Designation: D4404 – 10

Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry[1]

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 (ε) 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 μm and 2.5 nm (0.0025 μm). 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:*[2]

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

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.

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

[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] 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.**

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:

$$d = -4\gamma(\cos\theta)/P \quad (1)$$

where:

d = apparent pore diameter being intruded,
γ = surface tension of the mercury,
θ = contact angle between the mercury and the pore wall, and
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 performance and in identifying a material that can be expected to perform in a particular manner **(1, 2)**.[3]

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

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.

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,

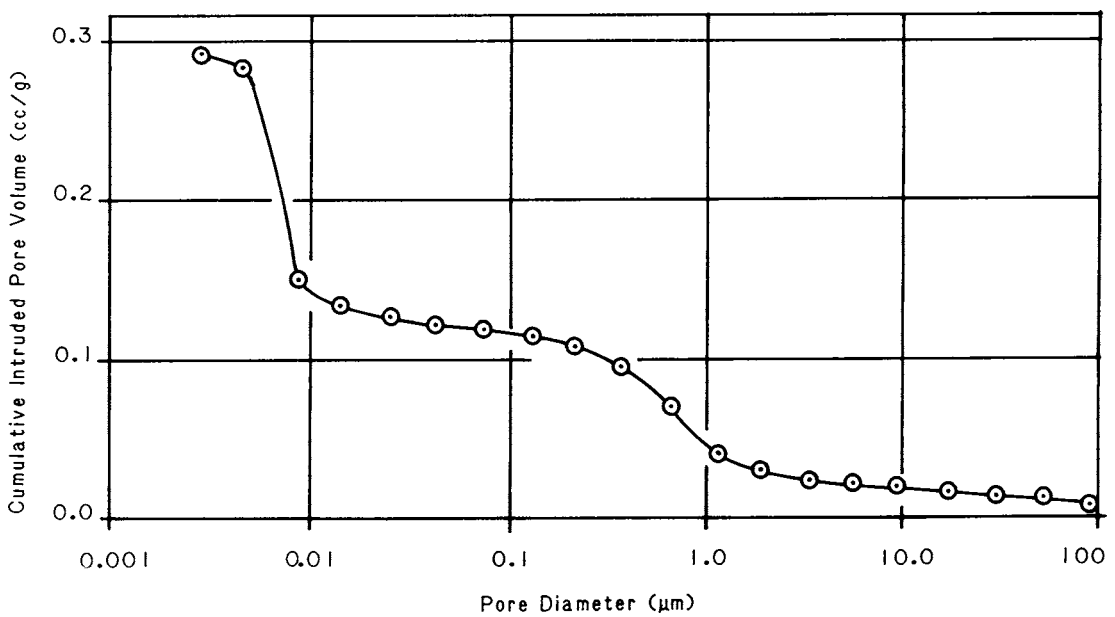

FIG. 1 Example of Cumulative Pore Volume Distribution Plot

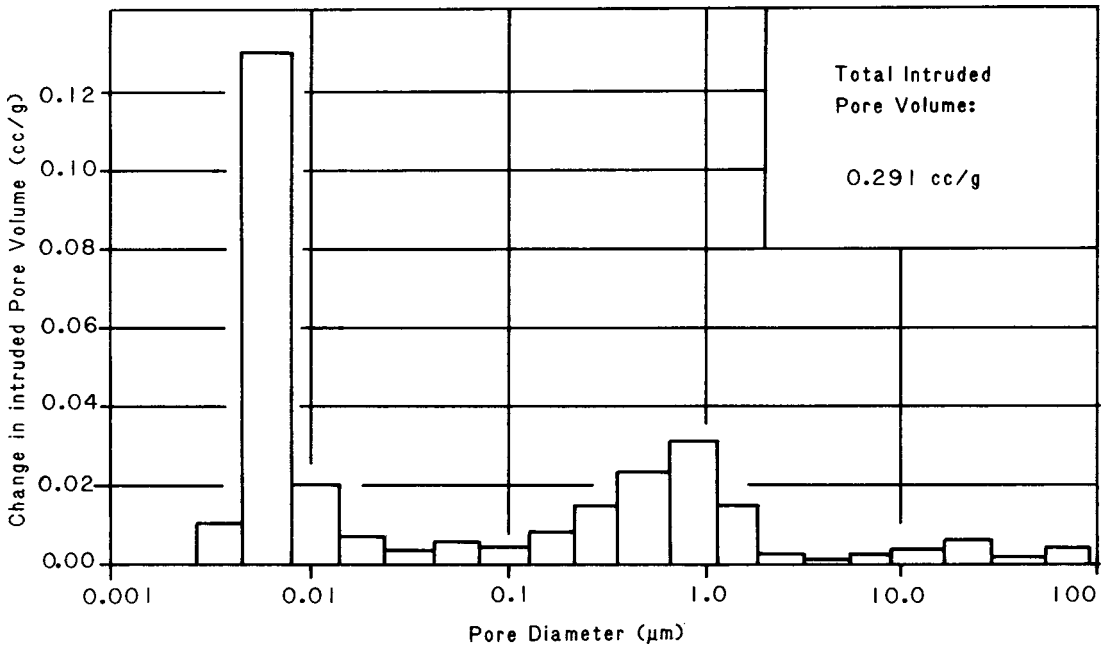

FIG. 2 Example of Differential Pore Volume Distribution Plot

a pressure generator to cause intrusion, pressure transducers, capable of measuring the intruding pressure with an accuracy of at least ±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 ±1 mm^3 ($±10^{-3}$ cm^3).

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 $±10^{-7}$ kg (±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