Designation: D 4525 – 90 (Reapproved 2001)

Standard Test Method for Permeability of Rocks by Flowing Air[1]

This standard is issued under the fixed designation D 4525; 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 coefficient of specific permeability for the flow of air through rocks. The procedure is to establish representative values of the coefficient of permeability of rocks or well-indurated soils.

1.2 This test method is limited to permeability values greater than 0.9869 nm^2(1.0 microdarcy), and is limited to rocks free of oil or unctuous matter.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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:*

D 2434 Test Method for Permeability of Granular Soils (Constant Head)[2]

D 3877 Test Methods for One-Dimensional Expansion, Shrinkage, and Uplift Pressure of Soil-Lime Mixtures[2]

2.2 *American Petroleum Institute Standards:*

RP-27 Recommended Practice for Determining Permeability of Porous Media[3]

RP-40 Recommended Practice for Core Analysis Procedure[3]

3. Summary of Test Method

3.1 The permeability of a rock sample is measured by flowing dry air through the specimen and measuring the pressure, the flow rate, and pressure differential of the air. Three or more tests are performed on a sample at different mean air pressure values. The permeability values are plotted as a function of the reciprocal mean pressure; those points lying on a straight line are extrapolated to a value corresponding to an infinite mean air pressure to obtain an equivalent permeability value for liquids.

4. Significance and Use

4.1 This test method is designed to measure the permeability to air of a small sample of rock. By extrapolation, this test method also determines an equivalent of the liquid permeability. This parameter is used to calculate the flow through rock of fluids subjected to a pressure differential.

5. Apparatus

5.1 *Permeameter*—The permeameter shall have a specimen holder; a pressure transducer or gage, or manometers, for measuring the air pressure differential across the ends of the specimen; a means for measuring the flow rate of the air; and a means for providing dry air to the flow stream (see Fig. 1).

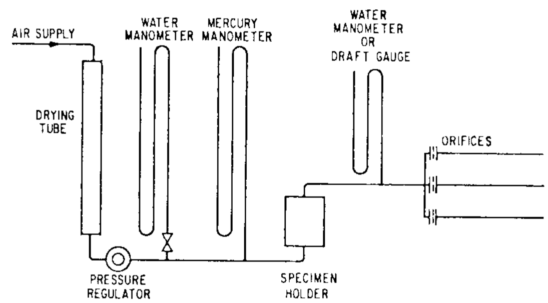

FIG. 1 Air Permeameter

5.1.1 *Specimen Holder*—The specimen holder shall have a diameter of at least ten times the diameter of the largest particle of the specimen. Where suitable, the preferred diameter is 2.54 cm. The entrance and exit flow ports shall be sufficiently large to prevent pressure loss at maximum flow rate. The length shall be 1.3 to 1.7 times the diameter.

5.1.2 *Preferred Apparatus*—In the preferred form, the specimen holder shall be an elastomer sleeve and have means for confining the sleeve and compressing it against the specimen so as to prevent bypassing of air under pressure between the sleeve and the specimen. The holder shall also have a means for confining the ends of the sample. In the preferred form, the end confining plugs will have two ports each, one for the flow of air, and the other for a static pressure line to measure pressure at the end faces of the specimen, as in Fig. 2. This type of holder is suitable for many types of flowing fluids and allows the simulation of overburden stress on the specimen.

5.1.3 *Alternative Apparatus*—An elastomer bushing may be used to confine the specimen, as in Fig. 3. This holder is

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] Available from American Petroleum Institute, 2101 L St., NW, Washington, DC 20037.

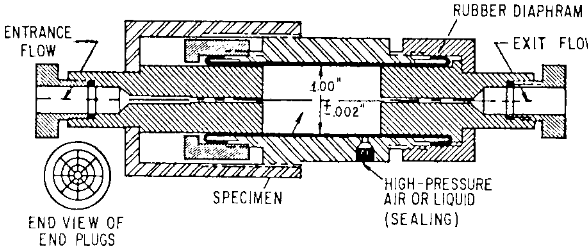

FIG. 2 Hassler Type Specimen Holder

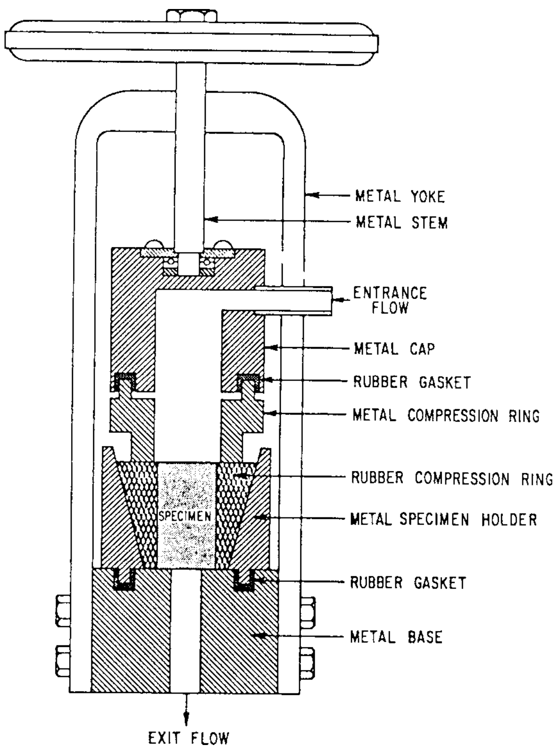

FIG. 3 Fancher-Type Specimen Holder

suitable for confining well-indurated specimens of a fine to moderate texture. This holder allows rapid operation; it cannot be used for simulating overburden stress.

5.1.3.1 Alternatively, a rigid bushing may be cast around the specimen (see Fig. 4). The casting material shall be one that will adhere well to both the specimen and the bushing, without penetration of the specimen beyond the superficial pores. Epoxies, polyesters, and sealing wax are suitable for this purpose. This method of mounting samples is particularly well suited for testing less well-indurated specimens. This technique is not applicable for tests requiring the simulation of overburden stress.

5.1.4 The flow rate of the air shall be sensed downstream from the specimen by means of calibrated orifices (Fig. 1), rotameters (Fig. 5), or a bubble meter (Fig. 6).

5.1.5 The preferred method of sensing pressure to obtain the pressure differential across the specimen is by means of pressure transducers located at the ends of the specimen. The transducers must operate over a range of 0 to 50 kPa (0 to 0.5 atmospheres) with a resolution of 250 Pa (0.0025 atmospheres) or better. Alternatively, the sensors may be connected to the end faces of the specimen with static lines, or placed in sufficiently large flow lines to cause less than 250 Pa (0.0025 atmospheres) loss of head at maximum flow rate. Pressure must be sensed between the downstream end of the specimen and the orifice if such a flow sensor is utilized.

5.1.5.1 Manometers may be utilized to measure the pressures of the flowing air. Both a mercury and water manometer must be provided, with a high-pressure cutoff valve to the water manometer as in Fig. 1, to provide the range of differential pressures required. The manometers must be 20 cm or more in height.

5.1.5.2 Alternatively, pressure gages with a range of 0 to 50 kPa (0 to 0.5 atmospheres) and a resolution of 250 Pa (0.0025 atmospheres) may be used to measure the pressure of the flowing air.

5.1.6 The dimensions of the column for drying the flowing air shall be a 2.54-cm inside diameter by a 30-cm or more length. The columns shall be filled with silica gel or anhydrous calcium sulfate, with indicator. There shall be a screen of 50 mesh on the downstream end of the filter to prevent particulate matter from reaching the specimen under test.

5.1.7 *Compressed Air Source*, with a regulator and gage, shall supply air pressure up to ½ atmosphere for the flow system.

5.1.7.1 The air shall be clean and free of particles that can plug the pores of the sample.

5.1.7.2 A compressed air supply with a separate regulator and gage, or a hydraulic pressure source with gage, shall

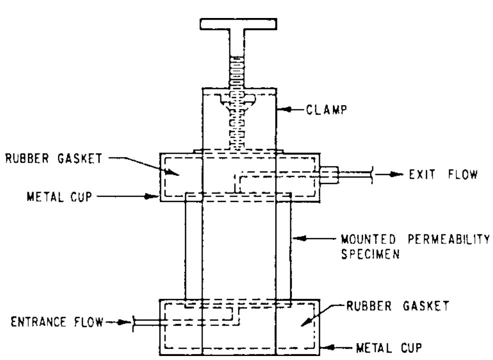

FIG. 4 Compression Cell for Ring-Mounted Specimens

FIG. 5 Shielded Microflowmeter

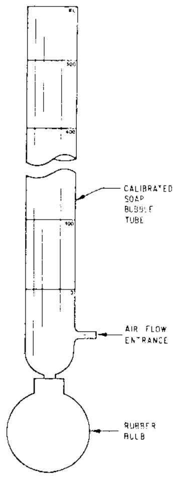

FIG. 6 Bubble Meter

supply pressure for seating the sleeve when that option for holding the specimen is used. A seating pressure of 700 kPa (7 atmospheres) or more shall be used for seating. Pressures up to 100 MPa (1000 atmospheres) may be required for simulating in situ stress.

5.1.8 *Small Vacuum Source*, for expanding the sleeve-type holder is required for specimen insertion when that holder option is utilized.

5.2 *Drilling Machine*, with a diamond bit and coolant circulating system to drill specimens from rock samples.

5.3 *Required Miscellaneous Implements*, including a stop watch for use with bubble meter, a metric scale graduated in millimetres for manometers, a caliper for measuring the length and diameter of the specimens in centimetres, ±0.05 cm, and a thermometer for measuring room temperature.

6. Sampling

6.1 An adequate supply of homogeneous material is necessary. A selection of samples shall be made using visual examination of the site of evaluation to provide a representative array of permeability values. Attention should be given to the in situ orientation of the sample when visual inspection indicates anisotropy is present. Dip and strike of bedding planes, if any, should be noted.

7. Test Specimens

7.1 Drill cylindrical specimens from the rock samples in orientations dictated by the in situ conditions and test goals, for example, parallel and perpendicular to the bedding planes. Drill the samples to a length between 1.3 and 1.7 times the diameter of the specimen.

7.2 The ends of the specimen shall be faced with a diamond saw to be approximately perpendicular to the axis of the specimen. Wash the end faces with clear water.

7.3 *Drying Specimens*:

7.3.1 If the specimen is free of swelling clay, dry in a conventional oven at a temperature of approximately 100°C until an equilibrium weight is obtained. Before weighing, cool specimens to room temperature in a desiccator. Drying time varies with specimen size and permeability; 4 h is generally sufficient for a permeable specimen of 2.54 cm in diameter.

7.3.2 If the specimen contains swelling clays, dry in a controlled humidity oven at 45 % relative humidity at 63°C until weight equilibrium is obtained. Drying time under these conditions is usually two to four days.

7.4 If necessary, clean engrained fines from the end faces of the specimen by mild wire brushing and air jetting.

8. Procedure

8.1 Measure and record the length and diameter of the specimen to ±0.1 cm. If the diameter is not uniform, measure at several positions, determine the mean diameter, and record.

8.2 Place the specimen in the specimen holder and add end pieces to the holder as necessary. Turn the wheel of the compression apparatus of the bushing-type holder, or increase the annulus pressure of the sleeve-type holder, until a seal against the periphery of the specimen is obtained. A pressure of 700 kPa (7 atmospheres) is usually found sufficient for sealing the sleeve-type holder. Apply additional pressure to the core holder if the simulation of in situ stress is required.

8.3 Open the entrance flow valve, allowing air to flow to the specimen. Adjust the entrance pressure upward until a suitable flow of air occurs, but do not exceed the critical velocity beyond which turbulent flow occurs or inertial effects become significant. Flow rates less than 2 cm^3/s per 1 cm^2 of specimen end face area usually are found to be satisfactory. Observe the flow rate until an equilibrium value is attained. Measure and record the flow rate and pressure differential across the specimen.

8.4 Lower the flowing pressure used in 8.3 to about two-thirds of the value and repeat the test of 8.3 and record.

8.5 Lower the flowing pressure used in 8.4 to about one-half of the value and repeat the test and record.

8.6 Make preliminary calculations of the flow rate divided by pressure differential of each step: 8.3-8.5. If the values are linearly related, proceed to Section 9. If the values are not linearly related, reduce the flowing pressure of 8.5, and repeat the test.

8.7 Repeat the procedures of 8.3-8.6 until a linear set of data is obtained.

9. Calculation

9.1 Calculate the coefficient of permeability, *k*, at each mean pressure, as follows:

$$k = [(2Q_e P_e \mu L)/(P_i^2 - P_e^2)A] \qquad (1)$$

where:

k = coefficient of permeability, m^2(=10^{12} Darcy),
Q_e = exit flow rate of air, m^3/s,
P_e = exit pressure of air, Pa,
L = length of specimen, m,
A = cross-section area of specimen, m^2,
P_i = entrance pressure of air, Pa, and
μ = viscosity of air at temperature of test, Pa·s.

9.2 Compute the mean pressure of each test for each specimen in Pa (atmospheres), and then calculate the reciprocal of each mean pressure, as follows:

$$2/(P_i + P_e) \qquad (2)$$

9.3 Plot the coefficient of permeability versus the reciprocal of the mean pressure for each test of a specimen, see Fig. 7.

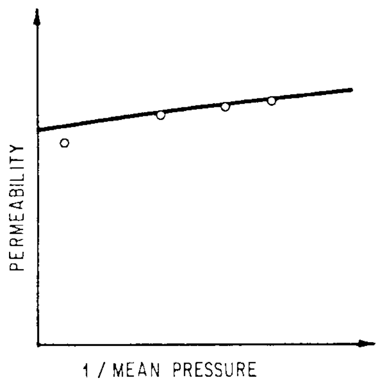

FIG. 7 Permeability versus Reciprocal Mean Pressure

Draw a straight line through at least three points (this will be at the lower values of reciprocal mean pressure) and extrapolate the line to intersect the ordinate line at zero reciprocal mean pressure. The value of *k* at the intersection is the equivalent liquid permeability of the specimen. If a straight line cannot be established through the data points, another test at a lower mean pressure may be required, or the complete test should be repeated.

10. Report

10.1 Report the following items for the particular test being performed:

10.1.1 Source of test specimen, including project name and location, as well as other pertinent data that help identify the specimen,

10.1.2 Date test is performed,

10.1.3 Physical description of the test specimen, including: rock type, such as sandstone, limestone, granite, etc.; location and orientation of inherent rock structural features; and any discontinuities and large inclusions or inhomogeneities, if any,

10.1.4 Length and diameter of the specimen (8.1), as well as flowing pressure during the test (8.3-8.7), and

10.1.5 The measured permeability (9.1), the mean pressures (9.2), and a permeability plot (9.3).

11. Precision and Bias

11.1 *Precision*—Due to the nature of rock materials tested by this test method, it is, at this time, either not feasible or too costly to produce multiple specimens that have uniform physical properties. Therefore, since specimens that would yield the same test results cannot be tested, Subcommittee D18.12 cannot determine the variation between tests since any variation observed is just as likely to be due to specimen variation as to operator or laboratory testing variation. Subcommittee D18.12 welcomes proposals to resolve this problem that would allow for development of a valid precision statement.

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

12. Keywords

12.1 flow and flow rate; permeability