Designation: D1411 – 09

Standard Test Methods for Water-Soluble Chlorides Present as Admixtures in Graded Aggregate Road Mixes[1]

This standard is issued under the fixed designation D1411; 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 are applicable to the determination of water-soluble calcium, magnesium, and sodium chlorides used as admixtures in the preparation of graded aggregate road materials.

1.1.1 *Method A, Total Chlorides*—Add an excess of a standard silver nitrate solution to the filtered sample; back titrate the excess silver with a standard ammonium thiocyanate solution to a red-orange endpoint using Volhard indicator.

1.1.2 *Method B, Calcium Chloride*—Calcium in an alkaline solution is titrated with standard EDTA solution using calcein modified as an indicator. The color change is from green to blue.

1.1.3 *Method C, Magnesium Chloride*—Calcium and magnesium, in a solution buffered to a pH of 10, are titrated with standard EDTA solution using eriochrome black T as an indicator. The color change is from red to blue. Magnesium chloride is calculated by subtracting the EDTA calcium equivalent volume determined in the analysis for calcium chloride from the total EDTA volume used, to obtain the magnesium EDTA equivalent by difference.

1.1.4 *Method D, Alkali Chloride*—Alkali chlorides are calculated from the difference of the total chloride and calcium and magnesium determinations.

NOTE 1—These test methods assume that the aggregate did not contain significant amounts of the water-soluble chlorides in question before the admixture was added. If significant amounts of these chlorides are known or suspected to be present, the aggregate shall be tested for these constituents according to these test methods and the proper corrections made.

1.2 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.2.1 The method used to specify how data are collected, calculated, or recorded in this standard is not directly related to the accuracy to which the data can be applied in design or other uses, or both. How one applies the results obtained using this standard is beyond its scope.

1.3 *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.*

1.4 This practice offers a set of instructions for performing one or more specific operations. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this practice may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.

1.5 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1193 Specification for Reagent Water

D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction

D6026 Practice for Using Significant Digits in Geotechnical Data

3. Terminology

3.1 Except as follows in 3.2, all definitions are in accordance with Terminology D653.

[1] These test methods are under the jurisdiction of Committee D18 on Soil and Rock and are the direct responsibility of Subcommittee D18.15 on Stabilization With Admixtures.

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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.

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *admixture—in road construction*, a material other than water, aggregates, hydraulic cement, and fiber reinforcement used as an ingredient and added immediately before or during the mixing of road materials.

4. Significance and Use

4.1 The percentage of calcium chloride, magnesium chloride, and alkali chloride (calculated as sodium chloride) in graded aggregate obtained from aggregate roads or aggregate bases under paved roads is of interest to highway departments using calcium chloride or sodium chloride stabilization. The percentages of calcium chloride or sodium chloride obtained in these test methods are compared with the quantities added to determine whether the road material and stabilizing agent were properly mixed, whether leaching of the stabilizing agent occurred, etc.

NOTE 2—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/sampling/inspection/etc. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; D3740 provides a means of evaluating some of those factors.

5. Purity of Reagents

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.[3] 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, reference to water shall be understood to mean distilled water or water of equal purity conforming to Specification D1193.

6. Preparation of Samples

6.1 Submit samples from the field to the laboratory in sealed containers. For each individual sample, break up any agglomerated particles in a mortar and quarter the sample. Accurately weigh approximately 300 g (dry weight) of a quartered sample and transfer it to a 1-L bottle. Add 479 mL of water, 20 mL of ferric ammonium sulfate (100 g/L) and 1 mL of ammonium hydroxide (sp gr 0.90). Agitate in a shaker overnight or for 12 to 15 h. Filter through fluted filter paper, discarding the first 50 mL and retaining the remainder. Determine calcium, magnesium, and chloride in the clear filtrate.

NOTE 3—The addition of ferric ammonium sulfate and ammonium hydroxide should give complete coagulation of most of the dispersible materials and give clear filtrates.

[3] "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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

TOTAL CHLORIDES

7. Summary of Test Method

7.1 Add an excess of a standard silver nitrate solution to the filtered sample; and back titrate the excess silver with a standard ammonium thiocyanate solution to a red-orange endpoint using Volhard indicator.

8. Interferences

8.1 In effect, this method is a "Total Halides" method as it cannot distinguish between chloride and other halides that may be present.

9. Reagents

9.1 *Ammonium Thiocyanate, Standard Solution* (*0.1 N*)—Dissolve 7.6 g of ammonium thiocyanate (NH_4SCN) in water and dilute to 1 L. Standardize against the 0.1 *N* $AgNO_3$ solution.

9.2 *Benzyl Alcohol, Chlorine-Free* .

9.3 *Nitric Acid (sp gr 1.42)*—Concentrated HNO_3.

9.4 *Silver Nitrate, Standard Solution* (*0.1 N*)—Dissolve 17.0 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

9.5 *Volhard Indicator Solution*—Dissolve 10 g of ferric ammonium sulfate ($FeNH_4(SO_4)_2 \cdot 12H_2O$) in 100 mL of water and add 1 mL of HNO_3.

10. Procedure

10.1 Acidify a suitable portion of the sample solution (see Section 6) (10-mL aliquot) with 3 to 5 mL of concentrated HNO_3, add a known volume of the 0.1 *N* $AgNO_3$ solution that is in excess of the amount required to precipitate the chloride, and heat to boiling to coagulate silver chloride (AgCl). Cool to room temperature, add 3 to 5 mL of benzyl alcohol, and shake vigorously. If help is needed to determine the point at which an excess of $AgNO_3$ solution is achieved, add 5 mL of Volhard indicator and 1 mL of 0.1 *N* ammonium thiocyanate prior to the addition of 0.1 *N* $AgNO_3$ solution. The red orange color of Volhard indicator will disappear when excess $AgNO_3$ solution has been added.

10.2 Add Volhard indicator solution to the solution and titrate the excess $AgNO_3$ with 0.1 *N* NH_4SCN solution.

11. Calculation

11.1 Calculate the percentage of total chloride ion as follows:

$$\text{chloride, \%} \frac{[(aN - a`N`) \times 0.0355]}{bw} \times 100 \quad (1)$$

where:

- a = millilitres of $AgNO_3$ solution added,
- $a`$ = millilitres of NH_4SCN solution required for the titration,
- N = normality of the $AgNO_3$ solution,
- $N`$ = normality of NH_4SCN solution,
- b = millilitres of solution in aliquot taken, divided by 500, and