

MIL-M-3281A  
 6 December 1982  
 SUPERSEDING  
 MIL-A-3281  
 25 August 1950

MILITARY SPECIFICATION  
 MANGANESE 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 two types of technical grade manganese dioxide.

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

- Type I - For use in pyrotechnics
- Type II - For use in smoke mixtures

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

RR-S-366 - Sieve Test

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: 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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FSC 6810

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## STANDARDS

## MILITARY

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
- MIL-STD-1168 - Ammunition Lot Numbering
- MIL-STD-1188 - Commercial Packaging of Supplies and Equipment

(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
- D3037 - Carbon Black - Surface Area By Nitrogen Adsorption
- E248 - Manganese In Manganese Ore By The Pyrophosphate (Potentiometric) Method

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

3.2 Physical characteristics.

3.2.1 Particle size distribution. Manganese dioxide shall conform to the applicable particle size distribution of table II when tested as specified in 4.2.4.4.

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TABLE I. Chemical characteristics

Characteristic	Percent by weight		Test paragraph
	Type I	Type II	
Moisture content, max	2.5	1.0	4.2.4.1
Manganese content, min	55.0	50.0	4.2.4.2
Available oxygen, min	15.6	14.5	4.2.4.3

TABLE II. Particle size distribution

Sieve No.	Percent by weight passing			
	Type I		Type II	
	Min	Max	Min	Max
50	100.0	--	99.0	--
100	--	--	20.0	--
200	40.0	60.0	--	25.0

3.2.2 Surface area. Type II manganese dioxide shall have a surface area of no more than 10.0 square meters per gram when tested as specified in 4.2.4.5.

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 manganese dioxide 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). Each lot shall be identified and controlled in accordance with MIL-STD-1168.

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105.

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4.2.2.2 For test (see 6.5). Sampling shall be conducted in accordance with table III. A representative specimen of approximately 1 pound (lb) 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

: Number of containers in batch in 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

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled container, ready for shipment. Sample containers shall be examined for the following defects using an AQL of 4.0 percent defective:

- (a) Container damaged or leaking
- (b) Marking incorrect, missing, or illegible

4.2.3.2 For test (see 6.5). Approximately equal portions of all of the specimens taken in 4.2.2.2 shall be thoroughly mixed to form a composited specimen of no less than 1 lb. The composited specimen shall be tested as specified in 4.2.4. Each test shall be conducted in duplicate analysis. Failure of either analysis of any test 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 Moisture content. Transfer approximately 5 grams (g) of the specimen to a tared weighing bottle. Weigh the bottle and specimen to the nearest milligram. Dry the sample for 1 hour at 120°C. Stopper the bottle and cool in a sulfuric acid desiccator. Loosen the stopper for an instant to equalize the pressure, immediately restopper and weigh. Calculate the percent by weight moisture as follows:

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

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

4.2.4.2 Manganese content. Determine the manganese content of the specimen in accordance with ASTM E248.

4.2.4.3 Available oxygen. Transfer 100 milliliters (ml) of 15-percent sulfuric acid solution and 1.000 g of pure sodium oxalate to a 300-ml Erlenmeyer flask. To this solution add a 0.5-g portion of the specimen, dried as described in 4.2.4.1. Shake the flask, cover with a small watch glass, and heat on the steam bath for 2 to 3 hours or until all black or brown particles are dissolved. Remove the flask from the steam bath and titrate the excess of sodium oxalate in the hot solution with standard 0.1N potassium permanganate solution. Determine the quantity of standard potassium permanganate solution required to titrate 1.000 gram of pure sodium oxalate. Calculate the percent by weight available oxygen in the sample on a dry basis as follows:

$$\text{Percent by weight available oxygen} = \frac{0.8(A - B)N}{C}$$

where: N = Normality of the potassium permanganate solution,  
 A = Milliliters of the potassium permanganate solution required to titrate 1.000 g of pure sodium oxalate,  
 B = Milliliters of potassium permanganate solution required to titrate the excess sodium oxalate after reduction of the available oxygen in the specimen, and  
 C = Weight of the dry specimen in grams.

4.2.4.4 Particle size distribution. Select sieves conforming to RR-S-366, as applicable (see table II). Weigh to the nearest 0.01 g and nest in order of fineness with the most coarse sieve on top. Place the assembly on a bottom pan. Weigh to the nearest 0.01 g approximately 100 g of the specimen and place it on the first (most coarse) sieve. Place two 1-inch metal washers on the top sieve, cover and shake for 5 minutes by means of a mechanical shaker geared to produce  $300 \pm 15$  gyrations and  $150 \pm 10$  taps of the striker per minute. Weigh the material retained on each sieve and calculate the percent material passing through each sieve as required by table II.

4.2.4.5 Surface area. Determine the surface area of approximately 0.3 g of the specimen in accordance with the method entitled Surface Area By Monosorb Surface Area Analyzer of ASTM D3037, except that all specimens shall be out-gassed for 30 minutes at 300°C, and the weight of the specimen after outgassing is the weight to be used in calculating the specific surface area of the specimen as follows:

$$\text{NSA} = \frac{AC}{M_1 - M_2}$$

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where: NSA = Nitrogen surface area in square meters per gram,  
 A = Attenuator setting,  
 C = Counter reading in square meters,  
 $M_1$  = Mass of specimen and cell (after outgassing) in grams, and  
 $M_2$  = Mass of empty specimen cell in grams.

## 5. PACKAGING

5.1 Industrial unit packing, packing, and marking. A specified quantity of manganese dioxide (see 6.2) shall be unit packed, packed, and marked in accordance with MIL-STD-1188. In addition, each container shall be durably and legibly marked with the following precautionary marking:

## HAZARDS!

Strong oxidizer  
 Toxic by Ingestion  
 Toxic by Inhalation

DANGER! STRONG OXIDIZER. CONTACT WITH OTHER MATERIALS MAY CAUSE FIRE.

Keep from contact with clothing and other combustible materials.

Do not store near combustible materials.

Keep away from heat.

Store in tightly closed container.

Remove and wash contaminated clothing promptly.

WARNING! HARMFUL IF SWALLOWED OR INHALED.

Avoid breathing dust.

Keep container closed.

Use with adequate ventilation.

Wash thoroughly after handling.

FIRST AID: If swallowed, induce vomiting by sticking finger down throat or by giving soapy or strong salty water to drink. Repeat until vomit is clear. Call a physician. Never give anything by mouth to an unconscious person. If inhaled, remove to fresh air. If not breathing, give artificial respiration, preferably mouth to mouth. If breathing is difficult, give oxygen. Call a physician.

## 6. NOTES

6.1 Intended use. Type I manganese dioxide is intended for use in pyrotechnic compositions. Type II manganese dioxide is intended for use in smoke mixtures.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification
- (b) Type of manganese dioxide required (see 1.2)
- (c) Unit quantity of manganese dioxide required (see 5.1)

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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. Manganese dioxide is a strong oxidizer, toxic by ingestion and toxic by inhalation. All applicable safety rules, regulations and procedures must be followed in the handling and processing of this material.

## Custodians:

Army - EA  
Navy - OS

## Review activities:

Army - MD  
DLA - GS

## User activity:

Army - AR

## Preparing activity:

Army - EA

Project No. 6810-B341

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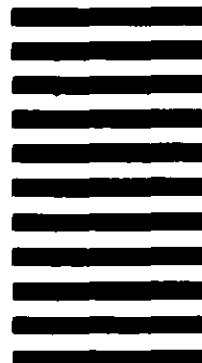


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*(See Instructions - Reverse Side)*

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3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION <i>(Mark one)</i> <input type="checkbox"/> VENDOR  <input type="checkbox"/> USER  <input type="checkbox"/> MANUFACTURER  <input type="checkbox"/> OTHER <i>(Specify):</i> _____
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a. Paragraph Number and Wording:	
b. Recommended Wording:	
c. Reason/Rationale for Recommendation:	
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