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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.

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.

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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.

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.

9.4 The samples to be used for DSC testing shall be excised from the material prepared as specified in 9.2.

## 10. Procedure

10.1 DSC testing shall be conducted on three samples prepared as specified in Section 9. The two tests and subsequent data analysis shall be as described in Test Method E2041, with exceptions as described in 10.1.1 and 10.1.2.

10.1.1 In one test, the sample shall be in an open container that is exposed to a pure dry air atmosphere.

10.1.2 In one test, the sample shall be in an open container that is exposed to an airflow that is saturated with water.

NOTE 4—Test Method E2041 specifies that a sample to be tested by DSC is to be contained within a hermetically sealed container. The two independent tests specified here place the sample in an open container, each of which is exposed to a different atmospheric condition. These two conditions represent extremes of environmental conditions that a firestop product might be exposed to during its design life: very dry air, and very humid air.

## 11. Calculation or Interpretation of Results

11.1 For each DSC test conducted, the conversion rate of the material shall be calculated for the following two time and temperature combinations:

11.1.1 Two hundred and seventy (270) days at 70°C.

11.1.2 Thirty (30) years (10 950 days) at 50°C.

11.2 The calculation of the conversion rate shall be as detailed below:

11.2.1 Calculate the rate of reaction using the calculations presented in the Calculation Section of Test Method E2041.

11.3 For inhomogeneous materials, the conversion rate calculations shall be performed using each of the DSC test results. The calculation that yields the highest conversion rate shall then be used in the evaluation of longevity.

11.4 Where the calculated conversion rate for each of the three tests (using the two environmental conditions 10.1.1 and 10.1.2) is at or below 5 %, the test can be reasonably assumed to indicate that the firestop material would provide acceptable performance over the course of a design life of 30 years, if environmental temperatures do not exceed 50°C for any extended period of time or for any extended recurrence of short intervals.

NOTE 5—Where the calculated conversion rate has produced a value in excess of 5 %, it cannot be concluded or assumed that the firestop material would have unacceptable performance over the course of a 30-year design life. Since this practice looks at all chemical and physical reactions, including those that might have no degradation of the firestopping abilities of the material, the finding of a conversion rate exceeding 5 % should not by itself be assumed to be an indicator of decreased firestopping ability during the service life. When a conversion rate in excess of 5 % is calculated, the user is encouraged to seek alternate longevity evaluation methods. Such methods include, but are not limited to, accelerated aging of samples using elevated temperature or elevated humidity, or both, or other longterm methods involving actual exposures in real time.

11.5 Where an assessment of longevity is desired for unusual conditions where the temperature is expected to exceed 50°C for an extended period of time or for short periods of time on a recurring basis, the 30-year conversion rates should be calculated using the highest temperature that is anticipated to expose the material on a regular basis.

11.6 For design situations where the material is expected to be exposed to temperatures significantly exceeding 50°C on a regular basis, but not continuously, the conversion calculation can be done in a multi-step fashion. This is done by calculating the conversion for each anticipated temperature over a fraction of the 30 years, and then adding the partial conversions together to obtain a total conversion over the full 30-year period. The 5 % conversion rate criterion can then be applied to assess expected longevity under the specified temperature conditions.

11.7 A situation might arise in which the test sponsor, the testing laboratory, or the agency asking for the testing has some doubt as to whether some important component (for example, an "active" ingredient within a carrier base) of a composite material has a conversion rate that is disproportionately high as compared to the rest of the material. In such a case, the testing could optionally be additionally performed on a sample consisting of only the single component that is speculated or known to be of most importance to the fire performance of the overall material. Unless a different value can be technically justified, the 5 % conversion rate acceptance threshold should be used for this one component alone to differentiate acceptable from unacceptable longevity.

NOTE 6—The fact that an important (for example, "active") component is contained as a small percentage of a larger amount of a carrier or binder material can often make the important component less subject to change over time than the bulk of the material would be. The carrier or binder will normally protect the important component from reacting with the air or moisture in the surrounding environment. Air or water vapor has to diffuse through the carrier or binder to reach the embedded component. In a different situation, if the reaction leading to change over time was loss of moisture from the important component, then the water would have to diffuse through the binder or carrier, thus also slowing down the degradation process. Since most chemical and physical reactions can be expected to occur at the surface of a material, rather than in depth, situations where a test is requested on only the important (or "active" component), rather than on the mixed/composite material, should be accordingly rare.

## 12. Report

12.1 The report shall include:

12.1.1 All information required by the Report section of Test Method E2041.

12.1.2 Time and conditions of curing or drying involved in sample preparation (if applicable).

12.1.3 Calculations showing conversion rate for 270 days at 70°C.

12.1.4 Calculations showing conversion rate for 30 years (10 950 days) at 50°C.

12.2 Calculations showing conversion rate for 30 years at temperature(s) other than 50°C, if desired by the test sponsor.

## 13. Precision and Bias

13.1 The precision and bias of this practice can be expected to be within the precision and bias boundaries specified in Test Method E2041 for any DSC testing.

13.2 It is not feasible to specify the precision of the DSC procedure for firestop materials specifically, as there is no test experience with this specific class of materials.

13.3 No information can be presented on the bias of the DSC procedure for firestop materials specifically because no material having an accepted reference value is available.

## 14. Keywords

14.1 Borchardt and Daniels kinetics; differential scanning calorimetry (DSC); fire stop; firestop; longevity

# APPENDIX

**(Nonmandatory Information)**

## X1. DSC METHOD DISCUSSION FOR EXPOSURE TEMPERATURE AND DURABILITY

X1.1 The DSC method is the best choice, if no reaction occurs in a DSC, let's say below 150°C. Here we can see the positive (and only) contribution in shortening the aging test. That means we have no indication of a thermal effect at temperatures under normal use of the products (with the restriction that a (quite unprobable) exact compensation of an exothermal by another endothermic reaction of exactly the same energy cannot be detected, as well as thermo-neutral reactions, for example, degradation by UV). We must mention and discuss the evaluation step of baseline definition, which can be a real problem if the situation is indifferent. Slightly different baselines will have a large effect on the result (kinetic parameters) as seen in the former done and evaluated experiments. I see the DSC at the moment as an "exclusion" method: that means, if nothing is detected (below 150°C), no further activities are necessary and the material is deemed to be durable "nothing" means below a certain threshold, because "zero" doesn't exist in any experiment.

X1.2 The 270 days at 70°C don't give any valuable durability – only over 70 days which is nothing. So better skip it completely. On the other hand, probably not always 30 years are necessary. As every time/temperature relation can be calculated: is it wise and necessary to prescribe a certain set of conditions or simply leave it to the needs of the applicant? What, in this case, has to be given is the set of parameters: time and temperature; it could also be a combination of several time/temperature sets, for example, a regular temperature increase for a limited time; the following chapters have to be amended then accordingly.

X1.3 The challenge of determining the long term performance of firestop materials has been an ongoing discussion, practically since the initial stages of modern firestopping with intumesent materials. The subject matter today is no less challenging, and efforts have been made by many in this industry to define the expected duration of firestop performance. In the preparation of this test method, the goal was to identify an estimation tool for long term performance of any given firestop material, and to minimize the required timeframe in order to ensure development of products with improved long term performance.

X1.3.1 Much research has been performed that provides insight into the advantages of using this method, and other methods, that at least provide some assurance that there is merit in these thoughts and actions. To that end, the purpose of this section is to provide the reader a reference to some of the research papers that are available for additional review.

X1.3.2 One research document that was referenced during the development of this standard is titled *Use of Differential Scanning Calorimetry Testing to Predict the Aging Behavior of Firestop Materials* and was authored by Udo Woersdoerfer, Ph.D., and John Valiulis, P.E. This document can be located on the International Firestop Council website (http://www.firestop.org) under the "Technical Library" header, "Papers" subheader.

X1.3.3 Another document that discusses alternative methods for longevity determination of firestop materials is titled *Aging & Environmental Exposure Properties of a Fire Protection Material* and was authored by Richard Ralph Licht. This document was published in *Journal of ASTM International*, September 2005, Vol. 2, No. 8, Paper ID JAI13028 and is available online at http://www.astm.org.

X1.3.4 As always, many sources are available in print and online to research chemical kinetics, the Arrhenius Equation, and differential scanning calorimetry.