



Designation: D501 – 03 (Reapproved 2023)

Standard Test Methods of Sampling and Chemical Analysis of Alkaline Detergents¹

This standard is issued under the fixed designation D501; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover procedures for the sampling and chemical analysis of inorganic alkaline detergents.

1.2 The procedures appear in the following order:

	Sections		Sections
Caustic Soda:		Total Alkalinity as Sodium Oxide (Na ₂ O)	69 – 71
Sampling	5	Matter Insoluble in Water	72 and 73
Total Alkalinity as Sodium Oxide (Na ₂ O)	6 – 8	Tetrasodium Pyrophosphate:	
Sodium Hydroxide (NaOH)	9 – 11	Sampling	74
Carbonate as Sodium Carbonate (Na ₂ CO ₃)	12	Tetrasodium Pyrophosphate	
Carbon Dioxide (CO ₂) by the Evolution Method	13 – 16	(Na ₄ P ₂ O ₇)	75 – 79
Soda Ash:		Matter Insoluble in Water	80 and 81
Sampling	17	Loss on Ignition	82 and 83
Matter Volatile at 150 °C to 155 °C	18 and 19	Borax:	
Total Alkalinity as Sodium Carbonate (Na ₂ CO ₃)	20 – 22	Sampling	84
Sodium Bicarbonate (NaHCO ₃)	23 – 25	Total Borate and Excess Alkalinity or Acidity	85 – 87
Sodium Bicarbonate (NaHCO ₃) by Potentiometric Titration	26 – 28	Matter Insoluble in Water	88 and 89
Matter Insoluble in Water	29 and 30	Sodium Triphosphate:	
Apparent Density	31 and 32	Sampling	90
Modified Soda (Sequicarbonate Type):		Trisratable Na ₂ O	91 – 94
Sampling	33	Total P ₂ O ₅ :	
Total Alkalinity as Sodium Oxide (Na ₂ O)	34 – 36	Preferred Method	95 – 97
Sodium Bicarbonate (NaHCO ₃) and Sodium Carbonate (Na ₂ CO ₃)	37 – 39	Alternative Method	98 – 101
Matter Insoluble in Water	40	pH Titration	102 – 107
Sodium Bicarbonate:		Quantitative Separation and Measurement of Various Phosphates:	
Sampling	41	Reverse-Flow Ion-Exchange Chromatography (Preferred	
Sodium Bicarbonate, Sodium Carbonate, and Free Moisture	42 – 45	Method)	108 – 119
Matter Insoluble in Water	46	Paper Chromatographic Method	120 – 127
Sodium Metasilicate, Sodium Sesquisilicate and Sodium Orthosilicate:		pH of 1 percent Solution	128
Sampling	47	Turbidity	129
Total Alkalinity as Sodium Oxide (Na ₂ O)	48 – 50	Temperature Rise	130 – 134
Total Silica as SiO ₂	51 – 53	Sulfate	135 – 137
Sodium Metasilicate (Na ₂ SiO ₃ ·5H ₂ O)	54	Ignition Loss	140 and 141
Sodium Sesquisilicate (3Na ₂ O·2SiO ₂ ·11H ₂ O)	55	Matter Insoluble in Water	142 – 144
Matter Insoluble in Water	56 and 57	Particle Size	145
Loss on Ignition of Sodium Sesquisilicate (3Na ₂ O·2SiO ₂ ·11H ₂ O)	58 and 59	Orthophosphate	146 – 151
Sodium Orthosilicate (Na ₄ SiO ₄)	60		
Trisodium Phosphate:			
Sampling	61		
Trisodium Phosphate (Na ₃ PO ₄) Content and Phosphorus			
Pentoxide (P ₂ O ₅)	62 – 64		
Trisodium Phosphate Calculated as Na ₃ PO ₄ ·12H ₂ O, Na ₃ PO ₄ ·H ₂ O, Na ₃ PO ₄ , and as P ₂ O ₅	65 – 68		

¹ These test methods are under the jurisdiction of ASTM Committee D12 on Soaps and Other Detergents and are the direct responsibility of Subcommittee D12.12 on Analysis and Specifications of Soaps, Synthetics, Detergents and their Components.

Current edition approved July 1, 2023. Published July 2023. Originally approved in 1938. Last previous edition approved in 2016 as D501 – 03 (2016). DOI: 10.1520/D0501-03R23.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards*:²

D459 Terminology Relating to Soaps and Other Detergents

D1193 Specification for Reagent Water

E1 Specification for ASTM Liquid-in-Glass Thermometers

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

3. Terminology

3.1 *Definitions*:

3.1.1 *inorganic alkaline detergent*—a water soluble inorganic alkali or alkaline salt having detergent properties, but containing no soap or synthetics.

3.1.2 For definitions of other terms used in these test methods, refer to Terminology **D459**.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 The term “inorganic alkaline detergent” in these test methods is defined in accordance with Terminology **D459**.

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.

4. Purity of Reagents

4.1 *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.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification **D1193**.

³ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

CAUSTIC SODA

5. Sampling

5.1 *Flake Caustic Soda*—Flake caustic soda shall be sampled by removing portions from various parts of the drum.

5.2 *Powdered Caustic Soda*—Powdered caustic soda shall be sampled by inserting a sampling tube through the contents of the drum in several places. The tube shall be dried by heating just before use.

5.3 *Fused Caustic Soda*—Fused caustic soda shall be sampled by taking chipped samples from the center and bottom of the drum and then mixing the gross sample in the approximate proportions in which the tops and bottoms occur in the drum.

5.4 *Precautions*—Caustic soda shall not be sampled in a moist atmosphere. In the case of fused caustic soda the portion taken for analysis shall have the surface layer of carbonate scraped off immediately before transferring to the weighing bottle. In all cases the sample shall be transferred to a thoroughly dried weighing bottle immediately after it is taken; the bottle shall be tightly stoppered at once.

TOTAL ALKALINITY AS SODIUM OXIDE (Na₂O)

6. Reagents

6.1 *Acid, Standard (1.0 N)*—Prepare and standardize a 1.0 N acid solution.

6.2 *Methyl Red Indicator Solution*.

7. Procedure

7.1 Weigh 10 g of the sample, dissolve in carbon dioxide (CO₂)-free water, wash into a 500 mL volumetric flask, and dilute to volume with CO₂-free water. Protect the solution from

the air as much as possible. Pipet a one-fifth aliquot into a 400 mL beaker and determine sodium oxide (Na₂O) by titrating the sample against 1.0 N acid, using methyl red as the indicator.

8. Calculation

8.1 Calculate the total alkalinity as sodium oxide (Na₂O) as follows:

$$\text{Total alkalinity as Na}_2\text{O, \%} = (A \times 5 \times 3.1) / W \quad (1)$$

where:

A = millilitres of acid required for titration of the Na₂O in the sample, and

W = grams of sample used.

SODIUM HYDROXIDE (NaOH)

9. Reagents

9.1 *Acid, Standard (1.0 N)*—Prepare and standardize a 1.0 N acid solution.

9.2 *Barium Chloride, Neutral Solution (100 g/L)*—Dissolve 100 g of barium chloride (BaCl₂·2H₂O) in water and dilute to 1 L. Make the solution neutral to phenolphthalein.

9.3 *Phenolphthalein Indicator Solution (10 g/L)*—Dissolve 1 g of phenolphthalein in 50 mL of ethyl alcohol and then mix with 50 mL of water.

10. Procedure

10.1 Determine the NaOH on a second one-fifth aliquot pipetted into a 250 mL Erlenmeyer flask. Add about 25 mL of