

MIL-L-20549B
12 January 1983
SUPERSEDING
MIL-L-20549A
15 January 1968

MILITARY SPECIFICATION

LEAD NITRATE, 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 one technical grade of lead nitrate.

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

(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.)

2.1.2 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.

: Beneficial comments (recommendations, additions, deletions) and any pertinent 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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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.")

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 Appearance. Lead nitrate shall be white in color when tested as specified in 4.2.4.1.

3.2 Chemical characteristics. Lead nitrate shall conform to the chemical characteristics of table I when tested as specified therein.

TABLE I. Chemical characteristics

Characteristic	Percent by weight		Test paragraph
	Minimum	Maximum	
Assay (as $Pb(NO_3)_2$)	98.0	---	4.2.4.2
Water content	---	1.25	4.2.4.3
Water-insoluble matter	---	0.20	4.2.4.4
Acidity (as HNO_3)	---	0.50	4.2.4.5
Copper	---	0.002	4.2.4.6

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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.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the lead nitrate 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 II. A representative specimen of approximately 200 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.

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 damaged or leaking
- (c) Unitization not as specified
- (d) Marking incorrect, missing, or illegible

TABLE II. Sampling for test

: Number of containers in batch or lot :	Number of sample containers :
: 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

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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 Appearance. Visually examine a portion of the specimen for color.

4.2.4.2 Assay. Weigh to the nearest milligram (mg) approximately 1 g of the specimen into a 250-milliliter (mL) beaker and dissolve in 100 ml of water. Add drop by drop, with stirring, 10 ml of saturated sodium sulfate solution containing 5 percent sulfuric acid and allow to stand for at least 2 hours. Filter through a tared filter crucible and wash with 1-percent alcohol. Heat the crucible and precipitate in a muffle furnace at $200^{\circ} + 10^{\circ}\text{C}$ to constant weight, cool in a desiccator, and weigh. Calculate the percent by weight lead nitrate as follows:

$$\text{Percent by weight lead nitrate} = \frac{109.2 (A - B)}{W}$$

where: A = Weight of crucible and precipitate in grams,
 B = Weight of crucible in grams, and
 W = Weight of specimen in grams.

4.2.4.3 Water content. Weigh to the nearest milligram approximately 10 g of the specimen into a tared weighing bottle. Heat the bottle and contents at $125^{\circ} + 2^{\circ}\text{C}$ for 5 hours, cool in a desiccator, and weigh. Calculate the percent by weight water content as follows:

$$\text{Percent by weight water content} = \frac{100 (A - B)}{W}$$

where: A = Weight of bottle and specimen before heating in grams,
 B = Weight of bottle and specimen after heating in grams, and
 W = Weight of specimen in grams.

4.2.4.4 Water-insoluble matter. Weigh to the nearest milligram approximately 25 g of the specimen into a 500-mL beaker. Add 250 mL of water and heat until the specimen is completely dissolved. Filter the hot solution through a tared filter crucible and wash thoroughly with hot water. Dry the crucible and residue at $110^{\circ}\text{C} + 2^{\circ}\text{C}$ for 2 hours, cool in a desiccator, and weigh. Calculate the percent by weight water-insoluble matter as follows:

$$\text{Percent by weight water-insoluble matter} = \frac{100 (A - B)}{W}$$

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

4.2.4.5 Acidity. Weigh to the nearest milligram approximately 25 g of the specimen and dissolve in 500-mL of water. Add a few drops of methyl red indicator and titrate with approximately 0.005N sodium hydroxide solution which has been standardized using methyl red indicator. Calculate the percent acidity as nitric acid as follows:

$$\text{Percent by weight acidity} = \frac{6.302 A B}{W}$$

where: A = Milliliters of sodium hydroxide solution used,
 B = Normality of sodium hydroxide solution, and
 W = Weight of specimen in grams.

4.2.4.6 Copper. Weigh 1.000 g of the specimen and dissolve in 15 mL of water. Add 5 mL of 10-percent ammonium acetate solution, 0.5 mL of ammonium thiocyanate solution (300 g of ammonium thiocyanate per liter), 2 mL of acetic acid, 0.5 mL of pyridine, and 10 mL of chloroform. Shake vigorously and allow to separate. Any greenish yellow color produced in the chloroform (lower) layer shall be no darker than that produced by 0.02 mg of copper ion (Cu) in an equal volume of solution containing the quantities of reagents used in the specimen test.

5. PACKAGING

5.1 Unit packing, level C. A specified quantity of lead nitrate (see 6.2) shall be unit packed to provide adequate protection against contamination, deterioration, and damage and to insure carrier acceptance and safe delivery to the first domestic destination. Containers shall be in compliance with Department of Transportation (DOT) regulations or the regulations of other common carriers applicable to the mode of transportation.

5.2 Packing, level C. Unit packs of 5.1 shall normally require no further protection for shipment, but if their consolidation is required, the shipping containers shall meet the same requirements as specified in 5.1.

5.3 Unitization. Identical quantities per non-combustible pallet, of identical unit packs, of lead nitrate shall be unitized in a manner to assure acceptance by common carrier and protection of contents and packing from supply source to first destination and for a minimum storage period of six months. Provision for stacking and stable supporting of superimposed loads shall be given prime consideration.

5.4 Marking. Containers and pallet loads shall be marked in accordance with DOT regulations and shall show the lot or batch number and date of manufacture of the lead nitrate. In addition, each container shall be durably and legibly marked with the following precautionary marking:

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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

Do not store near combustible materials. Store in a tightly closed container.

Remove and wash contaminated clothing promptly.

Avoid breathing dust.

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 nitrate is intended for use in the manufacture of lead styphnate and lead azide.

6.2 Ordering data. Acquisition documents should specify the title, number, and date of this specification, and the unit quantity per shipping container required.

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 nitrate is a strong oxidizer, toxic by ingestion and inhalation. All applicable safety rules, regulations, and procedures must be followed in the sampling and testing of this material.

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Custodians:

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Project No. 6810-B348

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