

# Standard Test Methods for Chemical Analysis of Sodium Chloride<sup>1</sup>

This standard is issued under the fixed designation E534; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

- 1.1 These test methods cover the chemical analyses usually required for sodium chloride.
- 1.2 The analytical procedures appear in the following sections:

Section
5 to 9
10 to 16
17 to 24
25 to 31
32 to 38
39 to 41

- 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 and health practices and determine the applicability of regulatory limitations prior to use.
- 1.5 Review the current material safety data sheets (MSDS) for detailed information concerning toxicity, first-aid procedures, handling, and safety precautions.

## 2. Referenced Documents

2.1 ASTM Standards:<sup>2</sup>

D1193 Specification for Reagent Water

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)<sup>3</sup>

E200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis

## 3. Significance and Use

3.1 Sodium chloride occurs in nature. It is a necessary article of diet as well as the source for production of many sodium compounds and chlorine. The methods listed in 1.2 provide procedures for analyzing sodium chloride to determine if it is suitable for its intended use.

## 4. Reagents

- 4.1 *Purity of Reagents*—Unless otherwise indicated, it is intended that all reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>4</sup>
- 4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II or III reagent water conforming to Specification D1193.

#### SAMPLE PREPARATION

#### 5. Scope

5.1 This test method covers preparation of a sample that will be as representative as possible of the entire bulk quantity. The results of any analysis pertain only to the sample used.

## 6. Apparatus

- 6.1 Coarse Grinder.
- 6.2 High-Speed Blender.
- 6.3 Oven.
- 6.4 Riffle Sampler.
- 6.5 Scale.

#### 7. Reagents

7.1 Hydrochloric Acid, Standard (1 meq/mL HCl)—Prepare and standardize in accordance with Practice E200.

<sup>&</sup>lt;sup>1</sup> These test methods are under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicalsand are under the direct responsibility of Subcommittee E15.02 on Product Standards.

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<sup>&</sup>lt;sup>2</sup> 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

<sup>&</sup>lt;sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

<sup>&</sup>lt;sup>4</sup> 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

#### **TABLE 1 Precision for Moisture Method**

Repeatability			Laboratory Precision			Reproducibility			
Level, %	Standard	Degrees of	95 % Limit, %	Standard	Degrees of	95 % Limit, %	Standard	Degrees of	95 % Limit, %
Level, /o	Deviation, %	Freedom		Deviation, %	Freedom		Deviation, %	Freedom	
0.003 to	0.0014	36	0.004	0.00223	18	0.006	0.00322	8	0.009
0.004									
0.025 to	0.0071	60	0.02	0.00428	30	0.01	0.0138	9	0.04
0.035									

#### 8. Rock and Solar Salt Stock Solutions

- 8.1 Mix and split sample to 500 g, using the riffle sampler.
- 8.2 If sample appears wet, dry at 110°C for 2 h.
- 8.3 Grind the sample to -8 mesh in the coarse grinder.
- 8.4 Mix ground sample well and weigh out a 25.0-g representative portion for rock salt or 50.0 g for solar salt.
- 8.5 Place 200 mL of water in the high-speed blender and start at low speed.
- 8.6 Slowly add the salt sample to the high-speed blender and blend for 5 min.
  - 8.7 Test for water insolubles as described in Sections 17-24.
- 8.8 Save filtrate from water insolubles test and dilute in a volumetric flask to 1 L with water as a stock solution for subsequent analyses.

# 9. Evaporated and Purified Salt Stock Solutions

- 9.1 Mix and split the sample to 100 g for evaporated salt, or 200 g for purified evaporated salt.
  - 9.2 Transfer to a 1-L volumetric flask.
  - 9.3 Add 800 mL of water and allow the salt to dissolve.
- 9.4 Add 2 mL of concentrated HCl to dissolve any water insoluble calcium salts, particularly calcium carbonate.
- 9.5 Dilute to volume with water and use as a stock solution for subsequent analyses.

## **MOISTURE**

## 10. Scope

10.1 This test method determines free moisture in the salt over a concentration range from 0.00 to 0.04 %. It does not determine occluded moisture trapped within the salt crystals. The procedure is based on weight loss after a sample is heated to volatize moisture.

## 11. Apparatus

- 11.1 Analytical Balance.
- 11.2 Desiccator.
- 11.3 Oven.

#### 12. Procedure, Rock and Solar Salt

- 12.1 Weigh 100 g of salt to the nearest 0.05 g into a previously dried and tared moisture dish.
  - 12.2 Dry at 110°C for 2 h.
  - 12.3 Cool in a desiccator and weigh.

## 13. Procedure, Evaporated and Purified Evaporated Salt

- 13.1 Weigh 20 g of salt to the nearest 0.001 g into a previously dried and weighed glass weighing bottle and cover.
  - 13.2 Dry at 110°C for 2 h.
  - 13.3 Cool in a desiccator, replace cover, and weigh.

#### 14. Calculation

14.1 Calculate the percentage of moisture as follows:

moisture, mass (m/m) 
$$\% = \frac{A}{R} \times 100$$
 (1)

where:

A = loss of mass on drying, g, and

B = mass of sample, g.

#### 15. Report

15.1 Report the moisture content to the nearest 0.001 %.

## 16. Precision and Bias

- 16.1 The following criteria should be used in judging the acceptability of results (Note 1):
- 16.1.1 Repeatability (Single Analyst)—The standard deviation for a single determination has been estimated to be the percent absolute values shown in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is the percent absolute values in Table 1.
- 16.1.2 Laboratory precision (Within-Laboratory, Between-Days Variability)—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the percent absolute values shown in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the percent absolute values in Table 1.
- 16.1.3 Reproducibility (Multilaboratory)—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be percent absolute values shown in Table 1. The 95 % limit for the difference between two such averages is the percent absolute values in Table 1.

Note 1—The preceding precision statements are based on an interlaboratory study performed around 1975 on five samples of sodium chloride containing covering the ranges of moisture in Table 1. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day.<sup>5</sup> Practice E180 was used in developing these precision estimates.

<sup>&</sup>lt;sup>5</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E15-1052.

**TABLE 2 Precision for Water Insolubles Method** 

		Repeatability			Reproducibility	
	Coefficient of Variation,	Degrees		Coefficient of Variation,	Degrees	
Level, %	% relative	of	95 % Limit, % relative	% Relative	of	95 % Limit, % relative
	/8 Telative	Freedom		/o neialive	Freedom	
0.002 to 0.005	22.0	20	62	91.7	9	257
0.01 to 0.04	21.9	18	61	42.2	8	118
0.15 to 0.35	22.0	20	62	20.5	9	57

16.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

#### WATER INSOLUBLES

#### 17. Scope

17.1 This gravimetric method determines only the amount of insolubles present in sodium chloride which will not dissolve in water.

## 18. Apparatus

- 18.1 Analytical Balance.
- 18.2 Desiccator.
- 18.3 Magnetic Stirrer with Stirring Bar.
- 18.4 Parabella Filter Funnel Assembly, 61000-mL, or its equivalent with 0.3-µm glass fiber filter disk.

#### 19. Reagents

19.1 Silver Nitrate, Standard Solution, 0.1 meq/mL AgNO<sub>3</sub>—Prepare and standardize in accordance with Practice E200.

# 20. Procedure, Rock and Solar Salts

- 20.1 Transfer a sample prepared in accordance with 8.1 to 8.6 to a 1-L Erlenmeyer flask, washing out the blender with 100 mL of water. Add 300 mL of water to give a total of 600 mL of water added.
- 20.2 Stir on a magnetic stirrer for 1 h. Adjust the stirrer speed to give maximum agitation without danger of losing any sample due to splashing. Place a beaker or watch glass over top of the flask while stirring.
- 20.3 Filter the solution by vacuum through a previously dried (110°C for 1 h) and accurately weighed filter disk using the Parabella funnel. Transfer all insolubles to the paper and wash free of chlorides with water until the filtrate shows no turbidity when tested with 0.1 meq/mL AgNO<sub>3</sub> solution.
- 20.4 Dilute filtrate and washings to 1 L with water in volumetric flask.
  - 20.5 Dry the filter disk at 110°C for 1 h.
- 20.6 Cool in a desiccator and weigh the disk on an analytical balance.
  - 20.7 Save the filtrate for subsequent analyses.

## 21. Procedure, Evaporated and Purified Evaporated Salts

- 21.1 Place a well mixed sample in a 2-L beaker. Use 100-g sample for evaporated or 200 g for purified evaporated salt.
  - 21.2 Add 750 mL of water.
- 21.3 Mix with a mechanical stirrer until solution is complete.
- 21.4 Filter the solution by vacuum through a previously dried (110°C for 1 h) and accurately weighed filter disk using the Parabella funnel. Transfer all insolubles to the paper and wash free of chlorides with water until the filtrate shows no turbidity when tested with 0.1 meq/mL AgNO<sub>3</sub> solution.
  - 21.5 Dry the filter disk at 110°C for 1 h.
- 21.6 Cool in a desiccator and weigh on an analytical balance.
- 21.7 Dilute the filtration and washings to 1 L with water in a volumetric flask and reserve for subsequent analyses.

## 22. Calculation

22.1 Calculate the percentage of water insolubles as follows:

insolubles, % mass 
$$(m/m) = \frac{A}{R} \times 100$$
 (2)

where:

A = increase in mass of filter disk, g, and

B = sample mass, g.

#### 23. Report

23.1 Report the percentage of water insolubles to the nearest percentage shown as follows:

Range, %	Report to, %
0.002 to 0.005	0.001
0.01 to 0.04	0.01
0.15 to 0.35	0.01

## 24. Precision and Bias

- 24.1 The following criteria should be used in judging the acceptability of results (Note 2):
- 24.1.1 Repeatability (Single Analyst)—The coefficient of variation for a single determination has been estimated to be the percent relative values shown in Table 2 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is percent relative values shown in Table 2.
- 24.1.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 23.5 % relative at 57

<sup>&</sup>lt;sup>6</sup> Fisher Scientific No. 9-730-200 has been found satisfactory.

df. The 95 % limit for the difference between two such averages is 66 % relative.

24.1.3 Reproducibility (Multilaboratory)—The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be percent relative values shown in Table 2 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is percent relative values shown in Table 2.

Note 2—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the ranges of water insolubles in Table 2. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day.<sup>5</sup> Practice E180 was used in developing these precision estimates.

24.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

#### CALCIUM AND MAGNESIUM

## 25. Scope

25.1 This test method covers the EDTA titrimetric determination of calcium and magnesium and the EDTA titrimetric determination of calcium. The magnesium content is determined by difference.

## 26. Apparatus

26.1 Magnetic Stirrer with Stirring Bar.

#### 27. Reagents

- 27.1 Eriochrome Black T Indicator Solution, Hydroxy Naphthol Blue, or its equivalent.
- 27.2 *Murexide (Ammonium Purpurate)* Indicator Solution, or its equivalent.
- 27.3 EDTA Standard Solution (1 mL = 0.400 mg calcium)—Dissolve 4.0 g of disodium dihydrogen ethylene diaminetetraacetate (EDTA) in 1 L of water. Standardize this solution against a standard calcium solution prepared by dissolving  $1.000~\rm g$  of  $CaCO_3~\rm and~2~mL$  of HCl in water and diluting to 1 L with water in a volumetric flask. Obtain an exact factor for the EDTA solution. This factor is equal to the milligrams of calcium equivalent to  $1.00~\rm mL$  of EDTA solution. See Practice E200.

$$factor = \frac{W}{V}$$
 (3)

where:

W = calcium in aliquot, mg, and

V = EDTA solution required for titration, mL.

27.4 Ammonium Chloride–Ammonium Hydroxide Solution—Add 67.5 g of ammonium chloride (NH<sub>4</sub>Cl) to 570 mL of ammonium hydroxide (NH<sub>4</sub>OH) contained in a 1-L volumetric flask. Reserve this solution for use as described in 27.6 and 27.7.

27.5 Potassium Cyanide Solution (50 g/L)—Dissolve 50 g of potassium cyanide (KCN) in water and dilute to 1 L with water. Store in a borosilicate glass bottle. (Warning—Potassium cyanide is extremely poisonous.)

TABLE 3 Stock Solutions (Calcium and Magnesium)

Stock Solution	Aliquot, mL
Kansas rock salt	10
Northern rock salt	25
Southern rock salt	50
Evaporated salt	50
Purified salt	200
Solar salt	100

27.6 Magnesium Sulfate Solution (2.5 g/L)—Dissolve 2.5 g of MgSO<sub>4</sub>·7H<sub>2</sub>O in water and dilute to volume with water in a 1-L volumetric flask. Determine the volume of EDTA solution equivalent to 50 mL of MgSO<sub>4</sub> solution as follows: Pipet 50 mL of MgSO<sub>4</sub> solution into a 400-mL beaker. Add 200 mL of water and 2 mL of NH<sub>4</sub>Cl:NH<sub>4</sub>OH solution (27.4). Add 1 mL of KCN solution and a sufficient amount of Eriochrome Black T Indicator solution or its equivalent. Titrate the solution with EDTA solution while stirring with a magnetic stirrer to the true blue end point. This gives the volume of EDTA solution equivalent to 50.0 mL of MgSO<sub>4</sub> solution.

27.7 Buffer Solution—Pipet 50 mL of MgSO<sub>4</sub> solution into the volumetric flask containing the remaining NH<sub>4</sub>Cl·NH<sub>4</sub>OH solution (27.4). Add the exact volume of EDTA solution equivalent to 50 mL of the MgSO<sub>4</sub> solution. Dilute to 1 L with water. Store the solution in a polyethylene bottle.

27.8 Potassium Hydroxide Solution (600 g/L)—Dissolve 150 g of potassium hydroxide (KOH) in 250 mL of water. Cool and store in a polyethylene bottle.

#### 28. Procedure

- 28.1 Using Table 3 as a guide, pipet two aliquots of stock solution into 400-mL beakers to give a titer between 2 and 10 mL of standard EDTA solution. One aliquot is used to determine total calcium and magnesium and the other for calcium.
- 28.2 Dilute to 200 mL with water, if necessary, and place on magnetic stirrer.
  - 28.3 Total Calcium and Magnesium:
- 28.3.1 Add 5 mL of buffer solution, 1 mL of KCN solution, and a sufficient amount of Eriochrome Black T Indicator Solution or its equivalent.
- 28.3.2 Titrate with standard EDTA solution to a true blue color.
  - 28.3.3 Record the millilitres used as Titration 1  $(T_1)$ .
  - 28.4 Calcium Only:
- 28.4.1 Add 2 mL of KOH solution, 1 mL of KCN solution to the other aliquot in 28.1 and 28.2, and stir for about 2 min to precipitate magnesium.
- 28.4.2 Add a sufficient amount of murexide solution or an equivalent calcium indicator solution.
- 28.4.3 Titrate with standard EDTA solution to a true blue color.
  - 28.4.4 Record the millilitres used as Titration 2  $(T_2)$ .

#### **TABLE 4 Precision for Magnesium Method**

Repeatability			Laboratory Precision			Reproducibility			
Range, %	Standard	Degrees of	95 % Limit, %	Standard	Degrees of	95 % Limit, %	Standard	Degrees of	95 % Limit, %
naliye, /o	Deviation, %	Freedom		Deviation, %	Freedom		Deviation, %	Freedom	
0.001 to	0.00071	56	0.002	0.00066	28	0.002	0.00147	8	0.004
0.003									
0.02 to	0.0035	52	0.010	0.033	26	0.092	0.0042	8	0.012
0.025									

#### 29. Calculation

29.1 Calculate the mass percent of calcium as follows:

Ca, % mass 
$$(m/m) = \frac{(T_2) (factor) (0.1)}{S}$$
 (4)

where:

 $T_2$  = EDTA used to titrate calcium only, mL.

S = mass of salt in aliquot, g.

29.1.1 See 27.3 for factor.

29.2 Calculate the mass percent of magnesium as follows:

$$Mg, \% \max (m/m) = \frac{\left(T_1 - T_2\right) \left(factor\right) \left(0.6064\right) \left(0.1\right)}{S} \qquad (5)$$

where:

 $T_1$  = EDTA used to titrate total calcium and magnesium,

S = mass of salt in aliquot, g.

## 30. Report

30.1 Report the percentage of calcium to the nearest 0.001 %. Report the percentage of magnesium to the nearest 0.001 %.

#### 31. Precision and Bias

- 31.1 The following criteria should be used in judging the acceptability of calcium results (Note 3):
- 31.1.1 Repeatability (Single Analyst)—The coefficient of variation for a single determination has been estimated to be 3.56 % relative at 100 df. The 95 % limit for the difference between two such runs is 10 % relative.
- 31.1.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 6.34 % relative at 40 df. The 95 % limit for the difference between two such averages is 18 % relative.
- 31.1.3 Reproducibility (Multilaboratory)—The coefficient of variation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be 9.82 % relative at 7 df. The 95 % limit for the difference between two such averages is 28 % relative.

Note 3—The preceding precision statements are based on an interlaboratory study performed around 1975 on five samples of sodium chloride covering the range from 0.036 to 0.909 % calcium. One analyst in each of eight laboratories performed duplicate determinations and repeated them on a second day.<sup>5</sup> Practice E180 was used in developing these precision estimates.

- 31.1.4 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.
- 31.2 The following criteria should be used in judging the acceptability of magnesium results (Note 4):
- 31.2.1 Repeatability (Single Analyst)—The standard deviation for a single determination has been estimated to be percent absolute values shown in Table 4 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is the percent absolute values shown in Table 4.
- 31.2.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The standard deviation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be the percent absolute values shown in Table 4 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the percent absolute values shown in Table 4.
- 31.2.3 Reproducibility (Multilaboratory)—The standard deviation of results (each the average of duplicates), obtained by analysts in different laboratories, has been estimated to be the percent absolute values shown in Table 4 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the percent absolute values shown in Table 4.

Note 4—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the ranges of magnesium given in Table 4. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day.<sup>5</sup> Practice E180 was used in developing these precision estimates.

31.2.4 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

#### **SULFATE**

# 32. Scope

32.1 This test method covers the gravimetric determination of the sulfate content of sodium chloride.

#### 33. Apparatus

- 33.1 Gooch Asbestos Slurry.
- 33.2 Gooch Filtering Crucible and Holder.
- 33.3 Muffle Furnace.
- 33.4 Oven.

**TABLE 5 Stock Solutions (Sulfate)** 

Stock Solution	Aliquot, mL
Rock salt	40
Evaporated salt	100
Purified evaporated salt	200
Solar salt	100

# 34. Reagents

- 34.1 Barium Chloride Solution (120 g  $BaCl_2$   $2H_2O/L$ )—Dissolve 120 g of barium chloride dihydrate in about 750 mL of water, filter, and dilute to 1 L.<sup>7</sup>
- 34.2 *Hydrochloric Acid Standard Solution* (1 meq/mLHCl)—Prepare and standardize in accordance with Practice E200.
- 34.3 *Methyl Orange Indicator Solution* (1 g/L)—Dissolve 0.1 g of methyl orange in 100 mL of water and filter if necessary.<sup>7</sup>

#### 35. Procedure

- 35.1 Using Table 5 as a guide, pipet the recommended aliquot of stock solution into a 400-mL beaker.
- 35.2 Dilute to 200 mL, add a few drops of methyl orange indicator solution and acidify with 1 mL of HCl (1+1) if necessary.
- 35.3 Heat solution gently to boiling and add 10 mL of BaCl<sub>2</sub> solution dropwise while stirring.
- 35.4 Digest on a hot plate below the boiling point for 30 min.
  - 35.5 Cool overnight.
- 35.6 Filter through a tared Gooch crucible previously prepared with an asbestos mat and ignited in a muffle furnace at 800°C for 30 min. Transfer all the precipitate to the crucible with a rubber policeman. Wash with portions of hot water until washings are free of chlorides.
- 35.7 Dry the crucible at  $110^{\circ}$ C for 15 min, then ignite in a muffle furnace at  $800^{\circ}$ C for 30 min.
  - 35.8 Cool in a desiccator and reweigh.

## 36. Calculation

36.1 Calculate percentage of sulfate as follows:

sulfate, % mass 
$$(m/m) = \frac{A}{R} \times 0.4115 \times 100$$
 (6)

where:

A = mass of precipitate, g, and

B = mass of salt in aliquot, g.

## 37. Report

37.1 Report the percentage of sulfate to the nearest 0.001 %.

## 38. Precision and Bias

- 38.1 The following criteria should be used in judging the acceptability of results (Note 5):
- 38.1.1 Repeatability (Single Analyst)—The coefficient of variation for a single determination has been estimated to be 7.14 % relative at 120 df. The 95 % limit for the difference between two such runs is 20 % relative.
- 38.1.2 Laboratory Precision (Within-Laboratory, Between-Days Variability)—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst on different days, has been estimated to be 8.03 % relative at 60 df. The 95 % limit for the difference between two such averages is 22 % relative.
- 38.1.3 *Reproducibility (Multilaboratory)*—The coefficient of variation of results (each the average of duplicates), obtained by the same analyst in different laboratories, has been estimated to be 8.01 % relative at 9 df. The 95 % limit for the difference between two such averages is 22 % relative.
- Note 5—The preceding precision statements are based on an interlaboratory study performed around 1975 on six samples of sodium chloride covering the range from 0.016 to 2.030 % sulfate. One analyst in each of ten laboratories performed duplicate determinations and repeated them on a second day. Practice E180 was used in developing these precision estimates.
- 38.1.4 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

#### REPORTING OF ANALYSES

#### 39. Scope

39.1 Analyses should be reported on a dry basis. If analyses are on an as received sample, correction should be made by converting to a dry basis. Sodium chloride purity is determined by subtracting the total percentage of impurities from 100. Moisture should be reported as a separate value.

## 40. Procedure

- 40.1 Convert sulfate to calcium sulfate and the unused calcium to calcium chloride unless the sulfate in sample exceeds the quantity necessary to combine with the calcium. In this case, convert the calcium to calcium sulfate and the unused sulfate first to magnesium sulfate, and the remaining sulfate, if any, to sodium sulfate. Convert the unused magnesium to magnesium chloride.
- 40.2 Report rock and solar salt impurities to the second decimal place and salt purity, by difference, to the first decimal place.
- 40.3 Report evaporated salt impurities to the third decimal place and salt purity, by difference, to the second decimal place.
- 40.4 Report purified salt impurities to the fourth decimal place and salt purity, by difference, to the third decimal place.

#### 41. Conversion Factors

 $BaSO_4 \times 0.4116 = SO_4$  $BaSO_4 \times 0.5832 = CaSO_2$ 

 $BaSO_4 \times 0.6086 = Na_2SO_4$ 

<sup>&</sup>lt;sup>7</sup> This reagent is also described in Practice E200.



Ca	×	$3.3967 = CaSO_4$
Ca	×	0.6064 = Mg
CaSO <sub>4</sub>	×	0.2944 = Ca
CaSO <sub>4</sub>	×	$0.8153 = CaCl_2$
CaSO <sub>4</sub>	×	$0.8842 = MgSO_4$
CaSO <sub>4</sub>	×	$1.0434 = Na_2SO_4$
Mg	×	$3.9173 = MgCl_2$
MgCl <sub>2</sub>	×	$1.4296 = CaSO_4$
MgCl <sub>2</sub>	×	$1.2641 = MgSO_4$

## 42. Keywords

42.1 calcium; evaporated salt; magnesium; moisture; purified salt; rock salt; sodium chloride; sulfate; water insolubles

#### SUMMARY OF CHANGES

Subcommittee E15.02 has identified the location of selected changes to this standard since the last issue (E534-08) that may impact the use of this standard.

(1) Deleted "in almost unlimited quantities" from 3.1.

(2) Added "or watch glass" to 20.2.

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