MIL-C-429D
2 June 1982
SUPERSEDING
MIL-C-429C
6 May 1969

MILITARY SPECIFICATION

CHLORINATED PARAFFIN, TECHNICAL

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers two types of technical grade chlorinated paraffin.

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

Type I - 40 percent chlorine
Type II - 70 percent chlorine

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. Unless otherwise specified, the following specifications, standards, and handbooks of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieve, Test
PPP-D-729 - Drums, Shipping and Storage, Steel, 55-Gallon (208 Liters)

FSC 6810

: Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed: to: Commander, US Army Armament Research and Development Command, ATTN: DRDAR-TSC-S, Aberdeen Proving Ground, MD 21010 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129 - Marking for Shipment and Storage

(Copies of specifications, standards, handbooks, drawings, and publications required by manufacturers in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. The issues of the documents which are indicated as DOD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

ASTM STANDARDS

D71 - Density of Solid Pitch and Asphalt (Displacement Method)
D95 - Water in Petroleum Products and Bituminous Materials by Distillation
D1064 - Iron in Rosin
D1193 - Reagent Water
D1544 - Color of Transparent Liquids (Gardner Color Scale)
D1638 - Urethane Foam Isocyanate Raw Materials
D1744 - Water in Liquid Petroleum Products by Karl Fischer Reagent
E28 - Softening Point by Ring-And-Ball Apparatus

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

(Industry association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Chemical and physical characteristics. Chlorinated paraffin shall conform to the applicable chemical and physical characteristics of table I when tested as specified therein.

TABLE I. Chemical and physical characteristics

Characteristic	Type I	Type II	Test paragraph
Color, Gardner scale	No darker than No. 5		4.2.4.1
Hydrochloric acid, maximum per 20 ml	1.0 mg		4.2.4.2
Total chlorine, percent by weight	41.0 to 43.0	68.8 to 73.0	4.2.4.3
Free chlorine	To pass test		4.2.4.4
Water content, percent by weight, maximum	0.1	0.5	4.2.4.5
Iron, percent by weight, maximum	0.01	0.01	4.2.4.6
Volatile matter, maximum, ml per 100g		0.5	4.2.4.7
Specific gravity, 25°/15.6°C, minimum		1.60	4.2.4.8
Acid number, maximum		0.5	4.2.4.9
Softening point, °C, minimum		85	4.2.4.10
Percent by weight passing through a No. 50 sieve, minimum		98	4.2.4.11

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.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the chlorinated paraffin 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. In the event the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105.

4.2.2.2 For test. Sampling shall be conducted in accordance with table II. A representative specimen of approximately 1 liter of type I chlorinated paraffin or approximately 1 kilogram of type II chlorinated paraffin shall be removed from each sample 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 containers in batch or lot	Number of sample containers
2 to 25	2
26 to 150	3
151 to 1,200	5
1,201 to 7,000	8
7,001 to 20,000	10
Over 20,000	20

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled container, ready for shipment. Sample containers shall be examined for the following defects grouped collectively using an AQL of 4.0 defects per hundred units:

(a) Contents per container not as specified
(b) Container not as specified
(c) Container closure not as specified
(d) Container damaged or leaking
(e) Marking incorrect, missing, or illegible
(f) Drum lining not as specified or missing

4.2.3.2 For test. Approximately equal portions of all of the specimens taken in 4.2.2.2 shall be thoroughly mixed to form a composited specimen of no less than 1 liter for type I chlorinated paraffin and no less than 1 kilogram for type II chlorinated paraffin. The composited specimen shall be tested as specified in 4.2.4. Each test shall be conducted in duplicate 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 D1193 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 Color. Determine color in accordance with ASTM D1544.

4.2.4.2 Hydrochloric acid. Pipet 20.0 milliliters (ml) of the specimen into a 250-ml separatory funnel. Add 50 ml of toluene and shake thoroughly. Add 30 ml of 95-percent ethanol and shake thoroughly. Add 100 ml of water and shake thoroughly. Allow the mixture to stand until the layers separate. Separate the two layers. To the alcohol-water (lower) layer, add a few drops of mixed indicator solution containing 0.050 gram (g) of methyl red and 0.075 g of bromcresol green dissolved in 100 ml of 95-percent ethanol. Titrate with 0.02N sodium hydroxide solution to the intermediate purple end point. Calculate the milligrams (mg) of hydrochloric acid per 20 ml of specimen as follows:

$$\text{Milligrams hydrochloric acid per 20 ml of specimen} = 36.5\ AB$$

where: A = Milliliters of sodium hydroxide solution used and
B = Normality of sodium hydroxide solution.

4.2.4.3 Total chlorine. Determine percent total chlorine in accordance with ASTM D1638, using the procedure for Total Chlorine by Schoniger Oxygen Flask.

4.2.4.4 Free chlorine. Prepare a starch-iodide solution by dissolving 5 g of potassium iodide in 100 ml of water containing 10 ml of 0.2-percent starch solution. Pipet 3 ml of the specimen into a test tube, add 2 ml of the starch-iodide solution, and shake vigorously for 1 minute. No blue color shall appear in the aqueous layer.

4.2.4.5 Water content. Determine percent water content in accordance with ASTM D1744 using chloroform as the solvent.

4.2.4.6 Iron. Prepare the required reagents and a calibration curve in accordance with the spectrophotometric method of ASTM D1064. Weigh to the nearest milligram approximately 10 g of the specimen and dissolve in 100 ml of toluene. Transfer the solution to an extractor and extract with 50 ml of concentrated hydrochloric acid by refluxing the acid and solution for 30 minutes. Remove from heat, allow to cool to room temperature, and transfer to a 250-ml volumetric flask. Wash the extractor with 20 ml of water and drain into the flask. Dilute to 250-ml with water. Pipet a 10-ml aliquot of the solution into a 50-ml Erlenmeyer flask and proceed as specified in the spectrophotometer method of ASTM D1064. Read the milligrams of iron from the calibration curve and calculate the percent iron as follows:

$$\text{Percent iron} = \frac{2.5\ A}{W}$$

where: A = Milligrams of iron from calibration curve and
W = Weight of specimen in grams.

4.2.4.7 Volatile matter. Determine percent volatile matter in accordance with the procedure in ASTM D95 except use 100 ml of water as the solvent.

4.2.4.8 Specific gravity. Determine specific gravity at 25°/15.6° C in accordance with ASTM D71.

4.2.4.9 Acid number. Weigh to the nearest 0.01 g approximately 25 g of the specimen into a 300-ml Erlenmeyer flask. Add 50 ml of toluene and warm on a steam bath with shaking until the specimen is well dissolved. Transfer to a separatory funnel. Rinse the flask with 50 ml of alcohol and add the alcohol to the separatory funnel. Add 100 ml of water to the separatory funnel and shake for 1 minute. Allow the layers to separate, draw off, and add phenolphthalein indicator to the aqueous layer. Titrate with 0.1N potassium hydroxide solution to the first permanent pink color. Calculate the acid number as follows:

$$\text{Acid number} = \frac{56.1\ AB}{W}$$

where: A = Milliliters of potassium hydroxide solution used,
B = Normality of potassium hydroxide solution, and
W = Weight of specimen in grams.

4.2.4.10 Softening point. Determine softening point in accordance with ASTM E28, paragraphs No. 6 and No. 8.

4.2.4.11 Percent passing through a No. 50 sieve. Weigh to the nearest 0.1 g approximately 100 g of the specimen and place on a No. 50 sieve conforming to RR-S-366 with a bottom pan. Cover and shake with a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Shake for 30 ± 1 minute, weigh the material remaining on the sieve and calculate the percent passing.

5. PACKAGING

5.1 Unit packing. Unit packing shall be level A or C as specified (see 6.2).

5.1.1 Level A.

5.1.1.1 Type I chlorinated paraffin. A quantity of 204 (+2 or -0) kilograms of type I chlorinated paraffin shall be packed in a 55-gallon metal drum conforming to type II of PPP-D-729. The interior of each drum, including the inner facing of the closures, shall be provided with an epoxy-phenolic lining of 0.038 millimeters minimum thickness in accordance with PPP-D-729. Cap seals shall be provided for closures.

5.1.1.2 Type II chlorinated paraffin. A quantity of 238 (+2 or -0) kilograms of type II chlorinated paraffin shall be packed in a removable head steel drum conforming to type IV of PPP-D-729. Interior surfaces shall be provided with an epoxy-phenolic lining of 0.038 millimeters minimum thickness in accordance with PPP-D-729.

5.1.2 Level C. Type I or type II chlorinated paraffin shall be packed as specified in 5.1.1.1 or 5.1.1.2, respectively, except that the exterior surface preparation and exterior coating of the drum shall not be required. In lieu thereof, any high baked enamel shall be applied.

5.2 Marking. Containers shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. Type I chlorinated paraffin is intended for use in clothing impregnation applications. Type II chlorinated paraffin is intended for use in flame retardation applications.

6.2 Ordering data. Acquisition documents should specify the following:

(a) Title, number, and date of this specification
(b) Type of chlorinated paraffin required (see 1.2)
(c) Level of unit packing required (see 5.1)

6.3 Batch. A batch is defined as that quantity of material which has been manufactured by some unit chemical process or subjected to physical mixing operation intended to make the final product substantially uniform.

6.4 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 E29.

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