Designation: D4542 – 15

Standard Test Methods for
Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer[1]

This standard is issued under the fixed designation D4542; 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 a rapid procedure for squeezing pore water from finegrained soils for the purpose of determining the amount of soluble salts present in the extracted pore water.

1.2 These test methods were developed for soils having a water content equal to or greater than approximately 14 %, for example, marine soils. An extensive summary of procedures for extracting pore water from soils has been presented by Kriukov and Manheim **(1)**.[2]

1.3 These test methods are not generally applicable for determining the soluble salt content of the pore water extracted from coarse-grained soils, such as clean sands and gravels.

1.4 Test Method A provides a procedure using a refractometer with a refraction index scale; Test Method B provides a procedure using a refractometer with a parts per thousand (ppt) scale.

1.5 *Units*—The values stated in SI units are to be regarded as the standard.

1.6 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026 unless superseded by these test methods.

1.6.1 The procedures used to specify how data are collected/recorded and calculated in the standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of these test methods to consider significant digits used in analysis methods for engineering data.

1.7 *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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

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

E832 Specification for Laboratory Filter Papers

2.2 *Federal Document:*

GG-S-945a Specification for Syringe and Needle, Disposable, Hypodermic, Sterile, Single Injection[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of common terms in this standard, refer to Terminology D653.

4. Significance and Use

4.1 The soluble salt content may be used to correct the index properties of soils such as water content, void ratio, specific gravity, degree of saturation, and dry density).

4.2 Minimize the time period between sampling and testing to reduce chemical changes, which may occur within the soil sample.

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.06 on Physical-Chemical Interactions of Soil and Rock.

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[2] The boldface numbers in parentheses refer to the list of references appended to this standard.

[3] 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.

[4] Available from Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, PA, 19120.

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

NOTE 1—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 ensure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

NOTE 2—Hulbert and Brindle (**2**) and Torrance (**3**) have shown that prolonged storage should be avoided as unpredictable and non-reproducible chemical changes may occur.

5. Apparatus

5.1 *Refractometer*—A temperature compensated refractometer scaled to either index of refraction or ppt (parts per thousand). A typical hand held refractometer is shown in Fig. 1.

5.2 *Soil Press*—The apparatus shall conform to the example shown in Fig. 2. It should be constructed from material appropriate for the application and chemically compatible with the material being tested.

5.3 *Syringe*—A 25-cm^3 syringe without needle, in accordance with Fed. Std. GG-S-945a.

5.4 *Balance*—A balance capable of determining mass with a readability of ±0.01 g.

5.5 *Filter Paper:*

5.5.1 A general purpose quantitative filter paper in accordance with Specification E832, Type II, Class F, for medium crystalline precipitates in the size range from 5 to 10 µm, with an ash content of 0.13 mg/12.5-cm circle. Cut filter paper to a diameter of 55 mm.

5.5.2 A general purpose quantitative filter paper in accordance with Specification E832, Type II, Class G, for fine crystalline precipitates in the size range from 0.45 µm, with an ash content of 0.13 mg/12.5-cm circle. Cut filter paper to a diameter of 25 mm.

5.6 *Refrigerator*—Cooling unit capable of maintaining a uniform temperature between 1 and 5°C.

FIG. 1 Typical Hand-Held Refractometer

5.7 *Micro-Syringe Filter Holder*—A device to filter a liquid directly from a syringe.[5]

5.8 *100-mL Polyethylene or Glass Bottle and Cap.*

5.9 *Miscellaneous Supplies*—Distilled water, alcohol, diluted HCl (1:10), detergent, and optional sterile bags for sample storage (see 8.6).

6. Reagents

6.1 *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 Analytic Reagents of the American Chemical Society, where such specifications are available.[6]

6.1.1 Hydrochloric Acid diluted 1:10
6.1.2 Alcohol
6.1.3 Chromic acid
6.1.4 Acetone

7. Preparation of Apparatus

7.1 Wash the parts of the press thoroughly. Rinse twice with distilled water and dry. Rust should not be present, especially inside or around the top of the cylinder. If rust is present, scrub gently with steel wool and soap or chromic acid. Rinse well with tap water and then twice with distilled water and dry.

7.2 If the press parts have been coated with rust preventer, wash them with alcohol and rinse once with tap water and twice with distilled water.

7.3 Dry by a method that will not contaminate the press. Clean compressed air, oven or air drying, or rinsing with acetone followed by air drying are acceptable.

7.4 Assemble the press.

NOTE 3—To prevent mud from circumventing the stainless steel wire screen, use flexible TFE-fluorocarbon gaskets on each side.

8. Sampling and Test Specimen Squeezing

8.1 Select a representative soil sample of approximately 50 g and place into the cylinder on top of a single sheet of 5 to 10-µm (55-mm) filter paper.

8.2 Apply pressure slowly until the first drops of water are expelled, then insert a clean, disposable, syringe (25 mL) in the effluent passage shown in Fig. 2. This process reduces the amount of air in the syringe and therefore, the amount of evaporation.

8.3 Apply pressure gradually to a maximum of 80 MPa, and hold until no more water is expelled or until the syringe is full (see Note 4 and Note 5).

8.4 Withdraw the syringe when the pressure is at a maximum and immediately expel the fluid from the syringe through

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[5] An apparatus such as the stainless steel Millipore Micro-Syringe Filter Holder XX30-025-00 is satisfactory for this purpose.

[6] Reagent Chemicals, American Chemical Society Specifications," American Chemical Society, Washington, DC. For suggestions on the testing of reagents lot listed by the America Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

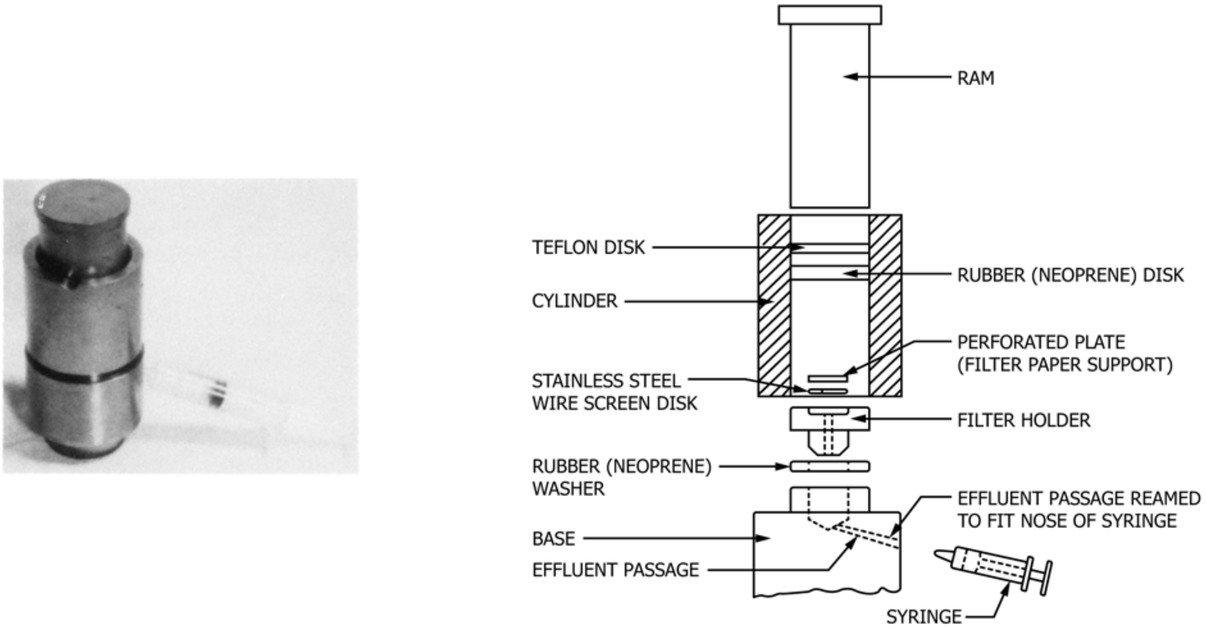

FIG. 2 Example of Heavy Duty Soil Press

a stainless steel micro-syringe holder, fitted with fresh 0.45-µm (25-mm) filter paper, into a clean 100-mL bottle (see Note 6). Cap the bottle. Expose the collected water to the atmosphere as little as possible.

8.5 Repeat 8.1 – 8.4, using the same syringe and filter if additional water is needed for experimentation and can be collected. Usually about 25 mL of pore water may be collected from 50 g of soil (see Note 7 and Note 8). Store the water at a temperature between 1 and 5°C (see Note 9 and Sections 9 and 10).

8.6 Remove the soil from the press. If additional tests are anticipated, store soil in a sterile plastic bag at a temperature between 1 and 5°C (see Note 9).

NOTE 4—Only a few drops (0.05 mL) of pore fluid are required to conduct the soluble salt determination by refractometer. It is recommended that 25 mL of pore water be collected, if possible, to allow for retesting or additional tests, or both.

NOTE 5—Kriukov and Komarova (**4**) have found that at a pressure of 59 MPa the chloride content drops in homogeneous soils. Manheim (**5**) reports using 101 MPa routinely. An average of these two recommendations is 80 MPa.

NOTE 6—Polyethylene or glass bottles should be washed with detergent and rinsed with tap water. They should then be rinsed once with diluted HCl (1:10) and twice with distilled water and then drained thoroughly.

NOTE 7—The amount of water expelled will depend on the initial water content of the sample. For example, using a 50-g sample of moist soil and assuming that 1 cm^3 of liquid is required to fill the apparatus, the following water contents are required to achieve the indicated amounts of expelled water:

Initial Water Content	*Amount of Water Expelled (mL)*
104	25
70	20
47	15
28	10
14	5
2	0

NOTE 8—The addition of fluid to a sample to increase its water content so that an increased amount of water can be expelled may result in the leaching of salts present in the soil and may distort the original salt content of the pore water.

NOTE 9—A storage temperature of about 5°C is recommended to reduce the growth of sulfate-reducing bacterium called Desulfovibrio.

9. Procedure

9.1 *Method A—Salinity Determination Using a Refractometer with a Refraction Index Scale:*

9.1.1 Filter specimen through a 0.45-µm filter.

9.1.2 Thoroughly wash with distilled water and dry the refractometer, shown in Fig. 1. Zero the instrument in accordance with the manufacturer's specifications.

9.1.3 Place a few drops of liquid on the refractometer platform and close the slide gently.

9.1.4 Hold the refractometer at right angles to a light source, and read and record the refractive index.

9.1.5 Obtain and record the salinity from the accompanying graph (Fig. 3), where n is the refractive index of the liquid and n_o the refractive index of distilled water (1.330 at 20°C).

NOTE 10—A typical temperature-compensated instrument is accurate to 0.1 % between 15.6 and 37.8°C; the instrument is most accurate between 18.3 and 21.1°C.

9.2 *Method B—Salinity Determination Using a Refractometer with a ppt Scale:*

9.2.1 Filter specimen through a 0.45-µm filter.

9.2.2 Thoroughly wash with distilled water and dry the refractometer, shown in Fig. 1. Zero the instrument in accordance with the manufacturer's specifications.

9.2.3 Place one to two drops of liquid into the semicircle of the white plastic area, which is held firmly against the glass platform. Allow the liquid to escape only under the white plastic area.

9.2.4 Hold the refractometer at right angles to a light source, and read and record the salinity. If the liquid is properly

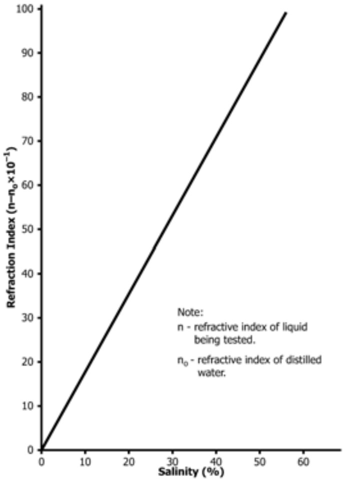

FIG. 3 Graph of Salinity versus Refraction Index

introduced, there should be a distinct black/white boundary. Read and record where the bottom of the hairline touches the beginning of the black boundary. Read to the nearest whole number.

NOTE 11—Salinity is given in parts per thousand parts (0/00). Salinity is the total amount of solid material, in grams, contained in 1 kg of sea water when all the carbonate has been converted to oxide, the bromine and iodine replaced by chlorine, and all organic matter completely oxidized (Sverdrup, Johnson, Fleming, (**6**)).

NOTE 12—Error in reading directly from the salinity scale is ±0.3 ppt.

NOTE 13—Actual sea water samples may have a black/yellow/white boundary when reading the refractometer. Readings are always taken at the beginning of the black boundary. Two to three readings should always be taken to ensure accuracy and precision.

10. Report: Test Data Sheet(s)/Forms(s)

10.1 Record the date and testing personnel.

10.2 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.8.

10.3 For each test, record as a minimum the following observations:

10.3.1 Location,
10.3.2 Depth,
10.3.3 Soil type,
10.3.4 ppt or percent soluble solids,
10.3.5 n, no, salinity, and
10.3.6 Refraction index or parts per thousand (ppt) from three locations recorded to whole numbers.

11. Precision and Bias

11.1 *Precision*—Test data on precision is not presented due to the nature of the soil materials tested by this test method. It is either not feasible or too costly at this time to have ten or more laboratories participate in a round-robin testing program. In addition, it is either not feasible or too costly to produce multiple specimens that have uniform physical properties. Any variation observed in the data is just as likely to be due to specimen variation as to operator or laboratory testing variation.

11.1.1 Subcommittee D18.06 is seeking any data from the users of this test method that might be used to make a limited statement on precision.

11.2 *Bias*—There is no accepted reference value for this test method, therefore bias cannot be determined.

12. Keywords

12.1 marine soils; porewater; refractometer; salt content; soluble salt; soluble salt content

APPENDIX

(Nonmandatory Information)

X1. MASS COMPOSITION

X1.1 Mass Composition Terms

X1.1.1 A mass composition diagram for partially saturated soils with soluble salts is shown in Fig. X1.1. Based on this figure the following terms and definitions are defined for describing various relationships among soil phases as proposed by Noorany (**7**). There are other phase relationship conventions proposed in several references below which offer different approaches to calculating corrections (**8-10**) to the related index properties.

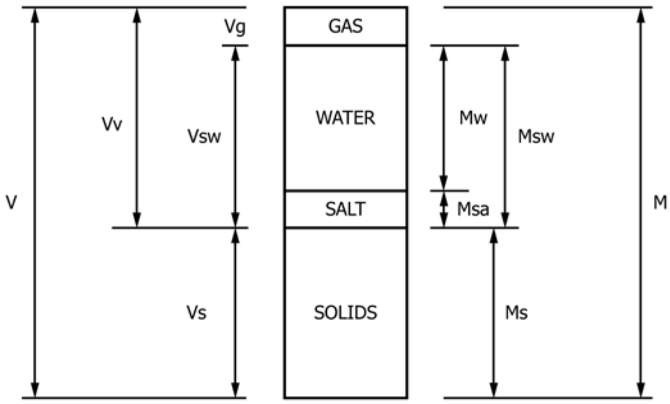

FIG. X1.1 Mass Composition Diagram for Soils Containing Soluble Salts

M = wet mass
M_d = oven dried mass (105°C)
M_s = mass of soil solids (excluding salt)
M_{sa} = mass of salt
M_{sw} = mass of sea water
r = salinity = M_{sa}/M_{sw} = mass of salt/mass of sea water
M_w = mass of distilled water = $M - M_d$
V = total volume
V_s = volume of soil solids (excluding salt)
V_{sw} = volume of sea salt
V_w = volume of distilled water
V_g = volume of gas
V_v = volume of voids = $V_{sw} + V_g$
ρ_s = density of solids excluding salt
ρ_o = density of distilled water at 4°C = 1 g/cm^3
ρ_{sw} = density of salt water at test temperature = 1.029 g/cm^3
G_s = specific gravity of solids excluding salts = ρ_s/ρ_o.

X1.2 Definitions and Phase Relations (7)

X1.2.1 Using the terms defined in X1.1.1 and referring to Fig. X1.1, $M_w = M - M_d = M_{sw} - M_{sa} = M_{sw}\ (1 - r)$ which yields the following:

$$M_{sw} = \frac{M - M_d}{1 - r} \quad \text{(X1.1)}$$

$$M_s = M - M_{sw} = \frac{M_d - rM}{1 - r} \quad \text{(X1.2)}$$

$$V_{sw} = \frac{M_{sw}}{\rho_{sw}} = \frac{M - M_d}{(1 - r)\rho_{sw}} \quad \text{(X1.3)}$$

$$V_s = \frac{M_s}{G_s\rho_o} = \frac{M_s}{G_s\rho_o} = \frac{M_d - rM}{(1 - r)G_s\,\rho_o} \quad \text{(X1.4)}$$

X1.2.2 The following terms are defined as follows:

$$\text{Density } \rho = \frac{M}{V} \quad \text{(X1.5)}$$

$$\text{Dry density } \rho_d = \frac{M_s}{V} = \frac{M_d - rM}{V(1 - r)} - \quad \text{(X1.6)}$$

$$\text{Water content}\,(\%)\ w = \frac{M_w}{M_d} \times 100 = \frac{M - M_d}{M_d} \times 100 \quad \text{(X1.7)}$$

$$\text{Fluid content }(\%)\ \bar{w} = \frac{M_{sw}}{M_s} \times 100 = \frac{M - M_d}{M_d - rM} \times 100 \quad \text{(X1.8)}$$

$$\text{Void ratio } \bar{e} = \frac{V_v}{V_s} = \frac{V(1 - r)G_s\rho_o}{M_d - rM} - 1 \quad \text{(X1.9)}$$

$$\text{Porosity } \bar{n} = \frac{V_v}{V} \times 100\,\% \quad \text{(X1.10)}$$

$$\bar{n} = \left[1 - \frac{(M_d - rM)}{v(1 - r)G_s\rho_o}\right] \times 100 \quad \text{(X1.11)}$$

Degree of saturation

$$\bar{s} = \frac{V_{sw}}{V_v} \times 100 \quad \text{(X1.12)}$$

$$\bar{s} = \frac{G_s(M - M_d)}{v(1 - r)G_s\rho_o - M_d + rM}\,\frac{\rho_o}{\rho_{sw}} \times 100 \quad \text{(X1.13)}$$

X1.3 Other Useful Relationships

$$M_d = \frac{M}{1 + w} \quad \text{(X1.14)}$$

$$M_s = \frac{M}{1 + \bar{w}} \quad \text{(X1.15)}$$

$$\rho_d = \frac{\rho}{1 + \bar{w}} \quad \text{(X1.16)}$$

$$\bar{n} = \frac{\bar{e}}{1 + e} \quad \text{(X1.17)}$$

$$\bar{w} = \frac{w}{1 - r - rw} \quad \text{(X1.18)}$$

$$\bar{e} = \frac{VG_s\rho_o\,(1 + \bar{w})}{M} - 1 \quad \text{(X1.19)}$$

or

$$\bar{e} = \frac{\bar{w}G_s}{S} \times \frac{\rho_o}{\rho_{sw}} \quad \text{(X1.20)}$$

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SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to this standard since the last edition (2007) that may impact the use of this standard. (November 1, 2015)

(1) Section 1.4 Spelled out "parts per thousand."
(2) Section 1.7 Relocated safety section from 1.6 to 1.7.
(3) Eliminated jargon and superlatives throughout. Improved syntax throughout.
(4) Section 5.2 – Clarified the construction of the soil press.
(5) Clarified Figure 2 – an example of a soil press.
(6) Added Section 6 – Reagents. Added a footnote on Reagent Chemicals from the American Chemical Society. Section 6.7.1 Clarified handling of the soil press.
(7) Section 8 – Procedure – clarified the operation of the two types of refractometers.
(8) Section 10 – Report: Test Data Sheet(s)/Form(s) – Added requirement to list date and testing personnel.Added requirement to use three test locations.
(9) Note 14 Added guidance allowing the use of non-standard equipment in place of a soil press depending on the material being tested.
(10) Renumbered sections as needed.
(11) Updated the Summary of Changes

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