MIL-F-23938A(AS)
1 September 1966
Superseding
MIL-F-23938
19 August 1964

MILITARY SPECIFICATION

FERRIC OXIDE

This specification has been approved by the Naval Air Systems Command, Department of the Navy.

1. SCOPE

1.1 This specification establishes the requirements for ferric oxide.

2. APPLICABLE DOCUMENTS

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

STANDARDS

Military

MIL-STD-129	Marking for Shipment and Storage
MIL-STD-414	Sampling Procedures and Tables for Inspection by Variables for Percent Defective

Federal

FED-STD-102	Preservation, Packaging and Packing Levels

(When requesting any of the applicable documents, refer to both title and number. All requests should be made via the cognizant Government Inspector. Copies of this specification and other unclassified specifications and drawings required by contractors in connection with specific procurment functions should be obtained upon application to the Commanding Officer, Naval Supply Depot (Code 105), 5801 Tabor Avenue, Philadelphia, Pennsylvania 19120. All other documents should be obtained from the procuring activity or as directed by the contracting officer.)

3. REQUIREMENTS

3.1 Preproduction. -The preproduction test sample shall be manufactured using the process methods and equipment proposed for production. In the event of significant changes in the process methods and equipment which in the opinion of the procuring activity may adversely affect the characteristics of the material, additional preproduction 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.

TABLE I

CHEMICAL AND PHYSICAL REQUIREMENTS

Characteristics	Minimum	Maximum
Iron as Fe_2O_3	98.70%	
Volatiles		0.20%
HCl Insolubles		0.20%
SiO_2		0.10%
H_2O Soluble		0.20%
Sieve Analysis - Retained on US Standard Sieve No. 325		0.20%

3.3 Workmanship. - The ferric oxide shall be a uniform product free from foreign materials. It shall be uniform in quality and manufactured in accordance with standard manufacturing procedures of the industry.

3.4 Data requirements. - No data is required by this specification or by applicable documents referenced in section 2 unless specified in the contract or order (see 6.2).

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. -Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any

commercial laboratory acceptable to 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 Classification of examination and tests. -Examinations and tests of the ferric oxide shall be classified as follows:

a. Preproduction tests (4.4)

b. Quality conformance inspection (4.5).

4.3 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.

4.4 Preproduction tests. -Preproduction tests shall consist of all of the Quality Conformance Inspections and requirements of this specification.

4.5 Quality Conformance Inspections. -The following procedures shall be performed 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 procuring activity.

4.5.1 Sampling. -Sampling for quality conformance inspections shall be in accordance with Standard MIL-STD-414, Inspection Level IV.

4.5.2 Determination of iron as Fe_2O_3. -The determination of iron as Fe_2O_3 shall be as follows:

4.5.2.1 Preparation of reagents.

a. 0.1 N Potassium Dichromate ($K_2Cr_2O_7$) - 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 (H_2SO_4) 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 H_2SO_4.

d. Stannous Chloride Solution ($SnCl_2$) - Dissolve 60 grams of $SnCl_2$ in 600 ml of concentrated HCl; add 400 ml of distilled water.

e. Saturated Mercuric Chroride ($HgCl_2$) - Place 100 grams of $HgCl_2$ in 1 liter of distilled water. Heat to dissolve.

4.5.2.2 Procedure.

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

b. Add Stannous Chloride ($SnCl_2$) solution to the dissolved sample dropwise until the solution is colorless; then add two drops of $SnCl_2$ in excess. Cool the solution in an ice bath.

c. To the cooled solution, add all at one time, 10 ml of saturated $HgCl_2$; 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:

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

$$\%\ Fe_2O_3 = \frac{(ml)\ (N)\ (7.985)}{\text{sample weight}}$$

(ml = milliliters of potassium dichromate)

4.5.2.3 Alternate procedure.

a. Weigh a 0.5 gram sample to the nearest 0.1mg 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% aqueous solution of Cupferron (ammonium N-nitrosophenylhydroxylamine). Do ant 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:

$$\text{percent } Fe_2O_3 = \frac{B - A}{W} \times 100$$

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

4.5.3 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.

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 $CaCl_2$ desiccator for 30 ± 5 minutes.

e. Weigh to the nearest 0.1 mg and record as C.

f. Calculate as follows:

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

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

4.5.4 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 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.

d. Heat to boiling and boil for 5 minutes. Again filter through a No. 00H filter paper 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:

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

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

4.5.5 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% 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 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 SO_3 fumes are evolved.

c. Place on a burner and evaporate the last traces of SO_3. Then, place in a muffle furnace at 1450 ± 50°F and 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 + reside before HF treatment
B = weight of crucible + residue after HF treatment
W = weight of sample

4.5.6 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.

4.5.7 Sieve analysis. -

4.5.7.1 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:

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

4.5.7.2 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 sieve has been weighed to the third 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's hair brush, 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.

5. PREPARATION FOR DELIVERY

5.1 Application. -The requirements of section 5 apply only to direct purchases by or direct shipments to the Government.

5.2 Preservation and packaging. -Preservation and packaging shall be Level C, as defined in Federal Standard FED-STD-102, or as specified (see 6.2).

5.2.1 Level C. -Preservation and packaging shall be in accordance with standard commercial practice to afford protection against damage, contamination of the product by moisture, corrosion, or other foreign matter.

5.3 Packing. -Packing shall be Level C as defined in Federal Standard FED-STD-102, or as specified (see 6.2).

5.3.1 Level C. -The product packaged as specified in 5.2 shall be packed in a manner to insure carrier acceptance and safe delivery at destination. Containers shall be in accordance with Uniform Freight Classification Rules or regulations of other carriers applicable to the mode of transportation.

5.4 Marking. -Unless otherwise specified by the contract or order, unit packages, intermediate packages, and shipping containers shall be marked in accordance with the requirements of MIL-STD-129 and shall include but not be limited to the following information (see 6.2)

a. Manufacturer's name and address
b. Gross and net weights
c. Lot number and date of manufacture
d. The number of this specification
e. Safety precautions or unusual storage requirements
f. Purchase order or contract number.

6. NOTES

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

6.2 Ordering data. -Procurement documents may specify but not be limited to the following information:

a. Title, number and date of this specification
b. Type and size of container desired
c. Place of inspection
d. Minimum lot size, if applicable
e. Place of delivery

f. Request for copies of inspection data, if source inspected
g. Selection of applicable levels of packing and packaging (see 5.1 and 5.2)
h. Marking requirements (see 5.3)
i. Data requirements..

6.3 Conflicting requirements. -Conflicting requirements arising between this specification and any specifications, publications, or drawings listed herein shall be referred in writing to the procuring activity or appointed agent for interpretation and clarification.

6.4 Request for deviation. -Request for deviation from this specification, applicable drawings, specifications, or materials or processes shall be forwarded to the procuring activity prior to incorporating the desired change into production. All deviations shall be limited to the contract under which they were granted. A deviation is a before-the-fact request.

6.5 Request for waiver. -Request for waiver from this specification, applicable drawings, specifications, or materials shall be forwarded to the procuring activity. A waiver is an after-the-fact request. A waiver request shall include the following:

a. Exact nature of the defect
b. CD number and defect classification, if any
c. Quantity of items involved.

6.6 Value engineering. -Manufacturers of material covered by this specification are encouraged to submit value engineering suggestions to the procuring activity in an effort to reduce cost.

6.7 Safety precautions. -All detailed safety precautions shall be strictly observed.

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004

INSTRUCTIONS

This sheet is to be filled out by personnel either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity *(as indicated on reverse hereof)*.

SPECIFICATION
MIL-F-23938A(AS) FERRIC OXIDE

ORGANIZATION *(Of submitter)*	CITY AND STATE

CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT
		$

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?

A. GIVE PARAGRAPH NUMBER AND WORDING.

B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES.

2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID

3. IS THE SPECIFICATION RESTRICTIVE?

☐ YES ☐ NO IF "YES", IN WHAT WAY?

4. REMARKS *(Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)*

SUBMITTED BY *(Printed or typed name and activity)*	DATE

DD FORM 1 APR 63 1426

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Naval Air Systems Command
Washington, D. C. 20360

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