

MIL-P-51431A(EA)
 19 July 1988
 SUPERSEDING
 MIL-P-51431(EA)
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MILITARY SPECIFICATION

PLASTIC MOLDING AND EXTRUSION MATERIAL, POLYETHYLENE COPOLYMER, HIGH DENSITY

This specification is approved for use within U.S. Army Chemical Research, Development and Engineering Center, Department of the Army, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers two types of high density polyethylene copolymer used for extrusion and molding processes.

1.2 Classification. Plastic material shall be of the following types as specified (see 6.2):

- Type I - Polyethylene butene-1 copolymer, high density
- Type II - Polyethylene hexene-1 copolymer, high density

2. APPLICABLE DOCUMENTS

2.1 Nongovernment publications. The following publications form a part of this specification to the extent specified herein. Unless otherwise indicated, the issues of the documents which are DOD adopted shall be those listed in the issue of the DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

ASTM STANDARDS

- D 638 - Tensile Properties of Plastics
- D 746 - Brittleness Temperature of Plastics and Elastomers by Impact
- D 1193 - Reagent Water
- D 1238 - Flow Rates of Thermoplastics by Extrusion Plastometer

: Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, U.S. Army Chemical Research, Development and Engineering Center, ATTN: SMCCR-SPT-S, Aberdeen Proving Ground, MD 21010-5423 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 9330

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- D 1505 - Density of Plastics by the Density-Gradient Technique
- D 1525 - Vicat Softening Temperature of Plastics
- D 1693 - Environmental Stress-Cracking of Ethylene Plastics
- D 1928 - Preparation of Compression-Molded Polyethylene Test Sheets and Test Samples
- D 2240 - Rubber Property-Durometer Hardness
- D 3951 - Commercial Packaging

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103).

(Nongovernment standards and other publications are normally available from the organizations which prepare or which distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.2 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein (except for associated detail specifications, specification sheets or MS standards), the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Material.

3.1.1 Material. The plastic material shall be a pellet form of type I or type II high density polyethylene copolymer of ethylene and butene-1 or hexene-1 monomers, respectively (see 6.3). No alcohols, glycols, sugars, organic acids, other compounds containing hydroxyl groups, mercaptans, or amines shall be used in the manufacture of the plastic material. The contractor shall certify that this requirement has been met.

3.2 Infrared spectrum characteristics. The plastic material shall have the infrared spectrum characteristics specified in 4.2.4.1 when tested as specified therein.

3.3 Antioxidant content. The plastic material shall contain no less than 0.08 percent by weight and no more than 0.12 percent by weight "Santnox R" (see 6.7) antioxidant when tested as specified in 4.2.4.2.

3.4 Flow rate. The plastic material shall have a flow rate of no more than 2.0 grams (g) per 10 minutes when tested as specified in 4.2.4.3.

3.5 Physical characteristics of compression-molded specimen. A compression-molded specimen of the plastic material, which has been made in accordance with procedure C, type III of ASTM D 1928, shall conform to the physical characteristics of table I when tested as specified in 4.2.4.4.

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TABLE I. Physical characteristics of compression-molded specimen

Characteristic	Requirement			
	Type I		Type II	
	Minimum	Maximum	Minimum	Maximum
Density, g per cubic centimeter	0.939	0.943	0.941	0.945
Environmental stress-cracking resistance, hours, F ₅₀ , full strength Igepal CO-630 as reagent	1000	----	1000	----
Hardness; type D durometer	60	66	62	68
Low temperature brittleness, °F	----	-180	----	-180
Tensile strength at yield, psi, 2 inches per minute	3000	----	3000	----
Elongation at break, percent, 2 inches per minute	600	----	600	----
Vicat softening point, °F	257	----	257	----

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.1.1 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the plastic material of one type, produced by one manufacturer, at one plant, from the same materials, and under essentially the same manufacturing conditions provided the operation is continuous.

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4.2.2 Sampling for test. Sampling for test shall be conducted in accordance with table II. A representative specimen of approximately 2.5 pounds shall be removed from each sample packing container and placed in a suitable clean, dry container labeled to identify the lot and container from which it was taken.

TABLE II. Sampling for test

: Number of packing containers in lot	: Number of sample packing containers :
: 2 to 25	: 2
: 26 to 150	: 3
: 151 to 1,200	: 5
: Over 1,200	: 8

4.2.3 Inspection procedure for test. Approximately equal portions of all the specimens taken in 4.2.2 shall be thoroughly mixed to form a composited specimen of no less than 5 pounds. The composited specimen shall be tested as specified in 4.2.4. Each test shall be conducted in duplicated analysis. Failure of either analysis of any test shall be cause for rejection of the lot represented.

4.2.4 Tests. Water in accordance with ASTM D 1193 and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted as follows:

4.2.4.1 Infrared spectrum characteristics. This test consists of two parts. Part I tests or infrared spectrum characteristics due to volatile effluents. Part II tests for infrared spectrum characteristics due to nonvolatile material.

(a) Background spectrum. Perform the manufacturer's specification tests on the double-beam infrared spectrometer to be used, and if necessary, repair or adjust the spectrometer to meet the manufacturer's specifications. Fit one window of a 5- or 10-centimeter (cm) path length infrared gas or liquid cell with an O-ring or gasket so that the window can be readily removed and replaced. A 34- to 50-millimeter (mm) bore cylindrical cell with at least one threaded end is recommended. A rectangular or other-shaped cell of equivalent cross-sectional area may be used if a window can be readily removed and replaced (see 6.4). (Note on cell preparation: Do not rinse or otherwise expose any part of the cell to chemical liquids or vapors for 1 hour prior to initial use for this test. If necessary, to assure freedom from previous samples, the cell should be disassembled and the individual parts heated in a vacuum oven at 35° to 45°C for 30 minutes at a moderate vacuum. If more than one sample is to be tested, two or more dry nitrogen flushes rather than solvent rinsing and oven drying between samples should ordinarily suffice to remove any residual particles of plastic or any adsorbed vapors from the cell. If it is necessary to polish the cell windows between samples, make sure the windows are free from solvent vapors before reassembling the cell.) Close or plug all cell valves

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and openings, remove the window, and flush the cell body with dry nitrogen gas. Replace the window, place the cell in the sample holder of the spectrometer with the infrared source on and the source beam shutters open, and leave the cell in place for 10 minutes. Use a screen or adjustable attenuator in the reference beam to give a 55 to 95 percent transmission spectral "baseline" over the 2.5 to 15 micron range. Record the 2.5 to 15 micron infrared spectrum of the cell plus nitrogen as a background using the spectrometer operating conditions recommended by the manufacturer for vapor phase survey spectra. (Avoid very fast or very slow scans. A 10- to 20-minute scan should suffice.) An essentially bandfree background spectrum should be obtained except that a small positive or negative atmospheric water vapor-carbon dioxide spectrum is permissible (all the water vapor bands at 2.5 to 2.9 and 5.5 to 7.5 microns and the carbon dioxide bands at about 4.26 and 14.98 microns covering 10 percent or less on the percent transmission scale). (See 6.5) If the atmospheric spectrum is too intense, continuously flush the spectrometer sample area with dry nitrogen. If extraneous bands (other than those of atmospheric water vapor-carbon dioxide) covering more than 3 percent on the percent transmission scale are observed, carry out or repeat the procedure given above under the note on cell preparation. Reflush the cell with dry nitrogen and repeat the background spectrum procedure. When a satisfactory background spectrum has been obtained, reflush the cell with dry nitrogen and lay the window in place on the O-ring or gasket, but do not tighten the holder.

(b) Preparation of sample, part I. Mill a sufficient amount of the specimen in a Wiley mill or equal (see 6.6) using a 10-mesh outlet screen so that 1.0 ± 0.2 g of milled specimen is obtained. Collect the specimen in a closed container. (A quick weighing may be employed, if time permits, to determine if the sample weight is in the specified range.) Rapidly transfer the reduced specimen to the recently reflushed cell prepared as specified in (a) through the window opening. (Some difficulty with static charge will probably be experienced.) Alternately, the milled sample may be collected directly into the cell. Replace the window and distribute the sample over the bottom of the cell so that the infrared beam is not obstructed. The milling, weighing, and cell-loading operations should be completed in 6 minutes or less total time. Place the cell in the sample holder of the spectrometer with the beam shutters open and the infrared source on and leave it in place for 15 minutes.

(c) Procedure, part I. Record the 2.5 to 15 micron infrared spectrum of the loaded cell prepared as specified in (b) using the same spectrometer conditions that were used for the background spectrum in (a). Remove the cell window, dump the milled sample into a suitable tared container, and use a brush to transfer as much milled sample as possible from the cell to the container. Weigh the container on an analytical balance and determine the sample weight to the nearest 0.01 g. If the sample weight is not between 0.80 and 1.20 g, repeat the procedure described in (b), making the necessary adjustments to obtain a sample weight of 0.80 to 1.20 g. Retain the milled sample for use in (e).

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(d) Calculation, part I. Read the percent transmission (I) to the nearest percent for the strongest extraneous band in the spectrum produced in (c) at the band absorption maximum. Read the percent transmission (I_0) to the nearest percent from the background spectrum produced in (a) at the same wavelength. Calculate the absorbance corrected to the second decimal place. [Absorbance = $\log_{10}(I_0/I)$.] Divide the absorbance by the sample weight in grams, calculating the absorbance per gram corrected to the second decimal place. The spectrum produced in (c) should be the same as that of the background spectrum in (a) except that changes are allowed in the intensities of the water vapor absorptions from 2.5 to 2.9 microns and from 5.5 to 7.5 microns and of the 4.26 and 14.98 micron carbon dioxide absorptions. The presence of extraneous absorption bands other than those permitted in the background spectrum and those described for water vapor-carbon dioxide shall constitute a failure of the infrared spectrum characteristics if the absorbance per gram of sample for the strongest extraneous band is 0.03 or greater.

(e) Preparation of sample, part II. Use two steel blocks of an adequate size to obtain a film large enough to cover the infrared beam of the spectrometer (3/4 to 1 inch thick and about 1 inch by 2 inches, if rectangular, or about 1.5 inches in diameter). Polish one face of each block to a smooth mirror finish. Heat the blocks to about 140°C on a hot plate or on the heated platen of a hydraulic press. (The temperature of the blocks can be measured by placing the stem of a bimetallic dial thermometer between the surfaces of the two blocks.) Pour the milled sample retained from (c) onto one of the polished surfaces in a roughly rectangular pile of uniform thickness. Place the polished surface of the other block on top of the milled sample and press the blocks together in a hydraulic press at 10,000 pounds total load for 5 seconds. A semitransparent film 0.25 to 0.35 mm thick should result. Remove the film immediately and measure its thickness at several places with a micrometer to the nearest 0.01 mm. If the thickness is 0.25 to 0.35 mm, choose an area as large as the spectrometer sample beam over which the thickness variation is least, and determine the average thickness. Place the film in a suitable holder using the area of the film measured above and determine the percent transmission at 3.05 microns. (The spectrometer should be set to read 0 to 100 percent transmission before the sample is inserted.) The percent transmission at 3.05 microns should be 40 or more. If the percent transmission at 3.05 microns is too low, or if the film thickness was not in the specified range, repeat the milling and pressing procedures specified in (b) and (e), respectively, making adjustments for proper thickness and clarity of the film. A slightly higher temperature of pressing (about 5°C) will produce a thinner, clearer film. A lower temperature will produce a thicker, but possible cloudier film. An adjustment of the sample weight may also be necessary.

(f) Procedure, part II. Place the film prepared as specified in (e) in the spectrometer. Use a screen or adjustable attenuator in the reference beam to produce an overall spectral "baseline" of 55 to 95 percent transmission over the 2.5 to 15 micron range. Record the 2.5 to 15 micron infrared spectrum of the film, using the manufacturer's recommended spectrometer conditions for condensed phase spectra (solids and liquids). Except for differences

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due to abscissa spectral presentation, spectrometer resolving power, and overall thickness of clarity of film, the spectrum shall show no appreciable deviation from the reference spectrum shown on figure 1 or figure 2, as applicable, with regard to positions of bands, relative intensities of bands, shapes of bands, and freedom from extraneous bands except as allowed in (g). Any spectrum which does not meet the above characteristics shall constitute a failure of the infrared spectrum characteristics.

(g) Intensities of extraneous bands. Read to the nearest percent the percent transmission value (I_0) of the spectral curve produced in (f) at the transmission maximum near 5.4 microns. Read to the nearest percent the percent transmission value (I) of the strongest extraneous band in the region 5.6 to 6.0 microns at its absorption maximum (transmission minimum). Determine the absorbance corrected to the second place. [Absorbance = $\log_{10}(I_0/I)$.] Calculate the absorbance per millimeter of film thickness using the average film thickness measured in (e). Draw a straight baseline between the points at which the spectral curve intersects the abscissa grid of the chart at 8.3 and at 10.5 microns. Draw another straight baseline for the spectrum between the 10.5 and 12.0 micron intersection points. Use the nearest whole percent transmission values of the intersection of each straight baseline with the abscissa grid at the wavelength of the band absorption maximum as the I_0 for the strongest extraneous band, with percent transmission I, in each of the two respective spectral regions. Calculate the absorbances as above. Calculate the absorbance per millimeter of film thickness corrected to the second decimal place for each extraneous band. The values of absorbance per millimeter when calculated as described above shall be 0.12 or less for an extraneous band in the 5.6 to 6.0 micron region, and 0.10 or less for extraneous bands in the 8.3 to 10.5 micron and 10.5 to 12.0 micron regions. Any spectrum which does not meet these criteria for intensities of extraneous bands shall constitute a failure of the infrared spectrum characteristics.

4.2.4.2 Antioxidant content.

(a) Standard "Santonox R" solution. Weigh 0.1000 ± 0.0001 g of "Santonox R" (see 6.7) and dissolve in spectro grade cyclohexane. Transfer quantitatively to a 1-liter volumetric flask and dilute to volume with spectro grade cyclohexane. This solution contains 0.1 milligram (mg) of "Santonox R" per milliliter (mL).

(b) Calibration. Transfer several aliquots of standard "Santonox R" solution containing from 0.1 to 1.6 mg of "Santonox R" to separate 125-mL separatory funnels fitted with "Teflon" stopcocks and dilute each aliquot to approximately 25 mL with spectro grade cyclohexane. Extract each solution three times with 15-mL portions of 1M sodium hydroxide solution. Combine the three aqueous extracts in a 50-mL volumetric flask and dilute to volume with 1M sodium hydroxide solution. Using a recording spectrophotometer equipped with 1-cm cells, measure the absorbance of the solutions versus a blank containing no "Santonox R" at 267 millimicrons. Prepare a calibration curve by plotting absorbance value versus concentration of "Santonox R" for each solution. Draw a straight line through the points which also passes through the origin.

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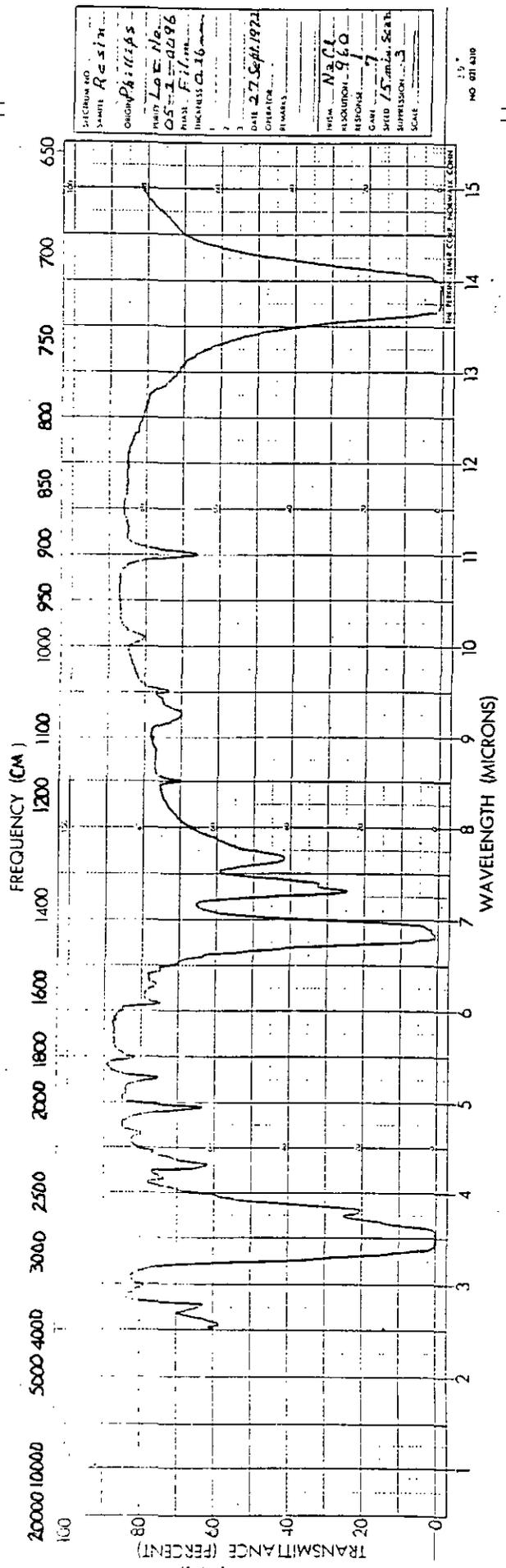


Figure 1. Infrared spectrum, type I

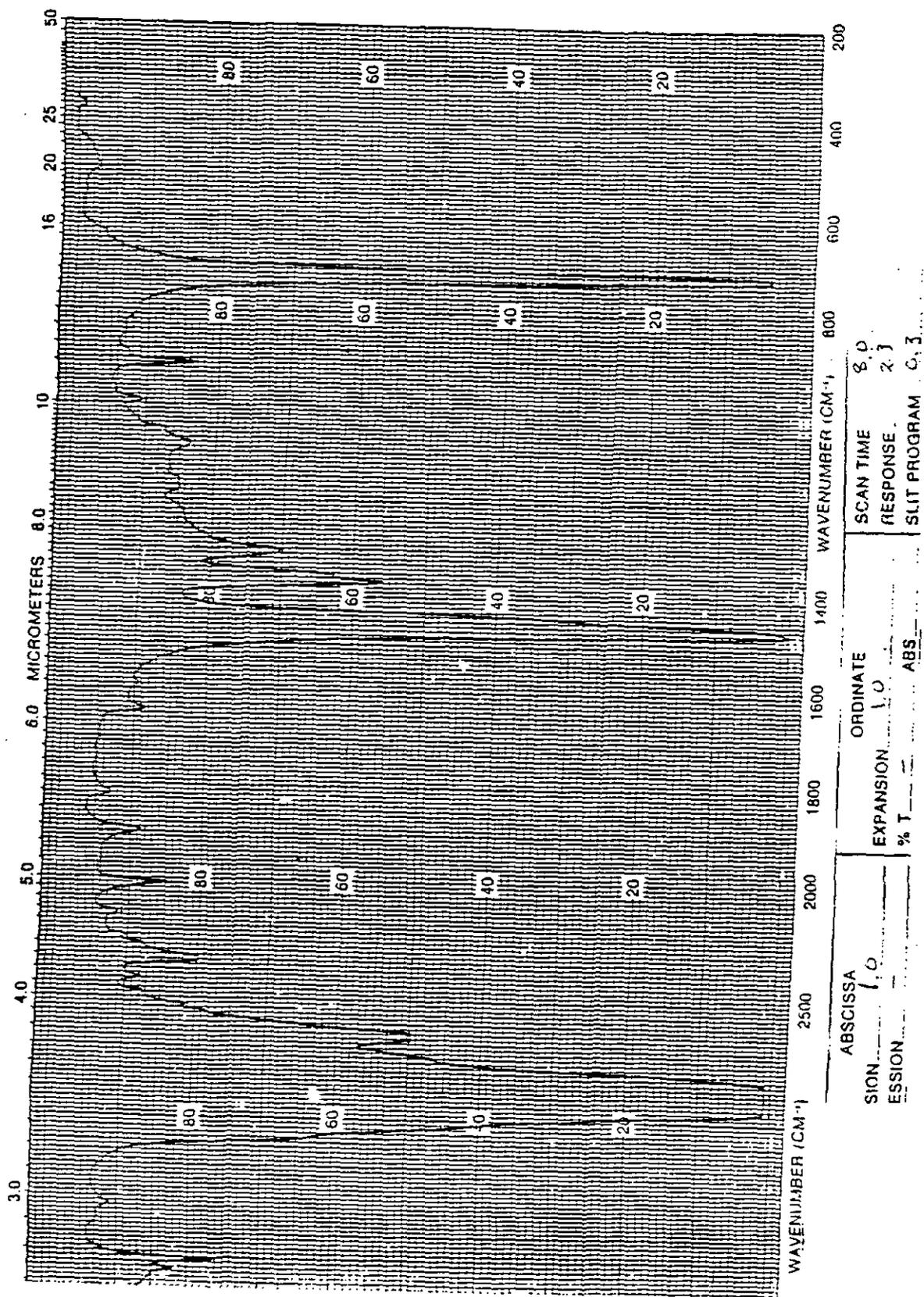


Figure 2. Infrared spectrum, type II

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(c) Procedure. Mill the specimen in a Wiley cutting mill or equal (see 6.6) equipped with a 10-mesh screen on the outlet tube. (If a milling apparatus other than a Wiley cutting mill is used, the extraction must be checked to determine the reflux time required for complete extraction.) Weigh to the nearest 0.1 mg a quantity of the milled specimen containing no more than 1.5 mg of "Santonox R". Transfer the sample to the bottom of a Wiley extractor tube or equal and add 30.0 mL of spectro grade cyclohexane. Place the extractor tube in a boiling water bath and reflux for 1 hour. Remove the tube from the water bath and cool to room temperature. Drain the clear extract into a small vial and securely cap to prevent evaporation loss. Transfer a measured aliquot to a separatory funnel fitted with a "Teflon" stopcock and extract three times with 15-mL portions of 1M sodium hydroxide solution. Combine the aqueous extracts in a 50-mL volumetric flask and dilute to volume with 1M sodium hydroxide solution. Measure the absorbance of the solution in a 1-cm cell versus a blank containing no specimen at 267 millimicrons. Using the calibration curve prepared as specified in (b), obtain the weight of "Santonox R" corresponding to the measured total absorbance. Calculate the percent by weight "Santonox R" as follows:

$$\text{Percent "Santonox R"} = \frac{A}{10BW}$$

where: A = Weight of "Santonox R" in milligrams obtained from calibration curve,
 B = Aliquot fraction extracted, and
 W = Weight of specimen in grams.

4.2.4.3 Flow rate. Determine the flow rate of the specimen in accordance with ASTM D 1238 using procedure A and condition F.

4.2.4.4 Physical characteristics of compression-molded specimen. Determine the physical characteristics of the compression-molded specimen specified in table I in accordance with the test methods specified in table III.

TABLE III. Test methods for compression-molded specimen

Characteristic	ASTM test method
Density	D 1505
Environmental stress-cracking resistance	D 1693, condition B
Hardness, type D durometer	D 2240
Low temperature brittleness	D 746, procedure A
	inspection
Tensile strength at yield	D 638, type IV specimen
Elongation at break	D 638, type IV specimen
Vicat softening point	D 1525, rate A

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5. PACKAGING

5.1 Commercial packing. A quantity of plastic material as specified (see 6.2) shall be packed in accordance with ASTM D 3951.

5.2 Marking. Containers shall be marked in accordance with ASTM D 3951. In addition, containers shall be marked to show the following information:

- (a) Manufacturer's lot number
- (b) Date of manufacture
- (c) Material identification

6. NOTES

6.1 Intended use. The materials covered by this specification are intended for use in the manufacture of plastic tubes and sheets.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Type of plastic material required (see 1.2)
- (c) Unit quantity required (see 5.1)

6.3 Plastic material composition. High density polyethylene copolymer made from an initial blend of approximately 96 percent by weight ethylene and 4 percent by weight butene-1 or hexene-1 has been found to produce satisfactory plastic material. However, the Government makes no warranty that plastic material made from the above initial blend will meet the requirements of this specification.

6.4 Infrared spectrometer cells. Beckman Cell No. 195480 and Barnes Cell No. 0004-025 have been found satisfactory for use in the infrared spectrum characteristics test.

6.5 Atmospheric infrared spectrum. See Perkin-Elmer Instrument Manual, Volume 3B for Model 21 instrument or comparable manufacturer's literature. Preferably, obtain an atmospheric spectrum on the spectrometer in use according to the manufacturer's instructions.

6.6 Wiley mill. A Wiley Intermediate Mill, Arthur H. Thomas No. 4276-M has been found satisfactory for use in preparing the sample.

6.7 "Santonox R". "Santonox R" is available from the Monsanto Chemical Company, St. Louis, MO. This material is chemically known as 4,4'-thiobis-(2-tert-butyl-5-methylphenol).

6.8 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E 29.

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6.9 Subject term (key word) listing.

Polyethylene butene-1 copolymer
Polyethylene hexene-1 copolymer

Custodian:

Army - EA

Preparing activity:

Army - EA

Project No. 9330-A009

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