



**American Water Works
Association**

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ANSI/AWWA B403-16
(Revision of ANSI/AWWA B403-09)

AWWA Standard

Aluminum Sulfate— Liquid, Ground, or Lump

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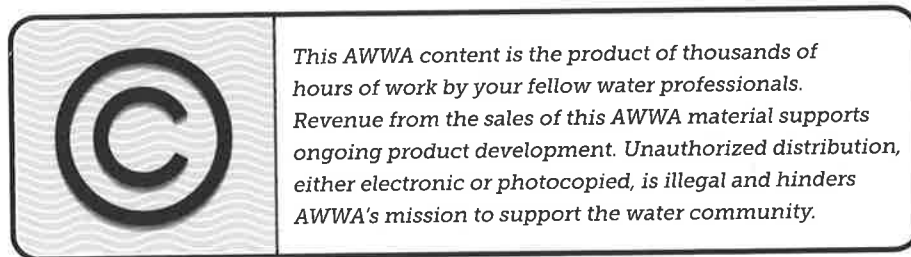
AWWA Standard

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Foreword

This foreword is for information only and is not a part of ANSI/AWWA B403.

I. Introduction.

I.A. *Background.* Coagulation of suspended and colloidal particles in water using various forms of aluminum sulfate has been known since the seventeenth century. Although historical details are lacking, in the early days of water purification by coagulation, lump or slab forms of aluminum sulfate were dissolved and added to the water in solution form. Sommerville, N.J., was probably the first utility in the United States serving potable water to use this treatment method on a more or less continuous basis. Shortly thereafter, numerous plants adopted this method, including the Hackensack (N.J.) Water Company and those of the former American Water Works and Electric Company.

The terms *filter alum*, *papermakers alum*, and *commercial alum* all refer to the same chemical: aluminum sulfate. Aluminum sulfate, as produced in tonnage quantities, has the formula $Al_2(SO_4)_3 \cdot nH_2O$, where n is approximately 14 waters of crystallization instead of 18, which is the true crystalline form. The purpose of producing a form of aluminum sulfate that has 14 waters of crystallization is twofold. First, it is stable and gains or loses water slowly under adverse storage conditions. Second, aluminum sulfate having 14 waters of crystallization is approximately 12 percent stronger than aluminum sulfate having 18 waters of crystallization. Using the more concentrated form of the chemical reduces the tonnage to be handled, reduces chemical storage requirements, and saves the consumer transportation costs.

Several other "dry alums" are used in treating swimming pool water and certain industrial waters. These products are "true alums," which are double salts containing 24 waters of crystallization, such as ammonia alum $Al_2(SO_4)_3(NH_4)_2SO_4 \cdot 24H_2O$ and potash alum $Al_2(SO_4)_3 \cdot K_2SO_4 \cdot 24H_2O$. These specialty alums are excellent coagulants but have a lower Al_2O_3 content and are not widely used compared with aluminum sulfate. Commercial liquid alum is shipped as a relatively clear, stable solution that contains up to 48.5 percent by weight dry product. In recent years, manufacturers have built production plants in areas of high consumption, and now the United States has many alum-producing plants.

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

Aluminum sulfate is prepared from aluminum hydroxide, alumina trihydrate, or alumina-bearing ores, such as clay and bauxite. The aluminum ore is ground to a required fineness and digested with sulfuric acid at elevated temperatures. Insoluble materials are removed by sedimentation. The supernatant solution is either decanted and sold in liquid form or concentrated and allowed to crystallize into a solid, dry hydrated product. The aluminum oxide content of the liquid is limited to about 8 percent Al_2O_3 to avoid crystallization in transit and storage. The solid, dry hydrated product usually contains 17 percent Al_2O_3 .

Liquid alum may be received and stored in corrosion-resistant tanks and transferred through appropriate liquid-metering devices directly into the water at the desired point of application.

I.B. *History.* A specification for Sulphate of Alumina was published in 1925 by the American Water Works Association (AWWA) in its *Water Works Practice Manual*.^{*} On June 25, 1942, AWWA adopted the Emergency Alternate Specifications for Sulphate of Alumina, which relaxed some provisions and allowed the chemical to be made from lower-quality raw materials. (As a wartime measure, higher-quality bauxite had been diverted to the aluminum-metals industry.) In 1952, AWWA's committee upgraded the standard under the guidance of J.E. Kerslake, with Paul Tamer acting as referee for the comments from producers and consumers. The standard was revised in 1958 to include liquid aluminum sulfate. Other revisions were made in 1964, 1969, 1970, 1982, 1988, 1993, 1998, 2003, and 2009. This edition was approved by the AWWA Board of Directors on June 19, 2016.

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 Water Research Foundation (formerly AwwaRF) 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.

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

^{*} *Water Works Practice Manual*. AWWA, New York (1925).

[†] Persons outside the United States should contact the appropriate authority having jurisdiction.

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 jurisdictions. 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 B403 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. 4.3.3 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 (http://www.nsf.org/newsroom_pdf/NSF-ANSI_60_watemarked.pdf). 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. *Safety and Spill Control.* Aluminum sulfate is a buffered acidic solution that causes irritation when it comes in contact with the eyes, skin, or mucous membranes. Protective clothing and equipment should be used when handling liquid alum. Good ventilation, good housekeeping procedures, and personal cleanliness are also recommended.

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

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

II.B. *Protective Equipment.* Dry aluminum sulfate dust can cause irritations to the respiratory system and eyes. Dust masks, goggles, and gloves are recommended for persons working in the presence of dry aluminum sulfate dust.

Normal precautions should be used to prevent spraying or splashing liquid alum. Face shields should be worn to protect the eyes. Outer coverings, such as vinyl or rubber rain jackets and pants, may be used to protect clothing from liquid alum.

II.C. *First Aid.* Aluminum sulfate should be flushed from the eyes using copious amounts of water. If irritation persists, a physician should be consulted promptly. Aluminum sulfate should be washed from the skin with copious amounts of water.

For additional safety information, refer to material safety data sheets available from the chemical supplier or manufacturer.

II.D. *Spill-Control Considerations.* Outdoor storage tanks should be suitably diked or otherwise provided with an adequate means of secondary containment. Appropriate secondary containment measures should be taken to prevent spills or leaks from indoor storage tanks and tank-car or tank-truck unloading stations from entering sewers or other channels that discharge directly to a water body or a municipal sewage system.

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.* When purchasing aluminum sulfate under the provisions of this standard, the following information should be provided by the purchaser:

1. Standard used—that is, ANSI/AWWA B403, Aluminum Sulfate—Liquid, Ground, or Lump, of latest revision.
2. Whether compliance with NSF/ANSI 60, Drinking Water Treatment Chemicals—Health Effects, is required.
3. Details of other federal, state or provincial, and local requirements (Section 4).
4. Type of aluminum sulfate required—liquid, ground, or lump (Sec. 4.1). Standard practice is to order liquid alum on a “dry basis” by specifying the quantity of aluminum sulfate as weight of dry chemical in a water solution.
5. If other than the listed percent of water-soluble aluminum is required (Sec. 4.2.1.2).
6. If basic or acidic alum is required (Sec. 4.2.2).

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. 5.3 through 5.10, demonstrate that the product meets specifications. Failure to meet specifications or the absence of, or irregularities in, seals may be sufficient cause to reject the shipment.

8. Quantity required and method of packaging and shipping (Section 6).

9. If less-than-carload quantities of liquid alum are ordered, the type and size of container required (Sec. 6.2.2.2).

10. If bulk shipments are to be accompanied by weight certificates or certificates issued by certified weighers (Sec. 6.2.4).

11. Whether alternative security measures have been adopted to replace or augment the security measures set out in Sec. 6.2.5 and 6.2.6.

12. Affidavit of compliance, if 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 made to the standard in this edition include the following:

1. Inclusion of definitions for potable water, reclaimed water, and wastewater (Section 3).

2. Revisions to the Notice of Nonconformance (Sec. 5.11).

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

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AWWA Standard

Aluminum Sulfate—Liquid, Ground, or Lump

SECTION 1: GENERAL

Sec. 1.1 Scope

This standard describes purified aluminum sulfate in liquid, ground, or lump form for use in the treatment of potable water, wastewater, or reclaimed water.

Sec. 1.2 Purpose

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

Sec. 1.3 Application

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

SECTION 2: REFERENCES

This standard references the following documents. In their latest editions, they form a part of this standard to the extent specified in this standard. In any case of conflict, the requirements of this standard shall prevail.

APHA,* AWWA, and WEF†—*Standard Methods for the Examination of Water and Wastewater*. Denver, Colo.: AWWA.

ASTM‡ E11—Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves.

ASTM E200—Standard Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis.

NSF§/ANSI¶ 60—Drinking Water Treatment Chemicals—Health Effects.

SECTION 3: DEFINITIONS

The following definitions shall apply in this standard:

1. *Acidic alum*: Acidic alum contains sulfuric acid, which is not combined as aluminum sulfate (expressed as “free H₂SO₄”).
2. *Aluminum sulfate*: The product of the reaction between sulfuric acid and a mineral rich in aluminum, such as bauxite.
3. *Basic alum*: Basic alum contains water-soluble aluminum, which is not combined as aluminum sulfate (expressed as “free Al₂O₃”).
4. *Constructor*: The party that provides the work and materials for placement or installation.
5. *Day*: A day is defined as a 24-hour period.
6. *Manufacturer*: The party that manufactures, fabricates, or produces materials or products.
7. *Potable water*: Water that is safe and satisfactory for drinking and cooking.

* American Public Health Association, 800 I Street, NW, Washington, DC 20001.

† Water Environment Federation, 601 Wythe Street, Alexandria, VA 22314.

‡ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

§ NSF International, 789 North Dixboro Road, Ann Arbor, MI 48113.

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

8. *Purchaser*: The person, company, or organization that purchases any materials or work to be performed.

9. *Purified aluminum sulfate*: Purified ground or lump aluminum sulfate that complies with the provisions of this standard and contains water-insoluble matter not exceeding 0.5 percent.

10. *Reclaimed water*: Wastewater that becomes suitable for beneficial use as a result of treatment.

11. *Supplier*: The party that supplies materials or services. A supplier may or may not be the manufacturer.

12. *Tamper-evident packaging*: Packaging having one or more indicators or barriers to entry that, 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 sealed plastic drums and tanks constitute two forms of tamper-evident packaging.

13. *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.

SECTION 4: REQUIREMENTS

Materials shall comply with the requirements of the Safe Drinking Water Act and other federal regulations for potable water and wastewater systems as applicable.

Sec. 4.1 Physical Requirements

Aluminum sulfate is the product of the reaction between sulfuric acid and a mineral rich in aluminum, such as bauxite. Solid aluminum sulfate shall be dry and clean, and shall be lump or ground, as specified. Liquid alum is a nearly saturated solution of aluminum sulfate.

4.1.1 *Lump aluminum sulfate*. Lump aluminum sulfate shall range in size so that 100 percent passes through a 3-in. (7.62-cm)* ring and at least 75 percent shall be retained on a ½-in. (13-mm) sieve.†

* Metric conversions given in this standard are direct conversions of US customary units and are not those specified in International Organization for Standardization (ISO) standards.

† ASTM E11.

4.1.2 *Ground aluminum sulfate.* Ground aluminum sulfate shall be of such size that not less than 90 percent shall pass a No. 10 sieve and 100 percent shall pass a No. 4 sieve.*

4.1.3 *Liquid aluminum sulfate clarity.* Liquid alum shall be of such clarity to permit the reading of flow-measuring devices without difficulty.

Sec. 4.2 Chemical Requirements

4.2.1 *Content of aluminum.*

4.2.1.1 Purified ground or lump aluminum sulfate shall contain water-soluble aluminum of not less than 9.0 percent as Al or 17.0 percent as Al_2O_3 .

4.2.1.2 Liquid alum shall contain water-soluble aluminum of not less than 4.23 percent as Al or 8.0 percent as Al_2O_3 , unless otherwise specified by the purchaser.

4.2.2 *Basic and acidic alum.* The range of basicity or acidity of alum shall be specified by the purchaser.

4.2.3 *Water-insoluble matter.*

4.2.3.1 In purified ground or lump aluminum sulfate, the water-insoluble matter shall not exceed 0.5 percent.

4.2.3.2 In liquid alum, the water-insoluble matter shall not exceed 0.2 percent.

Sec. 4.3 Impurities[†]

4.3.1 *General.* Aluminum sulfate that meets the requirements of this standard shall contain no soluble material or organic substances in quantities capable of producing deleterious or injurious effects to the health of those consuming a water that has been treated properly with the aluminum sulfate.

4.3.2 *Specific impurity limits.* The total water-soluble iron (expressed as Fe_2O_3) content of aluminum sulfate shall be no more than 0.75 percent,[‡] on a basis of 17 percent Al_2O_3 (9 percent as Al), in dry aluminum sulfate; or 0.35 percent, on a basis of 8.0 percent Al_2O_3 (4.23 percent as Al), in liquid aluminum sulfate.

4.3.3 *Product certifications.* Aluminum sulfate is a direct additive used in the treatment of potable water. This material should be certified as suitable

* ASTM E11.

† See Sec. I.C of the foreword.

‡ There are a number of water treatment plants in which aluminum sulfate of a higher iron content can be used without increasing the iron content of the treated water. If a purchaser determines that aluminum sulfate having a higher iron content can be used satisfactorily and, therefore, specifies a higher permissible iron content, aluminum sulfate with a higher iron content may be provided.

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. 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 *Lump or ground aluminum sulfate.*

5.1.2.1 If the aluminum sulfate is handled by conveyor or elevator, a mechanical sampling arrangement may be used.

5.1.2.2 If the aluminum sulfate is packaged, 5 percent of the packages shall be sampled. No sample shall be taken from a broken package.

5.1.2.3 In sampling lump material in bulk cars, sampling points shall be selected uniformly over the surface of the bulk material. Whenever possible, a minimum of 10 points at regular intervals shall be selected. At least 7 lb (3.2 kg) (about a shovelful) shall be taken at each point.

5.1.2.4 The sample must include a proportionate amount of lumps and fines. Overburden should be removed as necessary to obtain access to representative material.

5.1.2.5 Ground aluminum sulfate in bulk carload shipments or in packages should be sampled using a sampling tube or other effective device that measures at least $\frac{3}{4}$ in. (19 mm) in diameter.

5.1.2.6 The gross sample, weighing at least 50 lb (23 kg), shall be crushed, if necessary, and mixed thoroughly and divided to provide three 1-lb (0.5-kg) samples. These samples shall be sealed in airtight, moisture-proof glass containers.

5.1.2.7 The 1-lb (0.5-kg) sample of lump aluminum sulfate delivered to the laboratory shall be quartered to approximately 100 g. This amount shall be reduced by mechanical grinding or grinding rapidly in a mortar by hand to such size that all of it passes a No. 30 sieve.* After thorough mixing, this sample shall be stored in an airtight glass container. Samples of ground aluminum sulfate are split in the

* ASTM E11.

same way, except that the samples need not be reduced by further pulverizing. The necessary amounts shall be weighed from the glass container rapidly to avoid change in moisture content.

5.1.2.8 Each sample container shall be labeled to identify it and shall be signed by the sampler. A chain-of-custody form shall accompany all samples and shall be properly completed by the individuals collecting samples.

5.1.3 *Liquid alum.*

5.1.3.1 Equal portions shall be taken at five equally spaced time intervals during the unloading of the tank truck or railroad car. The total sample shall equal 2 qt (2 L).

5.1.3.2 The gross sample (2 qt [2 L]) should be thoroughly mixed, and three 1-pt (0.5-L) samples retained. They shall be sealed in airtight glass containers.

5.1.3.3 Each sample container shall be labeled to identify it and shall be signed by the sampler. A chain-of-custody form shall accompany all samples and shall be properly completed by the individuals collecting samples.

Sec. 5.2 Test Procedures—General

5.2.1 *Laboratory examination.* Laboratory examination by the purchaser of one of the three samples shall be completed within five days after receipt of the shipment.

5.2.2 *Safety.* Those unfamiliar with the hazards of any chemicals used in the test procedures that follow should consult current manufacturers' material safety and health data sheets or other appropriate material safety and health references.

Sec. 5.3 Specific Gravity

The specific gravity of aluminum sulfate solution may be determined with reasonable accuracy by using a hydrometer that meets or exceeds the ASTM standards for 117-H or by using a specific-gravity balance or a pycnometer. The aluminum sulfate manufacturer should provide information showing percentage of aluminum as Al or Al₂O₃ for aluminum sulfate solutions of different specific-gravity readings.

Sec. 5.4 Water-Insoluble Matter

5.4.1 *Procedure.*

5.4.1.1 Place a glass-fiber filter disc in a Gooch crucible, dry at 105°C to 110°C, cool in a desiccator, and weigh to the nearest 0.001 g.

5.4.1.2 Weigh out 20 g of dry aluminum sulfate or 40 g of liquid alum to the nearest 0.01 g. Dissolve the dry aluminum sulfate in approximately 150 mL of

hot distilled water and stir for a few minutes. Dilute the liquid alum in 150 mL of hot distilled water and stir.

5.4.1.3 Filter through the prepared crucible. Wash the residue with hot distilled water in at least three 25-mL rinses or until the filtrate is free from sulfates.*

5.4.1.4 Dry in an oven to constant weight (nearest 0.001 g) at 105°C to 110°C and calculate the percentage of insoluble matter using the following formula:

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

Sec. 5.5 Total Soluble Alumina and Aluminum

5.5.1 *Application.* This method is applicable to regular and iron-free grades of aluminum sulfate, liquid and solid. Provisions are made for application to acidified and high-basic liquid alums. Application is restricted to alums in which the ratio of percent of free H₂SO₄ to percent of total Al₂O₃ does not exceed 0.85.

5.5.2 *Principle.* The aluminum in the sample is complexed with an excess of standard ethylenediaminetetraacetic acid (EDTA) solution by boiling in the presence of an ammonium acetate buffer. Xylenol orange is used as an indicator.

5.5.3 *Interference.* Iron, if present, is also titrated; a correction must be made for it.

5.5.4 *Apparatus.*

5.5.4.1 Balance: analytical, readable to 0.0001 g.

5.5.4.2 Balance: triple beam or equivalent, readable to 0.1 g.

5.5.4.3 Beakers: one 1,500-mL, one 400-mL, one 250-mL, one 150-mL, one 100-mL.

5.5.4.4 Cylinders: graduated, one 500-mL, one 100-mL, one 50-mL, one 25-mL.

5.5.4.5 Bottles: dropping, polyethylene or ground-glass pipette, one 125-mL, one 60-mL.

5.5.4.6 Bottles: polyethylene, two 1-L.

5.5.4.7 Burettes: class A, one 25-mL, one 10-mL.

5.5.4.8 Flasks: Erlenmeyer, one 500-mL, one 250-mL.

5.5.4.9 Flasks: volumetric, one 1-L, one 500-mL.

5.5.4.10 Pipette: volumetric/transfer, class A, one 50-mL.

* No white precipitant after 5 percent barium chloride solution addition.

5.5.4.11 Syringe: disposable, Luer tip, without needle, 1-cm³, one syringe for each liquid-alum sample.

5.5.4.12 Other apparatus: boiling chips, glass rods, beaker tongs.

5.5.5 Reagents.

5.5.5.1 Water. Water used in the preparation of the reagents and in the procedure is either distilled or deionized.

5.5.5.2 Ammonium acetate buffer. Dissolve 500 g of ammonium acetate in 500 mL of water in a 1,500-mL beaker. Add 20 mL of glacial acetic acid.

5.5.5.3 EDTA solution, standard 0.05M. Prepare and standardize according to ASTM E200.

5.5.5.4 Sulfuric acid solution 1:1. To 25 mL of water, carefully add, while stirring, 25 mL of sulfuric acid. Allow to cool. Store in a polyethylene or ground-glass pipette dropping bottle.

5.5.5.5 Xylenol-orange indicator solution, 0.1 percent. Dissolve 0.1 g of xylenol-orange tetrasodium salt in 100 mL of water. Store in a dropping bottle. Shelf life is approximately 6 months.

5.5.5.6 Zinc sulfate solution, standard 0.05M. Dissolve 15 g of zinc sulfate heptahydrate in water, transfer to a 1-L volumetric flask, and dilute to the mark with water. Standardize the zinc solution as follows: pipette 25.0 mL of standard 0.05M EDTA solution into a 250-mL Erlenmeyer flask. Add 50 mL of water, 24 to 25 mL of ammonium acetate buffer, and 3 to 4 drops of xylenol-orange indicator solution. Titrate the EDTA with the zinc sulfate solution from a 25-mL burette to a sharp color change from a tan-yellow to red-violet. Calculate the factor F (see Eq 6) as follows:

$$F = \frac{\text{mL EDTA used}}{\text{mL ZnSO}_4 \text{ used}} \quad (\text{Eq 2})$$

5.5.6 Sample preparation for standard alum liquor and iron-free alum liquor.*

5.5.6.1 To a 500-mL Erlenmeyer flask or 400-mL beaker, add 50 mL of water.

5.5.6.2 Fill a 1-cm³ disposable syringe to the 1-cm³ mark with the liquor sample. Wipe the Luer tip of the syringe with tissue and weigh the syringe plus sample to the nearest 0.1 mg.

5.5.6.3 Transfer the sample to the beaker or flask. Reweigh the syringe to the nearest 0.1 mg and obtain the sample weight by difference.

* Where Al₂O₃ is 8.0 to 9.4 percent and free Al₂O₃ or free H₂SO₄ is less than 0.5 percent.

5.5.6.4 Add two drops of 1:1 sulfuric acid and mix.

5.5.6.5 Continue with step 1 of the procedure (Sec. 5.5.8).

5.5.7 *Sample preparation for all other alum products.*

5.5.7.1 Calculate the size sample to be used, as follows:

$$\text{grams sample} = \frac{11.5}{\text{expected \% total Al}_2\text{O}_3} \quad (\text{Eq 3})$$

Weigh a sample of this size (± 0.1 g), weighing with an accuracy of ± 0.001 g. Quantitatively transfer to a 400-mL beaker and dilute to about 300 mL with water.

5.5.7.2 Add sulfuric acid in the amounts specified below.

1. For an alum product containing no more than a few tenths of a percent of either free Al_2O_3 or free H_2SO_4 , add 18 to 20 drops of 1:1 sulfuric acid to the beaker.

2. For an acidified alum product, calculate the quantity of sulfuric acid to be added as follows (see Sec. 5.5.10.1):

$$\text{drops H}_2\text{SO}_4 = \frac{19 - \text{grams sample} \times \% \text{H}_2\text{SO}_4 \text{ in sample}}{5} \quad (\text{Eq 4})$$

Add this quantity of 1:1 sulfuric acid to the beaker.

3. For a special high-basic alum product (alum containing more than a few tenths of a percent of free Al_2O_3), calculate the quantity of sulfuric acid to be added as follows:

$$\text{drops H}_2\text{SO}_4 = 19 + (\text{grams sample} \times \% \text{free Al}_2\text{O}_3 \text{ in sample} \times 0.06) \quad (\text{Eq 5})$$

Add this quantity of 1:1 sulfuric acid to the beaker.

5.5.7.3 For dry alum samples, stir with a glass rod and heat, if necessary, to completely dissolve the sample. Cool the solution to room temperature and transfer quantitatively to a 500-mL volumetric flask. Dilute to the mark with water.

5.5.7.4 Pipette 50.0 mL of the sample solution prepared in Sec. 5.5.7.3 into a 500-mL Erlenmeyer flask or 400-mL beaker (see Sec. 5.5.10.2).

5.5.7.5 Continue with step 1 of the procedure (Sec. 5.5.8).

5.5.8 *Procedure.*

5.5.8.1 Pipette 50.0 mL of the standard 0.05M EDTA solution into the prepared sample solution. Add 25 mL of ammonium acetate buffer. Gently swirl to mix and add a boiling chip.

5.5.8.2 Heat the contents of the flask to boiling on a hot plate and continue the boiling for 2 to 3 minutes (see Sec. 5.5.10.3). Cool the solution to about room temperature.

5.5.8.3 Add 3 to 4 drops of xylenol-orange indicator solution to the flask. Titrate the excess EDTA with standard 0.05M zinc sulfate solution from a 25-mL burette to a sharp color change from a tan-yellow to a red-violet.

5.5.8.4 Determine the total iron in the sample as Fe₂O₃ using the method described in Sec. 5.6.4. (Omit this step when analyzing iron-free alum.)

5.5.9 Calculations.

$$\frac{[(\text{mL EDTA}) - (\text{mL ZnSO}_4)] \times \left(\frac{\text{moles}}{\text{L}} \text{ of EDTA} \times 5.098 \right)}{\text{grams of sample titrated}} = \begin{matrix} \% \text{ Al}_2\text{O}_3 \text{ and total Fe} \\ \text{expressed as Al}_2\text{O}_3 \end{matrix} \quad (\text{Eq 6})$$

NOTE: mL EDTA corrected to 20°C, *F* from Eq 2.

$$\% \text{ Al}_2\text{O}_3 = \% \text{ Al}_2\text{O}_3 \text{ and total Fe as Al}_2\text{O}_3 - (\% \text{ total Fe as Fe}_2\text{O}_3 \times 0.6385) \quad (\text{Eq 7})$$

5.5.10 Notes.

5.5.10.1 The computation given for the quantity of sulfuric acid to be added to acidified alum solutions will result in the addition of 0 to 19 drops of the 1:1 acid for alums being produced currently. If a particular computation should result in a small negative value for the number of drops (that is, one to three), the number of drops to be added is taken as zero. If a significant negative value (that is, four or more drops) results from the computation, add three drops of 6N NaOH solution for each drop of 1:1 H₂SO₄ computed.

5.5.10.2 The aliquot of sample solution used for titration should contain 0.110 to 0.125 g of aluminum calculated as Al₂O₃. If too large a sample size is taken for the test, the color change may occur when the first drop of zinc sulfate is added. In this event, rerun the analysis using a 25-mL aliquot of the prepared sample solution.

5.5.10.3 The sample solution, the added standard excess EDTA solution, and the ammonium acetate buffer solution are boiled together to ensure quantitative complexation of the aluminum with EDTA. A boiling time of 2 to 3 minutes is adequate. Close control over the volume of liquid is not required; however, do not let the sample evaporate to dryness.

5.5.10.4 EDTA chelates with multivalent cations on a 1:1 basis. Thus, the valence state of iron, for example, is of no consequence, and a correction is made only for total Fe expressed as Al₂O₃.

Sec. 5.6 Total Water-Soluble Iron

5.6.1 *Apparatus.* Spectrophotometer.

5.6.2 *Reagents.*

5.6.2.1 Potassium thiocyanate (KCNS) solution, 10 percent by weight.

5.6.2.2 Sulfuric acid, 1:1 by volume or 18*N*. Carefully mix, while stirring, equal volumes of sulfuric acid and water in a borosilicate glass (Pyrex) beaker. Allow to cool to room temperature. NOTE: Always add acid to water.

5.6.2.3 Potassium permanganate (KMnO₄), approximately 1.0 percent. Store in an amber glass bottle. Shelf life is approximately 6 months.

5.6.2.4 Standard iron solution, 1.0 mg Fe/mL.

5.6.2.5 Standard iron solution, 0.10 mg Fe/mL. Prepare by dilution of 1.0 mg Fe/mL standard. Include 10 mL 1:1 H₂SO₄ per liter of standard solution.

5.6.3 Calibration.

5.6.3.1 To a 100-mL volumetric flask, using a pipette, add 1.00 mL of 0.10 mg Fe/mL standard iron solution, 50 mL of water, 10.0 mL of 1:1 sulfuric acid, and 1 percent potassium permanganate, drop by drop, to obtain a persistent pink color. Add 10.0 mL of 10 percent potassium thiocyanate and dilute to the mark. Measure in a spectrophotometer at 475 nm within 10 minutes against a blank. Prepare the blank solution by transferring 10 mL of a 10 percent potassium thiocyanate solution and 10 mL of 1:1 sulfuric acid to a 100-mL volumetric flask. Dilute to the mark with water and mix.

5.6.3.2 Repeat, using 2.00, 3.00, 4.00, and 5.00 mL of 0.10 mg Fe/mL standard iron solution in successive flasks. Measure each within 10 minutes of preparation.

5.6.3.3 If the spectrophotometer is not direct-reading, plot the absorbance versus milligrams of iron on rectangular coordinate paper or plot percent transmittance versus milligrams of iron on semilog paper. Reducing the relationship to an equation is preferable.

5.6.4 Procedure.

5.6.4.1 Weigh out (accurate to 0.01 g) 10 g of liquid alum or 5 g dry alum and dilute to 1 L in a volumetric flask.

5.6.4.2 To a 100-mL volumetric flask, add 10.00 mL of sample solution, 50 mL water, 10.0 mL of 1:1 sulfuric acid, and 1 percent potassium permanganate, drop by drop, to obtain a persistent pink color.

5.6.4.3 Cool to room temperature.

5.6.4.4 Add 10.0 mL of 10 percent potassium thiocyanate and dilute to the mark.

5.6.4.5 Measure in a spectrophotometer at 475 nm within 10 minutes against a blank.

5.6.4.6 This procedure is designed to determine iron in liquid alum in the range of 0.1 to 0.7 percent Fe_2O_3 . If the iron content is outside of this range, the aliquot taken from the 1-L volumetric flask should be changed from the 10 mL stated in the previous procedure. This will change B in the equation for calculating percent Fe_2O_3 (see Sec. 5.7.2).

Sec. 5.7 Ferric Iron

5.7.1 *Procedure.* Use the procedure above for testing total iron, except do not use the potassium permanganate.

5.7.2 *Calculations.*

$$\frac{(M)(B)(C)(100)}{(W)(D)} = \% \text{ iron as } \text{Fe}_2\text{O}_3 \text{ (as is)} \quad (\text{Eq 8})$$

Where:

M = milligrams iron

B = total volume of sample solution divided by portion used:

$$1,000 \text{ mL}/10 \text{ mL} = 100$$

C = conversion factor: Fe to Fe_2O_3 = 1.4297

W = weight of sample, in grams

D = conversion factor: sample weight in grams to milligrams = 1,000

Using the values given above, Eq 8 becomes

$$\frac{(M)(100)(1.4297)(100)}{(W)(1,000)} = \frac{(M)(1.4297)}{(W)} = \% \text{ iron as } \text{Fe}_2\text{O}_3 \text{ (as is)}$$

If an equation is used to convert absorbance to milligrams, it should be combined with the above equation.

Example:

M = 0.106 mg Fe

W = 10.02 g

$$\frac{(0.106)(14.297)}{(10.02)} = 0.15\% \text{ iron as } \text{Fe}_2\text{O}_3$$

Sec. 5.8 Ferrous Iron

Subtract the ferric iron (Sec. 5.7) as ferric oxide (Fe_2O_3) from the total iron (Sec. 5.6) as Fe_2O_3 . This difference is the ferrous iron in terms of Fe_2O_3 . Multiply this figure by 0.90 to obtain the ferrous iron in terms of ferrous oxide (FeO).

Sec. 5.9 Basicity or Free Acid

5.9.1 *Direct method.** A direct method to determine basicity or acidity follows. The method is based on the decomposition of aluminum salts by an excess of neutral potassium fluoride to form two stable compounds neutral to phenolphthalein, whereas any free acid remains unaltered.

5.9.2 Reagents.

5.9.2.1 0.1 percent alcohol phenolphthalein solution.

5.9.2.2 Potassium fluoride solution. Dissolve 1,000 g of pure potassium fluoride in 1,200 mL of hot, carbon-dioxide-free distilled water and 0.5 mL phenolphthalein solution (Sec. 5.9.2.1), and neutralize with potassium hydroxide or sulfuric acid (or hydrofluoric acid[†]) until 1 mL of the solution in 10 mL of distilled water shows a faint pink color. Filter out any insoluble matter without washing and dilute to 2,000 mL with carbon-dioxide-free water. Store in a plastic bottle.

5.9.2.3 0.5*N* sulfuric acid and 0.5*N* sodium hydroxide. Standardize the alkali against the acid in approximately 40 mL of distilled water to which 10 mL of potassium fluoride solution (Sec. 5.9.2.2) has been added, using phenolphthalein (Sec. 5.9.2.1) as an indicator.

5.9.3 Procedure.

5.9.3.1 Use a solution of the sample equivalent to approximately 3.5 g of dry aluminum sulfate (or 7.0 g of liquid) or dissolve that amount in approximately 100 mL of distilled water and heat to boiling.

5.9.3.2 Add 10 mL of 0.5*N* sulfuric acid to the hot solution.

5.9.3.3 Cool to room temperature.

5.9.3.4 Add 18 to 20 mL of potassium fluoride solution and 0.5 mL phenolphthalein solution.

5.9.3.5 Titrate with 0.5*N* sodium hydroxide, drop by drop, until a slight pink color persists for 1 minute. The titration shows whether the sample is basic or acidic.

5.9.4 Calculations.

$$\frac{(\text{mL of } 0.5N \text{ H}_2\text{SO}_4 - \text{mL of } 0.5N \text{ NaOH}) \times 0.0085 \times 100}{\text{weight of sample}} = \frac{\% \text{ free alumina (Al}_2\text{O}_3)}{\text{alumina (Al}_2\text{O}_3)} \quad (\text{Eq 9})$$

* In this method, potassium hydroxide may be used as the alkali in place of sodium hydroxide on an equivalent basis.

† Hydrofluoric acid is an extremely dangerous and hazardous chemical capable of causing serious injury or death. Users of this chemical must familiarize themselves with all the necessary safety and handling precautions available from the chemical supplier.

$$\frac{(\text{mL of } 0.5N \text{ NaOH} - \text{mL of } 0.5N \text{ H}_2\text{SO}_4) \times 0.0245 \times 100}{\text{weight of sample}} = \begin{matrix} \% \text{ free sulfuric} \\ \text{acid (H}_2\text{SO}_4) \end{matrix} \quad (\text{Eq } 10)$$

5.9.5 *Interpretation of results.* Basic alumina exists if the sodium hydroxide back-titration is less than the amount of sulfuric acid added (that is, less than 10 mL). Free acid exists if the sodium hydroxide back-titration is more than the amount of sulfuric acid added (that is, greater than 10 mL). The sample is neutral if the sodium hydroxide back-titration is equal to the sulfuric acid added.

Sec. 5.10 Total Alumina Alternative Method

5.10.1 *Reagents.*

5.10.1.1 Deionized water.

5.10.1.2 25 percent potassium chloride solution. Dissolve 125 g of reagent-grade potassium chloride (KCl) in 500 mL with deionized water in a 500-mL volumetric flask.

5.10.1.3 Concentrated, reagent-grade nitric acid, HNO₃.

5.10.1.4 Stock aluminum standard, 1,000 mg/L as Al.*

5.10.1.5 Working Al standard solutions. Into a series of separate 100-mL volumetric flasks, pipette 0.0, 1.0, 2.5, and 5.0 mL of the 1,000 mg/L stock Al standard. Add 2.0 mL of 25 percent KCl solution and 1.0 mL of concentrated nitric acid to each flask. Dilute to volume with deionized water and mix. These standards have concentrations of 0, 10, 25, and 50 mg/L as Al, respectively.

5.10.2 *Apparatus.*

5.10.2.1 Atomic absorption spectrometer (AAS).

5.10.2.2 Aluminum hollow cathode lamp.

5.10.2.3 Volumetric flasks, assorted sizes.

5.10.2.4 Balance, accurate to 0.001 g.

5.10.2.5 Volumetric pipettes, assorted sizes.

5.10.3 *Instrument operating conditions.*

5.10.3.1 Wavelength: 309.3 nm.

5.10.3.2 Slit width: 0.7 nm.

5.10.3.3 Lamp energy: 15 mA.

5.10.3.4 Burner: N₂O single slot, parallel.

5.10.3.5 Flame type: N₂O-acetylene oxidizing (lean, blue).

* ThermoFisher Scientific Inc., Waltham, Mass., or equivalent.

5.10.3.6 Gas-pressure settings: Follow manufacturer's recommendations depending on the type of gas control in use.

5.10.4 *Procedure.*

5.10.4.1 Obtain the test sample and make any necessary dilutions with deionized water to allow the final dilution to be in the linear working range of the instrument (<50 mg/L as Al). Typically, the first dilution is 1:50. All sample weights should be obtained to the nearest 0.001 g. Pipette 5.0 mL of above solution into a 100-mL volumetric flask for the second dilution.

5.10.4.2 Pipette 2.0 mL of 25 percent KCl solution and 1.0 mL of concentrated HNO₃ into the flask and dilute to 100 mL with deionized water. Mix well. Prepare a blank by repeating this step in a clean and dry volumetric flask.

5.10.4.3 Optimize the instrumental parameters, such as wavelength, burner position, lamp alignment, fuel-oxidant ratios, and nebulization rate, following manufacturer's instructions.

5.10.4.4 Aspirate and measure the absorbance of the blank and a set of working standard solutions. The concentration range of the standards is 0 to 50 mg/L as Al.

5.10.4.5 Aspirate and measure the absorbance of the diluted test-sample solutions.

5.10.4.6 Prepare a calibration curve by plotting on linear graph paper absorbance versus concentration of Al in mg/L for the standards. Obtain the concentration of Al in mg/L in the diluted test samples from sample absorbance values and the corresponding concentrations on the calibration curve. Alternatively, a curve can be fit to the data using a scientific calculator with linear regression capability. Sample concentration can be read directly from some instruments with automatic calibration.

5.10.5 *Calculations.*

5.10.5.1 Concentration in mg/L of Al in test sample:

$$\text{mg/L as Al} = (\text{mg/L in diluted test sample}) (D)$$

Where:

D = any appropriate dilution factors

$$5.10.5.2 \text{ Percent Al} = \frac{\text{mg/L as Al}}{10,000}$$

$$5.10.5.3 \text{ Percent Al}_2\text{O}_3 = (\text{percent Al}) (1.89)$$

5.10.6 *Performance data.*

5.10.6.1 Range: 0 to 50 mg/L as Al in aspirated solutions.

5.10.6.2 Precision. Sample deviation is 1.18 percent relative as determined by three replicates of four samples by one operator.

Sec. 5.11 Notice of Nonconformance

If the aluminum sulfate 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 5 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 on retesting do not agree with the 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.

Material not complying with the requirements of this standard and the purchaser's documents may be rejected. Replacement and retesting shall be accomplished in accordance with the purchaser's documents.

Because the exact concentration of the active ingredient in specific shipments of some materials can vary within an acceptable range (thereby conforming to the standard), the purchase documents between purchaser and supplier should address treatment of concentration variation.

SECTION 6: DELIVERY*

Sec. 6.1 Marking

Each shipment of material shall be identified as to product, grade, net weight, name and address of manufacturer, and brand-name. Packages or containers shall show a lot number and identification of manufacturer. All markings on packages, containers, or bulk shipments shall conform to applicable laws and regulations,

* Governmental packaging and marking references reflect US requirements. Users of this standard outside the United States should verify applicable local 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.

including requirements established by the US Occupational Safety and Health Administration (OSHA) and US Department of Transportation (DOT).

Sec. 6.2 Packaging and Shipping

Packaging and shipping of aluminum sulfate shall conform to current federal, state or provincial, and local regulations.

6.2.1 *Lump or ground.* Lump or ground aluminum sulfate may be shipped in bulk, in lined steel or fiber drums, or in multiwall paper bags. The net weight of the packages shall not deviate from the recorded weight by more than 2.5 percent. If exception is taken to the weight of the material received, the certified unit weight shall be based on at least 10 percent of all the packages shipped and selected at random.

6.2.2 *Liquid.* Liquid alum may be shipped in tank trucks or railroad tank cars.* The tanks shall be rubber-lined or made of stainless steel or any other suitable material that will not be attacked by the acidity of the liquid.

6.2.2.1 Tank trucks or tank cars shall be in suitable condition for hauling liquid alum and shall not contain any substances that might affect the use or usefulness of the liquid alum in treating municipal or industrial water supplies.

6.2.2.2 If less-than-carload quantities are purchased, the supplier and the purchaser shall agree on the size and type of container.

6.2.3 *Contamination.* Bulk or semibulk containers, such as railcars, tank cars, trailers, tank trucks, and unit hoppers, shall be carefully inspected before loading of the chemical by the supplier to ensure no contaminating material exists.

6.2.4 *Certificate of weight.* The purchaser may require that bulk shipments be accompanied by weight certificates of certified weighers or the weights may be checked by the purchaser on delivery.

6.2.5 *Security requirements for nonbulk shipments.* Packaged product shall be stored, shipped, and delivered in tamper-evident packaging as defined in Section 3, item 12, or an alternative method or methods may be agreed on by the manufacturer and purchaser that provide a reasonable assurance of protection against tampering.

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

* Tank cars used in areas where air temperatures below 5°F (–15°C) prevail should be provided with insulation.

6.2.6.1 Seals. Bulk quantities of product may be sealed with 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.

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

6.2.6.3 Alternative method. An alternative method (or methods) agreed on by the manufacturer and purchaser may be used that provides reasonable assurance of protection against tampering.

Sec. 6.3 Affidavit of Compliance

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

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