



Designation: **E534 – 08 E534 – 13**

Standard Test Methods for Chemical Analysis of Sodium Chloride¹

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 (ϵ) 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:

Sample Preparation	Section 5 to 9
Moisture	10 to 16
Water Insolubles	17 to 24
Calcium and Magnesium	25 to 31
Sulfate	32 to 38
Reporting of Analyses	39 to 41

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Water Insolubles	17 to 24
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Reporting of Analyses	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.

<https://standards.iteh.ai/catalog/standards/sist/d4cc441a-3b46-4f81-8889-a6a85b8133cd/astm-e534-13>

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

[E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals \(Withdrawn 2009\)](#)³

[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 in almost unlimited quantities. 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.

¹ These test methods are under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicals and are under the direct responsibility of Subcommittee E15.02 on Product Standards.

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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 The last approved version of this historical standard is referenced on www.astm.org.

*A Summary of Changes section appears at the end of this standard

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.⁴

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**.

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 volatilize moisture.

⁴ *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

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

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

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:

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

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.⁵ Practice **E180** was used in developing these precision estimates.

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- μ m glass fiber filter disk.

19. Reagents

19.1 *Silver Nitrate, Standard Solution,* 0.1 meq/mL AgNO₃—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 AgNO₃ 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 AgNO₃ 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:

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

where:

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

⁵ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E15-1052.

⁶ Fisher Scientific No. 9-730-200 has been found satisfactory.

TABLE 2 Precision for Water Insolubles Method

Level, %	Coefficient of Variation, % relative	Repeatability		Reproducibility		
		Degrees of Freedom	95 % Limit, % relative	Coefficient of Variation, % Relative	Degrees of Freedom	95 % Limit, % relative
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

TABLE 2 Precision for Water Insolubles Method

Level, %	Coefficient of Variation, % relative	Repeatability		Reproducibility		
		Degrees of Freedom	95 % Limit, % relative	Coefficient of Variation, % Relative	Degrees of Freedom	95 % Limit, % relative
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

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
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 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.⁵ 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.

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

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

27.3 *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 g of CaCO₃ 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 mL of EDTA solution. See Practice E200.

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

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 (NH₄Cl) to 570 mL of ammonium hydroxide (NH₄OH) 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.)

27.6 *Magnesium Sulfate Solution* (2.5 g/L)—Dissolve 2.5 g of MgSO₄·7H₂O 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 MgSO₄ solution as follows: Pipet 50 mL of MgSO₄ solution into a 400-mL beaker. Add 200 mL of water and 2 mL of NH₄Cl:NH₄OH 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 MgSO₄ solution.

27.7 *Buffer Solution*—Pipet 50 mL of MgSO₄ solution into the volumetric flask containing the remaining NH₄Cl·NH₄OH solution (27.4). Add the exact volume of EDTA solution equivalent to 50 mL of the MgSO₄ 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.