



Designation: D 4404 – 84 (Reapproved 1998)^{e1}

Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry¹

This standard is issued under the fixed designation D 4404; 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.

^{e1} NOTE—Keywords were added editorially in December 1998.

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 operant 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 The intrusion process proceeds from the outside of a fragment toward its center. Comparatively large interior pores can exist that have smaller pores 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 sample, inter-fragment pores can exist in addition to intra-fragment 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 inter-fragment pores can have the same apparent diameter as some intra-fragment 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 multfragment samples. 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 Fig. 1 and Fig. 2).

1.4 Mercury intrusion may involve the application of high pressures to the sample. This may result in a temporary, or permanent, or temporary and permanent alteration 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.

1.5 *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 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:

C 699 Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of, and Physical Tests on, Beryllium Oxide Powder²

3. Terminology

3.1 Definitions:

3.1.1 *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 4.1.

3.1.2 *inter-fragment pores*—those pores between fragments when they are packed together and that are intruded during the test.

3.1.3 *intra-fragment pores*—those pores lying within the exterior outlines of the individual soil and rock fragments.

3.1.4 *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

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² *Annual Book of ASTM Standards*, Vol 12.01.

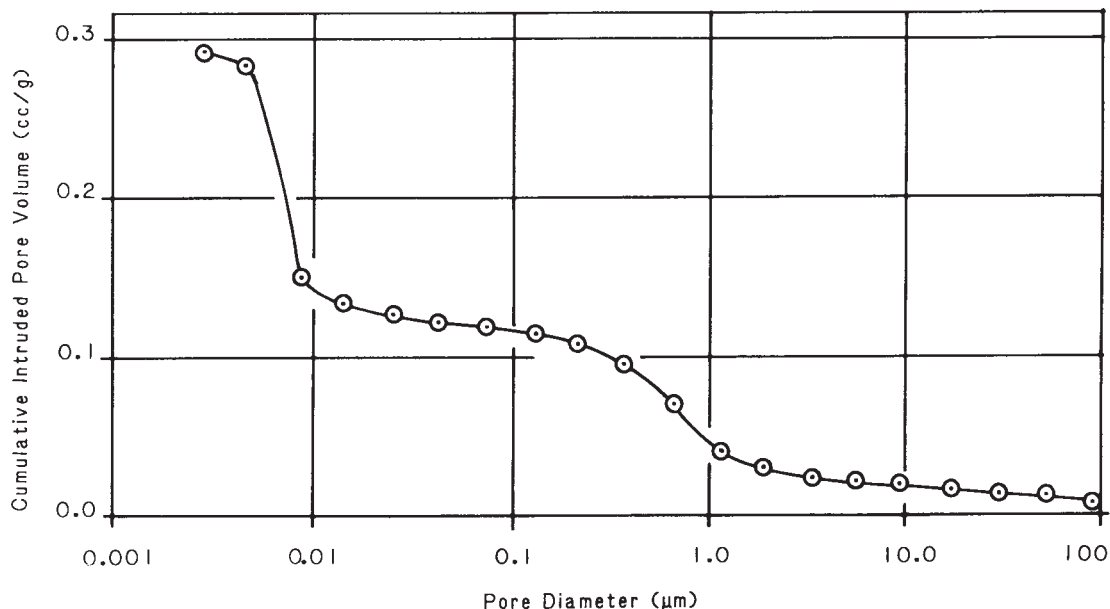


FIG. 1 Example of Cumulative Pore Volume Distribution Plot

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,
- 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 soil or rock performance. 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

6. Apparatus

6.1 *Mercury Intrusion Porosimeter*— This shall be equipped with a sample holder capable of containing one or several soil or rock fragments. This sample 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 ±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 mm³ (±10⁻³ cm³).

6.2 *Vacuum Pump*, if not part of the porosimeter, to evacuate the sample holder.

6.3 *Analytical Balance*, with an accuracy of at least ±10⁻⁷ kg (±0.1 mg).

7. Reagent

7.1 *Triple-Distilled Mercury*.

8. Safety Precautions

8.1 Mercury is a hazardous substance that can cause illness and death. Store mercury in closed containers to control its evaporation and use only in well ventilated rooms. Mercury can also be absorbed through the skin so avoid direct contact. Wash hands immediately after any operation involving mercury; the use of gloves is advocated. Exercise extreme care to avoid spilling mercury. Clean up any spills immediately using procedures recommended explicitly for mercury. Handle intruded samples with great care and dispose of in a safe and environmentally acceptable manner immediately after completion of the test.

9. Sampling, Test Specimens, and Test Units

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

NOTE 1—Sample size is limited by the pore-measuring capacity of the penetrometer, which is currently (1984) slightly more than 1 cm³. The small sample size may prevent the measurement of porosity represented by relatively large cracks and fissures in the material. Judgement is

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

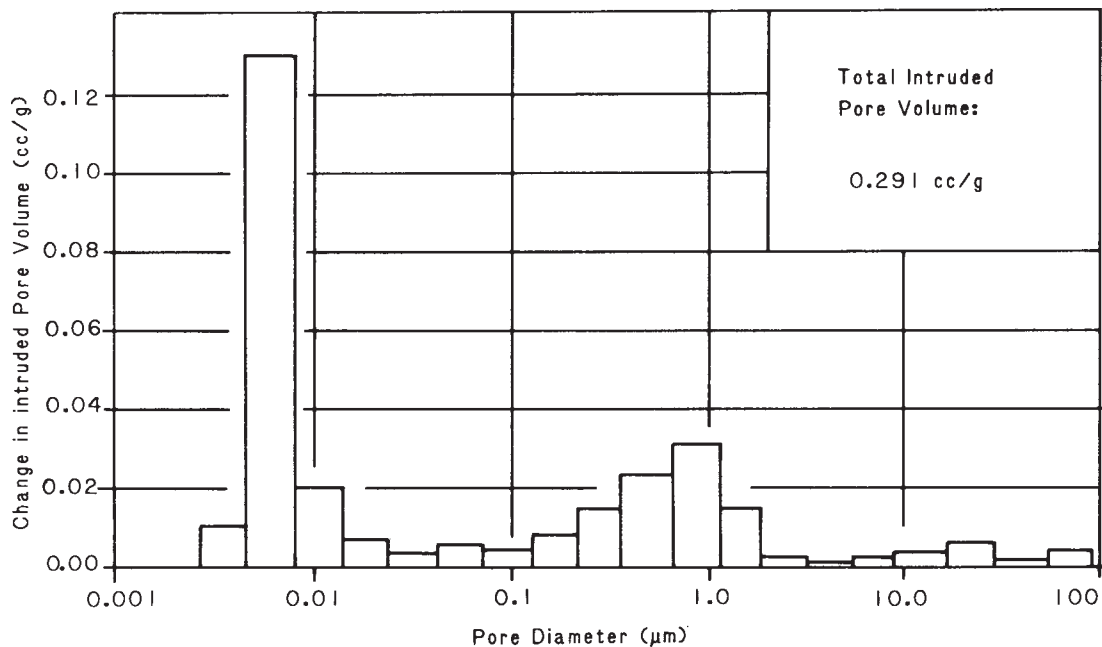


FIG. 2 Example of Differential Pore Volume Distribution Plot

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 that removes all foreign substances 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 Where the procedure described in 10.1 is not practical, rock or coarse-grained soil without fines shall be outgassed in a vacuum at 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 2—When performing the operation described in 11.2 and 11.3, the outgassed or dried material is exposed to the laboratory atmosphere and can reabsorb vapors. Thus, this operation should be carried out as rapidly as possible.

11.4 Place the penetrometer containing the sample in the appropriate chamber of the porosimeter, following the manufacturer’s instructions, and evacuate to a pressure of at least 1.3 Pa (10 µmHg).

11.5 Fill the penetrometer with mercury, in accordance with the manufacturer’s instructions, by pressurizing to some suitably low pressure.

NOTE 3—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 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 and prepare the instrument for pressurization and intrusion readings in accordance with the manufacturer’s instructions.

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.

NOTE 4—When raising the pressure incrementally, the pressure shall be maintained during the pause and not allowed to decrease.

NOTE 5—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 6—Use of the equation in 4.1 requires the absolute pressure, *P*. With some instruments it may not be possible to read the absolute pressure directly. In this case the gage pressure shall be recorded at each step, and