

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

This standard is issued under the fixed designation E 534; 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 ( $\epsilon$ ) 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  |
|-----------------------|----------|
| Sample Preparation    | 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 |

1.3 The values stated in SI units are to be regarded as the 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:

D 1193 Specification for Reagent Water<sup>2</sup>

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals<sup>3</sup> E 200 Practice for Preparation, Standardization, and Storage of Standard and Reagent Solutions for Chemical Analysis<sup>3</sup>

## 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.

#### 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 D 1193.

## 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 *N* HCl—Prepare and standardize in accordance with Practice E 200.

# 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.

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<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 15.05.

<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.

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#### **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, % |
|          | 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    |               |            |               |                      |            |               |                 |            |               |

8.8 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  $110^{\circ}$ 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, weight % = 
$$\frac{A}{B} \times 100$$
 (1)

where:

A = loss of weight on drying, g, and

B = weight 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 E 180 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*,<sup>6</sup> 1000-mL, or its equivalent with 0.3-µm glass fiber filter disk.

<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.

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