

# Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry<sup>1</sup>

This standard is issued under the fixed designation D4404; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

- 1.1 This test method covers the determination of the pore volume and the pore volume distributions of soil and rock by the mercury intrusion porosimetry method. The range of apparent diameters of pores for which this test method is applicable is fixed by the operating pressure range of the testing instrument. This range is typically between apparent pore entrance diameters of about  $100~\mu m$  and 2.5~nm ( $0.0025~\mu m$ ). Larger pores must be measured by another method.
- 1.2 Mercury intrusion porosimetry is useful only for measuring pores open to the outside of a soil or rock fragment; mercury intrusion porosimetry will not give the volume of any pores completely enclosed by surrounding solids. This test method will give only the volume of intrudable pores that have an apparent diameter corresponding to a pressure within the pressurizing range of the testing instrument.
- 1.3 Mercury intrusion may involve the application of high pressures to the specimen. This may result in a temporary or permanent alteration or both in the pore geometry. Generally, soils and rocks are composed of comparatively strong solids and are less subject to these alterations than certain other materials. However, the possibility remains that the use of this test method may alter the natural pore volume distribution that is being measured.
- 1.4 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.
- 1.5 **Warning**—Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website

(http://www.epa.gov/mercury/faq.htm) for additional information. Users should be aware that selling mercury or mercury-containing products or both into your state may be prohibited by state law.

- 1.6 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. For specific precaution statements, see Section 8.

#### 2. Referenced Documents

- 2.1 ASTM Standards:<sup>2</sup>
- D653 Terminology Relating to Soil, Rock, and Contained
- 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 For definitions of terms used in the test method, refer to Terminology D653.
  - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 apparent pore diameter—the diameter of a pore that is assumed to be cylindrical and that is intruded at a pressure, *P*, given by the equation in 13.1.
- 3.2.2 *inter-specimen pores*—those pores between particles when they are packed together and that are intruded during the test.
- 3.2.3 *intra-specimen pores*—those pores lying within the exterior outlines of the individual soil and rock fragments.

<sup>&</sup>lt;sup>1</sup> 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.

Current edition approved May 1, 2010. Published June 2010. Originally approved in 1984. Last previous edition approved in 1998 as D4404–84(2004). DOI: 10.1520/D4404-10.

<sup>&</sup>lt;sup>2</sup> 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.

3.2.4 *intruded pore volume*—the corrected volume of mercury intruded during the test.

## 4. Summary of Test Method

4.1 When a liquid does not wet a porous solid, it will not enter the pores in the solid by capillary action. The non-wetting liquid (mercury in this test method) can be forced into the pores by the application of external pressure. The size of the pores that are intruded is inversely proportional to the applied pressure. When a cylindrical pore model is assumed, the relationship between pressure and size is given as follows:

$$d = -4\gamma(\cos\theta)/P \tag{1}$$

where:

d = apparent pore diameter being intruded,

 $\gamma$  = surface tension of the mercury,

 $\theta$  = contact angle between the mercury and the pore wall,

P = absolute pressure causing the intrusion.

Any set of convenient and compatible units may be used.

4.2 The volume of the intruded pores is determined by measuring the volume of mercury forced into them at various pressures. A single determination involves increasing the pressure, either continuously or step-wise, and recording the measured intruded volume at various pressures.

#### 5. Significance and Use

5.1 This test method is intended for use in determining the volume and the volume distribution of pores in soil and rock with respect to the apparent diameter of the entrances of the pores. In general, both the size and volume of the pores affects the performance of soil and rock. Thus, the pore volume distribution is useful in understanding soil and rock perfor-

mance and in identifying a material that can be expected to perform in a particular manner (1, 2).<sup>3</sup>

5.2 The intrusion process to determine the volume of a pore proceeds from the outside of a specimen toward its center. Comparatively large interior pores can exist that have smaller outside openings as the only means of access. Mercury intrusion porosimetry will incorrectly register the entire volume of these "ink-bottle" pores as having the apparent diameter of the smaller access pores. In a test specimen, exterior specimen pores can exist in addition to intra-specimen pores (see Section 3 for definitions). The inter-fragment pores will vary in size and volume depending on the size and shape of the soil or rock fragments and on the manner in which the fragments are packed together. It is possible that some exterior specimen pores can have the same apparent diameter as some intra-specimen pores. When this occurs, this test method cannot distinguish between them. Thus, the test method yields an intruded pore volume distribution that is in part dependent upon the packing of multifragment specimens. However, most soils and rocks have intra-fragment pores much smaller than the inter-fragment pores. This situation leads to a bi-modal pore size distribution and the distinction between the two classes of pores can then be made (see Figs. 1 and 2).

Note 1—Notwithstanding the statement on precision and bias contained in this test method: The precision of this test method is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies which meet the criteria of Practice D3740 are generally considered capable of competent and objective testing. Users of this test method are cautioned that compliance with Practice D3740 does not in itself assure reliable testing. Reliable testing depends on several factors; Practice D3740 provides a means of evaluating some of those factors.

<sup>&</sup>lt;sup>3</sup> The boldface numbers in parentheses refer to the list of references appended to this standard

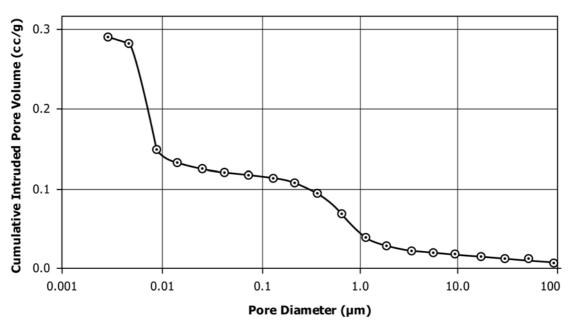


FIG. 1 Example of Cumulative Pore Volume Distribution Plot

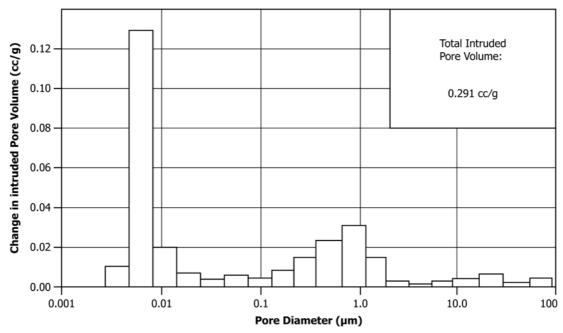


FIG. 2 Example of Differential Pore Volume Distribution Plot

#### 6. Apparatus

- 6.1 Mercury Intrusion Porosimeter—This shall be equipped with a specimen holder capable of containing one or several soil or rock fragments. This specimen holder is frequently called a penetrometer. The porosimeter shall have a means of surrounding the test specimen with mercury at a low pressure, a pressure generator to cause intrusion, pressure transducers, capable of measuring the intruding pressure with an accuracy of at least  $\pm 1$  % throughout the range of pressures over which the pores of interest are being intruded, and a means of measuring intruded mercury volumes to an accuracy of at least  $\pm 1$  mm<sup>3</sup> ( $\pm 10^{-3}$  cm<sup>3</sup>).
- 6.2 *Vacuum Pump*, if not part of the porosimeter, to evacuate the specimen holder.
- 6.3 Analytical Balance, with an accuracy of at least  $\pm 10^{-7}$  kg ( $\pm 0.1$  mg).
  - 6.4 Flume Hood

# 7. Reagent

- 7.1 Triple-Distilled Mercury.
- 7.2 Other grades or amalgams 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.
- 7.3 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.

#### 8. Safety Precautions

8.1 Mercury is a hazardous substance that can cause illness and death.

- Note 2—Mercury is found in several chemical forms, such as: elemental, inorganic, and organic:
- A) Elemental Mercury: Elemental mercury, also referred to as metallic mercury, is a shiny, silver-white, odorless liquid. Elemental mercury readily evaporates at room temperature to form a colorless, odorless gas. In an enclosed space, even a relatively small amount of mercury can result in the accumulation of a very high level of mercury vapor in indoor air. Therefore, exposure via inhalation of elemental mercury is a particular concern when mercury is spilled in laboratories or in other enclosed areas.
- B) Inorganic Mercury: Mercury combines with other elements, such as chlorine, sulfur, or oxygen, to form inorganic mercury compounds or "salts," which are usually in the form of white powders or crystals. Inorganic mercury does not readily evaporate and is not easily inhaled; however inorganic mercury can be absorbed across the gastrointestinal tract and the surface of the skin. Therefore, ingestion and skin contact can result in exposure.
- C) Organic Mercury: Mercury also combines with carbon to make organic mercury compounds. The most common form is methyl mercury, which is produced mainly by small organisms in the water, soil, and sediment. Increasing emissions of mercury into the environment can increase the levels of methyl mercury that these small organisms make. The most significant source of human exposure to organic mercury is through diet, particularly from fish products. Since organic mercury is easily absorbed through the gastrointestinal tract and through the skin, ingestion and skin contact can result in exposure.
- 8.2 Store mercury in closed containers to control its evaporation and use only in conjunction with a flume hood in a well-ventilated room.
- 8.3 Mercury can also be absorbed through the skin, so avoid direct contact. Wash hands immediately after any operation involving mercury; the use of mercury approved gloves is advocated.
- 8.4 Exercise extreme care to avoid spilling mercury. Clean up any spills immediately using procedures recommended explicitly for mercury.
- 8.5 Handle intruded specimens with great care and dispose of in a safe and environmentally acceptable manner immediately after completion of the test (See Note 3).

Note 3—USEPA conditionally exempts small quantity generators (CESQC). Small quantity generators (CESQC) generate 100 kg or less per month of hazardous waste, or 1 kg or less per month of acutely hazardous waste. Requirements for CESQGs include (see also 40 CFR 261.5<sup>4</sup>):

CESQGs must identify all the hazardous waste.

CESQGs must identify all the hazardous waste generated.

CESQGs may not accumulate more than  $1\,\,000$  kg of hazardous waste at any time.

CESQGs must ensure that hazardous waste is delivered to a person or facility that is authorized to manage it.

#### 9. Sampling, Test Specimens, and Test Units

9.1 The sample from which the test specimen is drawn shall be representative of the soil or rock. The test specimen shall be as large as practicable considering the test apparatus.

Note 4—A minimum of 3 specimens should be run on samples from similar material. If a statistical analysis of the results is desired, a minimum of 10 specimens is required.

Note 5—Specimen size is limited by the pore-measuring capacity of the penetrometer, which is currently (1984) slightly more than 16 387 mm. The small specimen size may prevent the measurement of porosity represented by relatively large cracks and fissures in the material. Judgement is required in the application of these measurements to the characterization of the soil or rock masses.

#### 10. Conditioning

10.1 The ideal preconditioning for the test specimen is an outgassing or drying procedure such as freeze drying and critical region drying that removes all foreign fluids from the pores and pore walls of the soil or rock and does not alter the soil or rock in any way. If possible, the appropriate combination of temperature and pressure and the required time of conditioning shall be experimentally determined for the specific soil or rock under test. This outgassing or drying technique shall then be the one specified and used.

10.2 Rock or coarse-grained soil without fines shall be outgassed in a vacuum at least 1.3 Pa (10 µmHg) and at a temperature of 150 °C for at least 24 h. Soil containing any plastic fines requires special drying procedures to avoid alteration of pore structure. Freeze drying has been successfully employed (3, 4) and is a simple procedure. Critical region drying may also be used (5), but is more complex and expensive than freeze drying.

### 11. Procedure

- 11.1 Outgas or dry the test specimen in accordance with 10.1 or 10.2.
- 11.2 Weigh the outgassed or dried specimen and record this weight.
- 11.3 Place the outgassed or dried material in the penetrometer.

Note 6—When performing the operation described in 11.2 and 11.3, the outgassed or dried material is exposed to the laboratory atmosphere and can readsorb vapors. Thus, this operation should be carried out as rapidly as possible.

- 11.4 Place the penetrometer containing the specimen in the pressure vessel of the porosimeter and evacuate to a pressure of at least 1.3 Pa (10  $\mu$ mHg).
- 11.5 Fill the penetrometer with mercury by pressurizing to the minimum pressure greater than 1.3 Pa (10  $\mu$ mHg) that will promote filling.

Note 7—The pressure required to fill the penetrometer with mercury is also capable of intruding sufficiently large pores of both the inter- and intra-fragment classes. Thus, the process can intrude into some pores and the volume distribution of these pores cannot subsequently be determined. This fact should be recognized, and where possible, a filling pressure should be selected that will not intrude pores in the diameter range of interest.

- 11.6 Place the filled penetrometer in the pressure vessel of the porosimeter, close and prepare the instrument for pressurization and intrusion readings. Information should be recorded in accordance with Practice D6026.
- 11.7 Raise the pressure, either continuously or incrementally, and record both the absolute pressure and the volume of intruded mercury until the maximum pressure of interest is reached. When raising the pressure incrementally, the pressure shall be maintained during the pause and not allowed to decrease.

Note 8—When raising the pressure incrementally, the pressure shall be maintained during the pause and not allowed to decrease.

Note 9—When testing some materials, the time required to achieve intrusion equilibrium will not be the same at all pressures. Often the equilibrium time is appreciably longer at pressures that cause an abrupt and large increase in intruded volume. Failure to record the equilibrium intrusion may result in some of the pore volume being incorrectly assigned to smaller pore diameters. The extent to which this may be a problem can be assessed by conducting two tests, each at a different pressure increase rate, and comparing the results.

Note 10—Use of Eq 2 requires the absolute pressure  $(P_{ABS} = P_G - P_L)$ . With some instruments it may not be possible to read the absolute pressure directly. In this case, the gauge pressure shall be recorded at each step, and the absolute pressures subsequently calculated.

11.8 The choice of pressure intervals at which data are to be recorded is left to the judgment of the operator. Normally, at least 10 to 15 intervals will be required to adequately define the pore volume distribution. In selecting these pressure intervals, a rough idea of the expected distribution is helpful, since the pressure interval can be larger in regions where little or no intrusion occurs and should be smaller in regions where a large volume of intrusion is expected. It is not necessary to continue the process up to the maximum pressurizing capability of the instrument if all of the pores of interest in a particular test specimen have been intruded at a lesser pressure.

11.9 Upon completion of the pressuring cycle, reduce the pressure and disassemble and clean the instrument in accordance with the manufacturer's instructions.

Note 11—User should refer to manufacturer's instruction for requirements for specific instruments.

# 12. Blank Test for Corrections

12.1 An intrusion test on a nonporous specimen is required to obtain values to use in correcting intrusion data for apparatus compressibilities and volume changes due to expansion/contraction because of temperature changes.

<sup>&</sup>lt;sup>4</sup> 40 CFR 261.5 "Special Requirements for Hazardous Waste Generated by Conditionally Exempt Small Quantity Generators." Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

- 12.2 Select a nonporous material that has approximately the same compressibility and bulk volume as the soil or rock specimen that is to be tested.
- 12.3 Test the nonporous specimen in exactly the same manner as outlined in Section 11. Raise the pressure in the same steps used for the soil or rock tests to ensure that temperature changes due to pressuring are the same.
- 12.4 Results of the blank run are a series of measured volume changes that can also be expected to occur along with actual intrusion in a test on a material. Such blank run results are used to correct the intruded volumes as discussed in 13.3.2.
- 12.5 Compressibilities of the various components in the system augment the measured intrusion values while the pressure-induced heating and consequent expansion of the system reduces the measured volumes. In a particular instrument, one of these effects will be dominant. Therefore, results of the blank test will be either an apparent intrusion (compressibility dominant) or an expulsion of mercury (heating dominant).
- 12.5.1 If results show apparent intrusion, they are to be subtracted from the values measured in the test on the soil or rock.
- 12.5.2 If the blank results show a mercury expulsion, they are to be added to the volumes in a test on the soil or rock.

#### 13. Calculation

- 13.1 Intruding pressures must be expressed as absolute pressures before they can be used to compute the corresponding pore diameters. If the recorded values are gauge pressures, they should be converted to absolute pressures. If the instrument reads directly in absolute pressure, omit this step.
- 13.2 Absolute pressures ( $P_{ABS}$ ) are converted to apparent intruded pore diameters using the Eq 2. This step requires that surface tension and contact angle be known. When a cylindrical pore model is assumed, the relationship between pressure and size is given as follows:

$$d = 4T(\cos\theta)/(P_{ABS}) \tag{2}$$

where:

d = apparent pore diameter being intruded,

T = surface tension of the mercury,

 $\theta$  = contact angle between the mercury and the pore wall,

 $P_L$  = absolute pressure causing the intrusion,

 $P_G$  = pressure of gas, and

 $P_{ABS} = P_{G} - P_{L}.$ 

Note 12—Since the technique is usually done under vacuum, the gas pressure begins at zero. The contact angle of mercury with most solids is between 135 and 142, so an average of 140 can be taken without much error. The surface tension of mercury at 20 °C under vacuum is 480 mN/m. With the various substitutions, the equation becomes: d = 213/ $P_{\rm L}$ .

- 13.2.1 Where triple-distilled mercury is used, the surface tension can generally be relied upon to be that reported in handbooks, that is, 0.484 N/m (19.055 d/mm) at 25  $^{\circ}$ C. Small deviations from this value are not significant as the surface tension enters the equation only as a linear term.
- 13.2.2 The contact angle enters the equation as a cosine function, and it is important to know the value of the angle accurately for the material under test. The contact angle of

mercury has been measured on a variety of solids by several different techniques; references to some of these measurements are given in Appendix X1. This appendix also lists references for several methods of contact-angle measurement that have been found useful. The ideal value for reducing the data is the one that has been determined for the material under test. If direct measurement is impractical, the use of an assumed value is customary. If mercury intrusion is being used for the comparison of similar materials for quality control purposes, then an assumed value is satisfactory; however, when different materials are being compared, the assumption of a single value for the contact angle may lead to errors.

- 13.3 The next step in the calculations is the correction of the intruded volume readings. Corrections fall into two categories: low-pressure corrections and high-pressure corrections.
- 13.3.1 Low-pressure corrections account for the fact that part of the apparent intrusion recorded at the lowest pressures is actually the compression of the air trapped in the penetrometer when it was filled with mercury. This correction is important only when the distribution of large pores must be measured accurately. When large pores are not of interest (pores larger than about 50- $\mu$ m diameter), the low pressure correction may be safely ignored.
- 13.3.2 High-pressure corrections to the intruded volume readings represent the compressibility of the specimen, penetrometer, and mercury, as well as temperature changes that occur as a result of pressurization. The value of this correction is experimentally determined with the blank intrusion test performed in Section 12. Values determined in the blank test are either added to or subtracted from the intrusion volumes discussed in 12.5.
- 13.4 The final calculation is the conversion of the corrected intruded volumes to a unit mass basis. Divide each corrected intrusion value by the specimen mass.

## 14. Report

- 14.1 The report shall include the following specific information:
  - 14.1.1 Specimen description,
  - 14.1.2 Specimen mass,
  - 14.1.3 Specimen outgassing or drying procedure,
  - 14.1.4 Contact angle used in calculations,
- 14.1.5 A table showing the corrected cumulative intrusion volumes on a per gram basis and the corresponding absolute intruding pressure (Note 13), and
- 14.1.6 A graphical, cumulative pore volume distribution having the intruded volumes per gram on the ordinate with an arithmetic scale, and the apparent pore diameters on the abscissa with a logarithmic scale.

Note 13—The pore size distribution may also be expressed in terms of intruded volume per cubic centimetre of total specimen volume, if this is a more convenient form for the results.

14.2 The report may also include a differential plot of the distribution. This plot may have either the slope of the cumulative plot taken at various points or the incremental increase in intrusion between various points plotted against the apparent pore diameters. The slope or change shall be on the

ordinate, with an arithmetic or logarithmic scale as appropriate, and the apparent pore diameters shall be on the abscissa with a logarithmic scale.

- 14.2.1 A differential plot can lead to a distorted image of the pore size distribution unless care is exercised in the selection of the diameter points at which the slope or change in intrusion is calculated. It is recommended that pore diameters be selected that are approximately equally spaced on the logarithmic diameter axis, and that these diameters be used consistently to avoid distortions in the appearance of the differential plot.
- 14.2.2 When a differential plot is presented, there shall be on the plot itself a statement of the cumulative total intruded pore volume. Typical examples of both cumulative and differential pore volume distribution plots are shown in Fig. 1 and Fig. 2. The intra-fragment pore concentration is shown to the left of the plot; the inter-fragment concentration is to the right.

#### 15. Precision and Bias

15.1 *Precision*—Test data on precision is 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. Also, 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.

- 15.1.1 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.
- 15.2 *Bias*—There is no accepted reference value for this test method, therefore, bias cannot be determined.
- 15.3 Appendix A of Ref (3) shows examples of single-laboratory variations for several compacted soils. No multi-laboratory comparisons are available, and statistical evaluations of variability are totally lacking.

## 16. Keywords

16.1 frequency distribution; hydraulic conductivity; mercury; permeability; pore distribution; pore volume; poresize; porosimetry; porosity; soil; soil physics; voids

#### **APPENDIX**

(Nonmandatory Information)

#### X1. DETERMINING CONTACT ANGLES

X1.1 The contact angle between mercury and the wall of a pore in a solid depends upon many factors. Among these are the nature of the solid, the cleanliness of the pore wall, the roughness of the pore wall, whether the mercury is advancing or retreating along the solid surface, and the purity of the mercury. For these reasons, the operant contact angle for a particular material will generally be different from that for another material. Thus, the best contact angle to use in reducing the porosimetry data is one that has been measured on the material in question with the mercury used for the

porosimetry experiment. Ref (6) gives a summary of a wide variety of experimental techniques for measuring contact angles.

X1.2 Some contact angles have been reported in the references. Some examples of contact angles are given in Table X1.1.

X1.3 If a published contact angle value is to be adopted, it is recommended that the reference be carefully studied to assess the validity of its use for reducing porosimetry data.

# REFERENCES

**TABLE X1.1 Examples of Contact Angles** 

Material	Contact Angle,°	Reference
Alkali borosilicate glass	153	(7)
Quartz	132-147	(8)
Clay minerals	139–147	( <del>9</del> )
Aluminum oxide	127	(10)

- (1) Garcia-Bengochea, I., and Lovell, C. W., "Correlative Measurements of Pore Size Distribution and Permeability in Soils," *Permeability and Groundwater Contaminant Transport, ASTM STP 746*, ASTM International, Oct. 1981, pp. 137–150.
- (2) Kaneuji, M., Winslow, D. N., and Dolch, W. L., "The Relationship Between an Aggregate's Pore Size Distribution and its Freeze/Thaw Durability in Concrete," *Cement and Concrete Research*, Vol 10, No. 3, 1980, pp 433.
- (3) Garcia-Bengochea, I., "Relation Between Permeability and Pore Size Distribution of Compacted Clayey Silts," *Joint High Research Project*, No. 78/4, School of Civil Engineering, Purdue University, W. Lafayette, IN, April 1978, 178 pp.
- (4) Zimmie, T. F., and Almaleh, L. J., "Shrinkage of Soil Specimens During Preparation for Porosimetry Tests," Soil Specimen Preparation for Laboratory Testing, ASTM STP 599, ASTM International, pp. 202–215.



- (5) Bhasin, R. N., "Pore Size Distribution of Compacted Soils After Critical Region Drying," Ph.D. Thesis, School of Civil Engineering, Purdue University, W. Lafayette, IN, May 1975, 222 pp.
- (6) Neumann, A. W., and Good, R. J., *Surface and Colloid Science*, Vol 11, Plenum Press, New York, 1979, Chapter 2.
- (7) Rootare, H. M., and Nyce, A. C., *International Journal of Powder Metallurgy*, Vol 7, No. 1, 1971, pp. 3–11.
- (8) Ellison, A. H., Klemm, R. B., Schwartz, A. M., Grubb, L. S., and Petrash, D. A., *Journal of Chemical and Engineering Data*, Vol 12,
- No. 4, 1967, pp. 607-609.
- (9) Diamond, S., "Pore Size Distributions in Clays," *Clays and Clay Minerals*, Vol 18, 1970, pp. 7–23.
- (10) Winslow, D. N., Journal of Colloid and Interface Science, Vol 67, No. 1, 1978, pp. 42–47.
- (11) Winslow, D. N., and Lowell, C. W., "Measurements of Pore Size Distributions in Cements, Aggregates, and Soils," *Powder Technology*, Vol 29, No. 1, May/June 1981, pp 151–165.

#### SUMMARY OF CHANGES

Committee D18 has identified the location of selected changes to this test method since the last issue, D4404–84(2004), that may impact the use of this test method. (Approved May 1, 2010)

- (1) Added new 1.4 requiring significant digits to be in accordance with Practice D6026.
- (2) Added mercury warning as new 1.5.
- (3) Moved old paragraph 1.3 to 5.2.
- (4) Added new Note 1 and renumbered subsequent notes.
- (5) Added new 6.4, requirement for flume hood.
- (6) Added new 7.2 and 7.3 on using other grades of mercury or amalgams and their purity.
- (7) Revised 8.1 and added Note 2 on forms of mercury and safety requirements.
- (8) Added Note 3 on disposal.

- (9) Added Note 4 on number of samples to be run.
- (10) Revised 11.5 on filling penetrometer.
- (11) Added requirement to use Practice D6026 in 11.6.
- (12) Deleted old Note 4 and added to section 11.7 as a requirement. "When raising the pressure incrementally, the pressure shall be maintained during the pause and not allowed to decrease."
- (13) Moved Eq 2 from Section 4 to 13.
- (14) Added Note 12.
- (15) Revised Section 15 Precision and Bias.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; http://www.copyright.com/