

NOT MEASUREMENT SENSITIVE

MIL-DTL-23938B(AS)
28 November 1997
SUPERSEDING
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DETAIL SPECIFICATION

FERRIC OXIDE

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 Scope. This specification establishes the requirements for ferric oxide.

2. APPLICABLE DOCUMENTS

(This section is not applicable to this specification.)

3. REQUIREMENTS

3.1 First article. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.3. If changes in the process methods and equipment adversely affect the characteristics of the material, additional first article samples may be required.

3.2 Chemical and physical requirements. Chemical and physical requirements of the ferric oxide shall be in accordance with table I.

3.3 Workmanship. The ferric oxide shall be free from foreign materials. It shall be manufactured in accordance with standard manufacturing procedures of the industry.

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 Warfare Center Aircraft Division, Code 414100B120-3, Highway 547, Lakehurst, NJ 08733-5100, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

FSC 1336

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TABLE I. Chemical and physical requirements.

Characteristics	Minimum	Maximum
Iron as Fe ₂ O ₃	98.70 %	-
Volatiles	-	0.20%
HCl solubles	-	0.20%
SiO ₂	-	0.10%
H ₂ O soluble	-	0.20%
Sieve analysis - Retained on US standard sieve No. 325	-	0.20%

4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as follows:

- a. First article inspection (see 4.3).
- b. Conformance inspection (see 4.4).

4.2 Lot size. A lot shall consist of material produced by one manufacturer in one continuous operation employing not more than one lot of each ingredient and with no change in formulation or process. If manufacture is by batch process, batches may be combined to form a lot provided that not more than one lot of each ingredient and no change in formulation or process is used (see 6.2).

4.3 First article inspection. First article inspection shall consist of all the conformance inspections and requirements of this specification, when a first article inspection is required (see 3.1).

4.4 Conformance inspection. Conformance inspection shall consist of all the test methods in 4.5 to determine compliance with section 3. Other test methods may be used if they offer assurance of equal results. If test methods differ from those specified herein, a copy of those methods or reference to available source for the methods shall be furnished to the acquiring activity.

4.4.1 Sampling. Sampling for conformance inspections shall be as specified in the contract or purchase order (see 6.2). All samples shall be selected at random from each lot. There shall be no defects.

4.5 Test methods.

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4.5.1 Determination of iron as Fe₂O₃. The determination of iron as Fe₂O₃ shall be as follows:

4.5.1.1 Preparation of reagents.

a. 0.1 N Potassium dichromate (K₂Cr₂O₇) - Weigh out exactly 4.9037 grams of reagent grade or analyzed potassium dichromate. Dilute to 1 liter with distilled water.

b. Sulfuric-phosphoric acid mixture - 150 mL of concentrated sulfuric acid (H₂SO₄) and 150 mL of concentrated phosphoric acid are mixed together. The mixture of acids is diluted to 1 liter with distilled water.

c. Diphenylamine indicator - One gram of diphenylamine is dissolved in 100 mL of concentrated H₂SO₄.

d. Stannous chloride solution (SnCl₂) - Dissolve 60 grams of SnCl₂ in 600 mL of concentrated HCl; add 400 mL of distilled water.

e. Saturated mercuric chloride (HgCl₂) - Place 100 grams of HgCl₂ in 1 liter of distilled water. Heat to dissolve.

4.5.1.2 Procedure.

a. Weigh 0.2 gram to the nearest 0.1 milligram (mg), of sample into a 250 mL beaker. Add 20 mL of concentrated HCl. Heat at 158 °-176 °F (70 °-80 °C) for one hour or until sample is completely dissolved.

b. Add stannous chloride (SnCl₂) solution to the dissolved sample dropwise until the solution is colorless; then add two drops of SnCl₂ in excess. Cool the solution in an ice bath.

c. To the cooled solution, add all at one time, 10 mL of saturated HgCl₂; let solution stand for approximately five minutes, then add 15 mL of the sulfuric-phosphoric acid mixture. Dilute the sample solution to 150-200 mL with distilled water. Add three drops of diphenylamine indicator and titrate immediately with the potassium dichromate solution. Near the end point, the green color changes to a dark blue-green. The dichromate is now added dropwise until the color changes to an intense violet blue.

d. Calculations:

$$N \text{ (Normality of Potassium Dichromate)} = \frac{(0.1000) (\text{Assay of Dichromate})}{100}$$

$$\text{Percent Fe}_2\text{O}_3 = \frac{(\text{mL}) (N) (7.985)}{\text{sample weight}}$$

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(mL = milliliters of potassium dichromate)

4.5.1.3 Alternate procedure.

- a. Weigh a 0.5 gram sample to the nearest 0.1 mg in a 250 mL beaker.
- b. Add 25 mL of distilled water and 15 mL of concentrated hydrochloric acid. Heat until the sample is completely dissolved.
- c. Transfer to a 250 mL volumetric flask. Wash the beaker carefully with 5 portions of distilled water. Add the washings to the volumetric flask. Make up to volume with distilled water.
- d. Pipette a 25 mL aliquot into a 250 mL beaker. Acidify the solution by addition of 5 mL of concentrated HCl.
- e. Add 30 mL of freshly prepared 6 percent aqueous solution of cupferron (ammonium N-nitrosophenylhydroxylamine). Do not allow the solution and the precipitate to get warm. Cold precipitation (about 50 °F) is preferred.
- f. Filter through a Munktell No. 00H or equivalent filter paper. Wash the precipitate with dilute hydrochloric acid solution.
- g. Transfer the filter paper containing the precipitate into a pre-ignited and weighed porcelain crucible.
- h. Dry in an oven before ignition. Ignite the material at first on a burner and then place in a muffle furnace, set at 1450 ± 50 °F, for 30 minutes.
- i. Cool in a desiccator and weigh.
- j. Calculate as follows:

$$\text{percent Fe}_2\text{O}_3 = \frac{B - A}{W} \times 100$$

where: A = weight of crucible
 B = weight of crucible plus precipitate
 W = weight of sample

4.5.2 Determination of volatiles. The determination of volatiles shall be as follows:

- a. Weigh, to the nearest 0.1 mg, 2 clean, dry (1 hour at 220 °F), stoppered weighing bottles and record weights as A.

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- b. Add approximately 5 gm of the undried sample to each. Stopper and weigh to the nearest 0.1 mg. Record weights as B.
- c. Tilt stopper and place in oven at 220 °F for at least 3 hours.
- d. Remove the bottles from the oven. Stopper and place in a CaCl₂ desiccator for 30 ±5 minutes.
- e. Weigh to the nearest 0.1 mg and record as C.
- f. Calculate as follows:

$$\text{Percent volatiles} = \frac{B - C}{B - A} \times 100$$

Note: Use tongs or cotton gloves to handle glassware. Make all weighings rapidly.

4.5.3 Total acid insolubles determination. The total acid insolubles shall be determined as follows:

- a. Weigh a 5 gram sample to the nearest 0.1 mg in a 250 mL beaker.
- b. Add about 50 mL of distilled water and 50 mL of concentrated hydrochloric acid.
- c. Heat until the material is dissolved. Filter through a Munktell No. 00H filter paper or equivalent, and wash 5 times with dilute HCl.
- d. Transfer the filter paper to the original beaker and add 25 mL of water and 10 mL of HCl; with the aid of a glass rod, tear the filter paper.
- e. Heat to boiling and boil for 5 minutes. Again filter through a No. 00H filter paper or equivalent and wash 5 times with dilute HCl.
- f. Transfer the filter paper containing the insoluble residue into a pre-ignited and weighed platinum crucible. Char the filter paper on a Meker burner and place the crucible in an electric muffle furnace at 1450 ±50 °F. Heat for 30 minutes.
- g. Cool in a desiccator and weigh.
- h. Calculate as follows:

$$\text{percent HCl insolubles} = \frac{B - A}{W} \times 100$$

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where: A = weight of crucible
 B = weight of crucible plus residue
 W = weight of sample

4.5.4 Silica content determination. The silica content shall be determined as follows:

- a. Wet the residue from HCl insolubles with two drops of concentrated sulfuric acid. Add about 15 mL of 48 percent hydrofluoric acid. Caution: Pour the acid directly from the polyethylene bottle and do not measure it in a glass container. If the acid gets on the skin, wash with water and soap to make sure that none of the material remains on the skin surfaces. This acid can cause severe burns.
- b. Place on a nichrome wire triangle or equivalent and place the triangle on a hot plate. The triangle should be so adjusted that the bottom of the crucible is about 1-2 mm from the surface of the hot plate. Heat until all of the acid is evaporated and heavy SO₃ fumes are evolved.
- c. Place on a burner and evaporate the last traces of SO₃. Then, place in a muffle furnace at 1450 ±50 °F. Heat for 30 minutes.
- d. Cool in a desiccator and weigh.
- e. Calculate as follows:

$$\text{percent SiO}_2 = \frac{B - A}{W} \times 100$$

where: A = weight of crucible plus residue before HF treatment
 B = weight of crucible plus residue after HF treatment
 W = weight of sample

4.5.5 Total water solubles determination. The total water solubles shall be determined as follows:

- a. Transfer about 10 grams of the sample weighed to the nearest 0.1 mg to a 250 mL beaker and add 100 mL distilled water. Bring to a boil and allow to boil for five minutes. Filter while hot using a close grained paper and collect the filtrate in a clean, dry, 250 mL beaker which has been previously weighed.
- b. Wash the residue once using hot distilled water. Place the beaker with the filtrate on a hot plate and evaporate to dryness. The sample may boil while evaporating but not so hard as to allow loss by spattering, especially near the end of the evaporation. Allow to cool and weigh. Calculate percent water soluble salts.

4.5.6 Sieve analysis.

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4.5.6.1 Procedure.

- a. Set up the following sieves in order:

No. 325 Sieve
Pan

- b. Place approximately 50.0 gm of the dry sample on the top sieve.

- c. Shake for 30 minutes.

- d. Transfer the material on the No. 325 sieve to a weighing container and weigh to the nearest 0.1 gm.

- e. Calculations:

$$\text{Percent retained on No. 325 Sieve} = \frac{\text{gms retained}}{\text{Sample wt.}} \times 100$$

4.5.6.2 Alternate procedure.

- a. Thoroughly wash and dry a US Standard No. 325 sieve and set aside for future use.

- b. Place 10 grams of the sample in a 600 mL beaker. Add enough of Triton 720 or Darvan No. 1 or Nacconal to form a heavy paste, and mix well to thoroughly incorporate the sample being tested.

- c. Dilute the mixture with water to a volume of approximately 300 mL and pour through the sieve set aside in step a, after the sieve has been weighed to the third decimal place.

- d. Wash the material on the sieve with a steady, gentle stream of water which has previously been passed through a US Standard No. 325 sieve. Using a small camel hair brush, or equivalent, brush the material through the sieve, running the water through at the same time. Continue this operation until no more ferric oxide passes through the sieve. Once or twice during the screening, the sieve may be removed from under the running water and a drop or two of the stock dispersing agent solution added, and spread over the sieve with the brush; after which, the washing and brushing operation can be continued.

- e. Dry the sieve in an oven at 212 °F to 220 °F for one hour. Cool and weigh the residue.

4.6 Acceptance criteria. All test results shall indicate compliance with the requirements of section 3. Failure to meet these requirements shall be cause for rejection of the lot.

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5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of material is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's System Command. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. Material purchased in accordance with this specification is intended for use as an ingredient in solid propellant grains.

6.2 Acquisition requirements. Acquisition documents must specify the following:

- a. Title, number and date of this specification.
- b. If first article is required (see 3.1).
- c. Minimum lot size, if applicable (see 4.2).
- d. Conformance sampling (see 4.4.1).
- e. Place of inspection.
- f. Packaging requirements (see 5.1).
- g. Place of delivery.

6.3 Subject term (key word) listing.

Ingredient
Propellant grains

6.4 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

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CONCLUDING MATERIAL

Preparing activity:
Navy - AS
(Project 1336-0019)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7.
3. The preparing activity must provide a reply within 30 days from receipt of the form.
NOTE: This form may not be used to request copies of documents, nor to request waivers, or clarification of requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

I RECOMMEND A CHANGE:

1. DOCUMENT NUMBER

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2. DOCUMENT DATE (YYMMDD)

971128

3. DOCUMENT TITLE

FERRIC OXIDE

4. NATURE OF CHANGE (Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.)

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME (Last, First, Middle Initial)

b. ORGANIZATION

c. ADDRESS (Include Zip Code)

d. TELEPHONE
(Include Area Code)

(1) Commercial:

(2) DSN:
(If Applicable)7. DATE SUBMITTED
(YYMMDD)

8. PREPARING ACTIVITY

a. NAME
COMMANDER
NAVAL AIR WARFARE CENTER
AIRCRAFT DIVISION

b. TELEPHONE NUMBER (Include Area Code)

(1) Commercial
(908) 323-2628(2) DSN
624-2628c. ADDRESS (Include Zip Code)
CODE 414100B120-3
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Suite 1403, Falls Church, VA 22041-3466
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