

MIL-R-51029C
 4 April 1983
 SUPERSEDING
 MIL-C-51029B
 15 November 1968

MILITARY SPECIFICATION

RIOT CONTROL AGENT, CS

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

1. SCOPE

1.1 Scope. This specification covers one type of CS, chemically known as o-chlorobenzalmalononitrile.

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

L-P-378 - Plastic Sheet and Strip, Thin Gauge, Polyolefin

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by
 Attributes

MIL-STD-650 - Explosive: Sampling, Inspection and Testing

: Beneficial comments (recommendations, additions, deletions) and any perti- :
 : nent 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. :

FSC 1365

MIL-R-51029C

2.1.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this specification to the extent specified herein.

CODE OF FEDERAL REGULATIONS (CFR)

49 CFR 171 to 179 - Department of Transportation Hazardous Materials Regulations

(The Code of Federal Regulations is available from the Superintendent of Documents, US Government Printing Office, Washington, DC 20402. Orders for the above publication should site "49 CFR 171 to 179.")

(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

D1193 - Reagent Water

(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 characteristics. CS shall conform to the chemical characteristics of table I when tested as specified therein.

MIL-R-51029C

TABLE I. Chemical characteristics

Characteristic	Requirement		Test paragraph
	Minimum	Maximum	
Assay, percent by weight	96.0	---	4.2.4.1
o-Chlorobenzaldehyde, percent by weight	----	1.0	4.2.4.2
Malononitrile, percent by weight	----	1.0	4.2.4.2
Volatile matter, percent by weight	----	1.0	4.2.4.3

3.2 Melting characteristics. CS shall begin to melt at no less than 93.0°C and shall be completely melted at no greater than 96.5°C when tested as specified in 4.2.4.4.

3.3 Particle size characteristics. When specified in the contract or purchase order (see 6.2), CS shall conform to the particle size characteristics of table II when tested as specified in 4.2.4.5.

TABLE II. Particle size characteristics

Sieve Size	Percent by Weight Passing
No. 16	100 minimum
No. 100	85 maximum
No. 200	25 maximum
No. 325	15 maximum

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.

MIL-R-51029C

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the CS 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 CS test (see 6.6). Sampling for test shall be conducted in accordance with table III. A representative specimen of approximately 500 grams (g) 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. Metal cans or plastic bottles with wide-mouth screw tops are recommended. Paper or plastic bags shall not be used.

TABLE III. 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 :
: :	: :

4.2.2.3 For container leakage test. Sampling shall be conducted in accordance with MIL-STD-105.

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled unit or shipping container, as applicable, ready for shipment. Sample unit and shipping containers shall be examined for the following defects using an AQL of 1.5 percent defective:

- (a) Contents per container not as specified
- (b) Container not as specified
- (c) Seams of plastic bag incompletely heat sealed
- (d) Container closure not as specified
- (e) Container damaged or leaking
- (f) Marking incorrect, missing, or illegible

MIL-R-51029C

4.2.3.2 For CS test (see 6.6). Each sample specimen taken in 4.2.2.2 shall be tested as specified in 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.3.3 For container leakage test. The sample unit shall be one container. The sample containers selected in 4.2.2.3 shall be tested as specified in 4.2.5 using an AQL of 1.5 percent defective.

4.2.4 CS 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 Assay.

(a) Ferric alum indicator. Dissolve 225 g of ferric ammonium sulfate and 100 milliliters (mL) of concentrated nitric acid in 1 liter of water.

(b) Procedure. Weigh to the nearest 0.1 milligram (mg) approximately 0.5 g of the specimen into a 250-mL Erlenmeyer flask and dissolve in 75 mL of absolute methanol. With vigorous swirling, add 25 mL of sodium cyanide solution (7.35 g of sodium cyanide per liter of water). Allow to stand for 10 to 15 seconds, add 2 mL of concentrated nitric acid, and swirl to mix. Immediately add 50 mL of silver nitrate solution (18.7 g of silver nitrate per liter of water) and swirl to mix. Filter through a medium pore qualitative filter paper on a Buchner funnel. To the filtrate, add 2.0 mL of ferric alum indicator prepared as specified in (a) and titrate with standardized 0.1N potassium thiocyanate solution to a pink end point. Run blank titers daily. Duplicate titers should agree within 0.1 mL. Calculate the percent by weight CS as follows:

$$\text{Percent CS} = \frac{18.8 (A - B) C}{W}$$

where: A = Milliliters of potassium thiocyanate solution used to titrate specimen,
 B = Milliliters of potassium thiocyanate solution used to titrate blank,
 C = Normality of potassium thiocyanate used, and
 W = Weight of specimen in grams.

4.2.4.2 O-chlorobenzaldehyde, and malononitrile.

4.2.4.2.1 Equipment and apparatus. Determine the percentages of o-chlorobenzaldehyde and malononitrile by means of gas-liquid chromatography with thermal conductivity detector using an internal standard (1, 10-dibromodecane has been found to be satisfactory for this application, but other purified chemicals may be employed). Isothermal or temperature programming may be used. Recommended column parameters and chromatographic conditions for an F&M Model

MIL-R-51029C

720 chromatograph are shown in table IV. Other equivalent instruments may be used but may require modification of conditions in order to obtain good peak shape, adequate resolution, and appropriate retention times.

TABLE IV. Chromatographic conditions

Characteristic	Condition
Column material	Pyrex glass
Column dimensions	6 feet (1/4-inch OD)
Column support	60/80 mesh "Gas-Chrom Q"
Column temperature	5°C/min from 65°C to 190°C
Column coating	10 percent QF-1
Injection port temperature	220°C
Detector temperature	200°C
Carrier gas	Helium
Carrier gas flow rate	90 ml/min
Attenuation	X4 (as needed)
Bridge current	135 ma

4.2.4.2.2 Calibration. Determine response factors for each of the two constituents as follows:

(a) Prepare a 50 mg per mL solution of each constituent of known purity in benzene, and a 50 mg per mL solution of the 1,10-dibromodecane of known purity in benzene.

(b) Under identical conditions, inject solutions of the constituent and of the internal standard in 5-microliter increments from 5-microliter to 50-microliter volumes.

(c) Plot concentration versus integrated area. Calculate the response factor from the ratio of the slopes, corrected for purity as follows:

$$R = \frac{SV}{TU}$$

where: R = Response factor,
 S = Area per unit weight of constituent,
 T = Area per unit weight of standard,
 U = Purity of constituent, percent by weight, and
 V = Purity of standard, percent by weight.

4.2.4.2.3 Procedure. Weigh approximately 0.5 g each of the specimen and the internal standard to the nearest milligram into a 10-ml volumetric flask. Dissolve in benzene and dilute to the mark with benzene. Prior to quantitation, saturate the column three times with 20-microliter injections of the

MIL-R-51029C

benzene solution. For the above conditions malononitrile elutes in 6 minutes at approximately 98°C, o-chlorobenzaldehyde in 7.5 minutes at 104°C, and 1,10-dibromodecane in 20 minutes at about 165°C.

4.2.4.2.4 Calculations. From the area ratios and predetermined response factors calculate the percentages of o-chlorobenzaldehyde and malononitrile as follows:

$$\text{Percent constituent} = \frac{ACP}{BDR}$$

where: A = Area of constituent,
 B = Area of standard,
 C = Weight of standard, in grams,
 D = Weight of specimen, in grams,
 P = Purity of standard, percent by weight, and
 R = Response factor for constituent involved.

4.2.4.3 Volatile matter.

(a) Apparatus. Determine percent volatile matter on a moisture balance type apparatus with an infrared lamp source, with the lamp being the reflector type and rated at 125 watts and 115 to 125 volts.

(b) Procedure. Place an amount of specimen (based on the amount recommended for the apparatus being used) into the apparatus and set the voltage regulator to 60 volts. Read every 15 minutes until a constant value is obtained.

4.2.4.4 Melting characteristics. Determine the temperature at which the specimen begins to melt and the temperature at which the specimen is completely melted in accordance with MIL-STD-650, method 209.1.

4.2.4.5 Particle size characteristics. Use sieves conforming to RR-S-366. Weigh the sieves specified in 3.3 to the nearest 0.01 g. Nest them in order of fineness with the coarsest sieve on top, and tape the sieves together to prevent loss of specimen. Place the assembly on a bottom pan in a well ventilated fume hood. Weigh to the nearest 0.01 g approximately 25 g of the specimen and place it on the top sieve. Cover the sieves and shake for 10 minutes by means of a mechanical shaker geared to produce 300 + 15 gyrations and 150 + 10 taps of the striker per minute. Weigh the material retained on each sieve and the bottom pan and calculate the percent by weight specimen passing through each sieve. NOTE: Do not brush material through screens.

4.2.5 Container leakage test. Place the container in each of the following positions, and leave it in each for a period of 15 minutes:

MIL-R-51029C

- (a) Upright
- (b) Upside down
- (c) On one side
- (d) On other side

Examine the container after each period for any evidence of leakage.

5. PACKAGING

5.1 Unit packing. CS, in a quantity as specified in the contract or purchase order (see 6.2), shall be unit packed level C in a polyethylene bag fitted in a removeable head steel drum in accordance with Department of Transportation (DOT) regulations. The bag shall have a minimum thickness of 0.006 inch and shall be formed from polyethylene conforming to type I, grade and finish optional of L-P-378. The seams of the bag shall be heat sealed. The closure shall be by double tying. There shall be no evidence of CS on the exterior of the closed drum when tested as specified in 4.2.5.

5.2 Packing. CS, unit packed level C as specified in 5.1, shall require no further protection for shipment.

5.3 Marking. Containers of CS shall be marked in accordance with DOT regulations and in a manner to assure safe and timely delivery from the supply source to destination. In addition, each container shall be marked to include the date of manufacture, lot number of the CS, and the following precautionary information:

HAZARD:

Irritant, Eye and Skin.

WARNING! CAUSES IRRITATION

Avoid contact with eyes, skin, and clothing.
Wash thoroughly after handling.

FIRST AID: In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician. Flush skin with water. (Wash clothing before reuse.) Do not rub eyes or skin if exposure occurs. Do not leave exposed individual unattended.

6. NOTES

6.1 Intended use. CS is intended for use in riot control.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification
- (b) Particle size characteristics required (see 3.3)
- (c) Unit quantity required (see 5.1)

MIL-R-51029C

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

6.4 Standards. Malononitrile may be purified by distillation. The fraction distilling at 92° to 94°C at 8 mm absolute pressure has been found to be satisfactory for use as a standard. O-chlorobenzaldehyde having a melting point of 9° to 11°C and a refractive index of N_D^{20} 1.566 has been found to be satisfactory for use as a standard.

6.5 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.

6.6 Sampling and testing precautions. This specification covers inspection and use of chemical material (CS - irritant, benzene - Carcinogen) which is potentially hazardous to personnel. These materials should be handled in well ventilated fume hoods. CS produces irritating physiological effects, such as stinging sensations to moist skin, running nose, sneezing, coughing, and burning of the eyes accompanied by a copious flow of tears. All applicable environmental and safety rules, regulations, and procedures must be followed in the handling and processing of these materials.

6.7 International standardization agreement. Certain provisions of this specification are the subject of international standardization agreement QSTAG 495. When amendment, revision, or cancellation of this specification is proposed which will modify the international agreement concerned, the preparing activity will take appropriate action through international standardization channels including departmental standardization offices to change the agreement or make other appropriate accommodations.

Custodians:

Army - EA
Air Force - 70

Review activities:

Army - MD

Preparing activity:

Army - EA
Project No. 1365-0221

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