

MIL-F-14580C
25 February 1975
SUPERSEDING
MIL-F-14580B
26 June 1968

MILITARY SPECIFICATION
FERRIC CHLORIDE, ANHYDROUS,
CRYSTALLINE, TECHNICAL

This specification is approved 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 Sheet and Strip, Thin Gauge, Polyolefin.
PPP-D-705	- Drum; Metal Shipping, Steel (Over 12 and Under 55 Gallon).

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.)

FSC 6810

MIL-F-14580C

2.2 Other publications. The following document forms 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, INC.

Standard Methods for Examination of Water and Wastewater.

(Application for copies should be addressed to the American Public Health Association, Inc., 1790 Broadway, New York, NY 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 (FeCl_3)	95.5	- - - -	4.4.2.4
Ferrous chloride (FeCl_2)	- - - -	1.0	4.4.2.3
Water insolubles, as Fe_2O_3	- - - -	4.0	4.4.2.2

3.1.1 Deleterious ingredients. When ferric chloride in the amount of 100 milligrams (mg) is added to 1 liter of water, the increase in the amount of the following deleterious ingredients in the water shall not exceed the amount specified:

Lead	- .025 mg/liter
Copper	- .5 mg/liter
Fluoride	- .80 mg/liter
Arsenic	- .025 mg/liter
Selenium	- .005 mg/liter
Hexavalent chromium	- .025 mg/liter

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 nor contain a large quantity of lumps that cannot be 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.

MIL-F-14580C

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 specified herein. Except as otherwise specified in the contract or order, the supplier 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 Classification of inspections. Inspections shall be classified as follows:

- (a) Quality conformance inspection (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 the process is a batch operation, each homogeneous batch or mix produced from one compounding formulation in one manufacturing and production operation shall constitute a lot.

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 tests. Sampling for tests shall be in accordance with MIL-STD-105, Inspection Level S-1. A specimen of not less than 10 grams (g) 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.

MIL-F-14580C

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 manganese 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). Cool solution to room temperature and dilute to 1 liter.

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 ($KMnO_4$) in approximately 1 liter of water, let stand in the dark for 2 weeks, and then 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 insolubles. 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° C (77° 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

MIL-F-14580C

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 the 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° C (212° F), heat for 15 minutes at 600° C (1112° F), cool in a desiccator to room temperature, and reweigh. All weighings shall be to the nearest milligram. Calculate the percent water insolubles 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 (FeCl₂). Pipette 100 ml of filtrate (see 4.4.2.2) into a 250 ml erlenmeyer flask, add 20 ml of preventive solution (see 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 FeCl₂ as follows:

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

where A = ml of KMnO₄ used in titration of sample,

B = ml of KMnO₄ used in titration of blank,

C = normality of KMnO₄ solution,

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

Percent FeCl₂ = (2.27) (percent ferrous iron).

Nonconformance to Table I shall constitute failure of this test.

4.4.2.4 Ferric chloride (FeCl₃). 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 1 ml of water) near the end point. Dilute the

MIL-F-14580C

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 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 KMnO_4 used in titration of sample,

B = normality of KMnO_4 solution,

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

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. Prepare a solution containing 100 milligrams per liter (mg/l), plus 5 mg/l minus 0 mg/l, of ferric chloride dissolved in distilled water. Coagulate the solution by raising the pH of the solution to between 7.5 and 8.5. Stir for 15 minutes and then let settle for an additional 15 minutes. Filter off the supernatant liquid. Test the supernatant liquid for lead, copper, fluoride, arsenic, selenium, and hexavalent chromium in accordance with the appropriate method outlined in Standard Methods for Examination of Water and Wastewater of the American Public Health Association. Nonconformance to 3.1.1 shall constitute failure of this test.

4.5 Inspection of preparation for delivery.

4.5.1 Quality conformance inspection of pack.

4.5.1.1 Unit of product. For the purpose of inspection, a completed pack prepared for shipment shall be considered a unit of product.

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

MIL-F-14580C

4.5.1.3 Examination. Samples selected in conformance to 4.5.1.2 shall be examined for the following defects. AQL shall be 2.5 percent defective.

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 1/4 inch.
108. Incomplete sealing (blisters or voids in seam).
109. Liner missing.
110. Liner not closed as specified.
111. Container not as specified.
112. Drum liners punctured or torn.
113. Holes in bags or liner.
114. Container closure not as specified.
115. Marking incorrect, missing or illegible.

5. PREPARATION FOR DELIVERY

5.1 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 to between 140° F and 170° F, even though very low absolute humidity conditions are present. This method will extend by approximately 10 times the time that the ferric chloride in the hopper may be exposed to the atmosphere without caking.

5.2 Packaging (see 6.4). The ferric chloride shall be packaged in 1-pound quantities in polyethylene (inner) bags and heat sealed. Each sealed inner bag shall be overpackaged in a polyethylene (outer) bag and heat sealed. The polyethylene inner and outer bags shall be fabricated from material conforming to L-P-378, Type I, in the form of extruded lay-flat tubing without side seams. The end seams shall be heat sealed not less than 1/4 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. Sealing for both inner and outer bags shall result in seams having no blisters or voids in the seam, nor shall the bags be punctured or torn.

MIL-F-14580C

Table II. Dimensions of Bags (inches)

Bags	Length	Width	Nominal Thickness of Film
Inner	9 \pm 0.25	5 \pm 0.25	0.0020
Outer	10 \pm 0.25	6 \pm 0.25	0.0060

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

5.3 Packing (see 6.4). The ferric chloride shall be packed in 1-pound bags or in bulk, as specified (see 6.2).

5.3.1 One-pound bags. The ferric chloride, packaged as specified in 5.2, shall be packed in 100-bag quantities in a drum conforming to PPP-D-705, Type VI, except that 22-gage steel is acceptable, and that overall height shall be such that, after filling, the outage shall not exceed 1-1/2 inches (see 6.3). Before packing, each drum shall be provided with a 5-mil-thick polyethylene bag liner fabricated from material and heat sealed as specified in 5.2. The drum bag liner shall be of sufficient length and width to formfit 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. The polyethylene drum liner shall not be punctured or torn in loading the drum or heat sealing the liner. The heat-seal requirements of 5.2 shall apply.

5.3.2 Bulk. One hundred and thirty-five pounds, plus 1 minus 0 pound, of ferric chloride shall be packed in the type of drum specified in 5.3.1, with the following exceptions and additions: Drum shall be made of 22-gage material and shall have a baked-on lining of a clear alkyd resin varnish of a minimum thickness of 0.0005 mil; the drum shall not exceed 18 gallons in capacity, with the inside diameter (I.D.) and outside diameter (O.D.) dimensions as specified in PPP-D-705. The drum shall be closed as specified in PPP-D-705.

5.4 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.

MIL-F-14580C

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 packed in 1-pound bags or in bulk (see 5.3).

6.3 Drum height. For packing 100 1-pound packages, a drum complying with the requirements of 5.3 will be approximately 24 inches in height. For packing 135 pounds bulk ferric chloride, drum height will vary depending on the density of the ferric chloride, which varies from batch to batch; however, the maximum size of the drum shall not exceed 18 gallons.

6.4 Level of packaging and packing (see 5.2 and 5.3). Only a single method of packaging and packing is suitable and adequate for this commodity. For purposes of the packaging/packing level, marking A/A shall be used. Level C packaging and packing is not applicable to this item, as the shipping container will also be used in the field with the water purification set as a storage container. As such, it is susceptible to exposure to all environmental conditions in addition to rough handling. A lesser level of pack would require a repack prior to troop issue.

Custodians:

Army - ME
Navy - YD
Air Force - 68

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Army - ME

Review activities:

Army - EA, MD
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User activity:

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Project No. 6810-B026

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DOCUMENT IDENTIFIER AND TITLE		
ML-P-141800 Ferric Chloride, Anhydrous, Crystalline, Technical		
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