

MIL-L-376C
 12 January 1983

 SUPERSEDING
 MIL-L-376B
 19 February 1968

MILITARY SPECIFICATION

LEAD DIOXIDE, 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 technical grade lead dioxide.

1.2 Classification. Lead dioxide shall be of the following types and
 classes as specified (see 6.2):

Type I - Low alkalinity
 Type II - High alkalinity
 Class 1 - Subsieve
 Class 2 - 100 mesh, nominal

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.

STANDARDS

MILITARY

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

: 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. :

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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 cite "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.

UNIFORM FREIGHT CLASSIFICATION RULES

(Application for copies should be addressed to the Uniform Classification Committee, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606.)

NATIONAL MOTOR FREIGHT CLASSIFICATION RULES

(Application for copies should be addressed to the American Trucking Associations, Inc., Traffic Department, 1616 P Street, NW, Washington, DC 20036.)

ASTM STANDARDS

- B214 - Sieve Analysis of Granular Metal Powders
- B293 - Subsieve Analysis of Granular Metal Powders by Air Classification
- B329 - Apparent Density of Powders of Refractory Metals and Compounds by the Scott Volumeter
- 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.)

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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. The lead dioxide shall conform to the applicable chemical characteristics of table I when tested as specified therein.

TABLE I. Chemical characteristics

Characteristic	Percent by weight		Test
	Type I	Type II	Method
Lead dioxide, min	95.0	95.0	4.2.4.1
Water-soluble salts, max	0.05	0.05	4.2.4.2
Acidity	None	None	4.2.4.3
Alkalinity as Na_2CO_3 , max	0.01	0.10	4.2.4.4
Ammonium salts	None	None	4.2.4.5

3.2 Particle size distribution. The lead dioxide shall conform to the applicable particle size distribution characteristics of table II when tested as specified in 4.2.4.6.

TABLE II. Particle size distribution

Characteristic	Percent by weight	
	Class 1	Class 2
Retained on a No. 100 sieve, max	---	0.1
Retained on a No. 140 sieve, max	---	10.0
Retained on a No. 200 sieve, max	0.0	---
Retained on a No. 325 sieve, max	5.0	---
Larger than 20 micrometers, max	20.0	---
Smaller than 5 micrometers, max	15.0	---

3.3 Apparent density (class 1 only). Class 1 material shall have a minimum apparent density of 1.20 grams (g) per cubic centimeter when tested as specified in 4.2.4.7.

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

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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 lead dioxide of one type and class, 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 (see 6.5). Sampling shall be conducted in accordance with table III. A representative specimen of approximately 400 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.

TABLE III. Sampling for test

<u>: Number of containers in batch or lot : Number of sample containers :</u>			
:	:	:	:
:	3 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 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 2.5 percent defective:

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

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4.2.3.2 For test. 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.

4.2.4 Tests (see 6.5). 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 Lead dioxide. Dry a portion of the specimen over sulfuric acid for 4 hours. Transfer approximately 0.5 g weighed to the nearest 0.1 milligram (mg) to a beaker and add 15 milliliters (mL) of 1 to 1 nitric acid. Add by pipet, 20 mL of 1 percent hydrogen peroxide. Stir until solution is complete, adding, if necessary, 100 mL of warm water to hasten solution. Titrate the excess hydrogen peroxide with 0.1N potassium permanganate. Run a blank. Calculate the percent by weight lead dioxide as follows:

$$\text{Percent by weight lead dioxide} = \frac{11.96 N (B - A)}{W}$$

where: N = Normality of potassium permanganate solution,
 B = Milliliters of potassium permanganate solution required for titration of the blank,
 A = Milliliters of potassium permanganate solution required for titration of the specimen, and
 W = Weight of specimen used in grams.

4.2.4.2 Water-soluble salts. Weigh approximately 3 g of the specimen to the nearest milligram, transfer to a beaker, add 75 mL of hot water, and boil for 5 minutes. Cool, dilute to 75 mL with water and filter, discarding the first few milliliters. Transfer 50 mL of the clear filtrate to a weighed platinum dish. Evaporate to dryness at 105° to 110°C, cool in a desiccator, and weigh. Calculate the percent by weight water-soluble salts as follows:

$$\text{Percent by weight water-soluble salts} = \frac{150A}{W}$$

where: A = Weight of residue in grams and
 W = Weight of specimen used in grams.

4.2.4.3 Acidity. Weigh approximately 10 g of the specimen and transfer to a 400-mL beaker. Add 200 mL of carbon dioxide-free water and boil for 15 minutes. Filter the mixture and wash with a small quantity of the neutral water. Test for acidity by immersing sensitive blue litmus paper in the filtrate for 3 minutes. Save the filtrate for the determination of alkalinity in 4.2.4.4.

4.2.4.4 Alkalinity. Add 3 drops of methyl orange indicator to the filtrate from 4.2.4.3 and titrate from a microburet containing 0.01N hydrochloric acid to a pink end point. Run a blank. Calculate alkalinity as follows:

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$$\text{Alkalinity as Na}_2\text{CO}_3 \text{ percent by weight} = \frac{5.3 N (A - B)}{W}$$

where: A = Milliliters of hydrochloric acid required for titration of the specimen,
 B = Milliliters of hydrochloric acid required for titration of the blank,
 N = Normality of the hydrochloric acid, and
 W = Weight of specimen used in grams.

4.2.4.5 Ammonium salts. Transfer 5 g of the specimen to a flask and add sufficient 20-percent sodium hydroxide solution to cover. Moisten a strip of red litmus paper with water and suspend it in the neck of the flask. Boil the solution gently for about 1 minute, taking care that no spray from the solution touches the paper. Note the color of the litmus paper. The appearance of a blue color indicates the presence of ammonium salts.

4.2.4.6 Particle size distribution. Determine the particle size distribution of class 1 material in accordance with ASTM B293 and that of class 2 material in accordance with ASTM B214.

4.2.4.7 Apparent density. Determine the apparent density of class 1 material in accordance with ASTM B329.

5. PACKAGING

5.1 Unit packing, industrial. A specified quantity of lead dioxide (see 6.2) shall be unit packed in accordance with Department of Transportation (DOT) regulations and in a manner to assure maintenance of specified purity and integrity of the container from supply source to destination and for a minimum storage period of 6 months.

5.2 Packing, industrial. Lead dioxide, unit packed as specified in 5.1, shall be packed in accordance with DOT regulations and in a manner to assure carrier acceptance and safe delivery to the first destination. Containers shall be in compliance with Uniform Freight Classification Rules, National Motor Freight Classification Rules and the rules of any other carrier applicable to the mode of transportation.

5.3 Unitization. Like quantities of packs of lead dioxide shall be unitized in a manner to assure carrier acceptance, protection of the contents, and integrity of the pack from supply source to first receiving activity, and to assure stability in stacking.

5.4 Marking. Containers and unitized loads shall be marked in accordance with DOT and any other applicable regulations. Each container shall be marked to show the batch or lot number and date of manufacture of the lead dioxide and the following precautionary information:

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HAZARDS:

STRONG OXIDIZER
TOXIC BY INHALATION AND INGESTION
DANGER! STRONG OXIDIZER
CONTACT WITH OTHER MATERIAL MAY CAUSE FIRE
WARNING! HARMFUL IF SWALLOWED OR INHALED
KEEP FROM CONTACT WITH CLOTHING AND OTHER
COMBUSTIBLE MATERIALS
STORE IN TIGHTLY CLOSED CONTAINER
REMOVE AND WASH CONTAMINATED CLOTHING PROMPTLY
KEEP CONTAINER CLOSED
USE ONLY WITH ADEQUATE VENTILATION
WASH THOROUGHLY AFTER HANDLING
KEEP AWAY FROM FOOD PRODUCTS

FIRST AID: If swallowed and victim is conscious, have victim drink water or milk and have victim induce vomiting by sticking finger down throat. If swallowed and victim is unconscious or having convulsions, do nothing except keep victim warm. Call for immediate medical attention. If in eyes, hold eyelids open and flush with plenty of water. If inhaled, remove to fresh air. If not breathing, give artificial respiration, preferably mouth-to-mouth. If breathing is difficult, give oxygen.

6. NOTES

6.1 Intended use. Lead dioxide is intended for use in pyrotechnic and primer formulations.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification
- (b) Type and class of material required (see 1.2)
- (c) Unit quantity 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 some 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.

6.5 Sampling and testing precautions. This specification covers inspection of chemical material which is potentially hazardous to personnel. Lead dioxide is a strong oxidizer, toxic by inhalation and ingestion. All applicable safety rules, regulations and procedures must be followed in the handling and processing of this material.

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Custodians:

Army - EA

Navy - AS

Review activities:

Army - MD

Navy - OS

DLA - GS

User activity:

Army - AR

Preparing activity:

Army - EA

Project No. 6810-B347

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL*(See Instructions - Reverse Side)***1. DOCUMENT NUMBER****2. DOCUMENT TITLE****3a. NAME OF SUBMITTING ORGANIZATION****4. TYPE OF ORGANIZATION (Mark one)**☐ **VENDOR**☐ **USER**☐ **MANUFACTURER**☐ **OTHER (Specify):** _____**b. ADDRESS (Street, City, State, ZIP Code)****5. PROBLEM AREAS****a. Paragraph Number and Wording:****b. Recommended Wording:****c. Reason/Rationale for Recommendation:****6. REMARKS****7a. NAME OF SUBMITTER (Last, First, MI) - Optional****b. WORK TELEPHONE NUMBER (Include Area Code) - Optional****c. MAILING ADDRESS (Street, City, State, ZIP Code) - Optional****8. DATE OF SUBMISSION (YYMMDD)**

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