



Designation: D1291 – 06

Standard Practice for Estimation of Chlorine Demand of Water¹

This standard is issued under the fixed designation D1291; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice provides a means of estimating the quantity of chlorine required to be added to a unit volume of water to accomplish a predetermined treatment objective or to completely react with all chlorine reactable substances in the water, or both.

1.2 Temperature, pH, and initial chlorine dosage are all variables in estimating the optimum chlorination practice. The effects of these variables can be evaluated using this practice.

1.3 Chlorine residual is determined using Test Method D1253.

1.4 This practice is applicable to all types of water in which the stated treatment objective can be evaluated or residual chlorine can be measured, or both.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*²

- D1129 Terminology Relating to Water
- D1193 Specification for Reagent Water
- D1253 Test Method for Residual Chlorine in Water
- D1293 Test Methods for pH of Water
- D3370 Practices for Sampling Water from Closed Conduits

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

¹ This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.1.1 *chlorine demand*—the amount of chlorine that must be added to a unit volume of water under specified conditions of pH, temperature, and contact time to completely react with all chlorine-reactable substances in the water. It is defined as the difference between the amount of chlorine applied and the amount of free chlorine remaining at the end of the contact period.

3.1.2 *chlorine requirement*—the amount of chlorine that must be added to a unit volume of water under specified conditions of pH, temperature, and contact time to achieve the objectives of chlorination.

3.2 *Definitions*—For definitions relating to this practice, see Terminology D1129.

4. Summary of Practice

4.1 Known amounts of chlorine are added to a series of 500 mL aliquots of sample. The treated sample aliquots are permitted to stand for a specified contact time (or a variety of different specified contact times) under specified conditions of pH and temperature. At the end of the contact time, the sample aliquots are either analyzed for chlorine content by Test Method D1253 or subjected to whatever evaluative technique is required to establish accomplishment of the treatment objective, or both.

5. Significance and Use

5.1 Chlorine is added to potable water, waste water, and industrial water for a variety of purposes. Some of these purposes are:

5.1.1 To eliminate or reduce the growth of microorganisms in water,

5.1.2 To destroy or modify decomposable organic substances so as to reduce the biochemical oxygen demand of the water,

5.1.3 To eliminate or reduce taste, odors, and color in the water,

5.1.4 To separate grease in waste water by eliminating the protective colloidal effect of proteins present, and

5.1.5 To destroy or modify substances in the waste water that react directly by oxidation, such as ammonia, cyanates, cyanides, ferrous iron, nitrites, phenol, phosphorus, sulfides, sulfites, thiocyanates, and other oxidizable constituents.

5.2 It is important to avoid over-chlorination in order to minimize chemical consumption, meet restrictions specified by regulatory agencies, and minimize equipment degradation.

6. Apparatus

6.1 All of the apparatus listed in Test Methods **D1293** and **D1253** may be required. Any other apparatus necessary to carry out the final evaluation of the effects of chlorination will be required.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**, Type III, which has been rendered chlorine demand-free. Chlorine demand can be removed by treating with excess chlorine and allowing this treated solution to stand in sunlight for several hours to destroy the chlorine residuals. Test Method **D1253** may be used to assure complete destruction of these residuals.

7.3 *Acetic Acid Solution (1 + 1)*—Mix equal volumes of glacial acetic acid and water.

7.4 *Calcium Hydroxide Solution (10.7 g/L)*—Weigh 10.7 g of 100 % hydrated lime, Ca(OH)₂, and suspend in water. Dilute the suspension to 1 L. Shake well each time before using.

7.5 *Chlorine Solution, Standard*—Prepare one of the standard solutions described in 7.5.1 and 7.5.2; standardize as described in 7.5.3.

7.5.1 *Chlorine Water*—Pass chlorine gas through reagent water until the solution contains from 0.5 to 10.0 mg/L Cl₂.

NOTE 1—**Warning:** Use a slow rate of addition and carry out the operation under a hood. Store in a glass stoppered amber bottle and standardize daily before use.

7.5.2 *Sodium Hypochlorite Solution, Standard*—Dilute a commercial sodium hypochlorite or bleach solution containing 10 to 100 g of available chlorine per litre with water to provide a solution containing from 0.5 to 10 mg available chlorine per litre, depending upon the maximum expected chlorine requirement for the sample. This solution must be standardized before use.

7.5.3 Standardize the chlorine water or sodium hypochlorite solution, standard, as follows:

7.5.3.1 Transfer 10 mL of the solution to be standardized to a porcelain dish.

7.5.3.2 Add 10 mL of acetic acid (1 + 1) (see 7.3) and 10 mL of potassium iodide solution (see 7.8).

7.5.3.3 Titrate with sodium thiosulfate solution (0.10 N) (see 7.11) until the yellow color of the liberated iodine is almost discharged.

7.5.3.4 Add 1 mL of starch indicator solution (see 7.12) and continue the titration to a colorless endpoint.

7.5.3.5 Calculate the concentration of available chlorine as follows:

$$\text{Available chlorine, mg/mL} = \frac{A \times B \times 35.45}{C}$$

where:

A = sodium thiosulfate solution, standard, mL,

B = normality of sodium thiosulfate solution, standard, and

C = chlorine solution titrated, mL.

7.6 *Hydrochloric Acid (1 + 1)*—Mix equal volumes of concentrated HCl (sp gr 1.19) and water.

7.7 *Potassium Dichromate Solution (0.100 N)*—Dissolve 4.904 g of anhydrous potassium dichromate (K₂Cr₂O₇) of primary standard quality in water and dilute to 1000 mL in a volumetric flask.

7.8 *Potassium Iodide Solution (50 g/L)*—Dissolve 50 g of potassium iodide (KI) in 1 L of freshly boiled and cooled water. Add 1 g of sodium bicarbonate (NaHCO₃) to stabilize the solution. Store in an amber bottle and avoid direct exposure to sunlight.

7.9 *Sodium Hydroxide Solution (10 g/L)*—Dissolve 10 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

NOTE 2—**Caution:** Heat is generated when dissolving sodium hydroxide in water.

7.10 *Sodium Thiosulfate Solution, Standard (0.10 N)*—Transfer 25 g of sodium thiosulfate pentahydrate (Na₂S₂O₃ · 5H₂O) to a 1-L volumetric flask containing about 800 mL water. Dissolve the compound in the water by shaking and periodic inversion. Add 1 g of sodium carbonate (Na₂CO₃) and dissolve. Dilute the solution to 1 L with water.

7.10.1 Standardize the sodium thiosulfate solution: Add 70 mL of water to a porcelain dish, and add, with constant stirring, 1 mL of concentrated sulfuric acid (H₂SO₄), 10.0 mL of 0.100 N K₂Cr₂O₇, and 20 mL of potassium iodide solution (see 7.8). Permit the reaction mixture to stand in the dark for about six minutes. Then titrate with the sodium thiosulfate solution until the yellow color of the liberated iodine is almost discharged. Add 1 mL of starch indicator solution (see 7.12) and continue the titration to a colorless endpoint.

7.10.1.1 The normality can be calculated as:

$$\text{Normality, Na}_2\text{S}_2\text{O}_3 = \frac{1}{\text{Na}_2\text{S}_2\text{O}_3 \text{ required in titration, mL}}$$

7.10.1.2 Perform this standardization in triplicate and average the results.

7.11 *Starch Indicator Solution*—Make a paste of 1 g of soluble starch and mix into 1 L of boiling water. Then, add 20 g of potassium hydroxide (KOH), mix thoroughly, and allow to stand for 2 h. Add 6 mL of glacial acetic acid (99.5 %). Mix

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.