

MIL-P-48240 (PA)
15 February 1974
SUPERSESSION
(see 6.4)

MILITARY SPECIFICATION
PYROTECHNIC
FIRST-FIRE COMPOSITIONS

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

1. SCOPE

1.1 This specification covers three types of non-hydroscopic first fire compositions (see 6.1).

Type I - yellow
Type II - red
Type III- green

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids form a part of this specification to the extent specified herein.

SPECIFICATIONS

MILITARY

MIL-B-162	Barium Nitrate
MIL-S-230	Silicon, Powdered
MIL-A-2550	Ammunition and Special Weapons, General Specification for
MIL-T-13723	Tetranitrocarbazole (TNC)
MIL-P-20307	Polyvinyl Chloride (For Use in Pyrotechnics)
MIL-S-20322	Strontium Nitrate, Non-Hydroscopic

STANDARDS

MILITARY

FSC: 1370

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- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes (ABC-STD-105)
- MIL-STD-109 - Inspection Terms and Definition
- MIL-STD-1168 - Lot Numbering of Ammunition
- MIL-STD-1234 - Pyrotechnics: Sampling, Inspection and Testing
- MIL-STD-1235 - Single and Multilevel Continuous
- Sampling Procedures and Tables for Inspection by Attributes

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

3. REQUIREMENTS

3.1 Material. The raw materials used in the manufacture of first-fire composition shall comply with the requirements of the applicable documents, The following materials shall be of the size and grade specified.

3.1.1 Barium nitrate. The barium nitrate shall comply with the requirements of Specification MIL-B-162, except that the average particle size shall be twenty(29) microns, maximum.

3.1.2 Strontium nitrate. The strontium nitrate shall comply with the requirements of Specification MIL-S-20322, Grade A or B.

3.1.3 Silicon. The silicon Shall be powdered and shall comply with the requirements of Specification MIL-S-230, except that the average particle size shall be 10 microns, maximum.

3.1.4 Tetranitrocarbazole. The tetranitrocarhazole shall comply with the requirements of' Specification MIL-T-13723.

3.1.5 Polyvinyl chloride.. The polyvinyl chloride shall comply with the requirements of MIL-P-20307.

3.2 Composition. The first-fire composition shall be as specified in Table I.

TABLE I

Percent by weight

First-fire composition	Type I	Type II	Type III
Barium nitrate	50 \pm 2	--	50 \pm 2
Strontium nitrate (non-hygrosopic)	--	50 \pm 2	-
Tetranitrocarbazole(TNC)	10 \pm 1	10 \pm 1	10 \pm 1
Silicon, powdered	20 \pm 1	16 \pm 1	13 \pm 1
Zirconium hydride (commercial)	15 \pm 1	15 \pm 1	20 \pm 1
Polyvinyl chloride	--	5 \pm 0.5	3 \pm 0.5
Laminac 4116 + 1 percent Lupersol DDM catalyst (see note)	5 \pm 0.5	4 \pm 0.5	4 \pm 0.5

(Note: Lupersol DDM catalyst is a 60 percent solution of methyl ethyl ketone peroxide in dirnethylpnthalate)

3.3 Moisture content. The moisture content of the first-fire composition shall be not more than 0.1 percent.

3.4 Burning rate. The burning rate of the first-fire composition shall be as follows:

	Type I	Type II	Type III
Burning rate inches/minute	7 \pm 1	5 \pm 1	5 \pm 1

3.5 Color of composition. The color of each shall be as follows:

	Type I	Type II	Type III
Color	yellow	red	green

3.6 First article inspection. This specification makes provisions for first article testing. Requirements for the submission of first article samples by the contractor shall be as specified in the contract.

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4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. 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 in the contract or order, the supplier 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. Reference shall be made to MIL-STD-109 to define terms used herein,

4.1.1 Submission of product. At the time each completed lot of items deliverable under the contract is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted:

a. A statement that the lot complies with all of the quality assurance provisions specified in this specification.

b. Specification number and date, together with identification and date of changes thereto.

c. Certificates of analysis on all materials used directly by the contractor when such materials are controlled by Government specifications shall be made available upon request by the Contracting Officer.

d. Quantity of product in the lot.

Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.2 First article inspection

4.2.1 Submission. Prior to the start of regular production the contractor shall submit a first article sample (see 6.2c) to a Government approved facility as designated by the contracting

officer for evaluation in accordance with the provisions of 4.2.2. The first article shall consist of eight(8) ounces of first-fire composition in accordance with instructions issued by the Contracting Officer. All samples submitted shall have been produced by the contractor using the same production processes, procedures, and equipment as will be used in fulfilling the contract. All materials, including packaging and packing, shall be obtained from the same sources of supply as will be used in regular production. The sample shall be accompanied by certificates of analysis. A first article quantity, or portion thereof, as directed by the Contracting officer, shall also be submitted whenever there is a lapse in production for a period in excess of 90 days, or whenever a change occurs in manufacturing process, material used, drawing, specification or source of supply as to significantly affect product uniformity as determined by the Government. Prior to submission, the contractor shall inspect the sample to the degree necessary to assure that it conforms to the requirements of the contract and submit a record of this inspection with the sample. A sample containing known defects will not be submitted unless specifically authorized by the Contracting Officer.

4.2.2 Inspections to be performed. The sample will be subjected by the Government to any or all of the examinations or tests specified in 4.4 of this specification.

4.2.3 Rejection. If any sample fails to comply with any of the applicable requirements, the first article quantity shall be rejected. The Government reserves the right to terminate its inspection upon any failure of a sample to comply with any of the stated requirements.

4.3 Inspection provisions

4.3.1 Lot formation, A lot shall consist of one or more batches of first fire composition produced by one manufacturer. in accordance with the same specification, or same specification revision, under one continuous set of operating conditions . Each lot shall consist of that quantity of first fire composition that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous. The product shall be submitted for inspection in accordance with MIL-STD-105 (or MIL-STD-1235 when applicable). The criteria and procedures for the assignment of lot numbers shall be in accordance with MIL-STD-1168 .

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4.3.2 Testing

4.3.2.1 Sampling. For the tests given in 4.4.1 thru 4.4.5 the containers in the lot shall be divided into two (2) equal size groups. For each of these groups an individual sample shall be prepared in such a manner that the sample obtained is truly representative of the contents of the containers sampled. Randomly select containers for sampling in accordance with MIL-STD-105. However, if each group contains less than five (5) containers, select all containers for sampling. Withdraw, approximately, equal portions of the powder from each selected container, in such quantity that the aggregate weight of the portions of the samples shall be approximately 200 grams. Use the method described in ASTM E300 for solids for withdrawing the samples. Combine these portions and thoroughly mix. Perform the tests given in 4.4.1 thru 4.4.5 on the two samples formed. If either sample fails to meet the specified requirements in Section 3, the lot shall be rejected. The classification and code numbers for the tests shall be as follows:

TABLE II

Test	Requirement	Test Method	Defect Classification	Code No.
Determination of Composition	3.2	4.4.1 thru 4.4.6	Major	01001
Moisture Content	3.3	4.5	Major	01002
Burning Rate	3.4	4.6	Major	03001
Color	3*5	4.7	Major	04001
Determination of raw materials	3.1	4.8	Major	05001

4.4 Test method and procedures. The tests in 4.4.1 thru 4.4.5 shall be performed using prescribed analytical

procedures for replicate determinations given in standard analytical textbooks.

4.4.1 Preparation of sample for determination of composition. The sample shall be prepared in accordance with method 102.1 in MIL-STD-1234 prior to the following determinations.

4.4.2 Determination of barium or strontium nitrate. The dried sample in the crucible (see 4.4.1) shall be extracted with ten 20 ml portions of distilled water, stirring each portion and allowing it to remain in contact with the residue for approximately 1 minute before applying suction. The sample after the tenth extraction shall be tested for complete extraction by treating a few drops of the extracting liquid on a white spot plate with a few drops of a 1 percent diphenylamine solution in concentrated sulfuric acid. If a blue coloration develops, additional washings shall be used and retest made. The crucible and contents shall be transferred to an oven at $100 \pm 5^{\circ}\text{C}$ for 1 hour, cooled to a room temperature in a desiccator and weighed. The crucible and contents shall be reserved for the tetranitrocarbazole determination (see 4.4.3). The percentage of barium or strontium nitrate in the sample shall be calculated as follows to determine compliance with the applicable requirement of 3.2.

$$\text{Percentage of barium or strontium nitrate} = \frac{100 (A-B)}{W}$$

Where:

- A = weight of the crucible and dried sample
- B = weight of crucible and contents after water extraction
- W = total weight of the original undried sample corrected for moisture only

4.4.3 Determination of tetranitrocarbazole. Tetranitrocarbazole shall be determined in accordance with the procedure in MIL-STD-1234 METHOD 420.1, using acetone as the solvent.

4.4.4 Determination of silicon. The residue retained from the tetranitrocarbazole determination shall be aspirated to remove any residual acetone and then the crucible and contents dried in an oven at $100 \pm 5^{\circ}\text{C}$ for 15 minutes. The crucible and contents shall be removed from the oven, cooled to room temperature, and the crucible (on its side)

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placed in a clean 250 ml beaker. Approximately 50 ml of concentrated sulfuric acid shall be added to the beaker containing the crucible. The beaker and contents shall be gently warmed on a steam bath and the crucible occasionally rotated until all of the zirconium hydride is in solution. This will be evidenced by a cessation of the evolution of hydrogen gas produced in the reaction. The beaker with crucible shall be transferred to a hot plate at high heat and concentrated nitric acid carefully added dropwise until all the organic material present is completely oxidized. This will be evidenced by a permanent clarity of the solution. The solution shall be allowed to fume until a volume of approximately 35 ml remains. The solution shall be cooled to room temperature and sufficient distilled water, to make a volume of approximately 100 ml, shall be added cautiously under a watch glass. The crucible shall be removed from the solution with the aid of forceps. Holding the crucible above the beaker, it shall be washed with 50 ml of distilled water so that the washings are quantitatively transferred to the beaker. The solution shall be stirred thoroughly with a glass rod and allowed to cool to room temperature. The crucible, thus washed, shall be placed on a Fisher Filtrator containing a 400 mL beaker. Suction shall be applied and the material retained in the 250 ml beaker quantitatively transferred to the original crucible. The 250 ml beaker and the crucible and contents shall be washed throughly with an additional 50 ml of water. into the 400 ml beaker. The filtrate shall be reserved for the zirconium hydride determination. The crucible and contents shall be placed in an oven at $100^{\circ} \pm 5^{\circ}\text{C}$ for 1 hour, cooled to room temperature in a desiccator and weighed. The percentage of silicon in the sample shall be calculated as follows,

$$\text{Percent silicon} = \frac{100B}{W}$$

Where :

B = gain in weight of tared crucible.

W = total weight of the original undried sample corrected for moisture only.

4.4.5 Determination of zirconium hydride. The filtrate retained from the silicon determination shall be cooled to

10 ± 2°C in an ice bath, The zirconium hydride shall be determined in accordance with method 416.1 of MIL-STD-1234.

4.4.6 Determination of laminac plus polyvinyl chloride. The percent of laminac plus polyvinyl chloride in the sample shall be calculated by subtracting from 100 the combined percentages of barium or strontium nitrate, silicon, tetranitrocarbazole and zirconium hydride.

4.5 Determination of moisture content. The moisture content shall be determined in accordance with the procedure specified in MIL-STD-1234, Method 101.3 using methanol as the special solvent.

4.6 Determination of burning rate. The first-fire composition shall be pressed, at 4000 psi, into a paper candle case not less than 1/2 inch in diameter and not less than 3 inches in length. The paper candle case shall be ignited by means of a piece of quickmatch, or other suitable means, and the rate determined by means of a stop watch.

4.7 Color of Composition. The color of the composition shall be determined by visual observation during the determination of the burning rate (see 4.6).

4.8 Determination of raw materials. The raw material shall be determined in accordance with the applicable specifications.

5. PREPARATION FOR DELIVERY

5.1 Packaging and packing not applicable to these mixtures.

6. NOTES

6.1 Intended use. Material purchased under this Specification is intended for use as an ingredient in pyrotechnic composition as follows:

a. Type I. Yellow first fire. composition is intended for use with yellow illuminant and signal composition.

b. Type II. Red first fire composition is intended for use with red illuminant and signal composition.

Type III. Green first fire composition is intended for use with green illuminant and signal composition.

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6.2 Ordering data. Procurement documents should specify the following:

- a. Title, number and date of this specification
- b. Type specified in the contract.
- c. Provisions for submission of first article samples.

d. Acceptance and description sheets - Acceptance and description sheets shall be prepared for each lot in accordance with MIL-STD-1171.

6.3 Inspection code numbers. The five digit code numbers assigned to the inspections herein are to facilitate future data collection and analysis by the Government.

6.4 Supersession Data. This specification includes the requirements of Purchase Description PA-PD-594, dated 15 September 1954;, with AMEND 2 dtd 6/8/73.

Custodian:
ARMY-MU

Preparing Activity:
ARMY-MU

project Number: 1370-A-912

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