Designation: D4535 – 13$^{\varepsilon 1}$

Standard Test Methods for Measurement of Thermal Expansion of Rock Using Dilatometer[1]

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.

ε[1] NOTE—Editorial corrections were made throughout in February 2014.

1. 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 should 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 *Units*—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. 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.

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 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

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:*

[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.

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[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 Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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, ε_t [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, δ_t, divided by the original specimen length.

3.2.2 *mean coefficient of linear expansion, α_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 α_m is as follows: α_m will be a positive value indicating an increase in the length of the rock specimen upon heating $(T_2 > T_1)$ and α_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.

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.

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, 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.

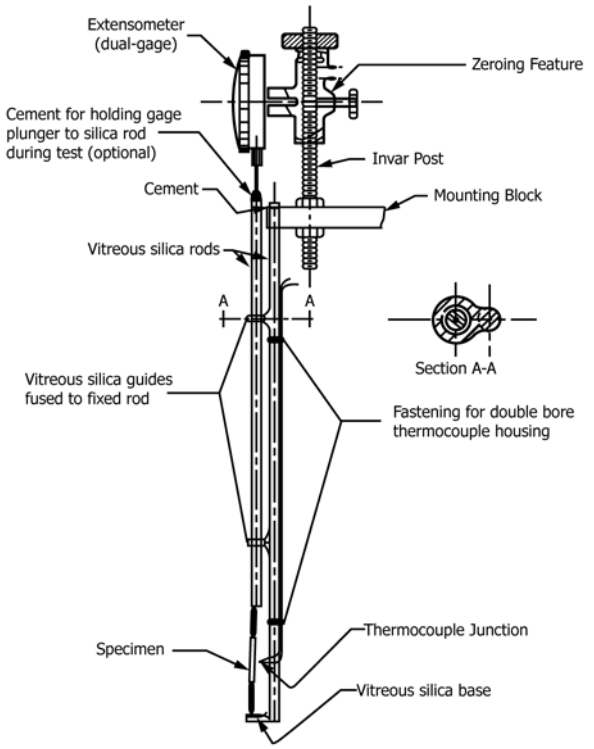

FIG. 1 Apparatus Commonly Used to Perform Bench Top (Test Method A) Thermal Expansion Measurements

5.2 The coefficient of thermal 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 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 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

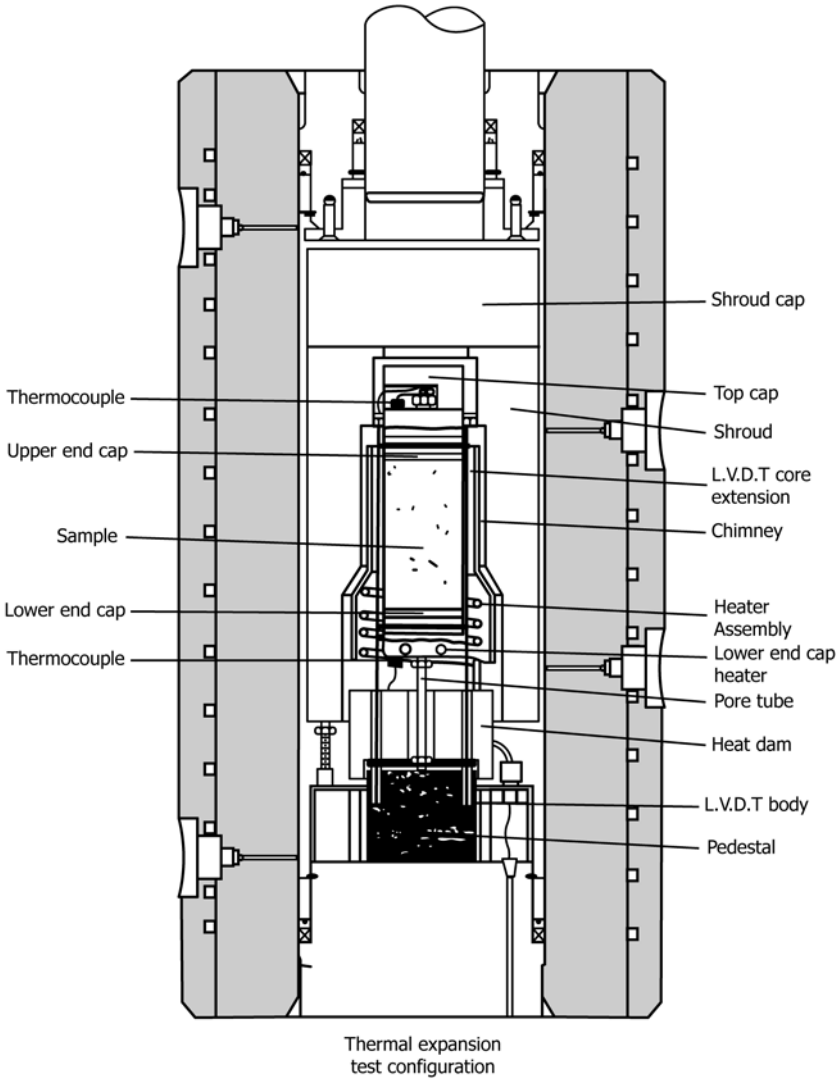

FIG. 2 Apparatus Commonly Used to Perform Confined (Test Method B) Thermal Expansion Measurements

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 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 *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 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; 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 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 ±1°C. The mean specimen temperature shall be controlled within ±1°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 should be measured to within ±0.5°C with a resolution of at least ±0.2°C. Make measurements at three locations on the axis of the specimen, one near each end and one at the specimen midpoint.

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.

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.

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 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.

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 change

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 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, the specimen 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.

9.3 *Moisture Condition of Specimens*—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 furnace 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 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 ± 2°C at a rate not greater than 1°C/min. Maintain this temperature for at least 24 h. Cool the specimen to ambient temperature at a rate no greater than 1°C/min.

10. Standardization

10.1 *Verification Specimen*—Prepare a verification 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 verification specimen using the same procedure and the same apparatus to be used to test the rock specimens. 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 verification specimen is subtracted from the verification expansion results as follows:

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

where:

$$\delta_s = \alpha \cdot l \cdot \Delta T \quad (2)$$

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 verification specimen, cm
α = coefficient of linear expansion for the verification specimen
l = gauge length of the verification specimen, cm
ΔT = temperature difference between a reference temperature (room temperature or slightly elevated above room temperature) and an elevated temperature, °C

10.5 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 15.

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 10.2.

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. 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). 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 3):

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

12.1.1 *Schedule A*—A series of constant temperatures.

12.1.2 *Schedule B*—Heating or cooling at constant rate.

12.2 *Schedule A*—Heat or cool the dilatometer assembly between any two temperatures at a maximum rate of 1°C/min, leaving it at each temperature until the output of the extensometer shows no significant change. A significant change would be 2 % of the displacement measured during any two temperature increments. Make measurements at a sufficient number of temperatures so the rock's thermal strain as a function of temperature is known. Usually, a minimum of eight measurements is required. The minimum holding time is 30 min. Read the extensometer and temperature at each hold temperature and record both.

12.3 *Schedule B*—Starting at room temperature, or some other slightly elevated temperature, heat the specimen at a rate less than 1°C/min. Heating or cooling rates in excess of 1°C/min are unacceptable since faster rates may produce thermal gradients which result in specimen damage and significant differences between measured specimen temperature and actual specimen temperature. During heating or cooling, read and record the extensometer and temperature.

12.4 Perform at least two complete heating and cooling cycles on each specimen to record the changes induced by heating. If large hysteresis is observed, additional cycles may be necessary.

12.5 For Test Method B confined experiments, exercise care to make sure the confining pressure and, if applicable, the pore pressure are maintained constant throughout the heating and cooling cycles. The use of gas backed hydraulic accumulators is a convenient and inexpensive method for maintaining constant stress and pore pressure.

NOTE 3—In general, Schedule A results in greater accuracy; however, it is more practical to use Schedule B because (*1*) a series of constant temperature holds is more time consuming, and (*2*) in temperature regions where the expansion of the material is time-dependent, the constant rate conditions specified in Schedule B usually lead to easier comparison of the data.

13. Calculations

13.1 Calculate the corrected thermal expansion, δ_t, as follows:

$$\delta_t = \delta_1 - \delta_2 \quad (3)$$

where:

δ_1 = apparent thermal expansion measured by the apparatus, cm
δ_2 = thermal expansion of the test apparatus, cm

13.1.1 Use the thermal expansion of the apparatus, δ_2, calculated as described in 10.4. Make this calculation for each discrete temperature if Schedule A was used. If Schedule B was used, make sufficient calculations so that a well-defined curve is described in δ_t versus temperature, T, space.

13.2 Calculate thermal strain, ε_t, and apparatus thermal strain, ε_1, using the following relationships:

$$\varepsilon_t = \delta_t / L_0 \quad (4)$$

$$\varepsilon_1 = \delta_1 / L_0$$

where:

δ_t = corrected thermal expansion, cm
L_0 = original specimen length at same reference temperature, cm
δ_1 = apparent thermal expansion measured by the test apparatus, cm

13.3 On the same chart, plot rock thermal strain and apparatus thermal strain as a function of temperature. An example of how the final plot may appear is shown in Fig. 3.

13.4 If desired, the mean coefficient of linear expansion between any two temperatures may be calculated as follows:

$$\alpha_m = (L_2 - L_1)/[L_0 \cdot (T_2 - T_1)] = (\varepsilon_{T2} - \varepsilon_{T1})/(T_2 - T_1) \quad (5)$$

where:

ε_{T1} = specimen thermal strain at temperature, T_1
ε_{T2} = specimen thermal strain at temperature, T_2

14. Report Test Data Sheet(s)/Form(s)

14.1 The methodology used to specify how data are recorded on the test data sheet(s)/form(s) as given below, is covered in 1.6 and Practice D6026.

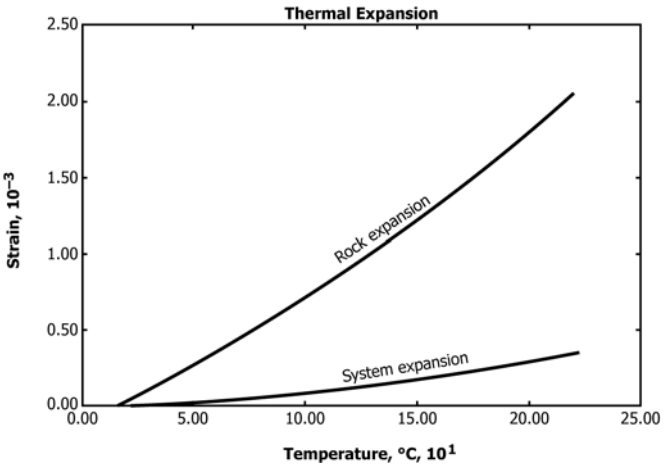

FIG. 3 Presentation of Rock and Apparatus Thermal Strain Versus Temperature

14.2 Record as a minimum the following general information (data):

14.2.1 Project information, such as project name, number, source of test specimens, including other pertinent data that helps identify the specimen.

14.2.2 Name of person(s) who prepared and tested the specimens, including the date(s) performed.

14.2.3 Description of the samples. Include the rock type, structure and fabric, grain size, discontinuities or voids, and weathering of the sample as applicable.

14.2.4 Describe special handling procedures, such as those used to maintain moisture content, to avoid damage during machining, and the like.

14.2.5 The thermal expansion of the test apparatus, δ_2, nearest 0.01 cm.

14.2.6 The apparent thermal expansion measured by the apparatus, δ_1, nearest 0.01 cm.

14.3 Record as a minimum the following test data:

14.3.1 The test method used, A or B, the heating schedule used, A or B. Include additional information regarding confining and pore pressures used during the test.

14.3.2 The average, initial length and diameter of the specimen(s) to the nearest 0.001 mm.

14.3.3 The moisture content of the sample(s).

14.3.4 The field orientation of each sample.

14.3.5 The temperatures, T_1, T_2, and the reference temperature T_0 to the nearest 0.5°C.

14.3.6 The specimen lengths, L_0, L_1, L_2, taken at their respective temperatures, to the nearest 0.001 mm.

14.3.7 The corrected thermal expansion, δ_t, to the nearest 0.01 cm.

14.3.8 The specimen thermal strain, ε_t.

14.3.9 If applicable the mean coefficient of linear expression, α_m.

14.3.10 A listing of the test equipment actually used, including the name, model number, if known, and basic specifications of each major piece of equipment, as applicable.

14.3.11 List any deviations from Section 12 or the equipment used. Discuss the effect of the variation upon the test results.

14.3.12 Plots of thermal strain versus temperature for each specimen. Include on each plot the sample designation, rock type, and temperature range. For tests performed under Test Method B, describe any special environmental conditions to which the specimen was subjected. These may include, but are not limited to, confining stress and pore pressure.

14.3.13 Summary tables may be presented. These may include sample designation, temperature ranges, average coefficients of thermal expansion, and uncertainties.

14.3.14 Each plot should have error bars indicating the magnitude of the estimated 95 % level of uncertainty. This uncertainty includes the combined effects resulting from transducer readout devices. Also add (in a statistical manner) the uncertainty resulting from the subtraction of the apparatus thermal strain from the measured thermal strain data.

15. Precision and Bias

15.1 The precision of thermal expansion measurements using the above methods has been estimated to be approximately 5 % for a specific rock type. This estimate is based on approximately 150 measurements on similar rocks.[3] However, the precision for any specific test is dependent on the thermal strain of the dilatometer and how large this apparatus thermal strain is in comparison to the rock thermal strain. Also of importance is the magnitude of the rock thermal strain in comparison to that of the apparatus verification sample (a large difference in thermal expansion between the two results in greater precision). The final precision, therefore, depends on the specific apparatus being used and the rock being tested.

15.2 *Bias*—There is no accepted reference value for this test method; therefore, bias cannot be determine

16. Keywords

16.1 rock; thermal expansion/contraction; thermal strain; dilatometer

[3] Van Buskirk, R., Enniss, D., and Schatz, J., "Measurement of Thermal Conductivity and Thermal Expansion at Elevated Temperatures and Pressures," Symposium on Measurement of Rock Properties at Elevated Pressures and Temperatures, *ASTM STP 869*, 1985, p. 108.