.9.3.2 Analysis of sample. Weigh 0.1 g accurate to  $\pm 1$  mg of sample into a 40 mL sample vial. Add 25 mL of cyclohexane and close the vial with a screw cap. Place the vial on the mixing device until the sample dissolves (preferably overnight). Pipet 20 mL of alcoholic potassium hydroxide (KOH) into the vial containing the dissolved sample and place the vial on the mixing device for 20 minutes. Remove the vial from the mixer, allow the phases to separate, then, using a pipet or syringe, remove enough alcoholic potassium hydroxide to fill a one centimeter (cm) spectrophotometer cell. Measure the absorbance at wavelengths 308, 330, and 292 nm versus alcoholic potassium hydroxide as reference.

4.5.9.4 Calculations. The  $\Delta A$  shall be calculated using equation 1 of 4.5.9.3.1. The concentration of antioxidant in the extract in  $\mu\text{g/mL}$  shall be read from the previously prepared calibration curve. The antioxidant content of the sample shall be determined as follows:

$$\text{Antioxidant, weight percent} = \frac{(C)(V)(10^{-4})}{W} \times 100$$

Where:  $C$  = Concentration of antioxidant in the extract,  $\mu\text{g/mL}$   
 $V$  = mL of alcoholic potassium hydroxide used in extraction  
 $W$  = Weight of sample in grams.

Duplicate determinations shall not differ from the mean by more than  $\pm 0.02$  percent antioxidant in the range of 0.1 to 2 percent.

4.5.10 Insoluble in trichloroethylene.4.5.10.1 Apparatus.

- a. Filter crucibles - Selas No. 3001, 30 mL capacity or equivalent.

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4.5.10.2 Reagents and materials.

## a. Trichloroethylene.

4.5.10.3 Procedure. Weigh 10 g of the sample into a 250 mL beaker. Add 100 mL of trichloroethylene and mix with a stirring rod to dissolve the sample. Filter the solution through a dried, tared filter crucible. Rinse all the materials from the beaker to the crucible using additional amounts of trichloroethylene. Thoroughly wash the contents of the crucible with additional amounts of trichloroethylene, then air dry by maintaining a vacuum 5 minutes after filtration is complete. Dry the crucible at least 30 minutes in an oven at 220°F. Remove the crucible from the oven and cool in a desiccator for 30 minutes and reweigh to obtain the weight of the insoluble material. Percent insolubles shall be calculated as follows:

$$\text{Insoluble in Trichloroethylene wt. percent} = \frac{\text{grams insoluble materials}}{\text{grams sample}} \times 100$$

4.5.11 Ash content. The ash content shall be determined as follows:4.5.11.1 Apparatus.

a. Muffle furnace capable of operating in the range of 450°F to 1,100°F and controlled within  $\pm 50^\circ\text{F}$ .

b. Weighing dishes or crucibles.

c. Desiccator.

4.5.11.2 Procedure. Weigh accurately, in duplicate, 2 g of polymer sample into a tared weighing dish or crucible. Ignite material over a low flame until all combustible matter has charred and no liquid remains. At this point, raise the furnace temperature to  $930 \pm 50^\circ\text{F}$  and maintain there until the carbonaceous material has completely burned away. DO NOT OPEN the furnace door for at least one hour after the sample is placed inside, or before it has reached maximum temperature. One hour at maximum temperature is required for complete ashing. After complete ashing has occurred, remove the weighing dish or crucible, cool, and weigh. If weighing is delayed more than 5 minutes, store the dish or crucible in a desiccator.

4.5.11.3 Calculations. Weight percent ash shall be calculated as follows:

$$\text{Wt. percent ash} = \frac{\text{grams ash}}{\text{grams sample}} \times 100$$

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grams sample

Duplicate determinations shall not differ from the mean by more than  $\pm 5$  percent.

4.5.12 Heat of combustion. The heat of combustion shall be determined in accordance with ASTM-D240.

4.5.13 Acceptance criteria. All test results shall indicate compliance with all of the requirements of section 3. Failure to meet these requirements shall be cause for rejection of the lot.

## 5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of material is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

## 6. NOTES

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

6.1 Intended use. The carboxyl terminated, linear polybutadiene furnished under this specification is intended for use in the manufacture of the solid propellant grains as a propellant ingredient and as a propellant burning restrictor ingredient, and for such other uses as may be specified by the acquiring activity.

6.2 Acquisition requirements. Acquisition documents must specify the following:

- a. Title, number, and date of the specification.
- b. Issue of DoDISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.2).



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- c. Whether first article inspection is required (see 3.1 and 4.3).
- d. With respect to first article inspection, the instructions for the testing of the first article sample (see 4.3).
- e. Conformance sampling (see 4.4.1).
- f. Instructions for disposition of first article sample, when required (see the contract or order).
- g. Packaging requirements (see 5.1).

6.3 Material Safety Data Sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313 and Code of Federal Regulation 29 CFR 1910.1200. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

6.4 Miscellaneous notes.

6.4.1 Carboxyl content determination supplemental information. When determining the carboxyl content of polybutadiene (see 4.5.5.3.1), the end point is difficult to determine if the polymer has a dark red color. If the air were not excluded from the flask, atmospheric carbon dioxide would react with the sodium methylate and cause the end point to fade.

6.4.2 Sample cells. Normally only one sample cell thickness, 1,500  $\mu\text{m}$ , will be needed for this method. Its thickness should be measured with an optical microscope equipped with a Vernier scale for measuring lens tube travel. A 500- $\mu\text{m}$  sample cell may be needed. The thickness of this cell should be measured accurately by interference fringes. A complete reserve set of cells should be on hand. Cells slowly lose transmission with use and require periodic rebuilding. There is also the possibility that an unnoticed piece of gel will clog the cell completely. A spare set of cells will eliminate loss of time in such cases. A reference cell should not show appreciable absorption at the 4.35, 4.65 and 11.65  $\mu\text{m}$  wavelengths (see 4.5.6.3a).

Purification of samples. The exact procedure for purification before analysis cannot be specified explicitly because it will depend to a large extent on the form in which the sample is received. In general, raw samples will contain two types of contaminants:

- a Inorganic materials, such as catalyst residue, which will not contribute much infrared absorption of their own, but which will cause the concentration of the solution to be in error.

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b. Organic materials, such as antioxidant, which may affect the trans and vinyl determinations if they have infrared absorption bands near 10.35 or 11.0  $\mu\text{m}$ .

6.4.3 Measurement of spectra. For the 10.35  $\mu\text{m}$  band, the baseline should be tangent to the transmission maxima at 10.2 and 10.7  $\mu\text{m}$ . For the 11.0- $\mu\text{m}$  band, the baseline should be tangent to the transmission maxima at 10.7 and 11.2  $\mu\text{m}$  (see 4.5.6.3.2b and 4.5.6.5).

6.4.4 Calibration. Accomplish calibration using as secondary standards samples of Cis-4 polybutadiene analyzed to the complete procedure in Analytical Chemistry, 31,529 (1959), and have known trans and vinyl concentrations. In general, low molecular weight olefins cannot be used for calibration because the absorptivities so determined will be lower than those appropriate for polybutadiene. This will lead to high trans and vinyl concentrations and consequently low cis concentrations (see 4.5.6.5).

6.4.5 Exclusion of atmospheric moisture. The importance of excluding atmospheric moisture and pretitrating the water in the titration solvent and that absorbed on the walls of the titration flask cannot be overemphasized (see 4.5.8.4).

6.5 Subject term (key word) listing.

Acid content  
 Burning restrictor  
 Polymer  
 Propellant  
 Trichloroethylene

6.6 Changes from the previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

## CONCLUDING MATERIAL

Custodians  
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Preparing activity:  
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(Project 1336-0635)

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**I RECOMMEND A CHANGE:**

1. DOCUMENT NUMBER  
MIL-PRF-23942A(AS)

2. DOCUMENT DATE (YYMMDD)  
970930

3. DOCUMENT TITLE

Polybutandiene, Linear, Carboxyl Terminated

4. NATURE OF CHANGE (Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)

5. REASON FOR RECOMMENDATION

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