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15 December 1967
SUPERSEDING
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26 April 1961

MILITARY SPECIFICATION

RESORCINOL

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers resorcinol for use in explosives.

1.2 Classification. The resorcinol shall be of the following types and classes.

Type I - Crystal

Class 1 - General
Class 2 - Special

Type II - Powder

Type III - Flake, Technical Grade

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 specification to the extent specified herein:

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieves, Standard for Testing Purposes
PPP-C-301 - Chemicals, Dry and Paste, Packaging and Packing of
PPP-D-723 - Drums, Fiber

FSC 6810

STANDARDS

FEDERAL

Fed. Test Method Standard No. 791 - Lubricants, Liquid Fuels, and Related Products; Methods of Testing

MILITARY

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

(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.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM D-1193-66 - Reagent Water
ASTM D-1631-58T - Method of Test for ASTM Color of Petroleum Products
ASTM D-1631-59T - Water in Phenol and Related Materials by the Iodine Reagent Method

(Application for copies of ASTM publications should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa., 19103.)

CONSOLIDATED CLASSIFICATION COMMITTEE

Uniform Freight Classification Rules

(Application for copies should be addressed to the Consolidated Classification Committee, 202 Chicago Union Station, Chicago 6, Illinois.)

3. REQUIREMENTS

3.1 Material. The materials used in the manufacture of the resorcinol shall be of high quality. The resorcinol shall conform to the applicable requirements as specified herein and shall be visibly free of contaminants and foreign material such as grit and dirt.

3.2 Chemical and physical properties. The chemical and physical properties of resorcinol shall conform to the requirements of table I.

Table I. Chemical and physical properties

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Property	Requirements				Paragraph
	Type I		Type II	Type III	
	Class 1	Class 2			
Resorcinol content, % min.	99.5	99.5	99.5	99.5	4.4.2
Ash, % max.	0.005	0.005	0.005	0.005	4.4.3
Moisture, % max.	0.2	0.2	0.2	0.2	4.4.4
Phenol	no perceptible odor	no perceptible odor	no perceptible odor	no perceptible odor	4.4.5
Catechol, % max.	0.1	0.1	0.1	0.1	4.4.6
pH, min.	4.4	6.0	4.4	4.4	4.4.7
Water insoluble, % max.	0.01	0.01	0.01	0.01	4.4.8
Color when molten	-	-	-	1.0 on ASTM Scale	4.4.9
Solidification or freezing point, min.	-	109.7°C	-	-	4.4.10
Melting range	-	109 to 111°C	-	-	4.4.10
Granulation (thru U.S. sieve No. 8) min. %	-	98	-	-	4.4.11

3.3 Workmanship. The resorcinol shall be uniform in quality and shall conform to the requirements of this specification.

4. QUALITY ASSURANCE PROVISIONS

4.1 Inspection responsibility. 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 or any other inspection facilities and services acceptable to 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 that supplies and services conform to prescribed requirements.

4.2 Acceptance inspection. Conformance of the resorcinol to the requirements of this specification shall be determined entirely by means of acceptance inspection. The acceptance inspection shall consist of an examination for acceptability of facilities and quality control methods used by the manufacturer, examining and testing the acceptance samples (4.3.2) for all of the requirements of this specification, and an examination of the sample of filled containers (4.3.3) for conformance to section 5.

4.3 Sampling.

4.3.1 Lot. A lot shall consist of all resorcinol of the same type and class, manufactured by the same process, packed as specified in the contract or order (see 6.2) and offered for acceptance at one time.

4.3.2 Sample for test. From each lot offered for acceptance under contract, a random sample shall be selected in accordance with MIL-STD-105, inspection level II. A primary sample of one pint of material shall be removed from each container selected and stored in a large mouth glass stoppered bottle. Each primary sample shall be labeled so that the container from which it was taken can be easily identified. A composite sample shall be made of equal portions of these samples and a minimum of one pint shall be used for acceptance tests as specified in 4.4. The sample shall be labeled to show the name and address of the manufacturer, plant, name of product, specification number, lot number, date of sampling, and contract number.

4.3.3 Sample for examination of filled containers. A random sample of filled containers shall be selected from each lot of resorcinol offered for acceptance under contract, in accordance with MIL-STD-105, inspection level I and acceptable quality level (AQL) = 2.5 percent defective, to determine compliance with the requirements of section 5.

4.4 Inspection methods. Unless otherwise specified, all examinations and tests shall be conducted as specified herein to determine compliance with the applicable requirements of section 3. Water in accordance with ASTM D-1193 and reagent chemicals shall be used.

4.4.1 Visual inspection. Conformance of the resorcinol to the requirements for material (3.1) and workmanship (3.3) shall be determined by visual inspection.

4.4.2 Resorcinol content.

4.4.2.1 Reagents.

4.4.2.1.1 Starch indicator solution. Mix 2 gm. of soluble starch and several milligrams of the mercuric iodide (which acts as a preservative) with a little water. Add the mixture slowly to 500 ml. of boiling water and boil for 5 minutes. Cool to room temperature.

4.4.2.1.2 Standard sodium thiosulfate solution (approximately 0.1N). Dissolve 26 gm. of sodium thiosulfate pentahydrate and 0.1 gm. of sodium carbonate in sufficient freshly boiled and cooled water to make 1 liter. Let stand at least 24 hours before standardization. Standardize the solution as follows: Dissolve 0.20 to 0.23 gm. of potassium dichromate, weighed to 0.1 mg. in 60 ml. of water in an iodine titration flask and add 20 ml. of 1 N hydrochloric acid solution. Stopper the flask, swirl for a few seconds, and let stand for 10 minutes in the dark. Titrate with sodium thiosulfate (swirling continuously) until a light greenish brown color appears. Add 5 ml. of starch indicator solution, and continue titration until the deep blue starch-iodide color changes to a pale bluish green. Calculate the normality of the sodium thiosulfate solution as follows:

$$\text{Normality of sodium thiosulfate solution} = \frac{A}{0.04904B}$$

where: A = weight of potassium dichromate, gm.
B = volume of sodium thiosulfate, ml.

4.4.2.1.3 Potassium bromate-bromide solution (approximately 0.2N). Dry potassium bromate in an oven for 2 hours at 100°C and weigh 5.6 gm. into a pyrex bottle. Add 30 gm. of potassium bromide. Dissolve in water and dilute to 1 liter.

4.4.2.2 Procedure. Dissolve 1.5 gm. of the sample (weighed to 0.1 mg.) in water and dilute to 500 ml. in a volumetric flask. Mix thoroughly. Transfer a 20 ml. aliquot to an iodine flask. Add 25.00 ml. of the bromate-bromide solution to the flask, dilute with 100 ml. of water, add 10 ml. of hydrochloric acid, and immediately stopper the flask. Shake the flask and allow it to stand for 3 minutes. Place 15 ml. of 15 percent potassium iodide solution in the lip of the flask and remove the stopper just sufficiently to allow the potassium iodide solution to enter the flask without allowing any bromine vapor to escape. With the stopper in place, shake the flask thoroughly. Wash down the stopper and the neck of the flask with about 20 ml. of water. Titrate the liberated iodine with standard sodium thiosulfate solution until the solution assumes a light yellow color. Add 5 ml. of starch solution and continue the titration carefully until the blue color in the solution disappears. Carry out a blank titration using 75 ml. of water, 25.00 ml. of bromate-bromide solution and 10 ml. of hydrochloric acid. After the 3 minute waiting period, add 15 ml. of potassium iodide solution, and titrate the liberated iodine by the same procedure used for the sample. Calculate the resorcinol content as follows:

$$\text{Percent resorcinol} = \frac{(A-B)N \times 1.835}{W}$$

where: A = volume of thiosulfate solution for titration of blank, ml.
B = volume of thiosulfate solution for titration of sample, ml.
N = normality of thiosulfate solution.
W = weight of sample in aliquot, gm.

4.4.3 Ash. Transfer a 5 gm. sample to a tared porcelain crucible and heat gently with a Bunsen burner until the material is ignited at the surface. Remove the burner and allow the material to burn completely. After all of the free carbon on the sides of the crucible has burned-off completely, heat the residue with a strong flame or in a muffle furnace at 700° to 750°C until all the carbonaceous material has disappeared, then heat for an additional 30 minutes. Cool in a desiccator and weigh. Calculate the percent ash as follows:

$$\text{Percent ash} = \frac{100A}{W}$$

where: A = weight of residue, gm.
W = weight of sample, gm.

4.4.4 Moisture. The moisture shall be determined in accordance with ASTM D-1631-59T of Fed. Test Method Std. No. 791, method 3253.

4.4.5 Phenol. Dissolve 1 gm. of the sample in 20 ml. of water and warm gently. Phenol is not present if its odor is not detectable.

4.4.6 Catechol.

4.4.6.1 Reagents. Lead acetate solution (35 percent). Dissolve 35 gm. of lead acetate trihydrate in water and dilute to 1 liter. This solution should be filtered through No. 40 Whatman filter paper just before using so that it is free from lead carbonate.

4.4.6.2 Procedure. Weigh a 10 gm. sample into a 400 ml. beaker. Add 100 ml. of carbon dioxide-free water and stir until dissolved. Add 75 ml. of clear, freshly filtered lead acetate solution, stir, cover, and allow to stand for 3 hours, protected from fumes of H_2S and SO_2. Filter through a tared fritted glass crucible of medium porosity, wash with water and dry for 2 hours at 105° to 110°C. Cool in a desiccator and weigh. Calculate the percent catechol as follows:

$$\text{Percent catechol} = \frac{34.9A}{W}$$

where: A = weight of precipitate, gm.
W = weight of sample, gm.

Note: It is essential that water used in the determination be free of carbon dioxide, which reacts with lead acetate to form insoluble lead carbonate.

4.4.7 pH. Weigh 5 gm. of the sample into a 150 ml. beaker. Add 50 ml. of water having a pH of 6.8 ± 0.2. Stir the mixture until the sample is dissolved and determine the pH by means of a pH meter equipped with glass-calomel electrodes.

4.4.8 Water insoluble. Weigh a 10 gm. sample into a 400 ml. beaker. Add 100 ml. of water and stir until dissolved. Filter through a tared fritted glass crucible of medium porosity, wash with water, and dry for 1 hour at 105° to 110°C. Cool in a desiccator and weigh. Calculate the percent insoluble as follows:

$$\text{Percent insoluble} = \frac{100A}{W}$$

where: A = weight of residue, gm.
W = weight of sample, gm.

4.4.9 Color when molten. The molten color shall be determined in accordance with ASTM D-1500-58T.

4.4.10 Solidification or freezing point and melting range.

4.4.10.1 Apparatus. As shown in figure 1. The thermometer shall include the range of 90° to 120°C and shall be corrected.

4.4.10.2 Procedure. First dry the sample of approximately 40 gm. on a watch glass overnight in a desiccator containing an efficient desiccant such as fresh Drierite. This drying is necessary because a small amount of water significantly affects the freezing point. Heat the inner test tube (fig. 1) moderately over a flame to remove moisture. Place about 35 gm. of sample in this test tube and heat carefully to effect liquefaction. After approximately half of the sample has liquefied, insert the stirrer, stir with a reciprocating action of the stirrer to effect an even distribution of the heat, and carefully heat further until complete fusion has been obtained. The temperature of the fused material may thus be kept below 120°C which is desirable. Insert the thermometer and stopper so that the liquid level is at the immersion line of the thermometer. Stir until the thermometer is free from crystals, heating further if necessary. Place the assembly in the insulating jacket and allow to cool while stirring at a rate of about 120 strokes per minute. Observe the temperature during cooling, meanwhile noting the sample for the appearance of crystals. Normally, the sample super-cools slightly, then as crystallization starts, the temperature rises to a plateau, which is maintained for at least one-half minute, and may be retained for several minutes if the assembly is sufficiently insulated. This plateau is the freezing point. In the case of impure resorcinol, the plateau may be difficult to observe. In such a case, seeding the sample near the estimated freezing point with a few crystals of pure resorcinol usually reduces super-cooling and results in a sustained plateau.

4.4.11 Granulation. Place 100 gm. of the sample on a No. 8 U. S. standard sieve (conforming to RR-S-366). Cover and shake for 10 minutes in a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Weigh the portion that is retained and calculate to percentage of sample that is retained.

5. PREPARATION FOR DELIVERY

5.1 Packing and packaging.

5.1.1 Level A. Resorcinol shall be packaged in a heat sealed polyethylene bag in accordance with PPP-C-301 inside a lined fiber drum conforming to PPP-D-723, type III, grade A. Each drum shall not exceed 200 pounds net (see 6.2).

5.1.2 Level B. Resorcinol shall be packaged in a heat-sealed polyethylene bag in accordance with PPP-C-301 inside a lined fiber drum conforming to PPP-D-723, type II, grade A. Each drum shall not exceed 100 pounds net (see 6.2).

5.1.3 Level C. Resorcinol shall be packed in water tight shipping containers in accordance with PPP-C-301 for shipment from the supply source to the first receiving activity for immediate use. Containers shall comply with Consolidated Freight Classification Rules or other common carrier regulations applicable to the mode of transportation (see 6.2).

5.2 Marking. In addition to any special marking specified, shipping containers shall be marked in accordance with MIL-STD-129.

5.2.1 Special labeling. Each container of resorcinol shall be labeled as follows:

"RESORCINOL
Do not take internally.
Avoid breathing dust.
Can cause skin irritation.
Can be absorbed through the skin.
Wash thoroughly after handling.

6. NOTES

6.1 Intended use. Type I (class 1), type II and type III are intended for use in various ordnance applications. Type I (class 2) is intended for use in the manufacture of lead styphnate.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number and date of this specification.
(b) Type and class required.
(c) Size and type of shipping container.
(d) Weight of resorcinol to be packed in a shipping container.
(e) Level of packaging and packing desired (see section 5).
(f) Special labeling (see 5.2.1)

Custodian:
Army - MU

Review activities:
Army - MD, MU

User activities:
Army - MI

Preparing activity:
Army - MU

Project No. 6810-A789

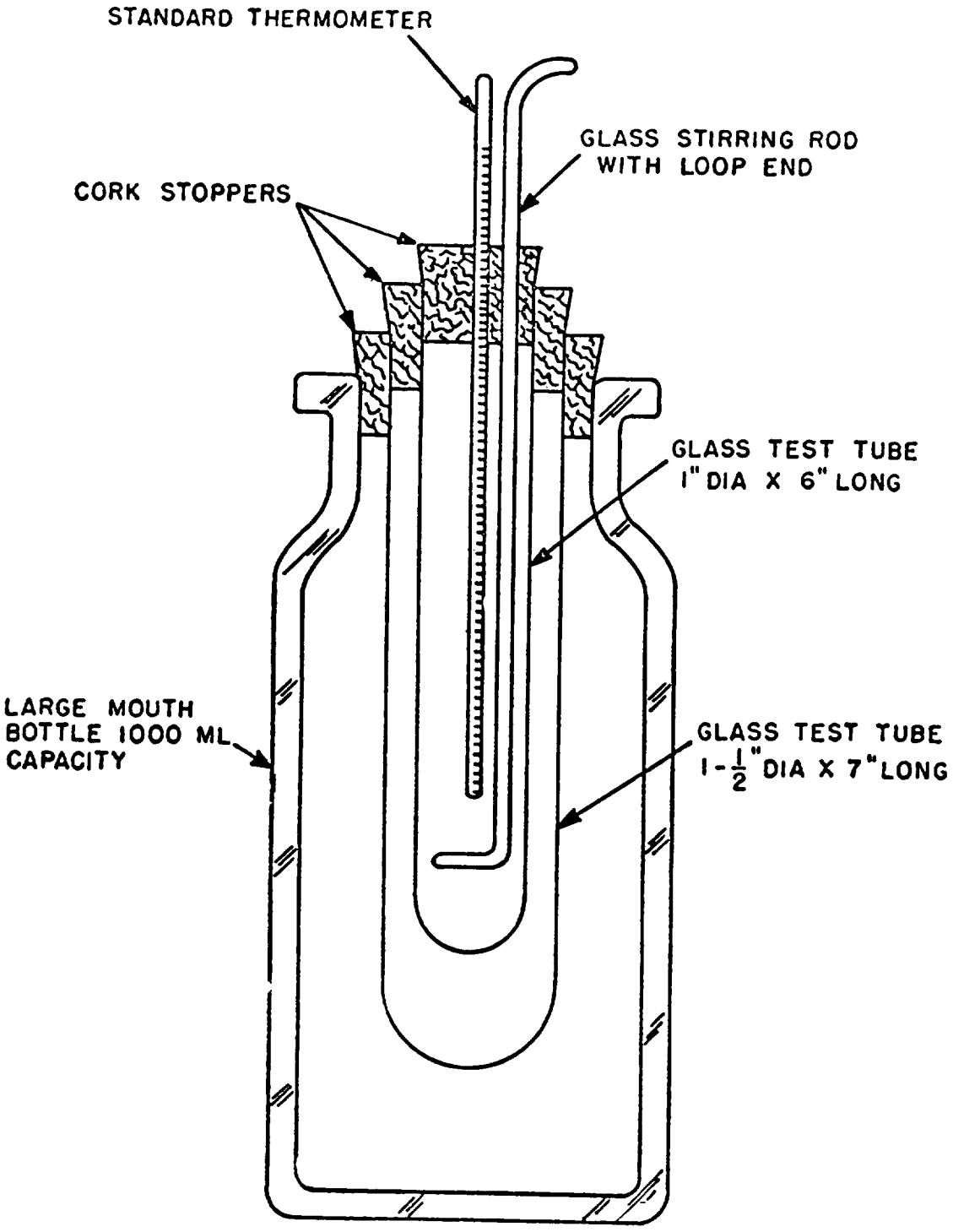

FIGURE 1. *Solidification point apparatus.*

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SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 22-R255

INSTRUCTIONS: 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. Comments and suggestions submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or serve to amend contractual requirements.

SPECIFICATION

ORGANIZATION

CITY AND STATE

CONTRACT NUMBER

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ 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?

☐ YES ☐ 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 - Optional)*

DATE

DD FORM 1 JAN 63 1426

REPLACES EDITION OF 1 OCT 54 WHICH MAY BE USED.