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Standard Practice for Longevity Assessment of Firestop Materials Using Differential Scanning Calorimetry[1]

This standard is issued under the fixed designation E2923; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a standardized procedure for quantitatively assessing the longevity of materials used in firestop systems, by the use of data obtained from differential scanning calorimetry.

1.2 This practice is intended to differentiate firestop materials that are expected to maintain performance characteristics over time from those that are expected to degrade in performance characteristics over time. DSC experimental curve evaluation can also deliver indifferent results, where an interpretation of sample properties is not possible without additional testing using conventional durability testing. It evaluates the extent of chemical reactions that will occur within the firestop material under specified conditions of temperature and humidity. This practice does not measure longevity under specific severe environmental conditions or building operation that might be experienced by an individual firestop system.

1.3 This practice is intended to be used to test the materials used within a firestopping system. The practice is not intended to be used to test the properties of assembled firestopping systems.

1.4 This practice is intended to evaluate the following types of materials used in through-penetration fire stops:
1.4.1 Endothermic,
1.4.2 Intumescent,
1.4.3 Insulation,
1.4.4 Ablatives, and
1.4.5 Subliming.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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. Some specific hazards are given in Section 8 on Hazards.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
E814 Test Method for Fire Tests of Penetration Firestop Systems
E2041 Test Method for Estimating Kinetic Parameters by Differential Scanning Calorimeter Using the Borchardt and Daniels Method

3. Terminology

3.1 *Definitions:*

3.1.1 *firestop material, n*—the part of a firestop system that provides the necessary seal to prevent the passage of flame and hot gases when tested in accordance with Test Method E814. This includes any material that serves the purpose of closing and sealing the gap(s) created in a fire-resistance rated wall or floor to accommodate a through-penetration.

3.1.2 *longevity, n*—a measure of the length of time a product meets specified performance requirements.

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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.

3.1.2.1 *Discussion*—

Longevity is not intended to be a measure of how long a product retains the precise properties that it had at the time of manufacture. Most materials will change over time to some extent, so a measurement of time before discernible change occurs would not generally be realistic or useful. Rather, longevity is intended to be a measure of how long a product retains its properties to a sufficient degree to be deemed as meeting the purpose(s) for which it was manufactured.

4. Summary of Practice

4.1 A small sample of the firestop material is tested by differential scanning calorimetry in accordance with Test Method E2041 to determine the following information:

4.1.1 Calculation of total released energy.

4.1.2 Determination of reaction order.

4.1.3 Determination of activation energy and Arrhenius frequency factor.

4.1.4 Calculation of the conversion rate for 270 days at 70°C.

4.1.5 Calculation of the conversion rate for 30 years (10 950 days) at 50°C.

4.2 Using the kinetic data, the chemical conversion rate for the material can be calculated for any time duration and temperature combination. The conversion rate for that time and temperature is then compared to the predetermined threshold of acceptability. That threshold shall be expressed as the largest fraction of the original material that shall be permitted to undergo change through chemical reaction(s) while still allowing the material to adequately perform its design function.

5. Significance and Use

5.1 Firestop systems are exposed to fire tests and classified using materials that have been, in all likelihood, quite recently manufactured. The testing provides a fire resistance rating for the firestop system that is measured in hours. The goal of firestop system testing is to identify and list firestop systems that will have a fire resistance rating that is no less than the fire resistance rating of the classified wall or floor assembly in which it is installed. A building fire that could put the firestop system to the test can occur at any time during the life of the building. By that time, the firestop system is composed of materials that have aged. Some assurance is desired to establish quantitatively that the firestop system will continue to have a fire resistance rating that is no less than that of the wall or floor assembly.

5.2 This practice provides one method for examining whether any changes are to be expected in the characteristics of a firestop material during its design life, as gauged by any chemical reactions that occur within the material to change it. The measurement of conversion rate provides a standard measure of how much a material will change over its design life. This provides an objective indication of whether the bulk of the material is likely to exhibit the desirable properties for which it was chosen in the firestop system.

5.3 Measurement of conversion rate allows different firestop materials used for similar purposes to be compared with respect to their ability to remain unchanged during their design life.

5.3.1 This allows materials with an unusually high conversion rate to be questioned and possibly rejected early on during the research and development process.

5.3.2 This allows materials to be screened by testing and listing agencies to ensure that they do not provide a listing for products that are not likely to have adequate performance for the full length of the intended design life.

5.3.3 This allows formulation changes that have no apparent impact on the results of the fire testing to be evaluated for any possible long-term consequences on performance.

5.3.4 Re-calculation of the conversion rate (other than for the standard time and temperature specified in Section 11) allows materials to be evaluated for suitability in applications where they may be regularly exposed to unusually high temperatures, or for suitability in installations which are intended to have an unusually long design life, or both.

5.4 Measurement of conversion rate allows longevity of firestop materials to be compared to the longevity of the classified wall or floor assemblies in which the firestop system is installed, by measuring the conversion rate for each. This comparison can ensure that the firestop system does not degrade significantly faster, thus possibly deeming it to be unacceptable. The comparison can also ensure that the firestop system is not unjustifiably held to a higher standard of longevity than the floor or wall itself.

5.5 The fundamental assumption inherent in making use of DSC conversion rate data for assessing longevity of firestop materials is that if the material has a chemical stability that keeps it from changing much over time in a certain environment, then it is reasonable to expect it to adequately perform its design function if subjected to an actual fire many years after installation.

6. Interferences

6.1 Because of its simplicity and ease of use, the Borchardt and Daniels method is often the method of choice for characterization of the kinetic parameters of a reaction system. The Borchardt and Daniels method, like all tools used to evaluate kinetic parameters, is not applicable to all cases. The user of this method is expressly advised to use this method and its results with caution.

6.2 Tabulated below are some guidelines for the use of the Borchardt and Daniels method.

6.2.1 The approach is applicable only to exothermic reactions.

NOTE 1—Endothermic reactions are controlled by the kinetics of the heat transfer of the apparatus and not by the kinetics of the reaction.

6.2.2 The reaction under investigation must have a constant mechanism throughout the whole reaction process. In practice, this means that the reaction exotherm upon heating must be smooth, well shaped with no shoulders, multiple peaks or discontinuous steps.

6.2.3 The reaction must be *n*th order. Confirmation of an *n*th order reaction shall be made by an isothermal experiment such as that described in Appendix X1 in Test Method E2041.

6.2.4 Typical reactions which are not *n*th order and to which Borchardt and Daniels kinetic shall not be applied for predictive purposes include many thermoset curing reactions and crystallization transformations.

6.2.5 The *n*th order kinetic reactions anticipate that the value of *n* will be small, non-zero integers, such as 1 or 2. Values of $n > 2$ or which are not simple fractions, such as ½ = 0.5, are highly unlikely and shall be viewed with caution.

6.2.6 The Borchardt and Daniels method assumes temperature equilibrium throughout the whole test specimen. This means that low heating rates, (that is, <10 K/min), small specimen sizes (<5 mg) and highly conductive sealed specimen containers, for example, aluminum, gold, platinum, etc., shall be used.

6.3 Since milligram quantities of specimen are used, it is essential that the specimen be homogeneous and representative of the test sample from which they are taken.

7. Apparatus

7.1 *Differential Scanning Calorimeter (DSC),* the instrumentation required to provide the minimum differential scanning calorimetric capability for this practice includes the following:

7.1.1 *DSC Test Chamber,* composed of the following:

7.1.1.1 *Furnace(s),* to provide uniform controlled heating of a specimen and reference to a constant temperature at a constant rate within the applicable temperature range of this practice.

7.1.1.2 *Temperature Sensor,* to provide an indication of the specimen/furnace temperature to ±0.01 K.

7.1.1.3 *Differential Sensor,* to detect heat flow difference between the specimen and reference equivalent to 1 µW.

7.1.1.4 A means of sustaining a test chamber environment of purge gas at a rate of 10 to 50 ± mL/min.

NOTE 2—Typically, 99.9+ % pure nitrogen, helium, or argon is employed. Use of dry purge gas is recommended and is essential for operation at subambient temperatures.

7.1.2 *Temperature Controller,* capable of executing a specific temperature program by operating the furnace(s) between selected temperature limits, that is, 170 to 870 K, at a rate of temperature change of up to 10 K/min constant to ±0.1 K/min.

7.1.3 *Recording Device,* capable of recording and displaying any fraction of the heat flow signal (DSC curve), including the signal noise, on the Y-axis versus temperature on the X-axis.

7.2 *Containers (pans, crucibles, vials, etc.),* that are inert to the specimen and reference materials, and which are of suitable structural shape and integrity to contain the specimen and reference in accordance with the specific requirements of this practice.

7.3 While not required, the user will find useful calculator or computer and data analysis software to perform the necessary least squares best fit or multiple linear regression data treatments required by this practice.

7.4 *Balance,* to weigh specimens, or containers, or both, to ±10 µg with a capacity of at least 100 mg.

8. Hazards

8.1 This practice uses equipment that alters a material's state that may create noxious gases that may be harmful. Care shall be taken to provide adequate ventilation for all equipment capable of producing this effect.

9. Sampling, Test Specimens, and Test Units

9.1 Material tested shall be as commercially supplied by the manufacturer.

9.2 Materials such as, but not limited to, sealants, putties, coatings, sprays, mortars and foams, which are normally shipped and dispensed at the time and place of final use from an air-tight or near air-tight container, shall be cast, formed, sprayed or otherwise applied as they normally would to create a sample of thickness which is considered by the test sponsor and laboratory to represent a typical field installation. The sample shall be allowed to cure or dry before testing. Curing or drying time shall be in accordance with manufacturer's published instructions for the product.

9.3 Inhomogeneous materials.

9.3.1 Due to the possibility that a milligram-sized sample might not include one or more constituents of an inhomogeneous material, multiple samples shall be taken and tested so as to ensure that the kinetic data (Arrhennius coefficients) of all constituents of the material have been measured.

NOTE 3—It is not intended that samples should be prepared and tested that would test each individual component as a pure material. The intent is that sufficient samples should be tested that each component has appeared in at least one test.