



**American Water Works
Association**

The Authoritative Resource on Safe Water®

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(Revision of ANSI/AWWA B407-05)

AWWA Standard

Liquid Ferric Chloride



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AWWA Standard

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Foreword

This foreword is for information only and is not a part of ANSI/AWWA B407.

I. Introduction.

I.A. *Background.* Ferric chloride is commercially available in two solid forms, hexahydrate and anhydrous, and in liquid form. This standard describes ferric chloride in aqueous (liquid) form for use in the treatment of municipal and industrial water supplies. Ferric chloride can be produced as a coproduct with titanium dioxide from natural ores containing iron and titanium oxides or by the controlled reaction of spent steel pickling solutions, hydrochloric acid, chlorine, and scrap iron. A high-purity product can be manufactured by reacting chlorine gas with iron, ferrous sulfate, or ferrous chloride. Recognizing that the purity of ferric chloride can vary with the manufacturing process, the purchaser may request that the supplier describe the manufacturing process used. The purchaser also may want to ask the supplier about potential impurities relative to the manufacturing process used.

This standard provides methods for the analysis of specific gravity, insoluble matter, total iron, ferrous iron, ferric chloride, and acidity.

I.B. *History.* In 1977, the AWWA Water Quality Division recommended to the AWWA Standards Council that a standard for ferric chloride be prepared. The Standards Council authorized the development of a ferric chloride standard on May 12, 1977. The first edition of ANSI/AWWA B407 was approved by the AWWA Board of Directors on June 5, 1983. Subsequent revisions were approved in 1988, 1993, 1998, and 2005. This edition of ANSI/AWWA B407 was approved on Jan. 22, 2012.

I.C. *Acceptance.* In May 1985, the US Environmental Protection Agency (USEPA) entered into a cooperative agreement with a consortium led by NSF International (NSF) to develop voluntary third-party consensus standards and a certification program for direct and indirect drinking water additives. Other members of the original consortium included the American Water Works Association Research Foundation (AwwaRF, now Water Research Foundation) and the Conference of State Health and Environmental Managers (COSHEM). The American Water Works Association (AWWA) and the Association of State Drinking Water Administrators (ASDWA) joined later.

* American National Standards Institute, 25 West 43rd Street, Fourth Floor, New York, NY 10036.

In the United States, authority to regulate products for use in, or in contact with, drinking water rests with individual states.* Local agencies may choose to impose requirements more stringent than those required by the state. To evaluate the health effects of products and drinking water additives from such products, state and local agencies may use various references, including two standards developed under the direction of NSF, NSF†/ANSI 60, Drinking Water Treatment Chemicals—Health Effects, and NSF/ANSI 61, Drinking Water System Components—Health Effects.

Various certification organizations may be involved in certifying products in accordance with NSF/ANSI 60. Individual states or local agencies have authority to accept or accredit certification organizations within their jurisdiction. Accreditation of certification organizations may vary from jurisdiction to jurisdiction.

Annex A, “Toxicology Review and Evaluation Procedures,” to NSF/ANSI 60 does not stipulate a maximum allowable level (MAL) of a contaminant for substances not regulated by a USEPA final maximum contaminant level (MCL). The MALs of an unspecified list of “unregulated contaminants” are based on toxicity testing guidelines (noncarcinogens) and risk characterization methodology (carcinogens). Use of Annex A procedures may not always be identical, depending on the certifier.

ANSI/AWWA B407 addresses additives requirements in Sec. 4.3 of the standard. The transfer of contaminants from chemicals to processed water or the residual solids is becoming a problem of great concern. The language in Sec. 4.3.2 is a recommendation only for direct additives used in the treatment of potable water to be certified by an accredited certification organization in accordance with NSF/ANSI 60, Drinking Water Treatment Chemicals—Health Effects. However, users of the standard may opt to make this certification a requirement for the product. Users of this standard should also consult the appropriate state or local agency having jurisdiction in order to

1. Determine additives requirements, including applicable standards.
2. Determine the status of certifications by parties offering to certify products for contact with, or treatment of, drinking water.
3. Determine current information on product certification.

II. Special Issues.

II.A. *Storage and Handling Precautions.* Liquid ferric chloride is an orange-brown aqueous solution that is acidic and corrosive to common metals. Suitable materials for construction of storage and handling facilities include titanium, tantalum,

* Persons outside the United States should contact the appropriate authority having jurisdiction.

† NSF International, 789 N. Dixboro Road, Ann Arbor, MI 48105.

synthetic-rubber-lined steel, corrosion-resistant fiberglass-reinforced plastics (FRP), ceramics, polytetrafluoroethylene (PTFE), polyvinylidene fluoride (PVDF), and polyvinyl chloride (PVC). Steel, aluminum, copper, and polyamides, such as nylon, are not suitable.

Ferric chloride solution may cause burns to the eyes, and acid-resistant goggles should be worn during handling. Contact with skin may cause irritation. This can be avoided by wearing rubber gloves, boots, jacket, and pants.

For additional safety aspects, refer to material safety data sheets (MSDS) available from the chemical supplier or manufacturer.

II.B. *Basis for Payment.* The basis for payment shall be the dry weight equivalent of ferric chloride supplied.

III. Use of This Standard. It is the responsibility of the user of an AWWA standard to determine that the products described in that standard are suitable for use in the particular application being considered.

III.A. *Purchaser Options and Alternatives.* This standard for liquid ferric chloride permits a wide range of ferric chloride concentration. The purchaser should be aware of the wide range of water weight relative to the shipping cost.

The following information should be provided by the purchaser:

1. Standard used—that is, ANSI/AWWA B407, Liquid Ferric Chloride, of latest revision.
2. Whether compliance with NSF/ANSI 60, Drinking Water Treatment Chemicals—Health Effects, is required.
3. Net weight to be supplied (Sec. II.B).
4. Details of other federal, state or provincial, and local requirements (Section 4).
5. Whether specific gravity of the solution is stipulated (Sec. 4.1).
6. Percentage of ferrous iron permitted. If ferrous iron in excess of 2.5 percent is permitted, the purchaser should state the maximum allowable ferrous iron concentration (Sec. 4.2).
7. Whether the purchaser will reject product from containers or packaging with missing or damaged seals. The purchaser may reject product from bulk containers or packages with missing or damaged seals unless the purchaser's tests of representative samples, conducted in accordance with Sec. 5.2–5.11, demonstrate that the product meets the standard. Failure to meet the standard or the absence of, or irregularities in, seals may be sufficient cause to reject a shipment.
8. Size and type of container to be used (Sec. 6.2.1).
9. If weight certificates are required (Sec. 6.2.2).

10. Whether alternative security measures have been adopted to replace or augment the security measures set out in Sec. 6.2.3 and 6.2.4.

11. Whether an affidavit of compliance, certified analysis, or both, is required (Sec. 6.3).

III.B. *Modification to Standard.* Any modification of the provisions, definitions, or terminology in this standard must be provided by the purchaser.

IV. Major Revisions. Major revisions to the standard in this edition include the following:

1. Inclusion of a requirement for compliance with the Safe Drinking Water Act and other federal regulations (Section 4).

2. Inclusion of a requirement for tamper-evident packaging (Sec. 6.2.3 and 6.2.4).

V. Comments. If you have any comments or questions about this standard, please call the AWWA Engineering and Technical Services Department at 303.794.7711, FAX at 303.795.7603, write to the department at 6666 West Quincy Avenue, Denver, CO 80235-3098, or e-mail the group at standards@awwa.org.



**American Water Works
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AWWA Standard

Liquid Ferric Chloride

SECTION 1: GENERAL

Sec. 1.1 Scope

This standard describes ferric chloride in aqueous (liquid) form for use in the treatment of potable water, wastewater, and reclaimed water. Applications of the chemical include (1) water softening with lime or a combination of lime and soda ash to improve hardness reduction and coagulation, and (2) water clarification, as a coagulant, followed by settling or filtration.

Sec. 1.2 Purpose

The purpose of this standard is to provide the minimum requirements for liquid ferric chloride, including physical, chemical, sampling, packaging, shipping, and testing requirements.

Sec. 1.3 Application

This standard can be referenced in documents for purchasing and receiving liquid ferric chloride and can be used as a guide for testing the physical and chemical properties of liquid ferric chloride samples. The stipulations of this standard apply when this document has been referenced and then only to liquid ferric chloride used in the treatment of potable water, wastewater, and reclaimed water.

SECTION 2: REFERENCES

This standard references the following document. In its latest edition, it forms a part of this standard to the extent specified in this standard. In any case of conflict, the requirements of this standard shall prevail.

NSF*/ANSI† 60, Drinking Water Treatment Chemicals—Health Effects.

SECTION 3: DEFINITIONS

The following definitions shall apply in this standard:

1. *Day*: A day is defined as a 24-hr period.
2. *Manufacturer*: The party that manufactures, fabricates, or produces materials or products.
3. *Potable water*: Water that is safe and satisfactory for drinking and cooking.
4. *Purchaser*: The person, company, or organization that purchases any materials or work to be performed.
5. *Reclaimed water*: Wastewater that becomes suitable for beneficial use as a result of treatment.
6. *Supplier*: The party that supplies materials or services. A supplier may or may not be the manufacturer.
7. *Tamper-evident packaging*: Packaging having one or more indicators or barriers to entry which, if breached or missing, can reasonably be expected to provide visible evidence to the purchaser that tampering has occurred. The tamper-evident features of the packaging shall be designed to, and shall, remain intact when handled in a reasonable manner during manufacture, storage, shipment, and delivery to the purchaser. Properly constructed, labeled, and closed carboys or drums constitute two forms of tamper-evident packaging.
8. *Wastewater*: A combination of the liquid and water-carried waste from residences, commercial buildings, industrial plants, and institutions, together with any groundwater, surface water, and stormwater that may be present.

* NSF International, 789 N. Dixboro Road, Ann Arbor, MI 48105.

† American National Standards Institute, 25 West 43rd Street, Fourth Floor, New York, NY 10036.

SECTION 4: REQUIREMENTS

Materials shall comply with the requirements of the Safe Drinking Water Act and other federal regulation for potable water, wastewater, or reclaimed water as applicable.

Sec. 4.1 Physical Requirements

Liquid ferric chloride (FeCl_3) is an orange-brown, acidic, corrosive, aqueous solution. The specific gravity of the solution shall be in the range of 1.3 to 1.5. The specific gravity will vary with the FeCl_3 concentration. Concentration may be varied with the season and point of destination to prevent crystallization during cold weather.

Sec. 4.2 Chemical Requirements

Liquid ferric chloride shall contain from 28 to 47 percent FeCl_3 by weight, which is 9.6 to 16.2 percent total iron as Fe. Not more than 2.5 percent of the total iron shall be in the ferrous state; however, the purchaser may specify a higher allowable ferrous-iron concentration. The free acid, expressed as HCl, shall not exceed 1.0 percent. The solution shall not contain more than 0.2 percent total insoluble matter by weight.

Sec. 4.3 Impurities*

4.3.1 *General.* The material supplied in accordance with this standard shall contain no soluble inorganic or organic substances in quantities capable of producing deleterious or injurious effects on the health of those consuming water that has been properly treated with the material. This standard applies to liquid ferric chloride produced by currently recognized methods of manufacture. If other methods or raw materials are used, impurities could be present that might be inconsistent with accepted water treatment practices. In such cases, additional tests by the manufacturer may be required to demonstrate that the product is suitable for water treatment purposes.

4.3.2 *Product certifications.* Liquid ferric chloride is a direct additive used in the treatment of potable water. This material should be certified as suitable for contact with or treatment of drinking water by an accredited certification organization in accordance with NSF/ANSI 60. Evaluation shall be accomplished in accordance with requirements that are no less restrictive than those listed in NSF/ANSI 60.

* See Sec. I.C of the foreword.

Certification shall be accomplished by a certification organization accredited by the American National Standards Institute.

SECTION 5: VERIFICATION

Sec. 5.1 Sampling

5.1.1 *Sampling point.* Samples shall be taken at the point of destination.

5.1.2 *Amount of shipment to be sampled.* Five percent of the containers shall be sampled, but a minimum of 5 and a maximum of 15 containers in any one shipment shall be sampled. No sample shall be taken from a leaking container. In the case of bulk shipments, a composite sample should be taken from the tank truck or barge at five equally spaced intervals during unloading of the bulk container.

5.1.3 *Sampling method, sample size, and handling.* The contents of each container to be sampled shall be mixed thoroughly, and a portion shall be taken of such size that the total gross sample shall consist of at least 10 qt (10 L). After thorough mixing of the gross sample, three 0.5-qt (0.5-L) samples shall be sealed in airtight bottles. Care must be taken during sampling not to introduce large quantities of air into the product. A chain-of-custody form shall accompany all samples and shall be properly completed by the sampler. Each sample container shall be labeled to identify it and shall be signed by the sampler.

5.1.4 *Distribution of samples.* One of the three sealed samples is for immediate use by the purchaser for testing of the shipment according to Sec. 5.2. The two remaining samples shall be retained for possible future use according to Sec. 5.12.

5.1.5 *Sample retention.* Samples shall be retained for at least 30 days after the date of receipt of the shipment before they are discarded.

5.1.6 *Test samples.* Test samples shall be obtained from sealed material samples delivered to the laboratory. Material samples shall be unsealed only when it is necessary to remove quantities of the material for testing. This shall be accomplished quickly, and the material samples shall then be resealed for future reference. Liquid ferric chloride shall be mixed thoroughly before a portion is withdrawn for examination. The bottle shall be kept sealed after the sample has been removed.

Sec. 5.2 Test Procedures—General

Sampling shall be conducted according to Sec. 5.1. The laboratory examination of the sample shall be completed within five days after receipt of the shipment.

Methods of testing ferric chloride for specific gravity, insoluble matter, total iron, ferrous iron, ferric iron, acidity, and impurities shall conform to the procedures described in the following sections. Use alternative procedures only with the written acceptance of the purchaser. In any case of conflict, the methods described in this standard shall prevail.

Sec. 5.3 Specific Gravity

The approximate amount of ferric chloride present in the sample can be determined by measuring the specific gravity with an accurate hydrometer at a known temperature. The supplier shall furnish a table showing the percentage of ferric chloride for different specific-gravity readings.

Sec. 5.4 Total Insoluble Matter

5.4.1 Apparatus.

1. Membrane filter holder—47 mm (or 110 mm).
2. Glass-fiber filters—Whatman GF/C (Whatman Inc., Clifton, N.J.) or equivalent.
3. Filter flask—1,000 mL.

5.4.2 Procedure.

1. Dry a filter at 103°C (217°F) for ½ hr. Cool in a desiccator and weigh to the nearest mg. This is the tare weight.
2. Place the filter, wrinkled surface up, in a filter holder. Apply a vacuum and wet with a small amount of distilled water.
3. Mix the sample thoroughly, and immediately pipette 25 mL into a tared 250-mL beaker. Rapidly determine the weight of the sample to the nearest 0.01 g. This weight will be used in the calculation for percent total insoluble matter (Sec. 5.4.3).
4. Add approximately 150 mL distilled water and filter.
5. Wash the residue in the beaker onto the filter.
6. Wash the filter repeatedly with distilled water until it is free of any yellow ferric chloride color. Perform at least six washings.
7. Remove the filter, dry 1 hr at 103°C (217°F), cool in a desiccator, and weigh to the nearest mg. Subtract the tare weight (from Sec. 5.4.2[1]); this is the weight of the residue to be used in the calculation for percent total insoluble matter (Sec. 5.4.3).

5.4.3 *Calculation.*

$$\% \text{ total insolubles} = \frac{\text{weight of residue} \times 100}{\text{weight of sample}} \quad (\text{Eq 1})$$

Sec. 5.5 Iron5.5.1 *Reagents.*

1. Concentrated sulfuric acid.
2. Concentrated hydrochloric acid.
3. Stannous chloride solution. Dissolve 100 g stannous chloride in 30 percent-by-volume hydrochloric acid solution (HCl/H₂O). Store over clean metallic tin. Discard if the solution becomes cloudy. The stannous chloride solution is not stable, and a fresh solution should be prepared each month.
4. Saturated mercuric chloride solution—poison. Dissolve mercuric chloride in 500 mL distilled water until no additional mercuric chloride will dissolve.
5. Sulfuric acid/phosphoric acid solution. Make 150 mL H₂SO₄ and 150 mL of 85 percent H₃PO₄ up to 1 L with distilled water. (Pour acids into water.)
6. Potassium dichromate solution. Dissolve 4.902 g reagent-grade potassium dichromate in distilled water and dilute to 1 L; this is a 0.1*N* solution.
7. Barium-diphenylamine sulfonate indicator. Dissolve 0.32 g barium diphenylamine sulfonate in 100 mL distilled water.

5.5.2 *Procedure.*

1. Mix the sample thoroughly. Quickly transfer approximately 10 mL sample to a previously tared stoppered weigh bottle. Weigh the sample to the nearest 0.01 g. The weight of the sample will be used in calculating percent total iron (Sec. 5.5.3). Transfer the sample to a 250-mL volumetric flask and dilute to the mark with distilled water. Invert 10 times and quickly pipette 50 mL sample from the 250-mL volumetric flask into a 500-mL Erlenmeyer flask.
2. Add 15 mL concentrated sulfuric acid and 10 mL concentrated hydrochloric acid to the contents of the flask and bring to a boil.
3. Stannous chloride reduction. When the solution has been heated to near the boiling point, 95° to 100°C (203° to 212°F), add stannous chloride solution, drop by drop, while gently swirling the flask with heat-resistant tongs until the yellow iron color in the solution is discharged. Add no more than one drop stannous chloride in excess. Allow solution to cool to room temperature and add 10 mL saturated mercuric chloride solution. A gray or black precipitate indicates an

excessive amount of stannous chloride. A white, silky precipitate should form; if it does not, discard and start over. Dilute to 150 mL with distilled water. Add 15 mL of the sulfuric acid/phosphoric acid solution and 0.3 mL of barium diphenylamine sulfonate indicator solution. Titrate at once with 0.1*N* potassium dichromate solution to a violet-blue end point.

5.5.3 Calculation.

$$\% \text{ total iron} = \frac{\text{mL } 0.1N \text{ K}_2\text{Cr}_2\text{O}_7 \times 0.5585}{\text{grams of sample} \times 50/250} \quad (\text{Eq } 2)$$

Sec. 5.6 Ferrous Iron

5.6.1 Reagents.

1. Concentrated sulfuric acid.
2. Sulfuric acid/phosphoric acid solution. Dilute 150 mL concentrated H₂SO₄ and 150 mL of 85 percent H₃PO₄ to 1 L with distilled water. (Pour acids into water.)
3. Barium diphenylamine sulfonate indicator. Dissolve 0.32 g barium diphenylamine sulfonate in 100 mL distilled water.
4. Potassium dichromate solution. Dissolve 4.902 g reagent-grade potassium dichromate in distilled water and dilute to 1 L. This is a 0.1*N* solution.

5.6.2 Procedure.

1. Pipette a 100-mL portion of the sample from the 250-mL volumetric flask (see Sec. 5.5.2[1]) and transfer to a 500-mL Erlenmeyer flask.
2. Add 15 mL concentrated sulfuric acid.
3. Dilute to 200 mL with distilled water.
4. Allow to cool to room temperature.
5. Add 15 mL of the sulfuric acid/phosphoric acid solution and 12 drops of barium diphenylamine sulfonate indicator solution.
6. Titrate at once with 0.1*N* potassium dichromate solution to a violet-blue end point.

5.6.3 Calculations.

$$\% \text{ ferrous iron} = \frac{\text{mL } 0.1N \text{ K}_2\text{Cr}_2\text{O}_7 \times 0.5585}{\text{grams of sample} \times 100/250} \quad (\text{Eq } 3)$$

$$\% \text{ total iron as ferrous} = \frac{\% \text{ ferrous iron}}{\% \text{ total iron}} \quad (\text{Eq } 4)$$

Sec. 5.7 Percent Ferric Chloride

5.7.1 *Procedure.* The percent ferric chloride is determined by subtracting the percent ferrous iron from the percent total iron and then converting the percent ferric iron to percent ferric chloride.

5.7.2 *Calculations.*

$$\% \text{ ferric iron} = \% \text{ total iron} - \% \text{ ferrous iron} \quad (\text{Eq 5})$$

$$\% \text{ ferric chloride} = \% \text{ ferric iron} \times 2.905 \quad (\text{Eq 6})$$

Sec. 5.8 Acidity

5.8.1 *Reagents.*

1. Potassium fluoride ($\text{KF} \cdot 2\text{H}_2\text{O}$)—analytical reagent grade.
2. Phenolphthalein indicator solution. Dissolve 5 g phenolphthalein in 1 L of 50 percent alcohol. Neutralize with sodium hydroxide.
3. Sodium hydroxide. To prepare a 1N solution, weigh out 40 g sodium hydroxide pellets and dissolve in approximately 200 mL CO_2 -free distilled water. Dilute to 1 L in a volumetric flask. Standardize against benzoic acid or potassium acid phthalate of known normality. Use modified methyl orange as an indicator. To prepare a 0.05N solution, dilute 50 mL 1N NaOH to 1 L with CO_2 -free distilled water.
4. Sulfuric acid. To prepare 0.05N sulfuric acid, dilute 50 mL concentrated sulfuric acid (36N) to 1 L. This solution is 1.8N. Take 28.0 mL of the 1.8N sulfuric acid and dilute to 1 L, thereby producing a 0.05N sulfuric acid. Standardize the 0.05N sulfuric acid against a known base before using.

5.8.2 *Procedure.*

1. Dissolve 20 g potassium fluoride in 40 mL of boiled distilled water. Add 0.2 mL phenolphthalein indicator solution and adjust to a faint pink color with 0.05N sodium hydroxide or sulfuric acid to obtain neutralized potassium fluoride.
2. Weigh 1 to 2 g (1 mL) ferric chloride solution and record weight to within 0.001 g. Wash into a 150-mL beaker with 50 mL of boiled distilled water.
3. Add 25 mL neutralized potassium fluoride and mix.
4. Add 0.2 mL phenolphthalein indicator solution and titrate to a faint pink color with 0.05N sodium hydroxide.

5.8.3 *Calculation.*

$$\% \text{ free acidity (as HCl)} = \frac{\text{mL } 0.05\text{N NaOH} \times 0.001825 \times 100}{\text{grams of sample}} \quad (\text{Eq 7})$$

Sec. 5.9 Iron Alternative Method

5.9.1 *Procedure.* The percent ferrous chloride and percent ferric chloride results are converted to percent iron and then added together. The calculation is performed with results from Sec. 5.10 and 5.11.

5.9.2 *Calculation.*

$$\% \text{ total iron} = \frac{\% \text{FeCl}_2}{2.2696} + \frac{\% \text{FeCl}_3}{2.905} \quad (\text{Eq 8})$$

Sec. 5.10 Ferrous Iron Alternative Method

5.10.1 *Reagents.*

1. Sulfuric acid/phosphoric acid solution. Use an ice bath to cool 600 mL distilled water in a beaker to $<10^{\circ}\text{C}$ (50°F). Add slowly, with stirring, 150 mL concentrated H_2SO_4 (95 to 98 percent by weight) and 250 mL 85 percent H_3PO_4 .

2. Diphenylamine sulfonate indicator. Dissolve 1.356 g diphenylamine sulfonic acid, sodium salt, in 500 mL distilled water.

3. Ceric sulfate solution. Cautiously add 30 mL concentrated H_2SO_4 (95 to 98 percent by weight) to 500 mL distilled water in a 1-L volumetric flask. Dissolve 63.255 g ceric ammonium sulfate dihydrate ($\text{Ce}[\text{NH}_4]_4 [\text{SO}_4]_4 \cdot 2\text{H}_2\text{O}$) in this acid solution. Cool to room temperature and dilute to the 1-L mark with distilled water. For best results, allow to stand two or more weeks and filter before use. This is a 0.1N solution. Ceric sulfate solution can also be purchased at a supplier-certified 0.1N concentration.

5.10.2 *Procedure.*

1. Pipette a 10-mL portion of the sample into a previously tared weighing cup. Record the weight to the nearest 0.001 g. Transfer into a 500-mL Erlenmeyer flask, with weighing cup rinses.

2. Dilute the sample to about 300 mL with distilled water. Add 15 mL sulfuric acid/phosphoric acid mixture and then 2 mL diphenylamine sulfonate indicator solution.

3. Titrate at once with 0.1N ceric sulfate solution from light green-gray to a purple-violet end point. It is useful to have a lighted magnetic stirrer under the flask.

5.10.3 *Calculations.*

$$\% \text{ ferrous chloride} = \frac{\text{mL } 0.1\text{N Ce}(\text{NH}_4)_4(\text{SO}_4) \cdot 2\text{H}_2\text{O} \times 1.2675}{\text{grams of sample}} \quad (\text{Eq 9})$$

$$\% \text{ ferrous iron} = \frac{\% \text{ ferrous chloride}}{2.2696} \quad (\text{Eq 10})$$

Sec. 5.11 Ferric Iron Alternative Method

5.11.1 Reagents.

1. 1.0*N* hydrochloric acid (HCl).
2. Potassium thiocyanate (KCNS) indicator solution. Dissolve 20 g KCNS in 100 mL of distilled water.
3. Disodium ethylene diamine tetraacetic acid (Na₂EDTA) solution. Dissolve 37.224 g Na₂EDTA · 2H₂O in distilled water and dilute to the mark in a 1-L volumetric flask. This is approximately a 0.1*M* solution, which should be standardized for accurate work. An appropriate iron reference for standardization is 1,000 ± 10 mg/L Fe in acidic solution. Na₂EDTA solutions can also be purchased at a supplier-certified 0.1*M* concentration.

5.11.2 Procedure.

1. Weigh 0.70 ± 0.05 g sample into a previously tared weighing cup. Record the weight to the nearest 0.0001 g.
2. Transfer contents into a 250-mL Erlenmeyer flask with rinses and add 135 mL distilled water.
3. While stirring, adjust solution pH to 1.7 ± 0.1 by dropwise addition of 1*N* HCl if the pH > 1.7 or 1*N* NaOH if the pH < 1.7.
4. Add 2 mL KCNS indicator solution.
5. Titrate with 0.1*M* Na₂EDTA solution from blood-red to a yellow end point. There is an intermediate orange color to the solution during this titration, but the actual end point is bright yellow. Best results are obtained using an automatic titrator with photometric end point detection.

5.11.3 Calculations.

$$\% \text{ ferric chloride} = \frac{\text{mL of } 0.1M \text{ Na}_2\text{EDTA} \times M \text{ Na}_2\text{EDTA} \times 16.22}{\text{grams of sample}} \quad (\text{Eq 11})$$

$$\% \text{ ferric iron} = \frac{\% \text{ ferric chloride}}{2.905} \quad (\text{Eq 12})$$

NOTE: The use of Na₂EDTA in this method may allow other impurities such as Mn, Cu, Zn, and possibly other metals to titrate as iron.

Sec. 5.12 Notice of Nonconformance

If the material delivered to the purchaser does not meet the chemical, physical, safety, or security requirements of this standard, the purchaser shall provide a

notice of nonconformance to the supplier within 10 days after receipt of the shipment at the point of destination. The results of the purchaser's tests shall prevail unless the supplier notifies the purchaser within five days after receipt of the notice of nonconformance that a retest is desired. On receipt of the request for a retest, the purchaser shall forward to the supplier one of the sealed samples taken according to Sec. 5.1. If the results obtained by the supplier do not agree with the test results obtained by the purchaser, the other sealed sample shall be forwarded, unopened, for analysis to a referee laboratory agreed on by both parties. The results of the referee analysis shall be accepted as final.

The supplier shall provide to the purchaser an adjustment that is agreed on between the supplier and the purchaser reflecting the diminished quality of the product.

SECTION 6: DELIVERY

Sec. 6.1 Marking*

6.1.1 *Required.* Each shipment of material shall be identified as to product, grade, net weight, the weight-percent ferric chloride, name and address of the manufacturer, and the brand name. Packages or containers shall show a lot number and identification of manufacturer. All markings on packaged, containerized, or bulk shipments shall conform to applicable laws and regulations, including requirements established by the US Occupational Safety and Health Administration (OSHA).

6.1.2 *Optional.* Packages may also bear the statement, "Guaranteed by (name of manufacturer) to meet the requirements of ANSI/AWWA B407, Standard for Liquid Ferric Chloride," provided that the requirements of this standard are met.

Sec. 6.2 Packaging and Shipping

Packaging and shipping of liquid ferric chloride shall conform to current federal, state or provincial, and local regulations.

* Governmental packaging, marking, and shipping references reflect US requirements. Users of ANSI/AWWA B407 outside the United States should verify applicable local, provincial, and national regulatory requirements. Because of frequent changes in these regulations, all parties should remain informed of possible revisions. Provisions of the purchaser's documents should not preclude compliance with applicable regulations.

6.2.1 *Shipping containers.* Liquid ferric chloride shall be shipped in carboys, drums, cars, tank trucks, or barges, as specified by the purchaser.

6.2.2 *Weight certificates.* The purchaser may require that bulk shipments be accompanied by weight certificates from certified weighers, or that the weights be checked by the purchaser on delivery.

6.2.3 *Security requirements for nonbulk shipments.* Packaged product shall be stored, shipped, and delivered in tamper-evident packaging as defined in Section 3, or an alternative method or methods may be agreed on by the manufacturer and purchaser that provide a reasonable assurance of protection against tampering.

6.2.4 *Security requirements for bulk shipments.* Bulk quantities of product shall be secured employing one of the following security measures or a combination of measures:

6.2.4.1 *Seals.* Bulk quantities of product may be sealed with a uniquely numbered tamper-evident seal(s). The seal numbers shall be recorded and disclosed on shipping documents such as the Bill of Lading. Seals shall be inspected upon receipt of product by the purchaser and evidence of tampering or removal should be reported to the carrier and supplier.

6.2.4.2 *Chain of custody.* A continuous chain of custody may be maintained between the manufacturer and the purchaser during storage and shipment if so specified by the purchaser.

6.2.4.3 *Alternative method.* An alternative method or methods may be agreed on by the manufacturer and purchaser that provide reasonable assurance of protection against tampering.

Sec. 6.3 Affidavit of Compliance

The purchaser may require either (1) an affidavit from the manufacturer or supplier that the material provided complies with applicable requirements of this standard, or (2) a certified analysis of the material at the time of delivery detailing the desired items.

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