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MIL-F-14580A  
11 May 1965

~~SUPERSEDING~~  
MIL-F-14580(CE)  
18 July 1957

MILITARY SPECIFICATION

FERRIC CHLORIDE, ANHYDROUS, CRYSTALLINE, TECHNICAL

This specification is mandatory for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 This specification covers crystalline, anhydrous ferric chloride.

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:

SPECIFICATIONS

Federal

L-P-378 - Plastic Film (Polyethylene Thin Gage).

Military

JAN-D-195 - Drums, Steel, Calcium Carbide (100-Pound Capacity).

STANDARDS

Military

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.  
MIL-STD-129 - Marking for Shipment and Storage.

(Copies of specifications and standards required by suppliers in connection with specific procurement functions should be obtained from the procuring 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. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

AMERICAN PUBLIC HEALTH ASSOCIATION

Standard Methods for Examination of Water, Sewage, and Industrial Wastes.

(Application for copies should be addressed to The American Public Health Association, Inc., 1790 Broadway, New York, N. Y. 10019.)

3. REQUIREMENTS

3.1 Chemical composition. The ferric chloride shall conform to table I.

Table I. Chemical composition

| Requirement                                  | Percent by weight |         | Test Paragraph |
|--|-------------------|---------|----------------|
|  | Minimum           | Maximum |                |
| Ferric chloride ( $\text{FeCl}_3$ )          | 95.5              | --      | 4.4.2.4        |
| Ferrous chloride ( $\text{FeCl}_2$ )         | --                | 1.0     | 4.4.2.3        |
| Water insolubles, as $\text{Fe}_2\text{O}_3$ | --                | 4.0     | 4.4.2.2        |
| Deleterious ingredients                      |                   |         |                |
| Lead   | --                | 0.025   | 4.4.2.5        |
| Copper                                       | --                | 0.5     | 4.4.2.5        |
| Fluoride                                     | --                | 1.7     | 4.4.2.5        |
| Arsenic                                      | --                | 0.025   | 4.4.2.5        |
| Selenium                                     | --                | 0.005   | 4.4.2.5        |
| Hexavalent chromium                          | --                | 0.025   | 4.4.2.5        |

3.2 Appearance. The color of the ferric chloride shall vary from black to brown. A yellowish tinge within 1/4 inch of the seam of the inner bag when packaged in 1-pound bags or within 1/4 inch of the top when packaged as bulk in drums will be acceptable. The material shall not be completely solidified or contain a large quantity of lumps that are not easily broken. A hard layer that is easily broken and does not extend more than 1/4 inch from the top will be acceptable for material packaged as bulk in drums.

3.3 Quantity. The ferric chloride shall be supplied in 1-pound units or in bulk, as specified (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 insure supplies and services conform to prescribed requirements.

4.1.1 Component and material inspection. The supplier is responsible for insuring that components and materials used are manufactured, examined, and tested in accordance with referenced specifications and standards.

4.2 Classification of inspection. Inspection shall be classified as follows:

- (a) Quality conformance inspection of the ferric chloride (see 4.3).
- (b) Inspection of preparation for delivery (see 4.5).

#### 4.3 Quality conformance inspection.

4.3.1 Lot. A lot shall consist of the ferric chloride offered for acceptance at one time which has been produced by one manufacturer under the same manufacturing conditions and with no change in materials provided the operation is continuous. In the event that the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.3.2 Sampling.

4.3.2.1 For examination. Sampling for examination shall be in accordance with MIL-STD-105.

4.3.2.2 For test. Sampling for tests shall be in accordance with MIL-STD-105, inspection level S-1. A specimen shall be removed from each container in the sample and placed in a clean, dry container labeled to identify the lot and the container from which it was taken.

4.4 Inspection procedure.

4.4.1 Examination. Samples selected in accordance with 4.3.2.1 shall be examined for the following defects. AQL shall be 2.5 percent defective.

101. Color not as specified.
102. Lumps or hard surface not easily broken.
103. Yellowish tinge extends beyond 1/4 inch of inner seam when packaged in 1-pound bags.
104. Yellowish tinge extends beyond 1/4 inch of top when packaged as bulk.

4.4.2 Tests. Samples selected in accordance with 4.3.2.2 shall be subjected to the tests specified in 4.4.2.2 through 4.4.2.5. Distilled water and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections shall be applied where significant. Tests shall be conducted as follows. AQL shall be 1.0 percent defective.

4.4.2.1 Reagent solutions for tests.

4.4.2.1.1 Preventive solution. Dissolve 67 g of manganous sulfate ( $MnSO_4 \cdot H_2O$ ) in 500 to 600 ml of water; add 138 ml of phosphoric acid ( $H_3PO_4$ , specific gravity 1.70) and 130 ml of sulfuric acid ( $H_2SO_4$ , specific gravity 1.84) and dilute to 1 liter. Cool to room temperature.

4.4.2.1.2 Mercuric chloride solution. Dissolve 5 g of mercuric chloride ( $HgCl_2$ ) in 95 ml of water.

4.4.2.1.3 Stannous chloride solution. Dissolve 150 g of iron-free stannous chloride ( $SnCl_2 \cdot 2H_2O$ ) in 1 liter of dilute hydrochloric acid (333 ml of concentrated hydrochloric acid to 667 ml of water).

4.4.2.1.4 Potassium permanganate solution (approximately 0.1 N). Dissolve approximately 3.25 g of potassium permanganate ( $\text{KMnO}_4$ ) in approximately 1 liter of water, let stand in the dark for 2 weeks, and filter, without washing, through a fritted glass crucible of fine porosity or a Gooch crucible with an acid washed pad. Store in a dark-colored glass-stoppered bottle. Standardize using sodium oxalate.

4.4.2.2 Water insoluble. Without undue exposure to air, transfer 4 to 5 g of ferric chloride to a tared weighing bottle, using a clean paper funnel to prevent ferric chloride from coming in contact with the ground joint. Stopper the bottle immediately, and reweigh the bottle and contents. Wash the specimen into a 500-ml volumetric flask with water, cool to approximately  $25^\circ\text{C}$ . ( $77^\circ\text{F}$ .), fill the bottle to the mark with water and shake by hand. Allow the contents to settle for about 10 minutes and then vacuum filter approximately 425 ml of the solution through a tared Gooch crucible, containing an acid washed asbestos pad, into a dry vacuum flask. Remove the flask containing the filtrate, and save the filtrate for succeeding analyses. Replace the vacuum flask with another vacuum flask, and filter the remaining solution. Wash the flask with water, and add washings to Gooch crucible. Repeat the washing procedure six times, allowing the filter to pull dry between each washing. Wash the precipitate once with ethyl alcohol. Dry the crucible and precipitate for 30 minutes at  $100^\circ\text{C}$ . ( $212^\circ\text{F}$ .), heat for 15 minutes at  $600^\circ\text{C}$ . ( $1112^\circ\text{F}$ .), cool in a desiccator to room temperature, and reweigh. All weighings are made to the nearest milligram. Calculate the percent water insolubles as  $\text{Fe}_2\text{O}_3$  as follows:

$$\text{Percent water insolubles} = \frac{100 A}{B}$$

where A = Weight of precipitate (weight of crucible with asbestos pad and precipitate minus weight crucible with asbestos pad), and

B = Weight of sample (weight of weighing bottle and sample minus weight of weighing bottle).

Nonconformance to table I shall constitute failure of this test.

4.4.2.3 Ferrous chloride (FeCl<sub>2</sub>). Pipette 100 ml of filtrate (see 4.4.2.2) into a 250 ml Erlenmeyer flask, add 20 ml of preventive solution (4.4.2.1.1) and titrate immediately to a 15-second pink end point with approximately 0.05 N potassium permanganate solution (50 ml of approximately 0.1 N solution diluted to 100 ml with water). Run a blank determination using water. Calculate the percent FeCl<sub>2</sub> as follows:

$$\text{Percent ferrous iron} = \frac{(0.0558) (100) (A-B) C}{0.2 D}$$

where A = ml of KMnO<sub>4</sub> used in titration of sample,

B = ml of KMnO<sub>4</sub> used in titration of blank,

C = normality of KMnO<sub>4</sub> solution,

D = weight of sample used in water insoluble determination (see 4.4.2.2),

$$\text{Percent FeCl}_2 = (2.27)(\text{percent ferrous iron}).$$

Nonconformance to table I shall constitute failure of this test.

4.4.2.4 Ferric chloride (FeCl<sub>3</sub>). Pipette 50 ml of filtrate (see 4.4.2.2) into a 250 ml Erlenmeyer flask and add 5 ml of concentrated hydrochloric acid. Place the flask on a hot plate and slowly evaporate the contents to 5 to 10 milliliters. Add stannous chloride solution (see 4.4.2.1.3) dropwise to the hot filtrate until the iron is reduced (color changes from yellow-red to a pale light green). Use diluted stannous chloride solution (1 ml of stannous chloride solution to one ml of water) near the end point. Dilute the contents to about 150 ml with water and cool. Add 10 ml of mercuric chloride solution (see 4.4.2.1.2). The formation of a slight, white, silky precipitate when the mercuric chloride solution is added indicates that the proper amount of stannous chloride solution has been added. Discard the specimen if no precipitate (insufficient stannous chloride solution has been added) or a heavy precipitate (excess of stannous chloride solution has been added) is formed. Repeat the test. If the proper amount of stannous chloride has been added, allow to stand 5 minutes, add 20 ml of preventive solution, and titrate immediately to a 15-second pink end point with approximately 0.1 N potassium permanganate solution. Calculate the percent ferric chloride as follows:

$$\text{Percent total iron} = \frac{(0.0558) (100) AB}{0.1 C}$$

where A = ml of  $\text{KMnO}_4$  used in titration of sample,

B = normality of  $\text{KMnO}_4$  solution,

C = the weight of the sample used in water insoluble determination (see 4.4.2.2),

percent ferric iron = D-E,

where D = Percent total iron,

E = Percent ferrous iron, and

Percent ferric chloride = (2.95) (percent ferric iron.)

Nonconformance to table I shall constitute failure of this test.

4.4.2.5 Deleterious ingredients. Determine the tests for lead, copper, fluoride, arsenic, selenium, and hexavalent chromium in accordance with the appropriate method outlined in Standard Methods for the Examination of Water, Sewage, and Industrial Wastes of the American Public Health Association using filtrate (see 4.4.2.2). Nonconformance to table I shall constitute failure of this test.

4.5 Inspection of preparation for delivery. The packaging, packing, and marking shall be inspected as follows.

4.5.1 Sampling for examination. Sampling for examination shall be by lots and shall be in accordance with MIL-STD-105. Lot acceptance or rejection shall be based on an AQL of 4.0 percent defective.

4.5.2 Examination. Samples selected in accordance with 4.5.1 shall be examined for the following defects.

105. Outer or inner bag missing when packaged in 1-pound quantities.
106. Ferric chloride or foreign matter in seam.
107. Width of seam is less than  $3/8$  inch.
108. Incomplete sealing (blisters or voids in seam).

- 109. Liner missing.
- 110. Liner not closed as specified.
- 111. Container not as specified.
- 112. Container damaged.
- 113. Holes in bags or liner.
- 114. Container closure not as specified.
- 115. Marking incorrect, missing, or illegible.

## 5. PREPARATION FOR DELIVERY

5.1 Packaging. Packaging shall be level A, B or C as specified (see 6.2).

5.1.1 Precautions. The following precautions shall be taken when packaging and packing the ferric chloride:

- (a) The time that the ferric chloride is exposed to the atmosphere shall be kept to an absolute minimum.
- (b) The air immediately above the loading hopper shall be heated between 140° F. and 170° F., even though very low absolute humidity conditions are present. This method will extend the time approximately 10 times that the ferric chloride in the hopper may be exposed to the atmosphere without caking.

### 5.1.2 Level A.

5.1.2.1 One-pound package. The ferric chloride shall be packaged in 1-pound quantities in polyethylene inner bags, sealed, and each sealed inner bag overpackaged in a polyethylene outer bag and the outer bag sealed. The polyethylene inner and outer bags shall be fabricated from polyethylene film conforming to L-P-378 and in the form of extruded lay-flat tubing without side seams. The end seams shall be heat sealed not less than 3/8-inch wide with a smooth polished finish. No type of parting agent shall be used in the fabrication of the seams. The dimensions of the bags shall be as shown in table II.

Table II. Dimensions of Bags (inches)

| Bag   | Length               | Width               | Nominal thickness |
|-------|----------------------|---------------------|-------------------|
| Inner | 9 <sup>±</sup> 0.25  | 5 <sup>±</sup> 0.25 | 0.0020            |
| Outer | 10 <sup>±</sup> 0.25 | 6 <sup>±</sup> 0.25 | 0.0060            |

After filling the inner bags and before heat sealing, precautions shall be taken that no ferric chloride dust or foreign matter is on the surfaces to be sealed. After filling and sealing, the inner bag shall be immediately transferred to a separate area for packaging in the outer bag.

5.1.2.2 Bulk quantity. Ferric chloride required in bulk quantity shall not be packaged.

5.1.3 Level B. The ferric chloride shall be packaged as specified in 5.1.2.1 for 1-pound packages. Bulk quantity shall not be packaged.

5.1.4 Level C. The ferric chloride shall be packaged as specified in 5.1.2.1 for 1-pound packages. Bulk quantity shall not be packaged.

5.2 Packing. Packing shall be level A, B, or C as specified (see 6.2).

5.2.1 Level A.

5.2.1.1 One-pound bags. The ferric chloride, packaged as specified in 5.1.2, shall be packed in 100-bag quantities in a 100-pound capacity steel drum conforming to JAN-D-195, except the height dimension shall be modified to contain the bags arranged in uniform layers. Each drum shall be provided with a 5-mil-thick polyethylene bag liner fabricated from material and heat sealed as specified in 5.1.2.1. The drum bag liner shall be of sufficient length and width to form-fit inside the drum with not less than a 3-inch length at the top for sealing. A tag with identification marking shall be attached to the top of the drum bag liner by covering the strings with a strip of the bag material and heat sealing to the bag. The drum shall be closed in accordance with the drum specification.

5.2.1.2 Bulk. One-hundred pounds of ferric chloride shall be packed in a polyethylene lined drum as specified in 5.2.1.1.

5.2.2 Level B. The ferric chloride shall be packed as specified in 5.2.1.

5.2.3 Level C. The ferric chloride shall be packed as specified in 5.2.1.

5.3 Marking. In addition to any special marking required by the contract or order, packages and shipping containers shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. The crystalline, anhydrous ferric chloride covered by this specification is intended for use as a coagulant for water purification.

6.2 Ordering data. Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Whether ferric chloride shall be packaged in 1-pound bags or in bulk (see 3.3).
- (c) Level of packaging and level of packing required (see 5.1 and 5.2). Level B preservation and packaging is intended to provide limited but economical protection and should be specified only when it is determined the ferric chloride will be held in covered storage approximately 1 year from date of initial preservation and packaging.

6.3 Batch. A batch is defined as that quantity of material which has been subjected to some unit chemical or physical mixing process intended to make the final product substantially uniform (see 4.3.1).

Custodians:

Army - MO(ERDL)  
Navy - SH  
Air Force - 68

Review activities:

Army - MU, MD  
Navy - YD, MC  
Air Force - 68

User activities:

Navy - SH

Preparing activity:

Army - MO(ERDL)  
Project No. 6810-0243

**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-14580A - Ferric Chloride, Anhydrous, Crystalline, Technical**

|                             |                |
|-----------------------------|----------------|
| ORGANIZATION (Of submitter) | CITY AND STATE |
|-----------------------------|----------------|

|              |                            |                     |
|--------------|----------------------------|---------------------|
| CONTRACT NO. | QUANTITY OF ITEMS PROCURED | DOLLAR AMOUNT<br>\$ |
|--------------|----------------------------|---------------------|

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 |
|---|------|

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