

Designation: D4535 - 13<sup>2</sup> D4535 - 21

# Standard Test Methods for Measurement of Thermal Expansion of Rock Using Dilatometer<sup>1</sup>

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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

ε¹ NOTE—Editorial corrections were made throughout in February 2014.

#### 1. Scope-Scope\*

- 1.1 These test methods cover the laboratory measurement of the one-dimensional linear thermal expansion of rocks using a dilatometer.
- 1.2 The methods are applicable between temperatures of 25°C to 300°C. Both bench top and confined measurement techniques are presented. Method A is used for unconfined or bench top measurements and 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 shouldshall be followed. If the results are to be reported as having been obtained by either test method, then the pertinent requirements prescribed by that test method shall be met.
- 1.4 These test methods do not establish details of construction and procedures 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.
- 1.5 <u>Units—</u>The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard. Reporting of test results in units other than SI 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 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.

ε<sup>2</sup> NOTE—Editorially updated units of measurement statement in April 2018.

<sup>&</sup>lt;sup>1</sup> 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 Nov. 1, 2013 June 1, 2021. Published December 2013 June 2021. Originally approved in 1985. Last previous edition approved in 2004 2013 as D4535 – 08:D4535 – 13<sup>e2</sup>. DOI: 10.1520/D4535-13E02.10.1520/D4535-21.



- 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 establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.
- 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.

#### 2. Referenced Documents

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

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

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:
- 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 specimen thermal strain,  $\varepsilon_{t\underline{TS}}$  [D], n—change in length,  $(L_2 L_1)$ , divided by the original length,  $L_0$ , of the specimen when the specimen is subjected to heat.
  - 3.2.1.1 Discussion—
- Specimen thermal strain is also equal to the corrected thermal expansion,  $\delta_{tTS}$ , divided by the original specimen length.
  - 3.2.2 mean coefficient of linear expansion,  $\alpha_m$ , n—a value, often expressed in parts per million per degree; obtained by dividing the linear thermal strain,  $((L_2 L_1)/L_0)$ , by the change in temperature  $(T_2 > T_1)$ .
    - 3.2.2.1 Discussion—

The sign convention used for  $\alpha_m$  is as follows:  $\alpha_m$  will be a positive value indicating an increase in the length of the rock specimen upon heating  $(T_2 > T_1)$  and  $\alpha_m$  will be a negative value indicating a decrease or contraction of the rock specimen.

#### 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 specimen 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 A is used when making unconfined or bench top measurements. The method and apparatus are similar to that described in Test Method E228. The rock specimen's thermal displacement is measured using a dilatometer as shown in Fig. 1. The specimen displacement is measured by a transducer located outside the heated area of the specimen; therefore, apparent strain due to apparatus expansion and contraction is minimized.reduced.
- 4.1.2 Test Method B is most suited for the measurement of rock thermal strain under confined conditions and employs a dilatometric device which is located inside the heated zone, as shown in Fig. 2. 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.

<sup>&</sup>lt;sup>2</sup> 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.



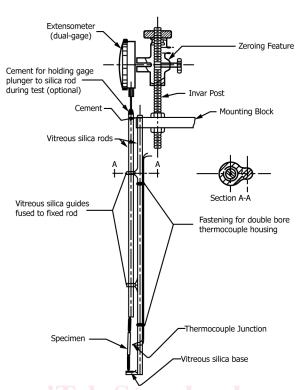


FIG. 1 Apparatus Commonly Used to Perform Bench Top (Test Method A) Thermal Expansion Measurements

- 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 requirednecessary for Test Method A.
- 4.2 In both test methods, specimen expansion is measured continuously as temperature is gradually increased or allowed to stabilize at discrete temperature points.

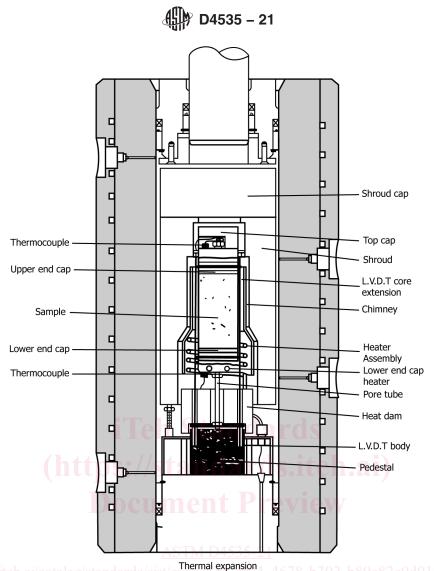
#### 5. Significance and Use

- 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 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,  $\alpha$ , 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 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, specimens with different orientations shall 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.

#### 6. Interferences

6.1 Care should be exercised in the interpretation of thermal strain data of rocks with significant moisture content. Under certain



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

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:

- 6.1.1 Evolved steam may change the pore pressure and thus the effective stress in the rock, resulting in anomalous strain readings.
- 6.1.2 Losing all the moisture may dehydrate clays in the pore space and thus change expansion characteristics, especially in layered rocksrocks.
- 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 A</u>—Test Method A—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 Test Method B—The entire dilatometer is exposed to elevated temperature; therefore, transducers, rods, and other components should be fabricated of materials with low thermal expansions. For example, fused silica, and super invar. When the apparatus is tested with a quartz calibration specimen, the apparatus strain should is recommended to be less than  $\frac{20 \% 20 \%}{20 \%}$  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; including dial gauges, linear variable differential transducers, or capacitive transducers. Whichever device is selected, it must have sufficient resolution to measure 0.01 % specimen strain (Refer to Practice E83).
- 7.2.1 Devices used in Test Method B must be fabricated of materials that allow direct exposure of the device to the anticipated temperature. Also, transducer bodies should be vented within the pressure vessel must be designed 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 specimen temperature shall be controlled within  $\pm 1^{\circ}$ C. The use of a programmable temperature controller that can slowly increase or decrease specimen 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 shouldshall be measured to within  $\pm 0.5$ °C with a resolution of at least  $\pm 0.2$ °C. Make measurements at three locations on the axis of the specimen, one near each end and one at the specimen midpoint near the midlength of the specimen.
- 7.5 Specimen Size Measurement Devices—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.
- 7.6 Verification Specimen—A specimen fabricated from fused silica or other material with a small, known coefficient of thermal expansion that is of the same dimensions and geometry as the rock specimens to be tested.

#### 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 thermomechanical investigation evaluation 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.
- 8.2 Statistical Requirements—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 <u>required\_necessary</u> to obtain a specific level of statistically valid results may be determined using Test Method E122. However, it may not be economically <u>possible\_practical</u> to achieve specific confidence levels and professional judgment <u>may be required-is advised.</u>
- 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 The orientation of each sample shall be recorded on the sample and carry suitable markings through each cutting until the final specimen is ready for testing. These If applicable, these markings should indicate compass direction and up/down directions, and other orientation with respect to geologic structures.
- 8.3 *Moisture Condition of Samples*—The moisture condition of the rock can influence the measured thermal expansion. The samples shall be preserved to prevent moisture changechange.
- 8.4 *Anisotropy*—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.
- 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 more suitable 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 Test Method A are given in Test Method E228. For Test Method B, thea specimen should be that is a right circular cylinder with a length to diameter ratio of 2 to 1. 1 is recommended. 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. specimen. 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.
- 9.3 Moisture Condition of Specimens—Test the specimens in a manner that bestadequately 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 furnace for 24 h. At no time during <u>During</u> the drying <u>process shall</u> <u>process</u>, the specimen <u>shall not</u> 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 furnace 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.

#### 10. Standardization

## **Document Preview**

- 10.1 Verification Specimen—Prepare a verification specimen of known thermal expansion from fused silica or other material of known low (~0.55 × 10–6 cm/cm/°C) thermal expansion. The specimen shall have the same geometry and dimensions as the rock specimens to be tested. The half eatloy/standards/sist/cadec | ee-db | 1-4678-b302-b89e82c9d0 | frastm-d4535-2 |
- 10.1 Test the verification specimen using the same procedure and the same apparatus to be used to test the rock specimens. Note: The resulting data set thus represents allows for the thermal expansion of the test apparatus and to be calculated, which will be subtracted from the rock test data.
- 10.2 Repeat the standardization test procedure three times, procedure, starting from the same initial condition, to verify the repeatability of the dilatometer. Variation from run to run should be no greater than 5 %.shall not exceed 5 %.
- 10.3 The calculated Thermal expansion of the verification specimen is subtracted from the verification expansion results as follows: apparatus:

 $\delta_2 = \delta_1 - \delta_s \tag{1}$ 

where:

$$\delta_{s} = \alpha \cdot l \cdot \Delta T \tag{2}$$

10.3.1 The calculated expansion of the verification specimen is subtracted from the verification expansion results as follows:

$$\delta_{TA} = \delta_{app} - \delta_{vs} \tag{1}$$

where:

$$\delta_{vs} = \alpha \cdot l \cdot \Delta T \tag{2}$$



where:

<u>=</u> thermal expansion of the test apparatus, cm

apparent thermal expansion measured by the apparatus, cm

thermal expansion of the verification specimen, cm

= coefficient of linear expansion for the verification specimen

 $\underline{\alpha}$ gauge length of the verification specimen at reference temperature, cm

 $\Delta T$ temperature difference between a reference temperature (room temperature or slightly elevated above room temperature)

and an elevated temperature, °C

where:

 $\theta_{2}$ thermal expansion of the test apparatus, em

apparent thermal expansion measured by the apparatus, em

= thermal expansion of the verification specimen, cm

= coefficient of linear expansion for the verification specimen

= gauge length of the verification specimen, cm

= temperature difference between a reference temperature (room temperature or slightly elevated above room temperature)

and an elevated temperature, °C

10.3.2 The thermal expansion of the apparatus is recommended to be less than 20 % of the measured thermal expansion of the rock.

10.4 The thermal expansion of the apparatus should be less than 20 % of the measured thermal expansion of the rock. The measured thermal expansion of the apparatus shall be reported as specified in Section 1514.

### 11. Preconditioning

11.1 Rock specimens shall not be thermally cycled before the actual testing unless drying is specified, in which case drying shall be performed in accordance with  $\frac{10.29}{.3.1}$ .

#### 12. Procedure

- 12.1 For either test method, clean the specimen with a non-chemical reactive solvent, such as acetone, and install the specimen in the dilatometer. Take special care to make sure the end surfaces of the specimen are free from foreign particles.
- 12.2 If Test Method B confined experiments are to be performed, jacket the specimen with an appropriate heat resistant jacketing material to prevent confining fluid intrusion. (Note 2):
- 12.3 For either test method, clean the specimen with a non-chemical reactive solvent, such as acctone, and install the specimen in the dilatometer. Take special care to make sure the end surfaces of the specimen are free from foreign particles. If Test Method B confined experiments are to be performed, jacket the specimen with an appropriate heat resistant jacketing material to prevent eonfining fluid intrusion (Note 2). Install all temperature measuring instrumentation and insert the specimen into the furnace. Heat the specimen in accordance with one of the following thermal schedules, A or B ((Note Note 3): 3):

Note 2—Silicone elastomers are often used for jacketing material.

- 12.3.1 *Schedule A*—A series of constant temperatures.
- 12.3.2 *Schedule B*—Heating or cooling at constant rate.
- 12.4 Schedule A—Heat or cool the dilatometer assembly specimen between any two target temperatures at a maximum rate of an average rate not exceeding 1°C/min, leaving it at each temperature until the output of the extensometer shows no significant change. A significant change would be 2 %- is 2 % of the displacement measured during any two temperature increments. For some combinations of temperature increments and specimen thermal expansion, fluctuations in the displacement readings may exceed the 2% criterion; in these cases, maintain the target temperature until the displacement rate oscillates about zero. Make measurements at a sufficient number of temperatures so the rock's thermal strain as a function of temperature is known. Usually,