Designation: E 359 – ~~95~~00

Standard Test Methods for Analysis of Soda Ash (Sodium Carbonate)[1]

This standard is issued under the fixed designation E 359; 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 Department of Defense.

1. Scope

1.1 These test methods cover the analyses usually required on commercial soda ash (sodium carbonate).

1.2 The analytical procedures appear in the following sections:

	Sections
Total Alkalinity, Titrimetric	8-15
Sodium Bicarbonate, Titrimetric	16-23
Loss on Heating, Gravimetric	24-30
Moisture, Calculation	31-35
Sodium Chloride, Titrimetric	36-42
Sodium Sulfate, Gravimetric	43-49
Iron, Photometric	50-58
Sieve Analysis	59-65

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 Review the current Materials Safety Data Sheets (MSDS) for detailed information concerning toxicity, first aid procedures, handling and safety precautions.

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.* Specific hazards statements are given in Section 6.

2. Referenced Documents

2.1 *ASTM Standards:*

C 429 Test Method for Sieve Analysis of Raw Materials for Glass Manufacture[2]

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[1] These test methods are under the jurisdiction of ASTM Committee E~~-~~15 on Industrial and Specialty Chemicals and are the direct responsibility of Subcommittee E15.~~5~~02 on ~~Alkalies.~~ Product Standards.

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D 1193 Specification for Reagent Water[3]
E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]
E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals[5]
E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[6]
E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens[4]
E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals[6]
E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis[6]
E 300 Practice for Sampling Industrial Chemicals[6]

3. Significance and Use

3.1 Soda ash is used in a number of manufacturing processes. The procedures listed in 1.2 are suitable for specification acceptance and manufacturing control of commercial soda ash.

4. Apparatus

4.1 *Photometers and Photometric Practice*—Photometers and photometric practice used in these test methods shall conform to Practice E 60.

4.2 *pH Meters*—pH meters and their use shall conform to Test Method E 70.

4.3 *Buret*—A calibrated 50-mL buret, or any standard 50-mL buret calibrated by either the National Institute of Standards and Technology or by the user. Alternatively, a 100-mL calibrated buret with a 50-mL bulb at the top and a 50-mL stem below may be used.

5. Purity of Reagents and Water

5.1 *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.[7] 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.

5.2 Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D 1193.

6. Hazards

6.1 Soda ash is a primary skin irritant. Dusts or mists are moderately irritating to the mucous membrane of the nose and eyes. The irritation is temporary and symptoms usually disappear shortly after contact is ~~ended. The Material Safety Data Sheet (MSDS) should be consulted for further information.~~ ended.

7. Sampling

7.1 The general principles for sampling solids are covered in Practice E 300. The following aspects of soda ash sampling must be considered:

7.2 *General*:

7.2.1 The selection of a representative sample is a necessary prerequisite for any accurate analysis, and this is particularly important with the alkalies, since they are susceptible to rapid contamination by moisture and carbon dioxide upon exposure to air. Also, some of them are not uniform in particle size and tend to segregate on handling.

7.2.2 The characteristics of soda ash that make proper sampling difficult at times are its tendency to absorb moisture and carbon dioxide from the air through any commercial container in which it is generally shipped, and the susceptibility of dense ash in bulk to segregate in relation to particle size as the result of normal transit vibrations.

7.2.3 Details of good sampling depend on: (*1*) the type of shipment, whether in containers or in bulk; (*2*) the type of product, whether light or dense soda ash; and (*3*) the type of analysis desired, whether chemical or physical.

7.3 *Bulk Shipments*:

7.3.1 Although bulk shipments are normally in transit a relatively short time, there is likely to be some absorption of moisture and carbon dioxide in exposed surface areas. If physical tests such as screen analysis are to be included, it is particularly important to avoid segregation that occurs on surface areas.

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[2] *Annual Book of ASTM Standards*, Vol 15.02.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 03.05.

[6] *Annual Book of ASTM Standards*, Vol 15.05.

[7] *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. Pharmacope~~utic~~al Convention, Inc. (USPC), Rockville, MD.

7.3.2 To sample boxcar shipments, brush aside the surface layer to a depth of 12 in. (305 mm) and take portions systematically from the newly exposed area to the bottom of the car by means of a sample thief.

7.3.3 Hopper cars and trucks are more difficult to sample adequately. Samples can be taken through the hatches with a sample thief, as for boxcar sampling. Preferably, samples should be taken during the unloading operation at the point of discharge to the bin, or from any open section of the conveyor.

7.4 *Bag Shipments*:

7.4.1 Packaged soda ash that has been in storage for some time can be sampled satisfactorily only by emptying the whole package and mixing thoroughly before taking the sample. Even such a portion is likely to represent only the package sampled rather than the stock of packages as a whole. The reason is that a bag or other container taken from an outer layer of the storage pile is subject to more air contact and consequently more moisture and carbon dioxide absorption than are packages buried farther back in the stock.

7.4.2 To get an idea of the quality of the soda ash as packed, it is the usual practice to take the sample from somewhere near the center of the package. This may be done by removing the top 6 or 8 in. (150 or 200 mm) of soda ash from the package, then removing the sample from the center of the remaining portion. Such a sample carefully taken will generally be found representative except in cases of long storage or unusually damp storage conditions.

7.5 *Sample Preparation*:

7.5.1 Thoroughly mix the total sample taken. Then quarter or riffle the entire sample to obtain the required size sample for analysis. Minimize exposure to moisture and carbon dioxide.

7.5.2 Store the sample for analysis in a glass or other suitable container that will not contaminate the sample and that can be sealed to prevent exposure of the sample to moisture or carbon dioxide.

TOTAL ALKALINITY

8. Scope

8.1 This test method covers the titrimetric determination of the total alkalinity of soda ash. This alkalinity is normally expressed as percent sodium oxide (Na_2O).

9. Summary of Test Method

9.1 Total alkalinity is determined by titration with standard hydrochloric (or sulfuric) acid using methyl orange or modified methyl orange indicator solution.

10. Interferences

10.1 Alkalies other than soda ash (sodium carbonate) and compounds that consume acid will affect the accuracy of this test method.

11. Reagents

11.1 *Hydrochloric (or Sulfuric) Acid* (1.0 *N)*—Prepare in accordance with Practice E 200 (record temperature of solution when standardized).

11.2 *Modified Methyl Orange Indicator Solution or Methyl Orange Indicator Solution*—See Practice E 200.

11.3 *Water*, carbon dioxide-free (freshly boiled and cooled).

12. Procedure

12.1 Weigh, to the nearest 0.1 mg, 4.4 ± 0.1 g (Note 1) of the sample and transfer to a 500-mL conical flask. Add 100 mL of water and swirl to dissolve the sample.

NOTE 1—Use of the specfied weight of sample requires a 100-mL buret for titration and is recommended. If a 50-mL buret is used, the sample weight should be halved.

12.2 Add 3 drops of modified methyl orange indicator solution (Note 2). Titrate this solution with standard 1.0 *N* acid to a gray end point (Note 3). Record the volume to the nearest 0.02 mL and temperature of the acid used. Correct the acid normality for any difference from the standardization temperature by use of the factor $\Delta N/°C = 0.00035$ between 20 and 30°C. Add the correction when the temperature of use is below and subtract when above the temperature of standardization (see Practice E 200).

NOTE 2—If desired, 0.1 % methyl orange indicator solution may be used.

NOTE 3—The analyst should end the titration at the same shade of color as was used for the end point in the standardization of the acid.

13. Calculation

13.1 Calculate the total alkalinity as percent sodium oxide (Na_2O) as follows:

$$\text{sodium oxide, wt \%} = \frac{(A \times B \times 0.030990) \times 100}{W} \tag{1}$$

where:
A = acid required for titration of the sample, mL,
B = corrected normality of the acid, and
W = sample used, g.

13.2 Alternatively, calculate the alkalinity as sodium carbonate as follows:

$$\text{sodium carbonate, wt \%} = 1.7101 \times Na_2O\text{, wt \%} \quad (2)$$

13.3 If actual sodium carbonate content is desired, the sodium bicarbonate content must be determined separately as described in Sections 16 and 23. Then:

$$\text{sodium carbonate (actual), \%} = A - (B \times 0.6308) \quad (3)$$

where:
A = Na_2CO_3, % (see 13.2), and
B = $NaHCO_3$, % (see 21.1).

14. Report

14.1 Report the percentage of sodium oxide to the nearest 0.01 %.

15. Precision and Bias

15.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 4):

15.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.032 % absolute at 52 DF. The 95 % limit for the difference between two such runs is 0.09 % absolute.

15.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.038 % absolute at 26 DF. The 95 % limit for the difference between two such averages is 0.11 % absolute.

15.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.154 % absolute at 8 DF. The 95 % limit for the difference between two such averages is 0.43 % absolute.

NOTE 4—These precision estimates are based on an interlaboratory study of analyses performed in 1967 on three samples of soda ash covering the range from 58.190 to 58.385 % sodium oxide. Ten laboratories analyzed the three samples, with one analyst in each laboratory performing duplicate determinations and repeating 1 day later.[8] Practice E 180 was used for developing these precision estimates.

15.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

SODIUM BICARBONATE

16. Scope

16.1 This test method describes the titrimetric determination of sodium bicarbonate in soda ash. The lower limit of determination is 0.02 % sodium bicarbonate.

17. Summary of Test Method

17.1 Bicarbonate is determined titrimetrically by adding a sample to an excess of standard sodium hydroxide solution (thus converting bicarbonate to carbonate), precipitating the carbonate with barium chloride solutions and back-titrating the excess sodium hydroxide with standard acid solution using a pH meter to determine the end point.

17.2 A primary standard is run simultaneously to correct the titration for adsorption or occlusion of sodium hydroxide on the barium carbonate.

18. Apparatus

18.1 *pH Meter*, with glass and calomel electrodes. Standardize the pH meter with commercially available pH 10 buffer solution.

18.2 *Magnetic Stirrer*, with TFE-fluorocarbon-covered stirring bar.

19. Reagents

19.1 *Barium Chloride Solution* (120 g/L)—See Practice E 200.

19.2 *Hydrochloric Acid, Standard* (0.1 *N*)—See Practice E 200.

19.3 *Sodium Carbonate, Primary Standard* Na_2CO_3—Dry about 10 g of anhydrous primary standard sodium carbonate (Na_2CO_3) in a platinum dish or low-form weighing bottle (70-mm diameter) for 4 h at 250°C (minimum) but do not exceed 300°C. Cool in a desiccator. Prepare fresh for use.

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[8] Supporting data are available from ASTM Headquarters. Request RR: E15–0046.

19.4 *Sodium Hydroxide, Standard Solution* (0.1 *N)*—See Practice E 200.

19.5 *Water*, carbon dioxide-free (freshly boiled and cooled).

20. Procedure

20.1 Perform the following steps of the procedure on equal weights of both the sample and the primary standard sodium carbonate (Na_2CO_3) (Note 5). Make duplicate determinations.

NOTE 5—To compensate for the adsorption or occlusion of NaOH by the precipitated $BaCO_3$ (20.5), the use of primary standard Na_2CO_3 as a blank is required.

20.2 Place 200 mL of $BaCl_2$ solution in a 400-mL beaker. Using a pH meter, adjust the solution to pH 8.8 by addition of 0.1 *N* NaOH solution (or HCl) as required.

20.3 Into a 600-mL beaker place 150 mL of CO_2-free water. Add by pipet 5.0 mL of 0.1 *N* NaOH solution.

NOTE 6—If, in 20.6, the pH of the sample solution is below 8.8 before titrating with 0.1 *N* HCl, repeat the test adding by pipet 10.0 mL of 0.1 *N* NaOH solution to the beakers being prepared for both the sample and the primary standard Na_2CO_3.

20.4 Weigh, to the nearest 1 mg, 4.0 g of the sample (or of the primary standard) and transfer to the solution in the 600-mL beaker. Place the beaker on a magnetic stirrer, insert a stirring bar, and stir to dissolve.

NOTE 7—The subsequent operations should be completed within 5 min to minimize absorption of atmospheric CO_2.

20.5 While continuing to stir, add the 200 mL of neutralized $BaCl_2$ solution by means of a 100-mL (or 200-mL, if available) pipet, allowing the reagent to run freely into the stirring solution.

20.6 Insert the electrodes into the solution and titrate slowly with 0.1 *N* HCl using a 10-mL buret, stirring continuously. When pH 8.8 is reached, allow the solution to stir for 1 min. If the pH remains at 8.8, the end point has been reached. If not, continue the titration until this pH is reached. Record the volume of titrant to the nearest 0.05 mL.

21. Calculation

21.1 Calculate the percentage of sodium bicarbonate as follows:

~~$\text{sodium bicarbonate, wt \%} = \frac{(B - A)N \times 0.084) \times 100}{W}$ (4)~~

$$\text{sodium bicarbonate, wt \%} = \frac{(B - A)N \times 0.084 \times 100}{W} \tag{4}$$

where:

A = acid for sample, mL,

B = acid for primary standard, mL,

N = normality of acid, and

W = sample used, g.

22. Report

22.1 Report the percentage of sodium bicarbonate to the nearest 0.01 %.

23. Precision and Bias

23.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 8):

23.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.030 % absolute at 60 DF. The 95 % limit for the difference between two such runs is 0.08 % absolute.

23.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.078 % absolute at 30 DF. The 95 % limit for the difference between two such averages is 0.22 % absolute.

23.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.084 % absolute at 9 DF. The 95 % limit for the difference between two such averages is 0.24 % absolute.

NOTE 8—These precision estimates are based on an interlaboratory study of analyses performed in 1967 on three samples covering the range from 0.23 to 0.98 % sodium bicarbonate. One analyst in ten laboratories performed duplicate determinations and repeated 1 day later.[8] Practice E 180 was used in developing these precision estimates.

23.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

LOSS ON HEATING

24. Scope

24.1 This test method covers the gravimetric determination of loss on heating of soda ash.

25. Summary of Test Method

25.1 Loss on heating is determined gravimetrically by heating a weighed sample under controlled conditions to expel moisture and thermally convert sodium bicarbonate to sodium carbonate by elimination of water and carbon dioxide.

26. Apparatus

26.1 *Drying Oven*, gravity-convection, Type IB. See Specification E 145.

26.2 *Weighing Bottle*, 70-mm diameter, low-form, glass, with cover.

27. Procedure

27.1 Dry the weighing bottle at 250°C minimum (270°C max) for 30 min. Cool in a desiccator and weigh to the nearest 0.1 mg.

27.2 Place 5 ± 0.1 g of the sample in the weighing bottle, cover and weigh to the nearest 0.1 mg. Determine the sample weight by difference.

27.3 Dry with the cover ajar for 4 h at 250°C minimum (270°C maximum). Cool in a desiccator with the cover ajar. Weigh to the nearest 0.1 mg with the cover closed.

28. Calculation

28.1 Calculate the percentage loss in weight as follows:

$$\text{loss in weight, wt \%} = \frac{(A - B) \times 100}{W} \tag{5}$$

where:

A = weight of bottle and sample before heating, g,

B = weight of bottle and sample after heating, g, and

W = sample used, g.

29. Report

29.1 Report the percentage loss in weight to the nearest 0.01 %.

30. Precision and Bias

30.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 9):

30.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.016 % absolute at 54 DF. The 95 % limit for the difference between two such runs is 0.04 % absolute.

30.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.015 % absolute at 27 DF. The 95 % limit for the difference between two such averages is 0.04 % absolute.

30.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.030 % absolute at 8 DF. The 95 % limit for the difference between two such averages is 0.08 % absolute.

NOTE 9—These precision estimates are based on an interlaboratory study of analyses performed in 1967 on three samples covering the range from 0.35 to 0.55 % loss on heating. One analyst in each of ten laboratories performed duplicate determinations and repeated 1 day later.[8] Practice E 180 was used in developing these precision estimates.

30.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

MOISTURE

31. Scope

31.1 This test method covers the calculation of moisture in soda ash.

32. Summary of Test Method

32.1 Moisture is determined by calculation. The percent of volatile products resulting from decomposition of sodium bicarbonate, 21.1, is subtracted from the percent loss on heating, 28.1, and the difference is reported as moisture.

NOTE 10—The test method makes the assumption that in commercial soda ash, nonvolatile matter other than moisture and products of sodium bicarbonate decomposition will not be evolved at 250 to 270°C.

33. Calculation

33.1 Calculate the percentage of moisture as follows:

$$\text{moisture, wt \%} = A - (B \times 0.369) \tag{6}$$

where:
A = loss on heating, % (see 28.1), and
B = sodium bicarbonate, % (see 21.1).

34. Report

34.1 Report the percentage of moisture to the nearest 0.01 %.

35. Precision and Bias

35.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 11):

35.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.014 % absolute at 54 DF. The 95 % limit for the difference between two such runs is 0.04 % absolute.

35.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.031 % absolute at 27 DF. The 95 % limit for the difference between two such averages is 0.09 % absolute.

35.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.044 % absolute at 8 DF. The 95 % limit for the difference between two such averages is 0.12 % absolute.

NOTE 11—These precision estimates are based on an interlaboratory study of analyses performed in 1967 on three samples covering the range from 0.18 to 0.34 % moisture in soda ash. One analyst in each of ten laboratories performed duplicate determinations and repeated 1 day later.[8] Practice E 180 was used in developing these precision estimates.

35.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

SODIUM CHLORIDE

36. Scope

36.1 This test method covers the titrimetric determination of sodium chloride in soda ash. The lower limit of detection is 0.005 % sodium chloride.

37. Summary of Test Method

37.1 Chloride is determined titrimetrically by the Volhard method. A sample is dissolved, acidified, and treated with a small excess of standard silver nitrate solution. The precipitated silver chloride is removed by filtration and the excess silver nitrate is titrated with standard ammonium thiocyanate solution using ferric ammonium sulfate indicator solution.

38. Reagents

38.1 *Ammonium Thiocyanate, Standard Solution* (0.1 *N)*—See Practice E 200.

38.2 *Ferric Ammonium Sulfate Indicator Solution*—See Practice E 200.

38.3 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).

38.4 *Silver Nitrate, Standard Solution* (0.1 *N)*—See Practice E 200.

39. Procedure

39.1 Weigh, to the nearest 1 mg, 10 g of sample (Note 12) and transfer to a 500 mL glass-stoppered conical flask, using about 100 mL of water to effect the transfer. Add 1 mL of ferric ammonium sulfate indicator solution and sufficient HNO_3 (sp gr 1.42) slowly to dissolve the reddish-brown ferric hydroxide precipitate. Then add 1 to 2 mL excess HNO_3 and cool to room temperature.

NOTE 12—If the sodium chloride content is less than 0.1 % use a 20-g sample.

39.2 Add, from a buret, 1 mL of 0.1 *N* NH_4CNS solution. Titrate the solution with 0.1 *N* $AgNO_3$ solution to the disappearance of the reddish-brown color. Then add 2 mL of 0.1 *N* $AgNO_3$ solution in excess. Stopper the flask and shake the solution vigorously.

39.3 Filter off the precipitated silver chloride using semiquantitative paper. Wash the paper with three 5-mL portions of water. Titrate the filtrate and washings with 0.1 *N* NH_4SCN solution until the first permanent reddish-brown color appears and persists after shaking for a minimum of 15 s.

39.4 Record the volumes of titrants used to the nearest 0.02 mL. Include the initial volume of 0.1 *N* NH_4SCN solution used in 39.2.

40. Calculation

40.1 Calculate the percentage of sodium chloride as follows:

$$\text{sodium chloride, wt \%} = \frac{[(A\times N_1) - (B \times N_2)] \times 0.05844}{W} \times 100 \tag{7}$$

where:

A = $AgNO_3$ solution added, mL,
B = NH_4CNS solution added, mL,
N_1 = normality of $AgNO_3$ solution used,
N_2 = normality of NH_4CNS solution used, and
W = sample used, g.

41. Report

41.1 Report the percentage of sodium chloride to the nearest 0.001 %.

42. Precision and Bias

42.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 13):

42.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0026 % absolute at 50 DF. The 95 % limit for the difference between two such runs is 0.007 % absolute.

42.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.0058 % absolute at 25 DF. The 95 % limit for the difference between two such averages is 0.016 % absolute.

42.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.0068 % absolute at 7 DF. The 95 % limit for the difference between two such averages is 0.019 % absolute.

NOTE 13—These precision estimates are based on an interlaboratory study of analyses performed in 1967 on three samples covering the range from 0.08 to 0.18 % sodium chloride. One analyst in each of ten laboratories performed duplicate determinations and repeated 1 day later.[8] Practice E 180 was used in developing these precision estimates.

42.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

SODIUM SULFATE

43. Scope

43.1 This test method covers the gravimetric determination of sulfate present in soda ash. The lower limit of detection is 0.005 % sodium sulfate.

44. Summary of Test Method

44.1 Sulfate is determined gravimetrically by precipitation with barium chloride solution, filtering, washing, igniting, and weighing as barium sulfate.

45. Reagents

45.1 *Barium Chloride Solution* (120 g $BaCl_2 \cdot 2H_2O$/L)—See Practice E 200.
45.2 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).
45.3 *Methyl Orange Indicator Solution* (1 g/L)—See Practice E 200.
45.4 *Silver Nitrate Solution* (17 g/100 mL)—See Practice E 200.

46. Procedure

46.1 Weigh 25 g of the sample to the nearest 0.1 g into a 600-mL beaker and dissolve with about 200 mL of water. Add 2 to 4 drops of methyl orange indicator solution and acidify carefully with HCl, adding 1 mL in excess of that required to neutralize the sample.

46.2 Examine the solution at this point. If it contains any insoluble matter, filter off same on a retentive filter paper washing the paper once with water.

46.3 Heat the solution (or filtrate) to boiling. Add slowly to the boiling solution 25 mL of $BaCl_2$ solution with constant stirring. Digest for 30 min on a steam bath and allow the precipitate to settle overnight at room temperature.

46.4 Filter on ashless, fine quantitative paper and transfer the precipitate to the paper with a fine stream of hot water from a wash bottle. Wash the precipitate with successive small portions of hot water until the washings are free of chloride on testing with 3 to 4 drops of $AgNO_3$ solution.

46.5 Heat a platinum or porcelain crucible 850 to 900°C for 15 min, cool in a desiccator, and weigh to the nearest 0.0001 g. Fold the washed filter paper with precipitate and place in the tared crucible. Dry and char carefully without flaming over a low flame, and then more strongly until the paper is burned off. Ignite at 850 to 900°C for a minimum of 30 min. Remove the crucible from the furnace, cool partially, place in a desiccator, and cool to room temperature. Reweigh to the nearest 0.0001 g.

47. Calculation

47.1 Calculate the percentage of sodium sulfate as follows:

$$\text{sodium sulfate, wt \%} = \frac{(A - B) \times 0.60854 \times 100}{W} \tag{8}$$

where:

A = weight of crucible and precipitate after ignition, g,
B = weight of empty crucible, g, and
W = sample used, g.

48. Report

48.1 Report the percentage of sodium sulfate to the nearest 0.001 %.

49. Precision and Bias

49.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 14):

49.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0031 % absolute at 60 DF. The 95 % limit for the difference between two such runs is 0.009 % absolute.

49.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 0.0043 % absolute at 30 DF. The 95 % limit for the difference between two such averages is 0.012 % absolute.

49.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 0.0070 % absolute at 9 DF. The 95 % limit for the difference between two such averages is 0.020 % absolute.

NOTE 14—These precision estimates are based on an interlaboratory study of analyses performed in 1967 on three samples covering the range from 0.013 to 0.23 % sodium sulfate. One analyst in each of ten laboratories performed duplicate determinations and repeated 1 day later.[8] Practice E 180 was used in developing these precision estimates.

49.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

IRON

50. Scope

50.1 This test method covers the photometric determination of iron in soda ash. The lower limit of detection is 0.1 ppm as Fe.

51. Summary of Test Method

51.1 Iron is determined photometrically as the orange-red complex of the ferrous form with 1,10-phenanthroline (orthophenanthroline) in an acetate-buffered solution at pH 5. The color develops within 15 min, is very stable, and follows Beer's law. Intensity of the color formed is measured at 510 nm in a photometer calibrated with standard iron solutions.

52. Interferences

52.1 Impurities normally found in soda ash do not cause any interference. Copper, if present to the extent of 0.5 mg/100 mL of final solution, changes the hue of the solution, but interferes only slightly when excess reagent is present. Zinc, cadmium, and nickel form complexes and consume reagent but do not interfere when sufficient reagent is present.

53. Reagents

53.1 *Ammonium Acetate-Acetic Acid Solution*—See Practice E 200.
53.2 *Ammonium Hydroxide* (1 + 1)—See Practice E 200.
53.3 *Congo Red Indicator Paper*.
53.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).
53.5 *Hydroxylamine Hydrochloride Solution* (100 g/L)—See Practice E 200.
53.6 *Iron, Standard Solution* (1 mL = 0.010 mg Fe)—See Practice E 200.
53.7 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3).
53.8 *1,10-Phenanthroline (Orthophenanthroline) Solution* (3 g/L)—See Practice E 200.

54. Preparation of Standard Curve

54.1 *Calibration Solutions*—Transfer 0.5, 1.0, 2.0, 3.0, and 5.0 mL of the standard iron solution (1 mL = 0.010 mg Fe) into a series of 100-mL volumetric flasks. Dilute the contents of each flask to about 50 mL with water. Proceed as directed in 54.3.

54.2 *Reference Solution*—Transfer 50 mL of water to a 100-mL volumetric flask and proceed in accordance with 54.3.

54.3 *Color Development*—To each flask add in order, with mixing after each addition, 5 mL of hydroxylamine hydrochloride solution, NH_4OH (1 + 1) as required to make the solution just alkaline to congo red paper used as external indicator, 5 mL of the acetate buffer solution, and 5 mL of the phenanthroline solution. Dilute to volume with water and mix thoroughly. Allow to stand for 15 min for complete color development.

54.4 *Photometry*—Transfer a suitable portion of the reference solution to an absorption cell with a 5-cm light path (Note 15) and adjust the photometer (see 4.1) to the initial setting using a light band centered at approximately 510 nm. While maintaining this adjustment, take the photometric readings (absorbances) of the calibration solutions.

NOTE 15—This test method has been written for cells having a 5-cm light path. Cells of other dimensions may be used, provided suitable adjustments can be made in the amounts of samples and reagents used.

54.5 *Calibration Curve*—Plot on coordinate paper the photometric readings (absorbances) of the calibration solutions versus milligrams of iron per 100 mL of solution.

55. Procedure

55.1 *Test Solutions*—Weigh 50 ± 0.1 g of sample and transfer to a 600-mL beaker. Add 100 mL of water and stir to dissolve. Acidify with HCl (sp gr 1.19) in increments until 100 mL have been added. Cover with a watch glass, heat to boiling and boil for 1 min. Remove from the heat and examine the bottom of the beaker for the presence of "mill scale" or other insoluble particles (Note 16). If present, decant the clear solution to a 500-mL volumetric flask. To the residue in the beaker add 5 mL of HCl and 2 mL of HNO_3 (sp gr 1.42), heat to boiling in a hood, and evaporate nearly to dryness. Cool and transfer the residual solution to the 500-mL volumetric flask, carefully rinsing the beaker. Cool the solution in the flask and make up to the mark with water and mix well. Pipet an aliquot containing 0.005 to 0.05 mg of iron into a 100-mL volumetric flask.

NOTE 16—The presence of such "mill scale" affects the precision of results obtainable by the test method because of the difficulty of obtaining a representative sample.

55.2 *Reference Solution*—Transfer 50 mL of water to a 100-mL volumetric flask, and add 1 mL of HCl.

55.3 *Color Development*—Develop the color of the test solution, 55.1, and the reference solution 55.2, as described in 54.3.

55.4 *Photometry*—Take the photometric reading of the test and reference solutions as described in 54.4.

56. Calculation

56.1 Convert the photometric reading (absorbance) of the test solution to milligrams of iron by means of the calibration curve. Calculate the parts per million of iron as follows:

$$\text{iron, ppm} = \frac{A \times 1000}{B} \qquad (9)$$

where:

A = iron found in 100 mL of final solution, mg, and
B = sample represented in the aliquot taken, g.

57. Report

57.1 Report the parts per million of iron to the nearest 0.1 ppm.

58. Precision and Bias

58.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 17):

58.1.1 *Repeatability (Single Analyst)*—The coefficient of variation for a single determination has been estimated to be 4.98 % relative at 54 DF. The 95 % limit for the difference between two such runs is 14 % relative.

58.1.2 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 3.60 % relative at 27 DF. The 95 % limit for the difference between two such averages is 10 % relative.

58.1.3 *Reproducibility (Multilaboratory)*—The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 13.3 % relative at 8 DF. The 95 % limit for the difference between two such averages is 37 % relative.

NOTE 17—These precision estimates are based on an interlaboratory study of analyses performed in 1967 on three samples covering the range from 10 to 30 ppm iron in soda ash. One analyst in each of ten laboratories performed duplicate determinations and repeated 1 day later.[8] Practice E 180 was used in developing these precision estimates.

58.2 *Bias*—The bias of this test method has not been determined because of the lack of acceptable reference material.

SIEVE ANALYSIS

59. Scope

59.1 This test method covers the sieve analysis of soda ash (sodium carbonate) to measure the particle size. *ASTM STP 447A*[9] may also be useful to obtain guidelines for sieve analysis in general.

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[9] *Manual on Test Sieving Methods, ASTM STP 447A*, ASTM, West Conshohocken, PA, 1969.

60. Summary of Test Method

60.1 The particle size distribution of soda ash is determined by passing the material through a series of sieves arranged in order of increasing fineness and calculating the cumulative percentage of each screen fraction.

61. Apparatus

61.1 *Testing Sieves*—Sieves shall conform to Specification E 11, with particular reference to Table 1 and Section 4. Sieves shall be designated by the U.S. standard sieves and consist of the following sieves stacked from coarsest on top to pan on the bottom: 20, 30, 40, 50, 70, 80, 100, 140 200, 235, pan.

61.2 *Sieve Shaker*—A mechanically operated sieve shaker that imparts to the set of sieves a rotary motion and tapping action of uniform speed shall be provided. Shaker shall conform to specifications of 4.2 provided in Test Method C 429.

61.3 *Balance*—Properly calibrated and capable of 0.1 g accuracy.

TABLE 1 Precision for Sieve Analysis

	Laboratory Precision			Reproducibility		
Cumulative %	Coefficient of Variation, %	Degrees of Freedom	95 % Range (% Relative)	Coefficient of Variation, %	Degrees of Freedom	95 % Range (% Relative)
<1	20	10	56.0	31	5	86.8
1 to 25	7.5	102	21.1	12.1	5	33.9
25 to 95	2.1	79	5.9	3.9	5	10.9
>95	0.29	60	0.8	0.33	5	0.9

62. Procedure

62.1 Weigh 100 g of sample to the nearest 0.1 g, and transfer it onto the top of the stacked sieves. Cover the top sieve and place the entire assembly on the sieve shaker. Shake the nest of sieves for 10 min.

62.2 Carefully remove the top sieve from the stack and pour its contents into a weighing pan. Tap the sieve or use a small stiff brush to gather the remaining sample particles into the weighing pan.

62.3 Record the weight to the nearest 0.1 g. Weigh the contents of the remaining sieves in the same manner.

63. Calculation

63.1 Calculate the cumulative percentage of each screen fraction as follows:

$$\text{cumulative wt \%} = \frac{A}{B} \times 100 \tag{10}$$

where:

A = weight of screen fraction plus the combined weights of the previous screen fractions in the sample, g, and

B = weight of sample, g.

64. Report

64.1 Report the cumulative percentage to the nearest 0.1 %.

65. Precision and Bias

65.1 *Precision*—The following criteria should be used for judging the acceptability of results (Note 18):

65.1.1 *Laboratory Precision (Within-Laboratory, Between-Days Variability, Formerly Called Repeatability)*—The coefficient of variation of results, obtained by the same analyst on different days, has been estimated to be the values shown in Table 1 for the indicated cumulative percent levels at the associated degrees of freedom. The 95 % limit for the difference between two such results is also shown in the table.

65.1.2 *Reproducibility (Multilaboratory)*—The coefficient of variation of results, obtained by analysts in different laboratories has been estimated to be the values shown in Table 1 for the indicated cumulative percent levels at the associated degrees of freedom. The 95 % limit for the difference between two such results is also shown in the table.

NOTE 18—These above precision estimates are based upon an interlaboratory study on two samples of soda ash (typical product and a coarser than normal material). One analyst in each of six laboratories performed single determinations on three different days for a total of 36 determinations using 10 sieves each. Practice E 180 was used as a general guide in developing these precision estimates. The research report for the sieve analysis is not on file at ASTM Headquarters and is considered missing. Because replicate determinations were not made on a single day, no estimate of ~~this,~~ repeatability ~~precision could not be included in Table 1.~~ is possible.

65.2 *Bias*—The bias of the test method has not been determined because of the lack of acceptable reference material.

66. Keywords

66.1 bicarbonate; chloride; iron; loss ~~of~~ on heating; mois~~u~~ture; sieve analysis; soda ash; sodium carbonate; sulfate

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