



Standard Test Methods of Sampling and Chemical Analysis of Alkaline Detergents¹

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This standard has been approved for use by agencies of the 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:		Tetrasodium Pyrophosphate:	
Sampling	5	Sampling	74
Total Alkalinity as Sodium Oxide (Na_2O)	6-8	Tetrasodium Pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$)	75-79
Sodium Hydroxide (NaOH)	9-11	Matter Insoluble in Water	80 and 81
Carbonate as Sodium Carbonate (Na_2CO_3)	12	Loss on Ignition	82 and 83
Carbon Dioxide (CO_2) by the Evolution Method	13-16	Borax:	
Soda Ash:		Sampling	84
Sampling	17	Total Borate and Excess Alkalinity or Acidity	85-87
Matter Volatile at 150 to 155°C	18 and 19	Matter Insoluble in Water	88 and 89
Total Alkalinity as Sodium Carbonate (Na_2CO_3)	20-22	Sodium Triphosphate:	
Sodium Bicarbonate (NaHCO_3)	23-25	Sampling	90
Sodium Bicarbonate (NaHCO_3) by Potentiometric Titration	26-28	Titratable Na_2O	91-94
Matter Insoluble in Water	29 and 30	Total P_2O_5 :	
Apparent Density	31 and 32	Preferred Method	95-97
Modified Soda (Sequicarbonate Type):		Alternative Method	98-101
Sampling	33	pH Titration	102-107
Total Alkalinity as Sodium Oxide (Na_2O)	34-36	Quantitative Separation and Measurement of Various Phosphates:	
Sodium Bicarbonate (NaHCO_3) and Sodium Carbonate (Na_2CO_3)	37-39	Reverse-Flow Ion-Exchange Chromatography (Preferred Method)	108-119
Matter Insoluble in Water	40	Paper Chromatographic Method	120-127
Sodium Bicarbonate:		pH of 1 percent Solution	128
Sampling	41	Turbidity	129
Sodium Bicarbonate, Sodium Carbonate, and Free Moisture	42-45	Temperature Rise	130-134
Matter Insoluble in Water	46	Sulfate	135-137
Sodium Metasilicate, Sodium Sesquisilicate and Sodium Orthosilicate:		Ignition Loss	140 and 141
Sampling	47	Matter Insoluble in Water	142-144
Total Alkalinity as Sodium Oxide (Na_2O)	48-50	Particle Size	145
Total Silica as SiO_2	51-53	Orthophosphate	146-151
Sodium Metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$)	54		
Sodium Sesquisilicate ($3\text{Na}_2\text{O} \cdot 2\text{SiO}_2 \cdot 11\text{H}_2\text{O}$)	55		
Matter Insoluble in Water	56 and 57		
Loss on Ignition of Sodium Sesquisilicate ($3\text{Na}_2\text{O} \cdot 2\text{SiO}_2 \cdot 11\text{H}_2\text{O}$)	58 and 59		
Sodium Orthosilicate (Na_4SiO_4)	60		
Trisodium Phosphate:			
Sampling	61		
Trisodium Phosphate (Na_3PO_4) Content and Phosphorus Pentoxide (P_2O_5)	62-64		
Trisodium Phosphate Calculated as $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$, $\text{Na}_3\text{PO}_4 \cdot \text{H}_2\text{O}$, Na_3PO_4 , and as P_2O_5	65-68		
Total Alkalinity as Sodium Oxide (Na_2O)	69-71		
Matter Insoluble in Water	72 and 73		

1.3 *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.* Material Safety Data Sheets are available for reagents and materials. Review them for hazards prior to usage.

2. Referenced Documents

2.1 ASTM Standards:²

- D 459 Terminology Relating to Soaps and Other Detergents
- D 1193 Specification for Reagent Water
- E 1 Specification for ASTM Thermometers
- E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode

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.

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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. 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 D 459.

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 D 459.

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 Commit-

tee 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 D 1193.

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. 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 BaCl₂ solution and titrate the sample with 1.0 N acid using phenolphthalein as the indicator.

11. Calculation

11.1 Calculate the percentage of sodium hydroxide (NaOH) as follows:

$$\text{NaOH, \%} = (B \times 5 \times 4.0)/C \quad (2)$$

where:

B = millilitres of acid necessary for titration of the NaOH in the sample, and

C = grams of sample used.

CARBONATE AS SODIUM CARBONATE (Na₂CO₃)

12. Calculation

12.1 Calculate the carbonate as sodium carbonate (Na₂CO₃) as follows:

$$\text{Na}_2\text{CO}_3, \% = [(A - B) \times 5 \times 5.3]/W \quad (3)$$

where:

A = millilitres of acid required for titration of the Na_2O in the sample,

B = millilitres of acid required for titration of the NaOH in the sample, and

W = grams of sample used.

NOTE 1—When more accurate results are desired, the evolution method for carbon dioxide as described in Sections 13-16 should be used.

CARBON DIOXIDE (CO_2) BY THE EVOLUTION METHOD

13. Apparatus

13.1 *Apparatus Assembly*—Place a 150-mL wide-neck extraction flask on a gauze over a burner. Fit the flask with a three-hole rubber stopper, one opening to carry a 25-cm reflux condenser, the second to carry a thistle tube with a twoway stopcock for the introduction of acid into the flask, and the third to carry a tube for the introduction of a continuous stream of carbon dioxide (CO_2)-free air into the flask. Draw out the ends of the thistle and air supply tubes to a small point, and place them in the stopper so that the points are very close to the bottom of the flask. Attach to the air supply tube, a U-tube containing soda-asbestos (Ascarite) so that the air admitted to the flask will be free from CO_2 .

13.2 *Preparation of Absorption Train*—Attach to the top of the reflux condenser a train consisting of the following:

13.2.1 A U-tube containing granulated zinc for the removal of acid gases,

13.2.2 A drying tube containing magnesium perchlorate, anhydrous calcium sulfate (Drierite), or anhydrous calcium chloride,

13.2.3 A weighed U-tube containing soda-asbestos in the first half and the same drying agent in the second half as used in 13.2.2, and

13.2.4 A protective U-tube containing any of the above mentioned drying agents.

13.2.5 Attach the final tube to an aspirator.

14. Reagents

14.1 *Methyl Orange Indicator Solution* (1 g/L)—Dissolve 0.1 g of methyl orange in water and dilute to 100 mL.

14.2 *Sulfuric Acid* (2 + 9)—Mix 2 volumes of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) carefully with stirring into 9 volumes of water.

15. Procedure

15.1 Aspirate with a stream of carbon dioxide (CO_2)-free air at a rate of approximately 20 to 30 mL/min until the train is free from CO_2 as determined by no further change in weight greater than 0.3 mg in the U-tube.

15.2 Weigh 10 g of the sample to the nearest 0.01 g directly into the extraction flask, cover with 50 mL of freshly boiled water, add 2 drops of methyl orange solution, and close the apparatus with the train in place. Start the aspiration at a rate of 20 to 30 mL/min, and slowly add through the thistle tube sufficient H_2SO_4 (2 + 9) to neutralize the NaOH and a sufficient excess to ensure the final acidity of the mixture as indicated by the methyl orange. Always leave some acid in the thistle tube as an air seal. Heat gently and continue until the contents of the flask have boiled for 5 min; remove the source of heat, and continue aspirating until the flask has cooled, or for about 30 min.

15.3 Remove the U-tube containing soda-asbestos and weigh using a tared U-tube as a counterpoise. The increase in weight represents CO_2 .

16. Calculation

16.1 From the increase in weight of the tube calculate the percentage of carbon dioxide (CO_2) as sodium carbonate (Na_2CO_3) as follows:

$$\text{Na}_2\text{CO}_3, \% = [(C \times 2.409)/W] \times 100 \quad (4)$$

where:

C = grams of CO_2 , and

W = grams of sample used.

NOTE 2—This test method for the determination of Na_2CO_3 as CO_2 is to be preferred when a procedure more accurate than that described in Section 12 is required.