



## Standard Test Method for Pore Water Extraction and Determination of the Soluble Salt Content of Soils by Refractometer<sup>1</sup>

This standard is issued under the fixed designation D 4542; 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 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).<sup>2</sup>

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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

E 832 Specification for Laboratory Filter Papers<sup>3</sup>

#### 2.2 Federal Document:

GG-S-945a Specification for Syringe and Needle, Disposable, Hypodermic, Sterile, Single Injection<sup>4</sup>

### 3. Significance and Use

3.1 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).

3.2 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—Hulbert and Brindle (2) and Torrance (3) have shown that prolonged storage should be avoided as unpredictable and nonreproducible chemical changes may occur.

### 4. Apparatus

4.1 *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.



FIG. 1 Typical Hand-Held Refractometer

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

4.3 *Syringe*—A 25-cm<sup>3</sup> syringe without needle, in accordance with Fed. Std. GG-S-945a.

4.4 *Balance*—A balance capable of weighing with a sensitivity of  $\pm 0.01$  g.

#### 4.5 Filter Paper:

4.5.1 A general purpose quantitative filter paper in accordance with Specification E 832, Type II, Class F, for medium crystalline precipitates in the size range from 5 to 10  $\mu\text{m}$ , with an ash content of 0.13 mg/12.5-cm circle. Cut filter paper to a diameter of 55 mm (2.25 in.).

4.5.2 A general purpose quantitative filter paper in accordance with Specification E 832, Type II, Class G, for fine crystalline precipitates in the size range from 0.45  $\mu\text{m}$ , with an ash content of 0.13 mg/12.5-cm circle. Cut filter paper to a

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<sup>2</sup> The boldface numbers in parentheses refer to the list of references appended to this standard.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>4</sup> Available from Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, PA, 19120.

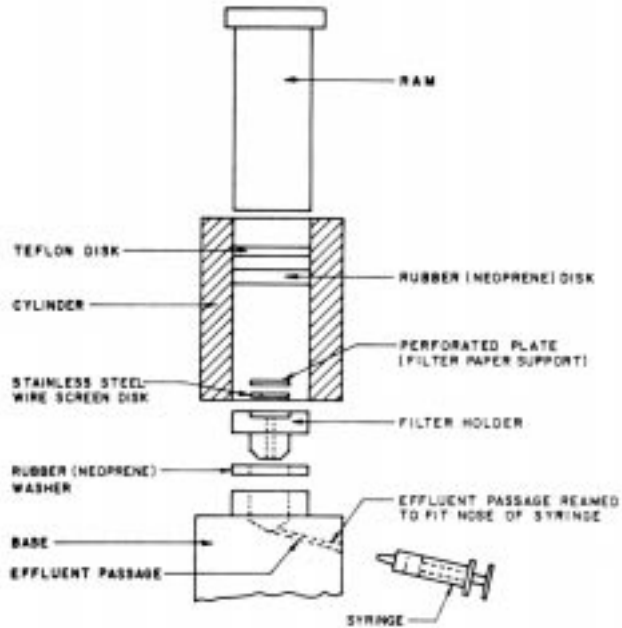


FIG. 2 Soil Press

diameter of 25 mm (0.98 in.).

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

4.7 *Micro-Syringe Filter Holder*—A device to filter a liquid directly from a syringe.<sup>5</sup>

4.8 *100-mL Polyethylene or Glass Bottle and Cap*.

4.9 *Miscellaneous Supplies*—Distilled water, alcohol, diluted HCl (1:10), detergent, and optional sterile bags for sample storage (see 6.6).

## 5. Preparation of Apparatus

5.1 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.

5.2 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.

5.3 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.

5.4 Assemble the press.

NOTE 2—To prevent mud from circumventing the stainless steel wire screen use flexible TFE-fluorocarbon gaskets on each side.

## 6. Sampling and Test Specimen Squeezing

6.1 Select a representative soil sample of approximately 50 g and place into the cylinder on top of a single sheet of 5 to

10- $\mu$ m (55-mm) filter paper.

6.2 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.

6.3 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 3 and Note 4).

6.4 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- $\mu$ m (25-mm) filter paper, into a clean 100-mL bottle (see Note 5). Cap the bottle. Expose the collected water to the atmosphere as little as possible.

6.5 Repeat 6.1-6.4, 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 6 and Note 7). Store the water at a temperature between 1 and 5°C (see Note 8 and Sections 7 and 8).

6.6 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 8).

NOTE 3—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 4—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 5—Polyethylene or glass bottles should be washed with detergent and rinsed with tap water. They should then be rinsed once with diluted

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

HCl (1:10) and twice with distilled water and then drained thoroughly.

NOTE 6—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<sup>3</sup> 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 7—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 8—A storage temperature of about 5°C is recommended to reduce the growth of sulfate-reducing bacterium called Desulfovibrio.

## 7. Procedure

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

7.1.1 Filter specimen through a 0.45-µm filter.

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

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

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

7.1.5 Obtain 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).

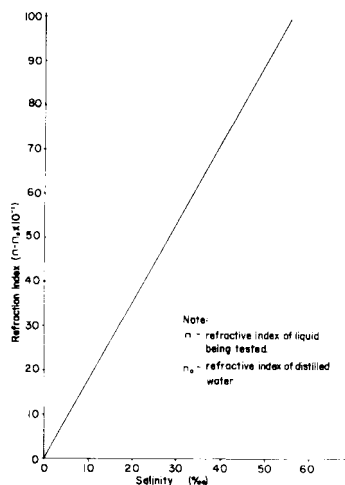


FIG. 3 Graph of Salinity versus Refraction Index

NOTE 9—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.

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

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

7.2.2 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.

7.2.3 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 10—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 11—Error in reading directly from the salinity scale is ±0.3 ppt.

NOTE 12—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.

## 8. Report

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

- 8.1.1 Location,
- 8.1.2 Depth,
- 8.1.3 Soil type,
- 8.1.4 ppt or percent soluble solids,
- 8.1.5 Water content, and
- 8.1.6 Total density of undisturbed sample.

## 9. Precision and Bias

9.1 *Precision*—Due to the nature of soil tested by this test method, it is too costly at this time to produce multiple specimens which 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.

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

## 10. Keywords

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

APPENDIX

(Nonmandatory Information)

X1. MASS COMPOSITION

**X1.1 Mass Composition Terms**

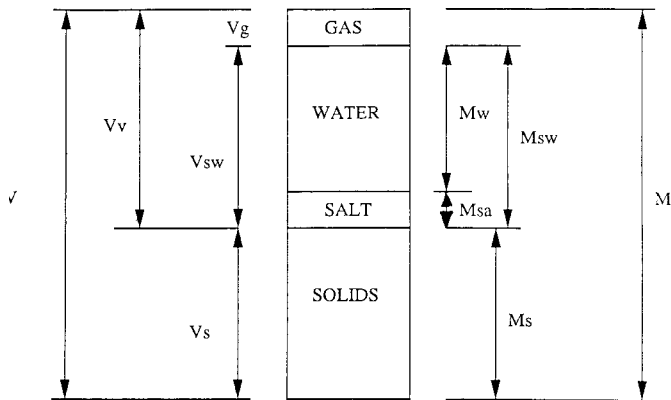
X1.1.1 A mass composition diagram for partially saturated soils with soluble salts is shown in Fig. X1.1. Based on this figure the following terms and definitions are defined for describing various relationships among soil phases.

- $M$  = 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 =  $V_{sw} + V_g$
- $\rho_s$  = density of solids excluding salt
- $\rho_o$  = density of distilled water at 4°C = 1 g/cm<sup>3</sup>
- $\rho_{sw}$  = density of salt water at test temperature = 1.029 g/cm<sup>3</sup>
- $G_s$  = specific gravity of solids excluding salts =  $\rho_s/\rho_o$ .

**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:

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



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

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

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

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

X1.2.2 The following terms are defined as follows:

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

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

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

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

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

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

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

Degree of saturation

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

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

**X1.3 Other Useful Relationships**

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

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

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

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

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

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

or

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

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