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MILITARY SPECIFICATION

POWDER, IGNITION, GASLESS, A-1A

This specification is approved for use by the Naval Air Systems Command, Department of the Navy, and is available for use by all departments and agencies of the Department of Defense.

1. SCOPE

1.1 This specification covers the minimum requirements for the procurement of A-1A gasless ignition powder.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

Federal

PPP-B-601	Boxes, Wood, Cleated-Plywood
PPP-B-621	Boxes, Wood, Nailed and Lock-Corner
PPP-C-96	Cans, Metal, 28 Gage and Lighter

Military

MIL-Z-399	Zirconium (Granular and Powdered)
MIL-E-463	Ethyl Alcohol (For Ordnance Use)

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: (Commander, Naval Air Systems Command, Engineering Division, Standardization Section (Code: AIR-52021), Department of the Navy, Washington, DC 20361) by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

MIL-I-706	Iron Oxide, Ferric Red Dry (Natural and Synthetic)
MIL-V-23776	Vermiculite, Expanded
MIL-I-24391	Insulation Tape, Electrical, Plastic Pressure Sensitive
MIL-B-26701	Bottles, Screw Cap and Carboys, Polyethylene Plastic

STANDARDS

Military

MIL-STD-129	Marking for Shipment and Storage
MIL-STD-1234	Pyrotechnics; Sampling, Inspection and Testing

DRAWINGS

Naval Sea Systems Command (Code Ident 53711)

LD 175031	Squib Mk 1 Mods
LD 480100	Multiple Delay Holder Board
LD 496332	Test Fixture for Ignition Powder
LD 486345	Delay and Ignition Powder Test Element Loading Fixture for the XB-71A Test Apparatus
1672881	Spacer
1280936	Primer Holder

Naval Air Systems Command (Code Ident 30003)

4904144	Pyrotechnic Delay Timing Circuit

PUBLICATIONS

Naval Air Systems Command (Code Ident 30003)

AD 1014	Operating Procedures for Pyrotechnic Delay Timing Apparatus

(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 document forms 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 proposals shall apply.

Code of Federal Regulations

49 CFR 170-190 Transportation

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402. Orders for the above publication should cite "the latest edition and supplements thereto.")

3. REQUIREMENTS

3.1 Materials. The materials used in the manufacture of the gasless ignition powder shall conform to the following.

3.1.1 Zirconium powder. The zirconium powder shall meet the requirements of MIL-Z-399, type II, class 1.

3.1.2 Ferric oxide powder. The ferric oxide powder shall meet the requirements of MIL-I-706, type I, class 2, with the following additions.

3.1.2.1 Total iron. Total iron shall be 69.7 ± 0.5 percent when determined in accordance with 4.6.1.1.

3.1.2.2 Average particle size. Average particle size shall be less than 1 micron when determined in accordance with 4.6.1.2.

3.1.2.3 Total oxygen content. Total oxygen content shall be 29.8 percent minimum when determined in accordance with 4.6.1.3.

3.1.3 Diatomaceous earth. The diatomaceous earth shall meet the following requirements.

3.1.3.1 Weight loss on ignition. Weight loss on ignition shall be a maximum of 0.4 percent when determined in accordance with 4.6.2.1.

3.1.3.2 Silica content. Silica content shall be a minimum of 90.0 percent when determined in accordance with 4.6.2.2.

3.1.3.3 Granulation. Granulation shall be a minimum of 99.0 percent and shall pass through a U.S. Standard Sieve 325 when tested in accordance with 4.6.2.3.

3.1.4 Ethyl alcohol. The ethyl alcohol shall meet the requirements of MIL-E-463.

3.2 Composition. The percentage of composition by weight of the A-1A powder shall be in accordance with table I. Acceptance of the powder under the tests of 4.6.4 and 4.6.5 shall be considered satisfactory compliance with the composition formulation.

3.2.1 Formulation procedure. The ingredients in the proportions by weight given in table I shall be mixed in ethyl alcohol (100 milliliters per 100 grams dry mix). The mixture shall be subdivided into approximately 250-gram increments and packaged. An advisory manufacturing procedure which has been found to produce satisfactory material is given in 6.4.

TABLE I. Composition of A-1A gasless ignition powder.

Ingredient	Composition (% by weight)
Zirconium powder	65.0 ± 1.0
Ferric oxide	25.0 ± 1.0
Diatomaceous earth	10.0 ± 1.0

3.3 Moisture content. The moisture content of the gasless ignition powder after drying and screening shall not exceed 0.20 percent by weight when determined in accordance with 4.6.3.

3.4 Heat output. The heat evolved by the gasless ignition powder when burned under an argon atmosphere of 25 atmospheres initial pressure shall be a minimum of 450 calories/gram when determined in accordance with 4.6.4.

3.5 Low-temperature functioning. All test elements shall function at -54°C when tested in accordance with 4.6.5, and the average weight loss of the powder in the test elements shall not exceed 30 percent of the original weight of the powder.

3.6 Workmanship. The mixing of the ingredients in each batch and the blending of the batches of ignition powders shall be of such quality as to produce a homogeneous lot free from lumps and foreign material.

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 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 Sampling.

4.2.1 Lot. A lot of gasless ignition powder shall be limited to a batch produced in one mixing operation. Maximum lot size shall be 50 pounds.

4.2.2 Sample. Individual samples shall be selected from 10 percent of the bottles after packaging or a minimum of two samples, whichever is greater. Heat output tests shall be performed on each sample. All other tests shall be conducted on a composite of these samples. Samples shall be identified in accordance with 5.3.1.

4.3 Test equipment and materials. In addition to standard laboratory equipment, the following special equipment will be required to perform the acceptance tests set forth in this specification.

(a) Adiabatic calorimeter - an automatic calorimeter with thermometers, motor stirrer drive, water cans and bomb (see 6.3)

(b) Standard sample for standardizing oxygen bomb calorimeter - benzoic acid, standard grade, prepared by National Bureau of Standards, Washington D.C. Benzoic acid pellets are recommended by Parr for the standardization of the calorimeter, as they minimize incomplete combustion. Powdered benzoic acid is not recommended. Instructions for standardizing the calorimeter are given in the Parr Instrument Co. Manual #130, Oxygen Bomb Calorimetry and Combustion Methods, pp 22-23.

(c) Ignition powder test element loading fixture - LD 496345

(d) Test fixture - LD 496332

(e) Primer squib Mk 1 Mods - LD 175031

(f) Multiple delay holder board - LD 480100

4.4 Test conditions. Unless otherwise specified, tests shall be conducted at a temperature of 25° ± 5°C.

4.5 Acceptance tests. Unless otherwise specified, the contractor shall conduct acceptance tests in the presence of the Government inspector to assure that the gasless ignition powder is in compliance with the requirements of 3.1, 3.3, 3.4, 3.5, and 3.6.

4.6 Test methods.

4.6.1 Ferric oxide.

4.6.1.1 Total iron determination. Take an approximate 2-gram sample from each container of the ferric oxide powder to be used in making the gasless ignition powder and dry at 110°C until successive weighings at 30-minute intervals agree to within ± 0.002 gram. Weigh 0.2 gram ± 0.002 gram of dried sample and place in a 250-milliliter beaker. Add 10 milliliters of 1:1 hydrochloric acid by volume. Cover the beaker with a watch glass and warm gently until all the sample is dissolved. Dilute with distilled water to about 50 milliliters. Pass the solution through a Jones reductor (or equivalent). The effluent from reductor is best collected under nitrogen. Wash the solution through the reductor with several 10-milliliter portions of water. Titrate to the appearance of a permanent blue-purple color. Record the number of milliliters of potassium dichromate solution required. Calculate the percentage of iron as follows:

$$\text{Percent Fe} = \frac{A \times N \times 0.05585}{SW} \times 100$$

where:

A= milliliters of potassium dichromate solution required.
N= normality of potassium dichromate solution
SW= grams of sample weight.

4.6.1.1.1 Indicator solution. Weigh 0.095 gram of barium diphenylamine sulfonate and add to 100 milliliters of water. Stir until solution is complete. Add 10 milliliters of dilute sulfuric acid (1:1). Let stand several hours and filter. Add 500 milliliters of 85 percent phosphoric acid and dilute to 1 liter.

4.6.1.2 Average particle size. The laboratory test for determining the average particle size of the ferric oxide powder shall be made in accordance with MIL-STD-1234, method 202-1.

4.6.1.3 Total oxygen determination. Take an approximate 2-gram representative sample from each container of the iron oxide powder to be used in making the gasless ignition powder. Dry the sample at 100°C until successive weighings at 30 minute intervals agree to within ± 0.002 gram. Weigh approximately 0.25 gram with an accuracy of ± 0.2 milligram of the dried iron oxide (Fe_2O_3) powder into a combustion train through which dry, oxygen-free hydrogen can be passed. Heat the tube by placing it in an electrically heated combustion furnace, gradually bringing the temperature to a dull red heat. The water evolved from the reduction of the Fe_2O_3 is collected in a tared tube containing a magnesium perchlorate water absorbent. After all moisture is swept into the absorbent, weigh the tube to obtain the weight of water absorbed. Calculate the percent oxygen as follows:

$$\text{Percent } O_2 = \frac{\text{Weight of water absorbed}}{\text{sample weight}} \times 88.88$$

Acceptable iron oxide powder shall contain a minimum of 29.8 percent total oxygen. (NOTE: Exact weight of tube and $Mg(ClO_4)_2$ must be recorded prior to experiment.)

4.6.2 Diatomaceous earth.

4.6.2.1 Weight loss on ignition. Take an approximate 2-gram sample from each container of diatomaceous earth to be used in making the gasless ignition powder. Dry the sample at 110°C until successive weighings at 30-minute intervals agree to within ± 0.002 gram. Weigh 0.15 gram ± 0.2 milligram of dry sample into a tared 30-milliter platinum crucible and cover with a platinum lid. Heat carefully over a Meker burner (or equivalent) to about 1100°C for 10 minutes. Cool the crucible in a desiccator and weigh. Calculate the percentage of weight loss based on the sample weight as the percentage of weight loss on ignition. Acceptable diatomaceous earth shall show a weight loss of 4.0 percent or less.

4.6.2.2 Silica content determination. Moisten the ignited material in the crucible with a few drops of water. Add 10 drops of 1:1 sulfuric acid. Fill the crucible about one-half full of 48 percent to 51 percent hydrofluoric acid and place on a sand bed on a hot plate in a hood. Heat until copious fumes of sulfur trioxide are evolved. Continue heating at higher heat until the residue appears dry. Then heat carefully over a flame until no further fumes are evolved. Heat the crucible to 1100°C for 10 minutes. Cool and weigh the crucible. Calculate the difference in determination as the percentage of silica. Acceptable test sample shall show a weight loss of 90.0 percent or more.

4.6.2.3 Granulation. Take a 100-gram sample from each container of diatomaceous earth to be used in making the gasless ignition powder. Transfer the sample to a U.S. Standard Sieve 325. Wash with a spray of water until no more material passes through. Recover the material retained on the screen by washing it into a beaker and filtering it through a tared filtering crucible of fine porosity. Wash the collected material several times with alcohol and dry at 100°C. Weigh the material and calculate the percentage passed through the sieve. Acceptable diatomaceous earth shall pass 99.0 percent or more through the U.S. Standard No. 325 Sieve.

4.6.3 Moisture content of gasless ignition powder. Transfer approximately 2 grams of gasless ignition powder from the representative sample of 4.2.2 to a tared aluminum weighing dish fitted with an aluminum lid. Weigh to the nearest 0.1 milligram and calculate weight of sample. Transfer weighing dish and sample to a vacuum drying oven set at 71° ± 2°C. Remove lid and dry sample under vacuum (28 inches Hg) until constant weight is obtained. Cool and weigh after drying. Calculate the weight loss as percentage of moisture in sample. Weighing shall be accurate to ± 0.2 milligram.

4.6.4 Heat output.

4.6.4.1 Apparatus. The test apparatus shall consist of a semimicro calorimeter complete with thermometer (19° to 25°C), spy glass, stirrer motor drive, ignition unit, and an adapter for pressurizing gas. (See 6.3)

4.6.4.2 Equipment and accessories. In addition to the apparatus described above, the following shall be required:

(a) Argon gas supply in pressure cylinder
(b) Standard pellets for standardizing calorimeter.

4.6.4.3 Procedure. The procedure for determining the calorific value of the gasless ignition powder shall be as described in the manufacturer's manual of instructions for operation of the calorimeter (see 6.3). Failure of a sample of the powder (4.2.2) to have a minimum heat output of 450 calories/gram shall be cause for rejection of the bottle from which the sample was taken. If a sample fails the test, each bottle in the lot shall be sampled and tested. Each bottle that fails the test shall be removed from the lot.

4.6.5 Low-temperature functioning test. The low-temperature functioning test is conducted on a sample from each lot. The specimens for test shall consist of 20 test elements loaded with the powder from the sample as specified in 4.2.2.

4.6.5.1 Loading of test elements. The total weight of the test elements shall be recorded before and after loading. Each test element is loaded with the use of funnel holder, cap, and spacer (LD 496345). The ignition powder is pressed into each element at 30,000 ± 500 psi in approximately three equal increments. The last increment shall be pressed with an excess of powder so that the igniter column protrudes slightly above the pressed end. The excess powder is then smoothed off with a soft cloth until the igniter column is flush with the end of the test element.

4.6.5.2 Assembly of test fixture. Insert each loaded test element into a holder with the baffles and spacers as shown in the assembly drawing (LD 496332). The baffle discs shall be mounted in the order shown in the assembly drawing to insure reproducible ignition of the powder surface. The remainder of the test fixture is assembled with the spacer (Drawing 1672881) and the primer holder (Drawing 1280936) which has been fitted with a Squib Mk 1 Mods (LD 175031). The 20 assembled test fixtures are mounted on a multiple delay holder board (LD 480100). The holder board is attached to a firing circuit (Drawing 4904144) with a cable. The attachment of the cable is arranged to enable each of the 20 test elements to be fired consecutively. Each test fixture shall be attached to the holder board so that the blowout from each test fixture is directed away from the others during firing. Detailed operation directions are described in AD 1014 with the exceptions noted in 6.5.

4.6.5.3 Test procedure. Place the test element in a cold box or low-temperature environment and condition for a minimum of 2 hours at -54° ± 1°C before test firing. Test firing shall be in accordance with procedure described in AD 1014 with the exceptions noted in 6.5.

4.6.5.4 Test element disassembly. After firing, each of the test fixtures shall be carefully disassembled and the test element examined for complete burning. Care shall be taken to insure that the slag lost during removal of the test fixture from the test board and during disassembly of the test fixture is recovered and included in the weight of the test element after firing.

4.6.5.5 Weight loss calculations. Weigh the 20 test elements after test firing for total weight. Subtract this total weight from original total weight of loaded test elements. The difference in weight indicates a loss of weight of the powder as a result of burning. The percentage weight loss of the powder as a result of burning is calculated as follows:

$$\text{Percent loss in weight} = \frac{\text{loss in weight of powder}}{\text{original weight of powder}} \times 100$$

5. PACKAGING

5.1 Preservation and packaging.

5.1.1 Level A. In addition to the following, Level A packaging shall be in accordance with 49 CFR 170-190.

5.1.1.1 Unit packaging. A mixture of approximately 250 grams maximum of gasless ignition powder and a minimum of 25 percent alcohol conforming to MIL-E-463 shall be packaged in a polyethylene bottle conforming to the requirements of MIL-B-26701. Inside diameter at the screw cap end of the bottle shall be a minimum of 1.0 inch. The bottle shall be sealed with a commercial type rubber stopper. The stopper shall be secured in place with not less than three turns of electrical tape conforming to MIL-I-24391 around the stopper and not less than two turns of tape around the length of the bottle.

5.1.1.2 Intermediate packaging. Unit containers shall be intermediately packaged in a container conforming to the requirements of PPP-C-96, type V - class 2, or type IV, with slip cover provided with leak-proof seal and locking tabs. The cover shall be held in place by soldering or crimping in at least four points. Size I, II, or IV expanded vermiculite material conforming to the requirements of MIL-V-23776 shall be used in the intermediate container to provide insulation shielding and to insure a tight pack.

5.2 Packing. Packing shall be level A or B as specified in the contract or purchase order.

5.2.1 Level A. In addition to the following, Level A packing shall be in accordance with 49 CFR 170-190.

5.2.1.1 Exterior containers. Intermediate containers shall be packed in an overseas-type, wood-cleated plywood box conforming to PPP-B-601 or in a class 2, style 2, 2½, 3, or 4, nailed wood box conforming to PPP-B-621. Vermiculite material, as specified in 5.1.1.2, shall be used within the selected shipping box to separate and provide insulation shielding of the intermediate containers and to insure a tight pack. The gross weight of a given shipping container shall not exceed 100 pounds.

5.2.2 Level B. In addition to the following, Level B packing shall be in accordance with 49 CFR 170-190.

5.2.2.1 Exterior containers. Intermediate containers shall be packed in a domestic-type, wood-cleated plywood box conforming to PPP-B-601 or in a class 1, style 2, 2½, 3, or 4, nailed wood box conforming to PPP-B-621. Vermiculite material, as specified in 5.1.1.2, shall be used within the selected shipping box to separate and provide insulation shielding of the intermediate containers and to insure a tight pack. The gross weight of a given shipping container shall not exceed 100 pounds.

5.3 Marking.

5.3.1 Identification. Each lot shall be identified by:

(a) Lot number
(b) Manufacturer
(c) Date of manufacture.

5.3.2 Special markings. Special marking requirements shall be in accordance with 49 CFR 170-190.

5.3.3 Normal markings. In addition to any special markings required by the contract or order, all markings shall be in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. The gasless ignition powder is used to transfer ignition from primers to gasless delays and from delays to detonators or propelling charges. The A-1A composition is an improved gasless ignition powder with reliable low-temperature functioning characteristics.

6.2 Ordering data. Procurement documents should specify the following:

(a) Title, number, and date of this specification
(b) Level of packing required (see 5.2)
(c) That the safety precaution requirements of the "Contractors' Safety Manual for Ammunition, Explosives, and Related Dangerous Material," DOD 4145.26M are applicable. NOTE: When this specification is used as part of the description of work to be accomplished by a Government activity, the safety precaution requirements of "Ammunition Ashore," OP 5, are applicable.

6.3 Test apparatus. An Adiabatic Calorimeter, Serial No. 1411, complete with thermometers, motor stirrer drive, and water cans, and oxygen bomb (Serial No. 1108), fabricated by the Parr Instrument Company, Inc., 211 53rd Street, Moline, Illinois 61625, has been found acceptable for this test. The test apparatus and method of operation is described in Manual No. 142, Instructions for the No. 1241 Combustion Calorimeter, which is furnished with the calorimeter.

6.4 Advisory manufacturing procedures.

6.4.1 Processing of zirconium. The zirconium powder is stored and shipped in water. Slurry zirconium in container by rolling on jar rolling mill until all zirconium particles are in suspension. Filter the zirconium slurry using a filter flask, Buchner funnel, and No. 2 Whatman filter paper or equivalent. Rinse filter cake twice with alcohol. When filter cake appears dry transfer to tared aluminum container. Immediately take random samples from the cake to make 50 grams. Dry the composite sample in a suitable oven at a temperature not to exceed 60°C (140°F) until two successive weighings agree within 0.2 percent. Calculate percent of zirconium from dry weight divided by wet weight of composite sample. Immediately weigh damp filter cake. Transfer weighed filter cake to container and slurry with alcohol on a jar roller mill. Calculate dry weight of zirconium which includes total of dry sample weights, and dry filter cake weights calculated from percent zirconium and corresponding damp filter cake weight. Other ingredient weights are calculated on the basis of the calculated dry weight of zirconium.

CAUTION: The operator should wear face shield, fire resistant clothing, asbestos gloves, safety glasses, and safety shoes. Operations such as weighing, filtering, and transferring the dry powder should be done behind a shield. Spatulas and containers should be nonsparking material and grounded.

6.4.2 Drying and Storage of ferric oxide and diatomaceous earth. The ferric oxide powder and diatomaceous earth shall be dried separately at 190° ± 5°F (85° to 90°C) in a suitable oven until two successive weighings at 1 hour intervals agree to within 0.5 percent. The dried ferric oxide powder and the diatomaceous earth shall be in a tighly closed container or in a suitable oven until ready for use.

6.4.3 Mixing of gasless ignition powder. The mixing procedure is based on a single batch of ignition powder. The operator must exercise care to maintain safety and reproducibility of mixing conditions to provide a homogeneous mixture of the desired composition of ignition powder. The mixer should be located in an isolated and barricaded area. Weighing and mixing should be performed in a shielded area.

(1) Add ethyl alcohol to a clean mixing bowl which is adapted to the mixer.
(2) Weigh diatomaceous earth and ferric oxide and add to the bowl.
(3) Lower mixing blade (fluid shear type) into bowl and allow to run.
(4) Stop mixer periodically and, using ethyl alcohol, wash accumulated material back into mixing area.
(5) Add zirconium slurry and repeat steps 3 and 4.
(6) Transfer mix into polyethylene bottles (a maximum of 250 grams per bottle). Seal bottles in accordance with 5.1.1.2.

6.4.4 Drying of ignition powder.

(1) Pour material to be dried into a metal drying tray.
(2) Dry at 60°C for 16 hours minimum.
(3) After drying, cool the powder for a minimum of 20 minutes.
(4) Transfer the dried and cooled ignition powder to a 30-mesh sieve with porcelain balls or rubber stoppers.
(5) Screen the powder on a Ro-Tap or other shaking apparatus operated by remote control and located in an isolated and barricaded area.
(6) When screened, remove the sieve pan behind a shield.
(7) Transfer the screened ignition powder to conductive rubber containers. The maximum quantity placed in each container shall be limited to 50 grams.

6.4.5 Storage of mixed ignition powder. When ignition powder manufactured and stored is over 200 grams, the powder shall be stored wet in well-stoppered polyethylene containers. Minimum weight of ethyl alcohol (conforming to MIL-E-463) added shall be 25 percent of weight of ignition powder.

6.5 Modifications to test procedures. Operating procedures for use of pyrotechnic delay timing apparatus in testing the ignition powder shall be as specified in AD 1014 with the following exceptions to 3.0 of that document.

(1) Load ignition element holder and primer holder in accordance with LD 496345.
(2) Screw primer holders and ignition element holders onto spacers.

6.6 Changes from previous issue. Asterisks are not used in this revision to identify changes with respect to the previous issue, due to the extensiveness of the changes.

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