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Designation: D4535 - 08 <u>D4535 - 13</u>

Standard Test Methods for Measurement of Thermal Expansion of Rock Using Dilatometer¹

This standard is issued under the fixed designation D4535; 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*Scope

1.1 These test methods cover the laboratory measurement of the <u>one-dimensional</u> linear (one-dimensional) thermal expansion of rocks using a dilatometer.

1.2 These test methods are applicable between temperatures of 25°C to 300°C. Both bench top and confined measurement techniques are presented. Test Method A is used for unconfined or bench top measurements and Test Method B is used for confined conditions. Rocks of varying moisture content can be tested.

1.3 For satisfactory results in conformance with these test methods, the principles governing the size, construction, and use of the apparatus described in these test methods should be followed. If the results are to be reported as having been obtained by this either test method, then all the pertinent requirements prescribed in this by that test method shall be met.

1.4 These test methods do not establish details of construction and <u>procedureprocedures</u> to cover all test situations that might offer difficulties to a person without technical knowledge concerning the theory of heat flow, temperature measurement, and general testing practices. Standardization of these test methods does not reduce the need for such technical knowledge. It is recognized also that it would be unwise, because of the standardization of this method, to resist in any way the further development of improved or new methods or procedures by research workers.

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

1.6 The procedures used to specify how data are collected/recorded or 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 this standard to consider significant digits used in analytical methods for engineering design.

1.7 The values stated in SI units are to be regarded as the standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.8 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:²

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D2216 Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass

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

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

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¹ These test methods are under the jurisdiction of ASTM Committee D18 on Soil and Rock and are the direct responsibility of Subcommittee D18.12 on Rock Mechanics. Current edition approved July 1, 2008Nov. 1, 2013. Published July 2008December 2013. Originally approved in 1985. Last previous edition approved in 2004 as D4535 - 85 (2004):D4535 - 08. DOI: 10.1520/D4535-08.10.1520/D4535-13.

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 Standardsvolume information, refer to the standard's Document Summary page on the ASTM website.

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E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

E83 Practice for Verification and Classification of Extensometer Systems E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 For definitions of common technical terms in this standard, refer to Terminology D653.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 samplespecimen thermal strain, ε_t —change in length of a unit length of sample when the sample is subjected to heat. The mathematical expression is:

 $\varepsilon_{\tau} = (L_{\tau} - L_{\tau})/L_{0} \tag{1}$

where:

 L_1 and L_2 = specimen lengths corresponding to temperatures T_1 and T_2 , and

 L_0 = the original specimen length at some reference temperature T_0 .

Thermal change in length, $(L_2 - L_1)$, divided by the original length, L_0 , of the specimen when the specimen is subjected to heat. Specimen thermal strain is also equal to the specimencorrected thermal displacement, expansion, δ_t , divided by the original sample length: specimen length.

 $\varepsilon_r = \delta_r / L_0$

3.2.2 mean coefficient of linear expression, expansion, α_m —between two temperatures, a value, often expressed in parts per million per degree. It is obtained by dividing the linear thermal strain, ($(TL_{12} and TL_{217})/L_0$ is defined as follows:), by the change in

$$\frac{\alpha_m = (L_2 - L_1) [L_0 (T_2 - T_1)]}{\text{temperature units cm/cm (in./in.) per temperature change in °C (°F).}$$
(3)

where:

 L_1 and L_2 = specimen lengths at temperatures T_1 and T_2 , respectively. Therefore, α_m is obtained by dividing the linear thermal strain, $(L_1 - L_2)/L_0$, by the change in temperature units are inch/inch or centimetre/centimetre per temperature change in °F or °C, respectively. α_m is often expressed in parts per million per degree.

3.2.3 Upon heating The sign convention used for α_m is as follows: α_m will be a positive value indicating an increase in the length of the rock specimen $(T_2 > T_1)$, an increase in the length of the rock sample will give a positive value of) and α_m . If a decrease in length (contraction) is observed, α will be a negative value indicating a decrease or contraction of the rock specimen methods, see Terminology D653.

4. Summary of Test Methods

4.1 The application of heat to a rock causes it to expand. This expansion divided by the original length of the rock <u>specimensspecimen</u> is the thermal strain from which coefficients of expansion can be calculated. This standard covers two methods for measuring rock expansion. The primary difference between the two methods is in the type of dilatometer used.

4.1.1 *Test Method I*—Test Method <u>IA</u> is <u>the procedure</u>-used when making unconfined or bench top measurements. The method and apparatus are similar to that described in Test Method E228. The rock <u>specimenspecimen's</u> thermal displacement is measured using a dilatometer as shown in Fig. 1. The <u>samplespecimen</u> displacement is measured by a transducer located outside the heated area of the <u>samplespecimen</u>; therefore, apparent strain due to apparatus expansion and contraction is minimized.

4.1.2 *Test Method II*—Test Method <u>II</u>—<u>B</u> is most suited for the measurement of rock thermal strain under conditions and employs a dilatometric device which is located inside the heated zone, as shown in Fig. 2. This test method is most suited for the measurement of rock thermal strain under confined conditions. Test Method B is amenable to confined thermal strain determinations; however, confined tests may be most appropriate when:

4.1.2.1 Pore pressure must be imposed in the pore space to maintain the liquid phase of water through the desired temperature range.

4.1.2.2 The thermal strain of the rock is sensitive to confining stress.

4.1.2.3 The specimen is fragile or friable, or both, and cannot be machined into the shapes required for Test Method A.

4.2 In both test methods, <u>samplespecimen</u> expansion is measured continuously as temperature is gradually increased or allowed to stabilize at discrete temperature points.

5. Significance and Use

5.1 Information concerning the thermal expansion characteristics of rocks is important in the design of any underground excavation where the surrounding rock may be heated. Thermal strain causes thermal stresses which ultimately affect excavation

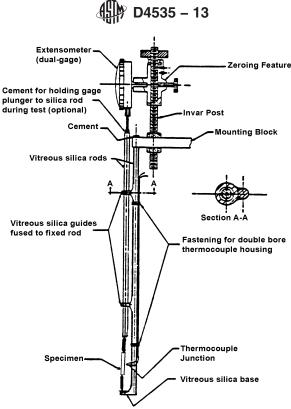


FIG. 1 Apparatus Commonly Used to Perform Bench Top (Method I) (Test Method A) Thermal Expansion Measurements

stability. Examples of applications where rock thermal strain is important include: nuclear waste repositories, underground power stations, compressed air energy storage facilities, and geothermal energy facilities.

5.2 The coefficient of thermal expansion or "alpha" or expansion, α , of rock is known to vary as the temperature changes. These methods provide continuous thermal strain values as a function of temperature, and therefore provide information on how alpha the coefficient of thermal expansion changes with temperature.

5.3 Rocks are also often anisotropic, thus displaying different thermal strains depending on the orientation of strain measurement. These methods allow for measuring strain in one direction only. If anisotropy is expected, samplesspecimens with different orientations should be prepared and tested.

NOTE 1—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 that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing. Users of this standard are cautioned that compliance with Practice D3740 does not in itself assure reliable results. Reliable results depend on many factors; Practice D3740 provides a means of evaluating some of those factors.

5.4 Care should be exercised in the interpretation of thermal strain data of rocks with significant moisture content. Under certain temperature and pressure conditions, steam may be produced in the pore space. Steam may cause errors because of microcrack production or changes in the pore pressure. The phase change from water to steam in the pore space can result in several phenomena which complicate data analysis, as follows:

5.4.1 Evolved steam may change the pore pressure and thus the effective stress in the rock, resulting in anomalous strain readings.

5.4.2 Losing all the moisture may dehydrate clays in the pore space and thus change expansion characteristics, especially in layered rocks.

5.5 The researcher using this standard must use best judgment as to how to make the thermal expansion measurement so that it accurately represents the conditions in the field.

5.6 Method II is amenable to confined thermal strain determinations. Confined tests may be most appropriate when:

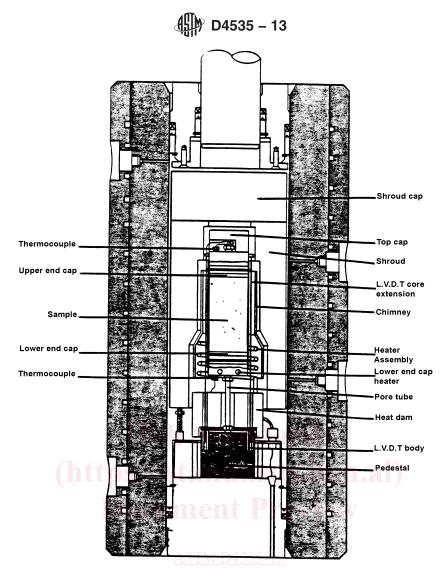
5.6.1 Pore pressure must be imposed in the pore space to maintain the liquid phase of water through the desired temperature range.

5.6.2 The thermal strain of the rock is sensitive to confining stress.

5.6.3 The sample is fragile or friable, or both, and cannot be machined into the shapes required for Method I.

6. Interferences

6.1 Care should be exercised in the interpretation of thermal strain data of rocks with significant moisture content. Under certain temperature and pressure conditions, steam may be produced in the pore space. Steam may cause errors because of microcrack



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FIG. 2 Apparatus Commonly Used to Perform Confined (Method II) (Test Method B) Thermal Expansion Measurements

production or changes in the pore pressure. The phase change from water to steam in the pore space can result in several phenomena which complicate data analysis, as follows:

6.1.1 Evolved steam may change the pore pressure and thus the effective stress in the rock, resulting in anomalous strain readings.

<u>6.1.2</u> Losing all the moisture may dehydrate clays in the pore space and thus change expansion characteristics, especially in layered rocks

6.1.3 Good judgment should be used when deciding how to make the thermal expansion measurement so that it accurately represents the conditions in the field.

7. Apparatus

7.1 Dilatometer:

7.1.1 <u>Test Method I—A</u>. The dilatometer used for bench measurements may be of the tube or rod type, as shown in Fig. 1. Those components of the dilatometer exposed to elevated temperatures should be fabricated of materials with coefficients of linear expansion that are as small as practicable.

7.1.2 <u>Test Method II—B—In Method II the The</u> entire dilatometer is exposed to elevated temperature. Therefore, temperature; therefore, transducers, rods, and other components should be fabricated of materials with low thermal expansions (for expansions. For example, fused silica, and super invar). When the apparatus is tested with a quartz calibration specimen, the apparatus strain should be less than 20 % of the anticipated rock strain (refer to Fig. 2).



7.2 *Extensometer*—Extensometers measure length change. In principle, any accurate length measuring device with good long-term stability may be used; this includes including dial gauges, linear variable differential transducers, or capacitive transducers. Whichever device is selected, it must have sufficient resolution to measure 0.01 % samplespecimen strain (Refer to Practice E83).

7.2.1 <u>Those devices Devices used in Test Method HB</u> must be fabricated of materials that allow direct exposure of the device to the anticipated temperature. Also, transducer bodies should be vented for operation in a pressure environment. At least two transducers are used, as shown in Fig. 2, and their outputs averaged.

7.3 *Furnace*—The furnace shall be large enough to contain the specimen and apparatus and maintain uniform temperature along the axis of the specimen with variations no greater than $\pm 1^{\circ}$ C. The mean sample temperature shall be controlled within $\pm 1^{\circ}$ C. The use of a programmable temperature controller that can slowly increase or decrease <u>samplespecimen</u> temperatures at rates at least as low as 0.1°C/min is recommended.

7.4 *Temperature Measuring Instruments*—Thermocouples or platinum resistant thermometers are recommended. The exact type will depend on the temperature range of interest. In general, the temperature should be measured to within $\pm 0.5^{\circ}$ C with a resolution of at least $\pm 0.2^{\circ}$ C. Make measurements at three locations on the axis of the sample, specimen, one near each end and one at the samplespecimen midpoint.

7.5 *Micrometer—Specimen Size Measurement Devices*—Calipers should have an index permitting direct reading of 0.025 mm for measuring the initial length of the specimen. A high grade screw micrometer customarily used in machine shop practice is satisfactory. Devices used to measure the length and diameter of the specimen shall be capable of measuring the desired dimension to within 0.1 % of its actual length.

8. Sampling

8.1 The number and types of rock cores tested depend partly on the intended application of the test results. For example, an initial mechanical characterization of a site might require several samples from a variety of formations, while a detailed thermo-mechanical investigation of a particular location may require many rock tests from a single formation. The final testing program will depend on the technical judgment and the experience of project personnel.

7.2 Statistical Requirements—It is recommended that the number of samples tested be sufficient to provide an adequate statistical basis for evaluation of the results. Rock types which are highly variable would require more tests than relatively uniform rocks, in order to evaluate the results with equal certainty.

7.3 Moisture Condition of Samples—The moisture condition of the rock can influence the measured thermal expansion. Test the specimens in a manner that best simulates the in situ conditions of interest. For natural conditions, the moisture content of the rock core and the chemical characteristics of the pore fluid shall be preserved between the time of recovery and testing; then determine the moisture content of core material contiguous to the test specimen.

7.4 Anisotropy—The thermal expansion coefficient of many rocks is different along various axes of the rock. Measure the thermal expansion in several directions in order to assess the degree of anisotropy.

8.2 *Documentation*—<u>Statistical Requirements</u>—Since the thermal expansion of most rock is anisotropic, it is important that the field orientation of each sample is recorded. Note the orientation of each sample on the sample and earry suitable markings through each cutting until the final specimen is ready for testing. These markings should indicate compass direction and up/down directions, and other orientation with respect to geologic structures. The number of samples and specimens tested shall be sufficient to provide an adequate statistical basis for evaluation of the results. Rock types that are highly variable will require more tests than relatively uniform rocks, in order to evaluate the results with equal certainty.

8.2.1 The number of samples and specimens required to obtain a specific level of statistically valid results may be determined using Test Method E122. However, it may not be economically possible to achieve specific confidence levels and professional judgment may be required.

8.2.2 Documentation—Since the thermal expansion of most rock is anisotropic, it is important that the field orientation of each sample is recorded. Note the orientation of each sample on the sample and carry suitable markings through each cutting until the final specimen is ready for testing. These markings should indicate compass direction and up/down directions, and other orientation with respect to geologic structures.

<u>8.3 Moisture Condition of Samples</u>—The moisture condition of the rock can influence the measured thermal expansion. The samples shall be preserved to prevent moisture change

<u>8.4 Anisotropy</u>—The thermal expansion coefficient of many rocks is different along various axes of the rock; therefore, in order to assess the degree of anisotropy, the thermal expansion must be measured in several directions.

9. Preparation of Test Specimens

9.1 Take the samples and machine them into the proper geometry as discussed in 9.2.

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9.1.1 Do not degrade the rock during machining. Prevent mechanical and fracture damage to the rock fabric by appropriately slow machining processes and the use of proper coolant. Select coolant fluids based upon chemical compatibility with the rock; for example, tap water may be adequate for granite, whereas a saturated brine or mineral oil may be best for salt.

9.2 Dimension and Geometry—In general, the proper geometry of a specimen is a right circular cylinder. The specific recommended dimensions for <u>Test</u> Method <u>HA</u> are given in Test Method <u>E228</u>. For <u>Test</u> Method <u>HB</u>, the <u>samplespecimen</u> should be a right circular cylinder with a length to diameter ratio of 2 to 1. For both methods the minimum dimension should be 10 times the largest grain size. Measure and record the length and diameter of the specimen to 0.001 mm. Take a minimum of three length measurements 120° apart and at least three diameter measurements at the quarter points of the height. Determine the average length and diameter of the specimen.

<u>9.3 Moisture Condition of Specimens</u>—Test the specimens in a manner that best simulates the in situ conditions of interest. For natural conditions, the moisture content of the rock core and the chemical characteristics of the pore fluid shall be preserved between the time of recovery and testing. Determine the moisture content of core material contiguous to the test specimen in accordance with Test Method D2216.

9.3.1 If the specimen is to be tested dry, dry at 80°C in a vacuum oven for 24 h. At no time during the drying process shall the specimen be subjected to heating or cooling rates greater than 1°C/min.

9.3.1.1 An alternative drying schedule may be used in those instances where a vacuum oven is not available and it is not of interest to know the test specimen response to the first application of heat. In such a case, heat the specimen to $105 \pm 2^{\circ}C$ at a rate not greater than $1^{\circ}C/min$. Maintain this temperature for at least 24 h. Cool the specimen to ambient temperature at a rate no greater than $1^{\circ}C/min$.

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10. Standardization

10.1 <u>Calibration Verification</u> Specimen—Prepare a <u>calibration verification</u> specimen of known thermal expansion from fused silica or other material of known low ($\sim 0.55 \times 10-6$ cm/cm/°C) thermal expansion. The specimen shall have the same geometry and dimensions as the rock specimens to be tested.

10.2 Test the <u>ealibrationverification</u> specimen using the same procedure (see the procedure section) and the same apparatus to be used to test the rock <u>samples</u>. The resulting data set thus represents the thermal expansion of the test apparatus and will be subtracted from the rock test data.

10.3 Repeat the standardization test procedure three times, starting from the same initial condition, to verify the repeatability of the dilatometer. Variation from run to run should be no greater than 5 %.

10.4 The calculated expansion of the <u>calibrationverification</u> specimen is subtracted from the <u>calibrationverification</u> expansion results as follows:

 $-\delta_2 = \delta_1 - \delta_s;$	-(1)
 $\delta_2 = \delta_1 - \delta_s$	(1)

 $\delta_{s} = \alpha \cdot l \cdot \Delta T$

where:

where:

 δ_2 = thermal expansion of the test apparatus, cm,

 δ_1 = apparent thermal expansion measured by the apparatus, cm

 δ_{s} = thermal expansion of the calibration specimen, em

 $\delta_s =$ thermal expansion of the verification specimen, cm

 α = coefficient of linear expansion for the calibration specimen,

 $\underline{\alpha}$ = coefficient of linear expansion for the verification specimen,

= gauge length of the calibration specimen, cm, and

(2)