Designation: D 345 – ~~97~~02

Standard Test Method for Sampling and Testing Calcium Chloride for Roads and Structural Applications[1]

This standard is issued under the fixed designation D 345; 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 This test method covers sampling of solid and liquid forms of calcium chloride, and sieve analysis of solid form calcium chloride. Referee procedures for chemical analysis are covered in Test Methods E 449.

1.2 A rapid method of chemical analysis is shown in Appendix X1. This rapid method is not to be used for determining chemical compliance of calcium chloride with specification requirements, such as in Specification D 98.

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

1.4 *This standard does not purport to address 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates[2]

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[1] This test method is under the jurisdiction of ASTM Committee ~~D-4~~ D04 on Road and Paving Materials and is the direct responsibility of Subcommittee D04.31 on Calcium and Sodium Chlorides and Other Deicing Materials.

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D 98 Specification for Calcium Chloride[3]
D 140 Practice for Sampling Bituminous Materials[3]
E 11 Specification for Wire- Cloth and Sieves for Testing Purposes[4]
E 449 Test Methods for Analysis of Calcium Chloride[5]

3. Significance and Use

3.1 This test method describes procedures to be used for sampling calcium chloride, and for determining grading of solid forms of calcium chloride for comparison with the requirements of a specification, such as Specification D 98.

4. Apparatus

4.1 *Sampling Equipment*:

4.1.1 *Sampling Tube*—Metal tube, approximately 25 to 40 mm (1 to 1½ in.) diameter, and of appropriate length (500 mm (20 in.), minimum) for sampling solid-form calcium chloride.

4.1.2 *Sampling Bottle*—Sampling devices such as described in Practice D 140 ~~may be used.~~ for sampling liquid calcium chloride.

4.1.3 *Sampling Containers*—Glass bottles or semi-rigid plastic containers with covers with air-tight seal for protecting the samples from time of sampling until the samples ae used in testing. The containers for solid-form calcium chloride shall have wide mouths or openings for solid-form calcium chloride.

4.2 *Sieves*, with the size openings required for the test, shall conform to the requirements of Specification E 11.

4.3 *Balances*, having a capacity of at least 250 g, shall be readable to 0.1 g, and shall be accurate to 0.1 g or 0.1 % of the test load, whichever is greater, at any point within the range of use.

5. Sampling

5.1 *Solid-Form Calcium Chloride*:

5.1.1 *Package Shipments*—Select not less than three containers at random from the shipment. For drums, sample each of the containers by scraping aside the top layer to a depth of approximately 25 mm (1 in.) and taking 0.5-kg (1-lb) samples by means of a sampling tube or other method that will ensure a sample that is representative of a cross section of the materials in the container to a depth of at least 150 mm (6 in.). For bags, take 0.5-kg (1 lb) samples by means of a sampling tube penetrating at least 300 mm (1 ft) into the bag. For small containers with capacity not more than 5 kg (10 lb), use the entire contents of the container.

5.1.2 *Bulk Shipments*—Select samples from at least three locations in the shipment. Scrape aside the top layer to a depth of approximately 300 mm (1 ft). Using a sampling tube, obtain a sample extending from the cleared surface to at least 50 % of the depth of the material in the container, or a depth of approximately 1 m (3 ft), whichever is less. Each sample shall contain at least 0.5 kg (1 lb) of material.

5.1.3 Use caution during the sampling operation to avoid exposing the sample unduly to atmospheric moisture. Immediately and thoroughly mix the individual samples to form a representative composite sample of material and store in a sealed glass or suitable plastic container.

5.2 *Liquid-Form Calcium Chloride, Bulk Shipments or Storage*—Obtain a sample of at least 500 mL (1 pt) from the bulk shipping container or storage tank or during discharge. Recirculate the solution in the tank until it is homogenous, then take one or more samples by means of an appropriate sampling device.

5.2.1 Use caution during the sampling operation to avoid exposing the sample unduly to atmospheric moisture. If more than one sample is taken, immediately and thoroughly mix the individual samples to form a representative composite sample of material and store in a sealed glass or suitable plastic container.

6. Procedure

6.1 *Sieve Analysis*:

6.1.1 Sieve approximately 200 g of calcium chloride, weighed to the nearest 0.1 g, in conformance with Method C 136, except that sieving shall be completed within 1 min ± 5 s (see Note 1). Use the sieve sizes necessary to provide the information required by the specification covering the material, and such other sizes as necessary to regulate the amount of material on a sieve, together with a bottom pan and a cover. Report the sieve analysis on the basis of the grading requirements in Specification D 98 or other applicable specification.

NOTE 1—It is important that the sieving be completed within approximately 1 min and weighing be completed quickly due to the hygroscopic nature of calcium chloride. When possible, the sieve analysis should be performed in an area with low relative humidity.

6.2 *Chemical Analysis*—Determine the chemical composition of the sample using Test Method E 449.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.
[3] *Annual Book of ASTM Standards*, Vol 04.03.
[4] *Annual Book of ASTM Standards*, Vol 14.02.
[5] *Annual Book of ASTM Standards*, Vol 15.05.

7. Precision and Bias

7.1 *Sieve Analysis*:

7.1.1 *Precision*—Precision indexes for sieving calcium chloride have not been determined. Use the precision indexes of Test Method C 136 as a guide for the sieve analysis carried out under this test method.

7.1.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedure for measuring the sieve analysis of calcium chloride, no statement on bias is being made.

8. Keywords

8.1 calcium chloride; grading; sampling; sieve analysis

APPENDIX

(Nonmandatory Information)

X1. RAPID METHOD OF ANALYSIS FOR CALCIUM CHLORIDE

X1.1 Scope

X1.1.1 This appendix covers a rapid method for chemical analysis of calcium chloride.

X1.2 Significance and Use

X1.2.1 The procedure for chemical analysis in this appendix determines total chlorides ($CaCl_2$, $MgCl_2$, NaCl, KCl, etc.) present in the sample and expresses that value as calcium chloride.

X1.2.2 This rapid method only determines total chlorides, and does not guarantee that the chloride is present as calcium chloride, therefore no material should be accepted or rejected under this method of analysis. However, when used in conjunction with Test Method E 449 on continuing shipments from a given source, the procedure may be used to determine changes in the composition of the material being furnished, especially with regard to moisture content. Thus the frequency of testing by Test Method E 449 may be reduced.

X1.3 Apparatus

X1.3.1 *Glassware*—Standard weighing bottles, volumetric flasks, and burets.

X1.3.2 *Balance*, as described in 4.3.

X1.4 Reagents

X1.4.1 *Nitric acid (HNO_3)*, 1 + 1 by volume.

X1.4.2 *Dichlorofluorescein*—0.1 % aqueous solution of the sodium salt of dichlorofluorescein.

X1.4.3 *Silver Nitrate Solution*—0.1 *N* $AgNO_3$.

X1.4.4 *Ammonium Hydroxide (NH_4OH)*.

X1.5 Procedure

X1.5.1 Weigh a 20.0 ± 0.2-g sample in a weighing bottle. Add distilled water to solid form calcium chloride to dissolve. Add sufficient nitric acid (HNO_3) (1 + 1) to clear, dilute to 1000 mL in a volumetric flask, and mix thoroughly. Titrate a 10-mL aliquot, containing five to seven drops of the dichlorofluorescein solution as indicator, with 0.1 *N* silver nitrate ($AgNO_3$) solution to a pink end point. Use of other suitable indicators is permitted.

X1.5.2 This procedure is not suitable for testing the material if more than a slight precipitate of magnesium hydroxide ($Mg(OH)_2$) appears when a 10 % filtered solution of the material is made alkaline with ammonium hydroxide (NH_4OH).

X1.6 Calculation

X1.6.1 Calculate the total chlorides expressed as percent $CaCl_2$, *C*, as follows:

$$C = \text{mL } 0.1\ N\ AgNO_3 \times 2.77 \quad \text{(X1.1)}$$

X1.7 Precision and Bias

X1.7.1 This procedure for chemical analysis is not intended for precise measurement or determination of specification compliance. Statements for precision and bias are therefore not necessary.