ANSI/AWWA B408-10
(Revision of ANSI/AWWA B408-03)

American Water Works Association

The Authoritative Resource on Safe Water®

*AWWA Standard*

# Liquid Polyaluminum Chloride

STANDARD
Since 1881

Approved American National Standard
ANSI

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# Foreword

*This foreword is for information only and is not a part of ANSI*/AWWA B408.*

## I. Introduction.

I.A. *Background.* This standard covers a variety of products based on the chemistry of aluminum salt solutions, where polyaluminum oligomers become the predominant and defining form of the aluminum cation. The characteristics of the corresponding anion in these oligomer products, although more commonly entirely chloride, may be instead sulfate or even some ratio of these two anions in combination. There are, additionally, formulations of these products wherein a polymer (organic polyelectrolyte) is blended in with inorganic oligomers to provide enhancements of specific functionalities.

This standard will focus on polyaluminum chloride (PACl) products—as they are the predominant presentation of this chemistry—commercially available as liquids in the United States and Canada. This standard covers liquid PACl for use in municipal and industrial water supplies. As a note, certain parts of this standard may be used wholly, or in part, to aid the user and producer to define and measure a specific related product chemistry being offered, though only as is appropriate and mutually agreed on beforehand by both the purchaser and supplier/manufacturer. Given these conditions, it may be necessary for the purchaser to acquire and utilize additional or different information and methodologies from that offered in this standard to allow the proper management of products in this group.

Some inorganic coagulants are mixed with polymers to produce blends.[†]

*Polyaluminum chloride* is a misnomer, however, because these products contain a mixture of polymers (actually aggregates of oligomers) of aluminum chloride hydroxide (Chemical Abstract Service [CAS] No. 1327-41-9) with the empirical formula $Al_n(OH)_mCl_{(3n-m)}$ for $0 < m < 3n$. At least the following five CAS numbers, 1327-41-9, 10284-64-7, 14215-15-7, 39290-78-3, and 12042-91-0, have been used for various types of PACl. *Basicity* (hydroxyl number) refers to the average number of hydroxide ions per aluminum atom in the PACl molecules or m/n. In some PACl products, an anion other

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

[†] Characterization of Inorganic Coagulant/Polymer Blends Using Refractive Index and Specific Gravity Measurements. 2004. Polyelectrolytes Standards Committee Report, B.S. Johnson, Chair. *J. AWWA* 96:170.

than chloride, such as sulfate, may also be present, but this anion should be present in much lower concentrations than the chloride ion if the product is being sold as PACl.

The basicity of PACl products can range from 0 to 2.5, though the basicity should not be 0 if the product is being sold as PACl. The basicity is converted to "percent basicity" using the following formula:

$$\text{percent basicity} = \frac{(\text{OH/Al}) \times 100}{3}$$

Where:

Al and OH are expressed as moles/liter

The values for percent basicity of liquid PACl products can range from about 10 to 83.3. The basicity of the product does not necessarily relate directly to product performance, so the highest basicity may not give the best performance in a particular application.

Polyaluminum chloride products contain varying amounts of PACl, the concentration of which is conventionally expressed as "percent by weight as Al" or "percent as Al." The range of PACl content in PACl products is about 2.5 to 13 percent as aluminum (5 to 25 percent as $Al_2O_3$).

Polyaluminum chloride can be commercially manufactured from a number of aluminum-containing raw materials, including aluminum metal, alumina trihydrate, aluminum chloride, aluminum sulfate, and combinations of these. The products can contain by-product salts, such as sodium/calcium/magnesium chloride or sulfate, depending on the manufacturing process. The presence of these salts is not harmful to product performance or to those handling the product. Recognizing that the purity of PACl can vary with the manufacturing process, the purchaser should ask the supplier for information concerning potential impurities.

This standard provides methods for analysis of active PACl expressed in percent as aluminum (or $Al_2O_3$), percent basicity, turbidity, and specific gravity.

I.B. *History.* In 1988, the AWWA Standards Committee on Iron Salts, Aluminum Salts, and Related Coagulant Aids organized a subcommittee to prepare a standard for PACl products. The first draft of the standard was reviewed in 1989, and the final draft was approved in 1992. The first edition of ANSI/AWWA B408, Standard for Liquid Polyaluminum Chloride, was approved by the AWWA Board of Directors on Jan. 31, 1993. The second edition was approved on June 21, 1998. The third edition was approved on June 19, 2003. This edition was approved June 20, 2010.

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.

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 B408 addresses additives requirements in Sec. 4.4 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. 4.4.2 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

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

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

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 PACl is a clear to slightly hazy solution that is acidic and corrosive to common metals. Suitable materials for construction of storage and handling facilities include synthetic rubber-lined steel, corrosion-resistant fiberglass-reinforced plastics (FRP), ceramics, tetrafluoroethylene polymer (PTFE), polyvinylidene fluoride (PVDF), polyethylene, polypropylene, and polyvinyl chloride (PVC). Steel (stainless and mild), aluminum, nickel, copper, or brass are *not* suitable. The supplier should be contacted for recommendations on appropriate materials of construction for the storage, handling, and packaging of any specific product.

Contact with PACl products may cause burns or irritation to the eyes or skin. Protective clothing, such as rubber gloves, boots, pants, and jackets, and eye protection, such as goggles or face masks, are recommended.

**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.* The following information should be provided by the purchaser:

1. Standard used—that is, ANSI/AWWA B408, Liquid Polyaluminum Chloride, of latest revision.
2. Details of other federal, state or provincial, and local requirements (Sec. 4.1).
3. Percent active PACl expressed as percent Al or as percent $Al_2O_3$.
4. Whether compliance with NSF/ANSI 60, Drinking Water Treatment Chemicals—Health Effects, is required.
5. 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 5.7, 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 the shipment.
6. Method of shipping, packaging, and container size (Sec. 6.2).

7. Whether alternative security measures have been adopted to replace or augment the security measures set out in Sec. 6.2.2 and 6.2.3.

8. An affidavit of compliance or certified analysis, or both, 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 changes 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 (Sec. 4.1).

2. Inclusion of a requirement for tamper-evident packaging (Sec. 6.2.2 and 6.2.3).

3. Additional information on the types of products covered in this standard (I.A).

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

ANSI/AWWA B408-10
(Revision of AWWA B408-03)

American Water Works
Association

*AWWA Standard*

# Liquid Polyaluminum Chloride

## SECTION 1: GENERAL

### Sec. 1.1 Scope

This standard describes polyaluminum chloride (PACl) in aqueous (liquid) form for use in the treatment of potable water, wastewater, and reuse or reclaimed water.

### Sec. 1.2 Purpose

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

### Sec. 1.3 Application

This standard can be referenced in specifications for purchasing and receiving liquid PACl and can be used as a guide for testing the physical and chemical properties of liquid PACl samples. The stipulations of this standard apply when this document has been referenced and then only to liquid PACl used in the treatment of potable water, wastewater, and reuse 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 within the standard. In any case of conflict, the requirements of this standard shall prevail.

*Analytical Methods for Atomic Absorption Spectrophotometry.* Perkin Elmer.

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

*Standard Methods for the Examination of Water and Wastewater.* APHA,[‡] AWWA, and WEF.[§]

## 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 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 drums and tank cars constitute two forms of tamper-evident packaging.

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

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

[‡] American Public Health Association, 800 I Street NW, Washington, DC 20001.

[§] Water Environment Federation, 601 Wythe Street, Alexandria, VA 22314.

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.

## SECTION 4: REQUIREMENTS

### Sec. 4.1 Materials

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

### Sec. 4.2 Physical Requirements

Liquid PACl products are clear to slightly hazy solutions, free of visible foreign matter or sediment. The specific gravity of the solution shall be in the range of 1.1 to 1.4. Specific gravity will vary with PACl content and by-product salt content.

### Sec. 4.3 Chemical Requirements

Liquid polyaluminum chloride–type products can have variable functions and characteristics. The determination of quality and conformance to agreed-upon requirements will often be product-specific, with key parameters being determined using various analytical methods. Accordingly, alternative methods can be utilized to determine key functions and constituents.

The polyaluminum chloride content in liquid PACl products shall be from 2.5 to 13 percent by weight expressed as aluminum (5 to 25 percent by weight PACl expressed as $Al_2O_3$). The percent basicity of the PACl shall be from 10 to 83 percent. The solution shall have turbidity less than 50 ntu.

### Sec. 4.4 Impurities*

4.4.1 *General.* The liquid PACl product supplied according to this standard shall contain no substances in quantities capable of producing deleterious or injurious effects to the health of those consuming water that has been properly treated with liquid PACl product. This standard applies to liquid PACl produced by currently recognized methods of manufacture and from suitably pure raw materials. If careless manufacturing procedures or other raw materials are used, impurities could be present that might be inconsistent with good water treatment

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* See Sec. I.C of the foreword.

practices. In such cases, additional tests by the manufacturer may be required to demonstrate that the product is suitable for water treatment purposes.

4.4.2 *Product certifications.* Liquid PACl 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. 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.* Sample at the point of destination.

5.1.2 *Liquid PACl.* Collect three 0.5-L (1-pint) samples from each tank car, tank truck load, semibulk container, or drum. Store these samples in airtight, moistureproof glass containers, each clearly identified with a label signed by the sampler.

5.1.3 *Distribution.* One of the three sealed PACl samples is for immediate use by the purchaser for testing of the shipment according to Section 5. Retain the two remaining material samples for future use, if necessary, according to Sec. 5.8.

5.1.4 *Retention.* Retain material samples for at least 30 days after the date of receipt of the shipment before discarding.

5.1.5 *Test samples.* Obtain test samples of liquid PACl from the sealed material sample delivered to the laboratory. Unseal the material sample container only when necessary to remove quantities of the product for testing. Seal the container immediately after the test sample is removed. Mix the PACl sample thoroughly before each test portion is withdrawn for analysis. Store the test sample in an airtight glass container.

CAUTION: PACl is irritating to skin and eyes.

## Sec. 5.2 Test Procedures—General

5.2.1 *Completion.* Laboratory examination of the sample by the purchaser shall be completed within five days after receipt of the shipment.

5.2.2 *Test procedures.* Methods of testing PACl 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 of this standard shall prevail.

While some material functions are based entirely on chloride anion, other materials can be based entirely on, or share anion concentration with, sulfate or other anions. While the ongoing concentration measurement of any of these constituents can provide an indication of product consistency, they may not be indicative of product effectiveness or ultimate functionality.

Often, simplified methods such as specific gravity and turbidity can provide quick or early indications of product quality problems or contamination.

## Sec. 5.3 PACl Content as Percent Al

5.3.1 *Reagents.*

1. Deionized (or distilled) water.

2. Aluminum standard (1 mg Al/mL). Weigh 1 g aluminum wire to the nearest 0.0001 g and transfer to a 1,000-mL volumetric flask. Add 50 mL 1:1 hydrochloric acid, swirl the flask, and allow the reaction to proceed until all of the aluminum has dissolved. (Warning: Hydrogen gas evolves during this reaction. Do not cap the flask. Keep the flask in a fume hood and away from spark, heat, and flame during this reaction.) Dilute with deionized water to the mark at 20°C and mix well. The concentration of the standard will be the actual weight of aluminum in milligrams divided by 1,000 mL. Store in a polyethylene bottle.

3. Ammonium acetate buffer. Weigh 500 g ammonium acetate in a 1,500-mL beaker and add 500 mL of deionized water. Stir with a glass rod to dissolve most of the ammonium acetate, then continue stirring with a magnetic stirrer until completely dissolved. Add 20 mL glacial acetic acid and mix. Store in a polyethylene bottle.

4. Xylenol orange indicator, dry powder. Weigh 60.0 g sodium chloride and transfer to a clean sheet of paper. Add 0.1 g xylenol orange tetrasodium salt. By lifting alternate corners of the paper, thoroughly mix the two solids. Transfer the mixture, a small quantity at a time, to a mortar and grind it to a fine powder. Transfer each ground portion to a 120-mL, wide-mouth, screw-cap bottle for storage. The dry powder is stable indefinitely. To prepare the xylenol orange indicator (1 g/L aqueous), dissolve 0.1 grams of xylenol orange tetrasodium salt in 100 mL of water. Store in a dropping bottle. Discard after one month.

5. Zinc sulfate, approximately 0.05*M*. Dissolve 15 g zinc sulfate heptahydrate in deionized water in a 1,000-mL volumetric flask, dilute to the mark with deionized water, and mix. Store in a polyethylene bottle.

Pipette 25.0 mL standardized ethylenediaminetetraacetic acid (EDTA) solution (Sec. 5.3.1[8]) into a 250-mL beaker or Erlenmeyer flask. Add 50 mL deionized water and 25 mL ammonium acetate buffer and mix. Add 0.1 to 0.2 g of the dry xylenol orange powder or 4 drops of the aqueous xylenol orange indicator, mix, and titrate with the zinc sulfate solution until the color changes from tan-yellow to red-purple. Calculate for factor $F_1$ as follows:

$$F_1 = \frac{25.0\ \text{mL EDTA}}{\text{mL ZnSO}_4} \qquad \text{(Eq 1)}$$

6. Sulfuric acid, concentrated.

7. Hydrochloric acid solution, 1:1. To 25 mL deionized water, add 25 mL concentrated hydrochloric acid and mix. Always add acid to water.

8. EDTA solution, approximately 0.05*M*. Add 18.6 g disodium ethylenediaminetetraacetate dihydrate to about 700 mL of deionized water in a 1-L beaker and stir on a magnetic stirrer until the EDTA is dissolved. Transfer to a 1,000-mL volumetric flask, dilute to the mark with deionized water, and mix. Store in a polyethylene bottle.

Standardize as follows: Pipette 25.0 mL aluminum standard (Sec. 5.3.1[2]) into a 250-mL beaker or Erlenmeyer flask. Pipette 50.0 mL EDTA solution into the same beaker or flask and mix. Add 25-mL ammonium acetate buffer (Sec. 5.3.1[3]) and a boiling chip and boil for 2 min. Cool, add 0.1 to 0.2 g of the dry xylenol orange powder or 4 drops of the aqueous xylenol orange indicator, mix, and titrate with zinc sulfate solution (Sec. 5.3.1[5]) using a 25-mL burette until the color changes from tan-yellow to red-purple.

$$\text{molarity of EDTA} = \frac{(25.0\ \text{mL Al standard})(\text{mg Al/mL})}{(B - A)(26.98)} \qquad \text{(Eq 2)}$$

Where:

$A = \text{mL ZnSO}_4 \times F_1$

$B = 50.0\ \text{mL EDTA}$

5.3.2 *Apparatus.*

1. Analytical balance accurate to 0.0001 g.
2. 1,000-mL volumetric flask.
3. 25-, 50-, 100-, 500-mL graduated cylinders.
4. 1,500-mL beaker, 1,000-mL beaker.
5. Glass rod.
6. Magnetic stirring plate and bar.
7. Mortar and pestle.
8. 120-mL, wide-mouth, screw-cap bottle.

9. 1,000-mL polyethylene bottles.
10. 150-mL dropping bottle.
11. Balance accurate to 0.01 g.
12. 25-mL pipette.
13. Boiling chips and hot plate.
14. 25-mL burette.
15. 250-mL beaker or Erlenmeyer flask.
16. 500-mL Erlenmeyer flask.

5.3.3 *Procedure.*

1. Calculate the size sample to be used, as follows:

$$\text{grams sample} = \frac{6.095}{\text{expected \% total Al}} \qquad \text{(Eq 3)}$$

Example: If expecting 5.3 percent Al, then

$$\frac{6.095}{5.3} = 1.15 \text{ g sample}$$

2. Weigh a sample of this size to the nearest 0.01 g. Quantitatively transfer it to a 500-mL Erlenmeyer flask and dilute with deionized water to about 200 mL.

3. Add 25 drops of concentrated sulfuric acid and mix.

4. Pipette 50.0 mL of the standardized 0.05*M* EDTA into the prepared sample solution. Mix.

5. Add 25 mL of ammonium acetate buffer.

6. Heat the contents to boiling and continue boiling for 2 to 3 min.

7. Cool the solution to room temperature.

8. Add a few drops of the aqueous xylenol orange indicator or 0.1 to 0.2 g of the dry xylenol orange powder indicator to the flask and mix.

9. Titrate the excess EDTA with 0.05*M* zinc sulfate until the color changes from a tan-yellow to red-violet.

NOTE: If too large a sample is taken, the color change may occur when the first drop of zinc sulfate is added. In this event, rerun the analysis using a smaller sample.

5.3.4 *Calculation.*

$$\text{\% total Al} = \frac{[\text{mL EDTA} - (\text{mL } ZnSO_4 \times F_1)]\ 2.698 \times \text{molarity of EDTA}}{\text{grams of sample titrated}} \qquad \text{(Eq 4)}$$

Sample calculation:

$$\frac{[50.0 - (5.7 \times 1.0546)]\ 2.698 \times 0.0505}{1.15 \text{ grams}} = 5.22\% \text{ total Al}$$

## Sec. 5.4 PACl Content as Percent $Al_2O_3$—Benchmark Method

Refer to *Analytical Methods for Atomic Absorption Spectrophotometry* when using this procedure.

5.4.1 *Reagents.*

1. Deionized water.

2. 25 percent potassium chloride solution. Dissolve 125 g reagent-grade potassium chloride, KCl, in 500 mL deionized water in a 500-mL volumetric flask.

3. Concentrated, reagent-grade nitric acid, $HNO_3$.

4. Stock aluminum standard, 1,000 mg/L as Al. Obtain pre-made from Fisher Scientific Corporation (Pittsburgh, Pa.), or equivalent.

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 25 percent KCl solution and 1.0 mL 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.

5.4.2 *Apparatus.*

1. Atomic absorption spectrophotometer (AAS).
2. Aluminum hollow cathode lamp.
3. Volumetric flasks, assorted sizes.
4. Balance, accurate to 0.001 g.
5. Volumetric pipettes, assorted sizes.

5.4.3 *Instrument operating conditions.*

1. Wavelength: 309.3 nanometers (nm).
2. Slit width: 0.7 nm.
3. Lamp energy: 15 milliamperes (mA).
4. Burner: $N_2O$ single slot, parallel.
5. Flame type: $N_2O$–acetylene oxidizing (lean, blue).
6. Gas-pressure settings: follow manufacturer's recommendations depending on the type of gas control in use.

5.4.4 *Procedure.*

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 the 1:50 diluted sample into a 100-mL volumetric flask for the second dilution.

2. Pipette 2.0 mL 25 percent KCl solution and 1.0 mL concentrated $HNO_3$ into the flask and dilute to 100 mL with deionized water. Mix well. Prepare a blank by repeating this step in a clean, dry volumetric flask.

3. Optimize the instrumental parameters, that is, wavelength, burner position, lamp alignment, fuel-oxidant ratios, and nebulization rate, following the manufacturer's instructions.

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

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

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.

5.4.5 *Calculations.*

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

2. Percent Al = (mg/L as Al)/10,000.

3. Percent $Al_2O_3$ = (percent Al) (1.89).

5.4.6 *Performance data.*

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

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

## Sec. 5.5 PACl Basicity

The basis for this method is neutralization of alkalinity in the sample with excess hydrochloric acid solution and back-titration with sodium hydroxide solution in the presence of potassium fluoride. Potassium fluoride prevents the hydrolysis of aluminum salts by the complexation of the aluminum cations. Phenolphthalein is used as an indicator. Using this alkalinity value (percent OH) and the percent aluminum, the percent basicity of the sample can be calculated. Alternatively, the determination of the molar ratio of aluminum and chloride, where no other anions are present, can be used to calculate the percent basicity. Sec. 5.7 addresses the determination of the chloride in PACl.

5.5.1 *Reagents.*

1. 1.0*N* hydrochloric acid solution, HCl. Fisher Scientific Corporation certified or equivalent.

2. 1.0*N* sodium hydroxide solution, NaOH. Fisher Scientific Corporation certified or equivalent.

3. Potassium fluoride, 50 percent solution. Dissolve 250 g anhydrous KF in 375 mL of hot, deionized water or dissolve 405 g $KF{\cdot}2H_2O$ in 220 mL of hot, deionized water. Cool and dilute to 500 mL with deionized water. Filter if necessary and store in a polyethylene bottle.

4. Phenophthalein indicator. Dissolve 1.0 g phenolphthalein in 100 mL ethanol.

5.5.2 *Apparatus.*

1. Erlenmeyer flasks, 250 mL.
2. Volumetric pipettes or Repipets, 25 and 50 mL.
3. Graduated cylinder, 50 mL.
4. Hot plate.
5. Burette, 50 mL with 0.1-mL subdivisions.
6. Magnetic stir bars, small.
7. Top-loading balance accurate to 0.01 g.
8. Magnetic stirring plate.

5.5.3 *Procedure.*

NOTE: The volume of sample used in this method is difficult to determine, as it depends on both percent Al and the percent basicity. In general, Table 1 provides guidelines.

1. Pipette 5.0 mL of the solution to be analyzed into a tared 250-mL Erlenmeyer flask. Reweigh and record weight difference to 0.01 g. Carefully add

**Table 1 PACl basicity sample size guideline**

| Sample Volume *(mL)* | Expected Aluminum *(percent)* | Expected Basicity *(percent)* |
|---|---|---|
| 5 | 5.3–8.0 | 00–50 |
| 3 | 8.0–10.6 | 50–70 |
| 2 | >10.6 | >70 |

magnetic stir bar. Select a second flask for a blank and add a stir bar. Mark the flasks for identification.

2. Add 50 mL deionized water to each Erlenmeyer flask.

3. Using a volumetric pipette, add 50.0 mL 1.0*N* HCl to each Erlenmeyer flask.

4. Place both flasks on a preheated hot plate (high heat). After boiling for 5 min, immediately remove the flasks from the hot plate and cool to room temperature.

5. Using a volumetric pipette or Repipet, add 25.0 mL 50 percent KF to each flask, and swirl liquid in flask to mix.

6. Add 4 drops phenophthalein indicator and titrate each flask with 1.0*N* NaOH while stirring to a faint pink end point. The end point should persist for 1 min. If the end point for the sample is detected without addition of titrant, repeat the above procedure using less sample.

5.5.4 *Calculations.*

$$\%\ \mathrm{OH} = \frac{(\text{titrant mL}_{\text{blank}} - \text{titrant mL}_{\text{sample}})(1.7)}{\text{weight of sample in grams}} \qquad \text{(Eq 5)}$$

$$\%\ \text{basicity} = \frac{\%\ \mathrm{OH}\ (52.91}{\%\ \mathrm{Al}\ \text{(see Sec. 5.3 or Sec. 5.4)}} \qquad \text{(Eq 6)}$$

5.5.5 *Performance data.*

Range: 0 to 20 percent OH or 0 to 83 percent basicity.

## Sec. 5.6 Product Turbidity

5.6.1 *Apparatus.*

1. Turbidimeter, Hach Company (Loveland, Colo.) Model 18900 or equivalent.

2. Sample tubes for turbidimeter.

3. Turbidity standards, 10 and 100 ntu.

5.6.2 *Procedure.*

1. See Section 2130B Nephelometric Method *Standard Methods for the Examination of Water and Wastewater* (latest edition).

## Sec. 5.7 Chloride

5.7.1 *Reagents.* Use reagent grade unless otherwise specified.

1. Water used in the preparation of reagents and in the procedure is deionized (or distilled).

2. Nitric acid, concentrated.

NOTE: Nitric acid is highly corrosive and can cause severe burns. Concentrated nitric acid emits hazardous nitrogen oxides. The acid is also a strong oxidant; spillage may cause a fire or liberate dangerous gases.

3. Sodium chloride solution, standard, 0.05*N*. Dry approximately 3.1 g of sodium chloride in a weighing dish in an oven at 105° to 110°C for 2 hr. Cool the crystals to room temperature in a desiccator.

Weigh 2.9221 g of the dried sodium chloride to the nearest 0.0001 g in a tared weighing bottle. Using a water-wash bottle, quantitatively transfer the crystals through a powder funnel into a 1,000-mL volumetric flask. Add about 150 mL deionized water and swirl to dissolve the crystals. Dilute to the mark at 20°C with deionized water and mix thoroughly. Store the solution in a polyethylene bottle.

4. Iron indicator solution. To a 1,500-mL beaker add 62 g ferric ammonium sulfate, $FeNH_4 (SO_4)_2 \cdot 12H_2O$, and 500 mL of deionized water. Stir until the crystals are dissolved. Add 438 mL concentrated nitric acid and mix. Store the solution in a polyethylene bottle.

5. Potassium thiocyanate solution, 0.05*N*. Weigh 4.86 g potassium thiocyanate to the nearest 0.01 g in a tared weighing bottle. Quantitatively transfer the crystals through a powder funnel into a 1,000-mL volumetric flask. Add about 250 mL deionized water and swirl to dissolve the crystals. Dilute to the mark with deionized water and mix thoroughly. Store the solution in a polyethylene bottle.

6. Benzyl alcohol. Dispense from an amber-glass dropping bottle. Benzyl alcohol vapor is irritating to the eyes, nose, and throat; the liquid can burn skin and eyes and also may be readily absorbed through the skin. It is combustible. Keep the reagent away from heat or flame.

7. Silver nitrate solution, standard 0.05*N*. Weigh 8.495 g silver nitrate to the nearest 0.001 g in a tared weighing bottle. Quantitatively transfer the crystals through a powder funnel into a 1,000-mL volumetric flask. Add about 250 mL deionized water and swirl to dissolve the crystals. Dilute to the mark with deionized water and mix thoroughly. Store the solution in a tightly stoppered amber-glass bottle. Standardize the solution as follows:

a. Pipette 25.0 mL of the standard 0.05*N* sodium chloride solution into each of three 500-mL glass-stoppered Erlenmeyer flasks. Determine the temperature of the sodium chloride solution and correct the volume pipetted to 20°C (Sec. 5.7.5[3], Table 2). Carry each flask through the remaining steps of the procedure.

**Table 2 Temperature corrections for volumetric solutions**

| | mL Correction per mL Used* | | | Factor† | | |
|---|---|---|---|---|---|---|
| Temperature °C | *N*/20 or less solutions | *N*/10 solutions | *N*/1 solutions | *N*/20 or less solutions | *N*/10 solutions | *N*/1 solutions |
| 15 | +0.0008 | +0.0008 | +0.0011 | 1.0008 | 1.0008 | 1.0011 |
| 16 | +0.0006 | +0.0007 | +0.0009 | 1.0006 | 1.0007 | 1.0009 |
| 17 | +0.0005 | +0.0005 | +0.0007 | 1.0005 | 1.0005 | 1.0007 |
| 18 | +0.0003 | +0.0004 | +0.0005 | 1.0003 | 1.0004 | 1.0005 |
| 19 | +0.0002 | +0.0002 | +0.0003 | 1.0002 | 1.0002 | 1.0003 |
| 20 | 0.0000 | 0.0000 | 0.0000 | 1.0000 | 1.0000 | 1.0000 |
| 21 | −0.0002 | −0.0002 | −0.0003 | 0.9998 | 0.9998 | 0.9997 |
| 22 | −0.0004 | −0.0004 | −0.0005 | 0.9996 | 0.9996 | 0.9995 |
| 23 | −0.0006 | −0.0006 | −0.0008 | 0.9994 | 0.9994 | 0.9992 |
| 24 | −0.0008 | −0.0009 | −0.0012 | 0.9992 | 0.9991 | 0.9988 |
| 25 | −0.0010 | −0.0011 | −0.0015 | 0.9990 | 0.9989 | 0.9985 |
| 26 | −0.0013 | −0.0013 | −0.0018 | 0.9987 | 0.9987 | 0.9982 |
| 27 | −0.0015 | −0.0016 | −0.0022 | 0.9985 | 0.9984 | 0.9978 |
| 28 | −0.0018 | −0.0019 | −0.0025 | 0.9982 | 0.9981 | 0.9975 |
| 29 | −0.0020 | −0.0021 | −0.0029 | 0.9980 | 0.9979 | 0.9971 |
| 30 | −0.0023 | −0.0024 | −0.0033 | 0.9977 | 0.9976 | 0.9967 |

*Apply correction to mL used to obtain corrected volume, or

†Multiply factor by mL used to obtain corrected volume.

b. Add 100 mL deionized water and 5 mL iron indicator solution. Swirl to mix. Using a 50-mL class A burette, add 27.0 mL the silver nitrate solution being standardized, while swirling vigorously. Record the exact volume of the silver nitrate solution added to the nearest 0.01 mL, determine its temperature, and correct the volume to 20°C (Sec. 5.7.5[3], Table 2).

c. Add 10 drops benzyl alcohol, stopper the flask, and shake it vigorously for 30 sec. Place a 500-mL Erlenmeyer flask beneath a long-stem funnel fitted with Whatman 42 or equivalent filter paper. Transfer the contents from the stoppered flask to the funnel. Rinse this flask with about 5 mL of deionized water, stopper the flask, and thoroughly shake the rinse water around in the flask. Transfer the rinse water to the filter. Repeat the rinsing and the transfer of the rinse water. A complete transfer of the precipitate to the filter is not necessary. Wash the precipitate on the filter twice with cold water and collect the washings with the filtrate. Using a class A burette, slowly and while constantly swirling, titrate the combined filtrate, rinse water, and washings with $0.05N$ potassium thiocyanate solution until a faint red color persists.

$$\text{normality of AgNO}_3 = \frac{(\text{mL NaCl at 20°C})(0.0500)}{(\text{mL AgNO}_3\text{ at 20°C})(\text{mL KSCN} \times F_2)} \qquad \text{(Eq 7)}$$

d. Determine the factor $F_2$ as follows: Add about 9 mL of the silver nitrate solution from the 50-mL burette to a 250-mL Erlenmeyer flask. Add 100 mL water and 5 mL iron indicator solution. Swirl to mix. Titrate slowly with the KSCN solution from the 10-mL burette, while swirling constantly, until a faint reddish color persists.

e. Average the three results.

f. Restandardize monthly.

$$F_2 \frac{\text{mL AgNO}_3}{\text{mL KSCN}} \qquad \text{(Eq 8)}$$

g. Repeat the determination and use the average factor rounded off to the nearest 0.001-mL $AgNO_3$/mL KSCN. Determine $F_2$ each time a new KSCN or $AgNO_3$ solution is prepared.

5.7.2 *Apparatus.*

1. Analytical balance accurate to 0.0001 g.
2. Weighing dish.
3. Drying oven.
4. Desiccator.
5. 1,500-mL beaker.
6. 1,000-mL volumetric flask.
7. 500-mL Erlenmeyer flask with stoppers.
8. 50-mL class A burette.
9. 50-mL pipette.
10. 250-mL Erlenmeyer flask.
11. 250-mL beaker.
12. 100-mL volumetric flask.

5.7.3 *Procedure.*

NOTE: Anions, such as bromide, iodide, ferricyanide, ferrocyanide, and thiocyanate, which form silver salts that are less soluble than silver chloride in dilute nitric acid solution, are also titrated and give rise to positive interference. None of these anions is expected to be present in the sample in significant amounts.

Bivalent mercury causes negative interference by forming a stable complex with thiocyanate ion.

1. Weigh 4 g of the sample to the nearest 0.001 g in a 250-mL beaker. Dilute to about 50 mL with deionized water. Acidify with 5 mL concentrated nitric acid and boil for 5 min. Cool and transfer the sample to a 100-mL volumetric flask and dilute to volume. Pipette 10.00 mL of this solution to a 500-mL glass-stoppered Erlenmeyer flask and dilute to about 150 mL with deionized water.
2. Add 5 mL iron indicator solution to the flask. Swirl to mix.
3. From a zeroed, 10-mL class A burette, add 1.0 mL 0.05*N* potassium thiocyanate solution and swirl to mix. Do not rezero this burette; the titration with thiocyanate solution will continue in Sec. 5.7.3(8).
4. Fill and zero a 50-mL class A burette with standard 0.05*N* silver nitrate solution. Titrate slowly while swirling vigorously until the red color is completely discharged. Add an additional 2-mL standard 0.05*N* silver nitrate solution. Record the exact volume of silver nitrate solution to the nearest 0.01 mL.
5. Add 10 drops benzyl alcohol, stopper the flask, and shake it vigorously for 30 sec (see note, Sec. 5.5.3).
6. Place a 500-mL Erlenmeyer flask beneath a long-stem funnel fitted with Whatman 42 or equivalent filter paper.
7. Transfer the contents from the stoppered flask to the funnel. Rinse this flask and thoroughly shake the rinse water around in the flask. Transfer the rinse water to the filter. Repeat the rinsing and the transfer of the rinse water. A complete transfer of the precipitate to the filter is not necessary. Wash the precipitate on the filter twice with cold water and collect the washings with the filtrate.
8. Using the 10-mL burette from which KSCN was added in Sec. 5.7.3(3), slowly and with constant swirling, titrate (without rezeroing) the filtrate with 0.05*N* potassium thiocyanate solution until a faint red color persists (see notes, Sec. 5.7.5).

5.7.4 *Calculation.*

$$\text{Chloride, as percent Cl} = \frac{[(\text{mL AgNO}_3) - (\text{mL KSCN} \times F_2)](\text{N of AgNO}_3)(3.545)}{\text{grams of sample} \times 10/100} \qquad \text{(Eq 9)}$$

5.7.5 *Notes and temperature corrections for volumetric solutions.*

1. Shaking with benzyl alcohol facilitates filtration by coagulating the precipitate.
2. A back-titration of at least 0.5 mL KSCN solution is necessary to ensure that an adequate excess of silver nitrate solution was present to completely precipitate the chloride present in the sample.

3. When volumetric solutions are standardized at 20°C, titrations should be made at this temperature or corrections applied to reduce the volume of the solution used in titrations to 20°C.

### Sec. 5.8 Notice of Nonconformance

If the liquid PACl product 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 in accordance with Sec. 5.1 . If the results obtained by the supplier on retesting 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 carry with it some means of identification. Each container of PACl shall be legibly marked with the net weight of the contents, the name of the manufacturer, the lot number, a brand name, if any, and other markings as required by applicable laws and regulations.

6.1.2 *Bulk material.* When shipped in bulk, the information required in Sec. 6.2 for packaged material shall accompany the bill of lading.

6.1.3 *Optional.* Containers may also bear the statement: "Guaranteed by (name of manufacturer) to meet the requirements of ANSI/AWWA B408, Liquid Polyaluminum Chloride," provided that the requirements of this standard are met.

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* Governmental packaging and marking references reflect US requirements. Users of ANSI/AWWA B408 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.

## Sec. 6.2 Packaging and Shipping

Packaging and shipping of PACl products shall conform to the current regulations of the US-Department of Transportation.

6.2.1 *Bulk containers.* Liquid PACl shall be shipped by tank car, tank truck, semibulk container, or drum, as specified by the purchaser. No contamination of PACl product by toxic substances must occur during shipping or storage, and the supplier shall ensure that the tank car, tank truck, semibulk container, or drum is suitably clean before loading. The supplier shall provide recommendations for appropriate materials of construction for the storage, handling, and packaging of each specific product.

6.2.2 *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.

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

6.2.3.1 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.

6.2.3.2 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.

6.2.3.3 Alternative method. An alternative method or methods 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 product provided according to the purchaser's order complies with all applicable requirements of this standard or (2) certified analysis of the product at the time of delivery detailing the desired items.

*AWWA is the authoritative resource for knowledge, information, and advocacy to improve the quality and supply of water in North America and beyond. AWWA is the largest organization of water professionals in the world. AWWA advances public health, safety, and welfare by uniting the efforts of the full spectrum of the entire water community. Through our collective strength, we become better stewards of water for the greatest good of people and the environment.*

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