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AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Methods for Measurement of Thermal Expansion of Rock Using a Dilatometer[1]

This standard is issued under the fixed designation D 4535; 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 These test methods cover the laboratory measurement of the 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. 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 methods should be followed. If the results are to be reported as having been obtained by this method, then all pertinent requirements prescribed in this method shall be met.

1.4 These test methods do not establish details of construction and procedure 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 *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:*

E 83 Practice for Verification and Classification of Extensometers[2]

E 228 Test Method for Linear Thermal Expansion of Solid Materials with Vitreous a Silica Dilatometer[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

$$\epsilon_t = (L_2 - L_1)/L_0 \quad (1)$$

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 strain is also equal to the specimen thermal displacement, δ_t, divided by the original sample length:

$$\epsilon_t = \delta_t/L_0 \quad (2)$$

3.1.2 *mean coefficient of linear expression, α_m*—between two temperatures, T_1 and T_2, is defined as follows:

$$\alpha_m = (L_2 - L_1)/[L_0(T_2 - T_1)] \quad (3)$$

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.

3.1.3 Upon heating ($T_2 > T_1$), an increase in the length of the rock sample will give a positive value of α_m. If a decrease in length (contraction) is observed, α_m will become negative.

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 specimens 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 I is the procedure used when making unconfined or bench top measurements. The method and apparatus are similar to that described in Test

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[1] These test methods are under the jurisdiction of ASTM Committee D-18 on Soil and Rock and are the direct responsibility of Subcommittee D18.12 on Rock Mechanics.

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

Method E 228. The rock specimen thermal displacement is measured using a dilatometer as shown in Fig. 1. The sample displacement is measured by a transducer located outside the heated area of the sample; therefore, apparent strain due to apparatus expansion and contraction is minimized.

4.1.2 *Test Method II*—Test Method II 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.

4.2 In both test methods, sample 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 or "alpha" or 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 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, samples with different orientations should be prepared and tested.

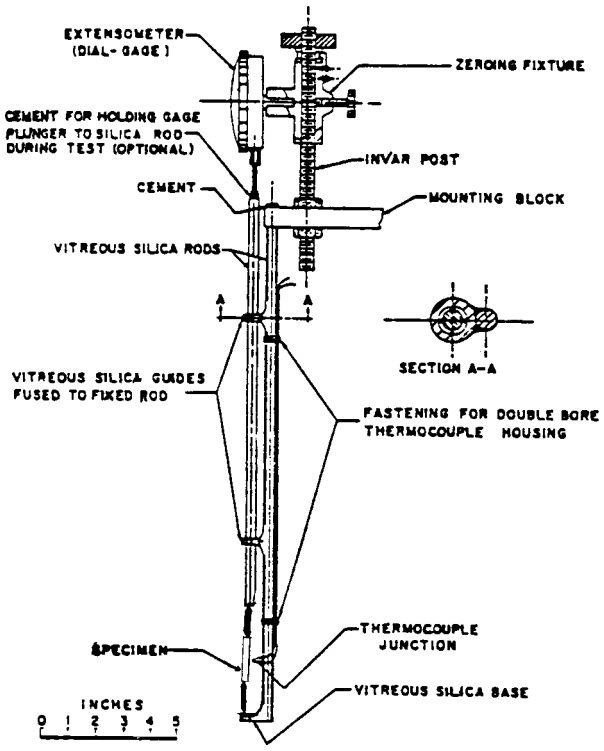

FIG. 1 Apparatus Commonly Used to Perform Bench Top (Method I) Thermal Expansion Measurements

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. Apparatus

6.1 *Dilatometer*:

6.1.1 *Method I*—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.

6.1.2 *Method II*—In Method II 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, 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).

6.2 *Extensometer*—Extensometers measure length change. In principle, any accurate length measuring device with good long-term stability may be used; this includes dial gages, linear variable differential transducers, or capacitive transducers. Whichever device is selected, it must have sufficient resolution to measure 0.01 % sample strain (Refer to Practice E 83).

6.2.1 Those devices used in Method II 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.

6.3 *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 sample temperature shall be controlled within ±1°C. The use of a programmable temperature controller that can slowly increase or decrease sample temperatures at rates at

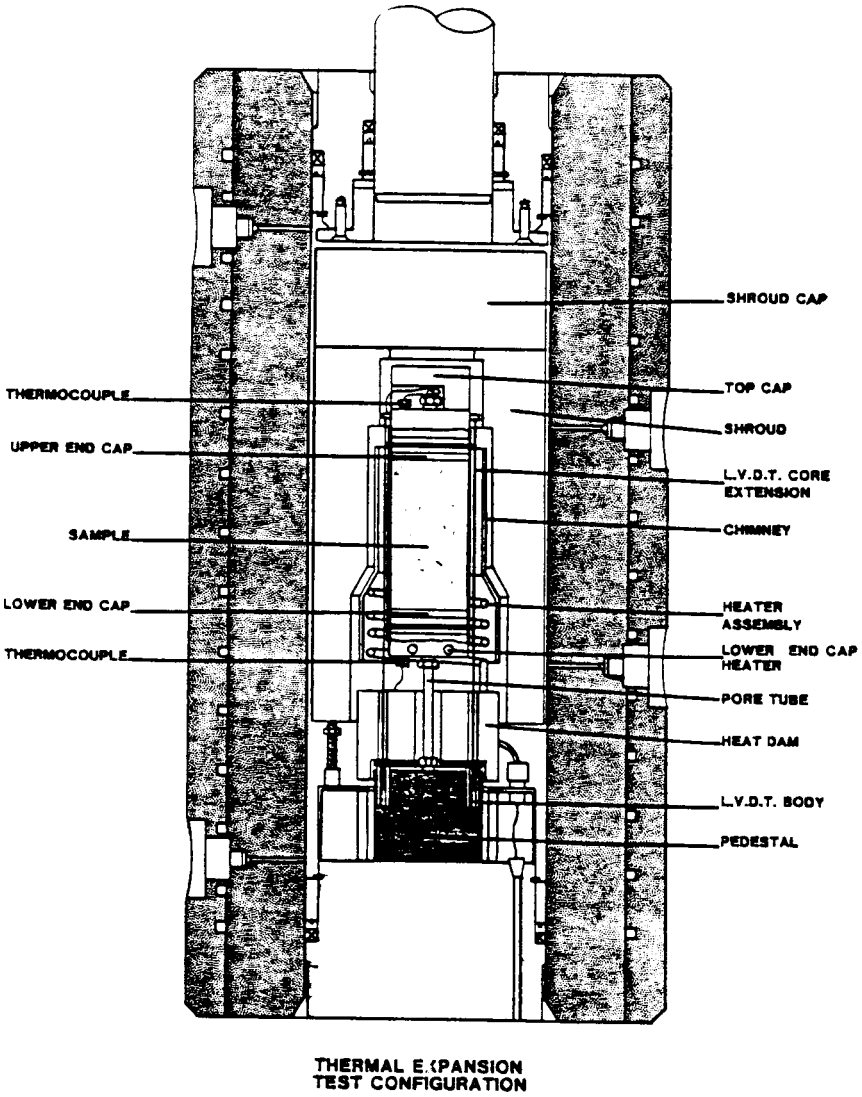

FIG. 2 Apparatus Commonly Used to Perform Confined (Method II) Thermal Expansion Measurements

least as low as 0.1°C/min is recommended.

6.4 *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 sample, one near each end and one at the sample midpoint.

6.5 *Micrometer*—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.

7. Sampling

7.1 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.

7.5 *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. Test Specimens

8.1 *Dimension and Geometry*—In general, the proper geometry is a right circular cylinder. The specific recommended dimensions for Method I are given in Test Method E 228. For Method II, the sample 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.

9. Preparation

9.1 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 *Drying*—If the sample 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 sample be subjected to heating or cooling rates greater than 1°C/min.

9.2.1 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 ± 2°C at a rate not greater than 1°C/min. Maintain this temperature for at least 24 h. Cool the sample to ambient temperature at a rate no greater than 1°C/min.

10. Standardization

10.1 *Calibration Specimen*—Prepare a calibration 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 calibration specimen using the same procedure (see the procedure section) and the same apparatus to be used to test the rock samples. 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 calibration specimen is subtracted from the calibration expansion results as follows:

$$\delta_2 = \delta_1 - \delta_s; \tag{4}$$

where:

$$\delta_s = \alpha \cdot l \cdot \Delta T \tag{5}$$

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, cm
α = coefficient of linear expansion for the calibration specimen,
l = gage length of the calibration specimen, cm, and
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 14.

11. Preconditioning

11.1 Rock samples shall not be thermally cycled before the actual testing unless drying is specified, in which case drying shall be performed in accordance with 9.2.

12. Procedure

12.1 Clean the sample with a non-chemical reactive solvent, such as acetone, and install the sample in the dilatometer. Take special care to ensure that the end surfaces of the specimen are free from foreign particles. If confined experiments are to be performed (Method II), jacket the specimen with an appropriate heat resistant jacketing material to prevent confining fluid intrusion (Note 1). 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 2):

NOTE 1—Silicone elastomers are often used for jacketing material.

NOTE 2—In general, Schedule A results in greater accuracy. 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.

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 that 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.

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 sample damage and significant differences between measured sample temperature and actual sample temperature. During heating or cooling, observe the extensometer reading and temperature. Calculate thermal

expansion as prescribed in 9.1 and report it as a function of temperature.

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

12.5 For confined experiments, exercise care to ensure that confining pressure and, if applicable, 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.

13. Calculations

13.1 Calculate the corrected thermal expansion, δ_t as follows:

$$\delta_t = \delta_1 - \delta_2 \tag{6}$$

where:

δ_1 = apparent thermal expansion measured by the apparatus, cm, and

δ_2 = thermal expansion of the test apparatus.

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 T space.

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

$$\epsilon_t = \delta_t / L_0 \tag{7}$$

$$\epsilon_1 = \delta_1 / L_0$$

where:

δ_t = specimen thermal displacement,

L_0 = original specimen length at same reference temperature, and

δ_1 = thermal expansion of the test apparatus.

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:

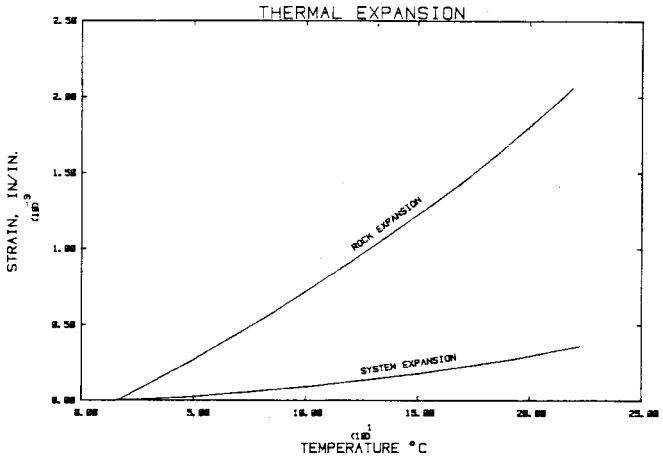

FIG. 3 Presentation of Rock and Apparatus Thermal Strain Versus Temperature

$$\alpha_m = (L_2 - L_1)/[L_0 \cdot (T_2 - T_1)] = (\epsilon_{T2} - \epsilon_{T1})/(T_2 - T_1) \tag{8}$$

where:

ϵ_{T1} = thermal strain at temperature T_1, and

ϵ_{T2} = thermal strain at temperature T_2.

14. Report

14.1 The report shall include the following:

14.1.1 Description of the samples and special handling procedures. The rock type, structure and fabric, grain size, discontinuities or voids, and weathering of the sample shall be described as a minimum. Further detail depends on the application of the results. Describe special handling procedures, such as those used to maintain moisture content, to avoid damage during machining, and the like.

14.1.2 Test sample dimensions.

14.1.3 A detailed listing of the equipment actually used for the test, including the name, model number, and basic specifications of each major piece of equipment.

14.1.4 If the actual equipment or procedure has varied from the requirements contained in this standard, each variation and the reasons for it shall be noted. Discuss the effect of the variation upon the test results.

14.1.5 The method, I or II, and the heating schedule, A or B, used. Fully discuss special procedures under Method II, such as the application of confining stress and pore pressure.

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

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

14.1.8 Each plot will have error bars indicating the magnitude of the estimated 95 % level of uncertainty. This 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.[4] 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 calibration 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.

[4] 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.

15.2 Bias is not known at this time since this is a new procedure. Estimates of the bias of this procedure must await (*1*) the results of round-robin testing of similar samples by different laboratories using this procedure, and (*2*) the availability of a recognized thermal expansion standard.

16. Keywords

16.1 destructive examination; rock; thermal expansion/contraction; thermal properties