



Designation: **D4404 – 10 D4404 – 18**

## 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 ( $\epsilon$ ) 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  $400\ \mu\text{m}$  and  $2.5\ \text{nm}$  ( $0.0025\ \mu\text{m}$ ). 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**.

1.4 **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.

1.5 **Units**—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. Reporting of test results in units other than SI, such as cgs, shall not be regarded as nonconformance with this test method.

1.6 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice **D6026**.

1.6.1 The procedures used to specify how data are collected/recorded and calculated in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of these test methods to consider significant digits used in analysis methods for engineering data.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. For specific precaution statements, see Section 8.

1.8 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

<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~~ Feb. 1, 2018. Published ~~June 2010~~ March 2018. Originally approved in 1984. Last previous edition approved in 1998 as **D4404-84(2004):D4404-10**. DOI: ~~10.1520/D4404-10~~ 10.1520/D4404-18.

\*A Summary of Changes section appears at the end of this standard

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

[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 common technical terms used in the test method, this standard, 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.

## 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:~~The diameter of the pores filled can be calculated from this applied pressure as described

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

in Section 13, Calculations.

where:

$d$  = ~~apparent pore diameter being intruded,~~

$\gamma$  = ~~surface tension of the mercury,~~

$\theta$  = ~~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.

4.2.1 The sample is contained in a sample cell frequently called a penetrometer. The sample is contained within a sealed bowl to which a small capillary is attached. The space in the penetrometer not occupied by the sample is first evacuated and then filled with mercury. One end of the penetrometer is open and sealed to the pressure system of the instrument. Pressure applied to the open end of the penetrometer capillary is then used to force the mercury into the pores, with larger pores filling at lower applied pressures, and smaller pores filling at higher applied pressures.

4.3 Additional information about the pore structure of the material may be determined by recording of the extrusion profile for the sample. This is performed by recording the cumulative quantity of mercury contained within the sample as pressure is reduced, again either continuously or step-wise.

4.4 Commercially available instruments include software for controlling sample evacuation, filling of sample penetrometer with mercury, pressurization of the sample according to a programmed scheme, allowance for equilibration of the pressure, recording of the pressure and amount of mercury intruded into the sample, and calculation of summary results.

## 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).<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

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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 33.2 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). The intr-fragment pore concentration is shown to the left of the plot; the inter-fragment concentration is to the right.

NOTE 1—Notwithstanding the statement on precision and bias contained in this test method: The precision of this test method. 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 which that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing/testing/sampling/inspection/etc. Users of this test method standard are cautioned that compliance with Practice D3740 does not in itself assure reliable testing results. Reliable testing depends/depends on several many factors; Practice D3740 provides a means of evaluating some of those factors.

6. Apparatus

6.1 *Mercury Intrusion Porosimeter*—This shall be a device 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 ±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<sup>3</sup> (±10<sup>-3</sup> cm<sup>3</sup>).

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

6.2 *Analytical Balance*, with an accuracy of at least ±10<sup>-6</sup> kg (±1 mg), and readable to at least ±10<sup>-7</sup> kg (±0.1 mg).

6.3 *Flume Hood/Fume Hood*—A ventilated enclosure designed to limit exposure to hazardous/toxic vapors, fumes, or dust by providing a means to move the air away from the user, preferably venting it outside.

6.4 *Thermometric Device*—A thermometric device capable of measuring the temperature range within which the test is being performed readable to 0.5°C or better and having an accuracy of at least ±1°. The thermometric device shall be standardized by comparison to a nationally or internationally traceable thermometric device and shall include at least one temperature reading within the range of testing. The thermometric device shall be standardized at least once every twelve months.

6.5 *Drying Oven*—Controllable at or above 150 ± 10°C.

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.

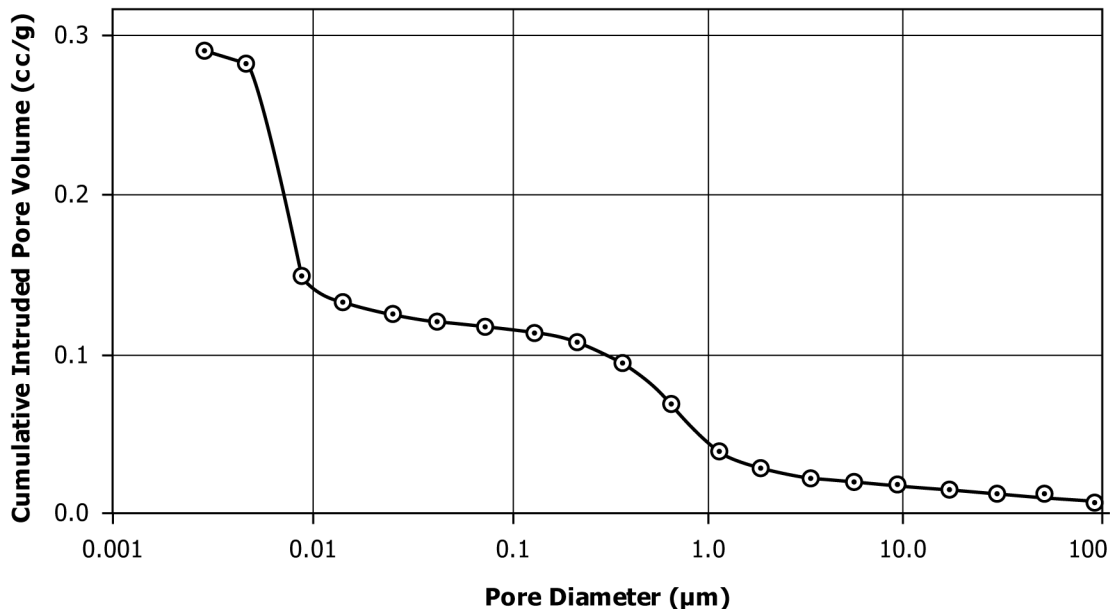


FIG. 1 Example of Cumulative Pore Volume Distribution Plot

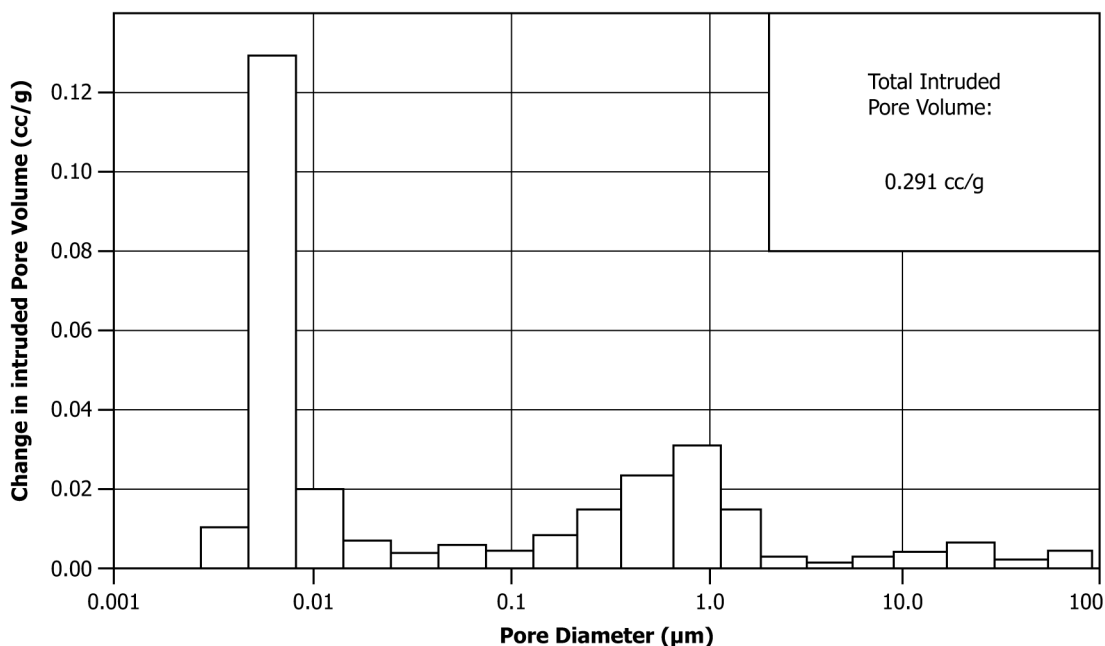


FIG. 2 Example of Differential Pore Volume Distribution Plot

7.2 *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.<sup>4</sup> 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.

## 8. Safety Precautions

8.1 **Mercury Warning**—Mercury has been designated by many regulatory agencies as a hazardous material that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable Safety Data Sheet (SDS) for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law (see Note 2), 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.

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(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.

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<sup>4</sup> 40 CFR 261.5 “Special Requirements—Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see for *Hazardous Waste Annual Standards for Laboratory Chemicals, Generated by Conditionally Exempt Small Quantity Generators*.” BDH Ltd., Poole, Dorset, U.K., and the *Available from United States Pharmacopeia and National Formulary*, U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, [http://www.access.gpo.gov/Pharmacopeial\\_Convention, Inc. \(USPC\), Rockville, MD](http://www.access.gpo.gov/Pharmacopeial_Convention, Inc. (USPC), Rockville, MD).

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8.2 Store mercury in closed containers to control its evaporation and use only in conjunction with a ~~flume~~fume 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).~~NOTE 3—USEPA conditionally exempts small quantity generators (CESQG). 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 CESQs include (see also See 40 CFR 261.5 ) for the eligibility requirements for CESQG's.~~

~~CESQs must identify all the hazardous waste.~~

~~CESQs must identify all the hazardous waste generated.~~

~~CESQs may not accumulate more than 1 000 kg of hazardous waste at any time.~~

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

## **9. ~~Sampling, Test Specimens,~~ Sampling and Test UnitsSpecimens**

9.1 The sample from which the test specimen is drawn shall be representative of the soil or ~~rock~~rock of interest. The test specimen shall be as large as practicable considering the test apparatus. Blank errors, discussed in Section 12, System Corrections, can be minimized when the volume of the sample under test occupies the majority of the sample cell space, minimizing the amount of mercury required to surround the sample. When adequate sample material is available, 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 necessary.

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~~NOTE 5—Specimen size is limited by the pore-measuring capacity of the penetrometer, which is currently (1984) slightly more than 16–387 mm.<sup>3</sup> 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.~~

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## **10. ~~Conditioning~~Preparation of Test Specimens**

10.1 The ideal preconditioning for the test specimen is an outgassing or drying procedure such as freeze drying ~~and/or~~ 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~~necessary 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 or below~~ 1.3 Pa (10 µmHg) and at a temperature of ~~±50~~150 ± 10 °C for ~~at least a minimum of~~ 24 h. Soil containing ~~any~~ plastic fines ~~requires~~needs 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.

10.3 After preconditioning, work quickly as the test specimen is exposed to the laboratory atmosphere where it can reabsorb vapors.

## **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.~~

11.1 ~~Place the outgassed or dried material~~ Determine and record the mass of the preconditioned test specimen to the nearest 0.0001 g and then quickly place the test specimen 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 reabsorb vapors. Thus, this operation should be carried out as rapidly as possible.~~

11.2 Place the penetrometer containing the specimen in the pressure vessel of the porosimeter and evacuate to a pressure of at least to 1.3 Pa (10  $\mu\text{mHg}$ )- $\mu\text{mHg}$ ) or below.

11.3 Fill the penetrometer with mercury by pressurizing to the minimum pressure greater than 1.3 Pa (10  $\mu\text{mHg}$ ) that 1.4 KPa or more such that it will promote filling.

NOTE 5—The pressure required used 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.

11.4 Place the filled penetrometer in the pressure vessel of the porosimeter, close and porosimeter if not done so in order to fill the penetrometer with mercury (commercially available instruments have different mercury filling means), then prepare the instrument for pressurization and intrusion readings. Information should be recorded in accordance with Practice Record the initial pressure used for mercury filling and base value corresponding to initial amount of mercury in the penetrometer. Most commercially available instruments use capacitance change to monitor mercury remaining in the penetrometer and not yet filling the sample pores, and thus the initial capacitance value must be recorded. Many of these commercially D6026. available instruments record the capacitance and pressure values automatically.

11.5 Raise the pressure, either continuously or incrementally, and record both the absolute pressure and the volume of intruded mercury to the nearest 0.1 mm<sup>3</sup> (0.0001 cm<sup>3</sup>) 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. When testing some materials, the time needed 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 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 ( $P_{ABS} = P_G - P_D$ ). 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.

11.6 When absolute pressure cannot be recorded directly, the gauge pressure shall be recorded at each step such that the absolute pressures can be calculated.

11.7 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 necessary 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.

11.8 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 System Correction

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

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 using the same steps increments used for the soil or rock tests to ensure make sure that temperature changes due to pressuring are the same.

12.4 Results Test results of the blank run nonporous material are a series of measured volume changes that can are also be expected to occur along with actual intrusion in a test on a material. Such blank run results are during an actual test. These results are then 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 nonporous specimen test will be either an apparent intrusion (compressibility dominant) dominant, appearing as additional recorded positive intrusion, or an expulsion of mercury (heating dominant) dominant, appearing as reduction in the amount intruded, also known as negative intrusion.

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.