



Designation: D4542 – 07

Standard Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer¹

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 This test method covers a rapid procedure for squeezing pore water from fine-grained soils for the purpose of determining the amount of soluble salts present in the extracted pore water.

1.2 This test method was 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 This test method is 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 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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.*

1.6 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

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.

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 Injection⁴

3. Terminology

3.1 Definitions:

3.1.1 For definitions of 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 (water content, void ratio, specific gravity, degree of saturation, and dry density).

4.2 It is necessary to minimize the time period between sampling and testing due to chemical changes which may occur within the soil sample.

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 assure 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 nonreproducible 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.

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



FIG. 1 Typical Hand-Held Refractometer

5.2 *Soil Press*—The apparatus shall conform to the requirements shown in Fig. 2.

5.3 *Syringe*—A 25-cm³ syringe without needle, in accordance with Fed. Std. GG-S-945a.

5.4 *Balance*—A balance capable of weighing with a sensitivity 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 (2.25 in.).

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 (0.98 in.).

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

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 7.6).

6. Preparation of Apparatus

6.1 Wash all parts of the press thoroughly. Rinse twice with distilled water and dry. Normally, rust should not be present, but if it is to be removed, especially inside or around the top of the cylinder, scrub gently with steel wool and soap or chromic acid. Rinse well with tap water and then twice with distilled water and dry.

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

6.3 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.

6.4 Assemble the press.

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

7. Sampling and Test Specimen Squeezing

7.1 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.

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

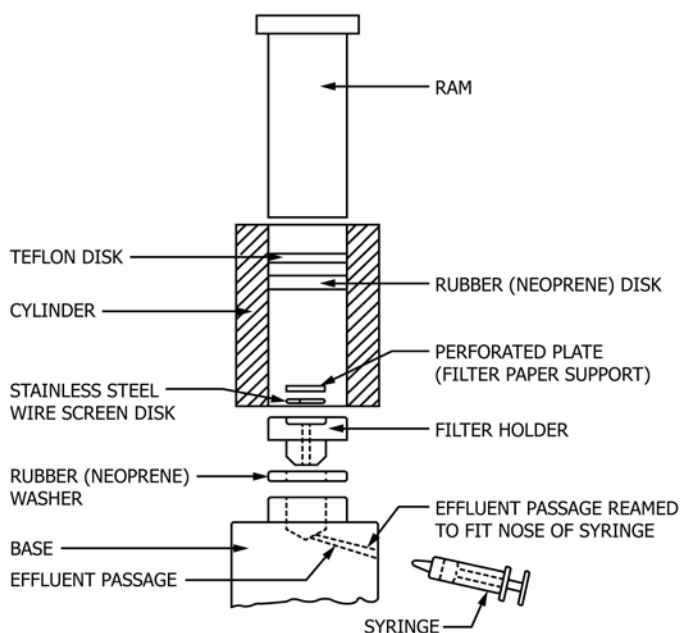


FIG. 2 Soil Press

7.2 Apply pressure slowly until the first drops of water are expelled, then insert a clean, disposable, plastic syringe (25 mL) in the effluent passage shown in Fig. 2. This is done to minimize the amount of air in the syringe and therefore, the amount of evaporation.

7.3 Apply pressure gradually to a maximum of 80 MPa (11 520 psi), and hold until no more water is expelled or until the syringe is full (see Note 4 and Note 5).

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

7.5 Repeat 7.1 - 7.4, 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 sediment (see Note 7 and Note 8). Store the water at a temperature between 1 and 5°C (see Note 9 and Sections 8 and 9).

7.6 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 (8500 psi) the chloride content drops in homogeneous soils. Manheim (5) reports using 101 MPa (14 700 psi) routinely. An average of these two recommendations is 80 MPa (11 520 psi).

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 cm³ 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.

8. Procedure

8.1 *Method A—Salinity Determination Using a Refractometer With a Refraction Index Scale:*

8.1.1 Filter specimen through a 0.45-µm filter.

8.1.2 Thoroughly wash with distilled water and dry the refractometer, shown in Fig. 1.

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

8.1.4 Hold the refractometer at right angles to a light source and read the refractive index.

8.1.5 Obtain salinity from the accompanying graph (Fig. 3), where n is the refractive index of the liquid and n_0 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.

8.2 *Method B—Salinity Determination Using a Refractometer With a ppt Scale:*

8.2.1 Thoroughly wash with distilled water and dry the refractometer, shown in Fig. 1.

8.2.2 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.

8.2.3 Hold the refractometer at right angles to a light source and read the salinity. If the liquid is properly introduced, there should be a distinct black/white boundary. Read 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.

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

9.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s), as given below, is covered in 1.6.

9.2 For each test, a record of the following observations is required:

- 9.2.1 Location,
- 9.2.2 Depth,
- 9.2.3 Soil type,

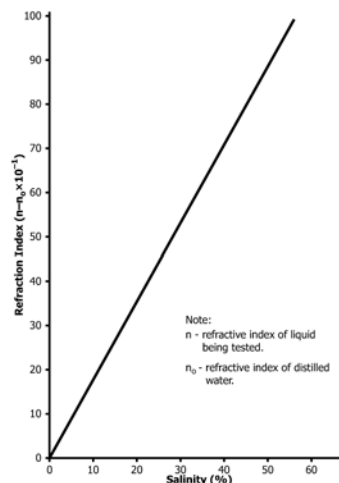


FIG. 3 Graph of Salinity versus Refraction Index