Designation: ~~E1877 – 11~~ E1877 – 13

Standard Practice for Calculating Thermal Endurance of Materials from Thermogravimetric Decomposition Data[1]

This standard is issued under the fixed designation E1877; 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 additional treatment of the Arrhenius activation energy data determined by Test Method~~describes the determination of thermal endurance, thermal index, and relative thermal index for ~~E1641 to develop a thermal endurance curve and derive a relative thermal index for materials.~~organic materials using the Arrhenius activation energy generated by thermogravimetry.

1.2 This practice is generally applicable to materials with a well-defined thermal decomposition profile, namely a smooth, continuous mass ~~change with a single maximum rate.~~change.

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

1.4 There is no ISO standard equivalent to this practice.

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:*[2]

E1641 Test Method for Decomposition Kinetics by Thermogravimetry Using the Ozawa/Flynn/Wall Method

E2550 Test Method for Thermal Stability by Thermogravimetry

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *failure, n*—change in some chemical, physical, mechanical, electrical or other property of sufficient magnitude to make it unsuitable for a particular use.

3.1.2 *failure temperature (T_f), n*—the temperature at which a material fails after a selected time.

3.1.3 *thermal index (TI), n*—the temperature corresponding to a selected time-to-failure.

3.1.4 *relative thermal index (RTI), n*—~~a measure of the thermal endurance of a material~~ the temperature corresponding to a selected time-to-failure when compared with that of a control with proven thermal endurance characteristics.

[1] This practice is under the jurisdiction of Committee E37 on Thermal Measurements and is the direct responsibility of Subcommittee E37.10 on Fundamental, Statistical and Mechanical Properties.

Current edition approved ~~Aug. 1, 2011~~Oct. 15, 2013. Published ~~September 2011~~December 2013. Originally approved in 1997. Last previous edition approved in ~~2010~~2011 as ~~E1877 – 00 (2010).~~E1877 – 11. DOI: ~~10.1520/E1877-11.~~10.1520/E1877-13.

[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.

3.1.4.1 *Discussion*—

The ~~*RTI*~~*TI* ~~is~~and ~~*also*~~*RTI* are considered to be the maximum temperature below which the material resists changes in its properties over a ~~defined~~selected period of time. In the absence of comparison data for a control material, a ~~time-to-failure~~ thermal endurance (time-to-failure) of 60 000 h has been arbitrarily selected for measuring ~~RTI. The RTI is therefore, the failure temperature, *TTI$_f$*, obtained~~and ~~*from*~~ *RTI.*~~the thermal endurance curve.~~

3.1.5 *thermal endurance, n*—the time-to-failure corresponding to a selected temperature. Also known as thermal lifetime.

4. Summary of Practice

4.1 The Arrhenius activation energy obtained from ~~Test~~ other Test Methods (such as Test Method E1641, Refs (**1, 2**),[3] etc.) is used to construct the thermal endurance curve of ~~a~~ an organic material from which an estimate of lifetime at ~~certain~~selected temperatures may be obtained.

5. Significance and Use

5.1 Thermogravimetry provides a rapid method for the determination of the temperature-decomposition profile of a material.

5.2 This practice is useful for quality control, specification acceptance, and research.

5.3 This test method is intended to provide an accelerated thermal endurance estimation in a fraction of the time require for oven-aging tests. The primary product of this test method is the thermal index (temperature) for a selected estimated thermal endurance (time) as derived from material decomposition.

5.4 Alternatively, the estimated thermal endurance (time) of a material may be estimated from a selected thermal index (temperature).

5.5 Additionally, the estimated thermal endurance of a material at selected failure time and temperature may be estimated when compared to a reference value for thermal endurance and thermal index obtained from electrical or mechanical oven aging tests.

5.6 This practice shall not be used for product lifetime predications unless a correlation between test results and actual lifetime has been demonstrated. In many cases, multiple mechanisms occur during the decomposition of a material, with one mechanism dominating over one temperature range, and a different mechanism dominating in a different temperature range. Users of this practice are cautioned to demonstrate for their system that any temperature extrapolations are technically sound.

6. Calculation

6.1 The following values ~~obtained by Test Method E1641~~are used to calculate thermal endurance, estimated thermal life and failure temperature.

6.1.1 The following definitions apply to ~~6.1~~6.1 – 6.4 ~~and 6.3~~:

6.1.1.1 E = Arrhenius activation energy (J/mol),

NOTE 1—E may be obtained from another methods (such as Test Method E1641, Ref (**1, 2**), etc.)

6.1.1.2 R = Universal gas constant ~~(= 8.314 510~~ (= 8.31451 J/(mol K)),

6.1.1.3 β = Heating rate (K/min),

NOTE 2—β may obtained from Test Method E2550 and is typically 5 K/min.

6.1.1.4 ~~β'~~TI = ~~Heating rate nearest the mid-point of the experimental heating rates (K/min),~~thermal index (K),

6.1.1.5 a = ~~Approximation~~ Doyle approximation integral ~~taken~~(taken from Table 1~~,~~),

6.1.1.6 α = Constant conversion ~~value,~~failure criterion,

6.1.1.7 t_f = Estimated thermal ~~life~~ endurance (thermal life) for a ~~given value of~~ constant conversion (α) ~~(min) (see Test Method~~taken as the failure ~~E1641),~~criterion (min),

6.1.1.8 T_c = Temperature for the point of constant conversion for β ~~(K),~~ (K) obtained from Test Method E2550, and

6.1.1.9 RTI = Relative Thermal Index (K).

6.1.1.10 ~~T_f~~σE_f ~~= Failure Temperature for a give value of conversion (α) (K) (see~~ standard deviation in activation energy (J/mol) obtained from Test Method E1641~~).~~, Ref (**1, 2**), etc.

NOTE 3—The precision of the calculation in this practice are exponentially dependent on the uncertainty of activation energy value used. Care should be taken to use only the most precise values of E.

6.1.1.11 TI = Thermal index (K),

6.1.1.12 σTI = Standard deviation of the thermal index (K),

6.1.1.13 σRTI = Standard deviation of the relative thermal index (K),

6.1.1.14 σt_f = Standard deviation of the thermal endurance (min),

6.1.1.15 t_r = Reference value for thermal endurance (min), and

6.1.1.16 T_r = Reference value for thermal index (K).

6.2 *Method 1 – Thermal Index:*

6.2.1 Using the activation energy (E) and failure temperature (T_c), determine the value for E/RT_c.

6.2.2 Using the value of E/RT_c, determine the value for the Doyle approximation intergral (a) by interpolation in Table 1.

6.2.3 Select the thermal endurance (t_f) and calculate its logarithm.

[3] ~~Krizanovsky, L., and Mentlik, V., *Journal of Thermal Analysis*, Vol 13, 1978.~~ The boldface numbers in parentheses refer to a list of references at the end of this standard.
~~[4] Flynn, J.H., and Wall, L.A., *Polymer Letters*, Vol 4, 1966 pp. 323–328.~~
~~[5] Flynn, J.H., *Journal of Thermal Analysis*, Vol 27, 1983, pp. 95–102.~~

TABLE 1 Numerical Integration Constants (3, 4)

E/RT	*a*
8	5.3699
9	5.8980
10	6.4157
11	6.9276
12	7.4327
13	7.9323
14	8.4273
15	8.9182
16	9.4056
17	9.8900
18	10.3716
19	10.8507
20	11.3277
21	11.8026
22	12.2757
23	12.7471
24	13.2170
25	13.6855
26	14.1527
27	14.6187
28	15.0836
29	15.5474
30	16.0103
31	16.4722
32	16.9333
33	17.3936
34	17.8532
35	18.3120
36	18.7701
37	19.2276
38	19.6845
39	20.1408
40	20.5966
41	21.0519
42	21.5066
43	21.9609
44	22.4148
45	22.8682
46	23.3212
47	23.7738
48	24.2260
49	24.6779
50	25.1294
51	25.5806
52	26.0314
53	26.4820
54	26.9323
55	27.3823
56	27.8319
57	28.2814
58	28.7305
59	29.1794
60	29.6281

6.2.4 Substitute the values for E,R, log(t_f), log(E/RT_c)) and a into Eq 1 to obtain the thermal index (*TI*) (**5**).

$$TI = E/(2.303\ R\ [\log(t_f) - \log\{E/R\beta\} + a]) \tag{1}$$

6.2.5 Determine the relative standard deviation (σ*TI*/*TI*) using Eq 2.

$$\sigma TI/TI \approx 1.2\sigma E/E \tag{2}$$

6.2.6 Report the thermal index (*TI*) and its relative standard deviation (σ*TI*/*TI*) along with the thermal endurance (t_f).

6.3 Use Eq 1 or Eq 2[3] and trial values of T_f to plot the logarithm of estimated thermal life (t_f) versus reciprocal of T_f as, by example, shown in Fig. 1.

$$\log t_f = E/(2.303\ R\ T_f) + \log[E/(R\beta)] - a \tag{1}$$

$$T_f = E/(2.303\ R\ [\log t_f - \log[E/(R\beta)] + a]) \tag{2}$$

Method B – Thermal Endurance Curve:

6.3.1 To calculate t_f, select the value for the temperature at the constant conversion point (T_c) for a heating rate (β) nearest the mid-point of the experimental heating rates. Use this value, along with the Arrhenius activation energy (E) to calculate the quantity

E/(R T_c) to select the value in Table 1.[4-6] Arbitrarily select a number of two or three temperatures in the region of the chosen percent mass loss, indicative of failure, in the mass change curve at the midpoint heating rate. Calculate the interest and calculate the corresponding logarithm of the thermal life from endurance ($\log[t_f]$) Eq 1. Plot the thermal endurance values at each temperature using Eq 3 curve, as shown in .Fig. 1, with thermal life on the ordinate and reciprocal of absolute temperature on the abscissa.

NOTE 2—The values for *E* and β may be obtained by the procedure described in Test Method E1641.

$$\log[t_f] = E/(2.303\ R\ T) = \log[E/(R\ \beta)] - a] \quad (3)$$

6.3.2 Prepare a display of logarithm of thermal endurance on the ordinate versus the reciprocal of absolute temperature on the abscissa (see Fig. 1).

6.3.3 Alternative thermal indexes (*TI*) and associated logarithm of thermal endurance ($\log[t_f]$ may be estimated from this display.

6.3.4 The standard deviation in the time-to-failure (t_f) may be estimated using Eq 4.

$$\sigma t_f/t_f = (1 - 0.052\ E/R\ T) \times (\sigma\ E/E) \quad (4)$$

6.4 The thermal endurance of two or more materials may be compared by calculating the RTI for each material. To compute RTI for each material; select some common thermal life for comparison, a typical value may be 60 000 h (6.8 years), insert that value (in minutes) and the appropriate activation energy for each material into Eq 2 to obtain T_f. This value of temperature is called the "relative thermal index (RTI) at the specified time". Materials with greater resistance to thermal decomposition will have a larger RTI.*Method C – Relative Thermal Index:*

6.4.1 Relative Thermal Index may be determined from the activation energy determined by thermogravimetry and the thermal index obtained by some other method (such as electrical or mechanical tests) using Eq 5.

$$RTI = E/R[\ln[t_f] - \ln[t_r] + E/(R\ T_r)] \quad (5)$$

6.4.2 The relative standard deviation of the relative thermal index (σ*RTI*/*RTI*) is estimate from Eq 6 where the reference values of thermal endurance (t_r) and corresponding reference temperature (*T*) are considered to be exact.

$$\sigma RTI/RTI = 1.4 \sigma E/E \quad (6)$$

7. Report

7.1 Report the following information:

7.1.1 If data other than that generated by Test Method The value, standard deviation (or relative standard deviation), and E1641 is used in these calculations, then include a description of the data source in the report, source for each value used in the determination;

7.1.2 Designation of the material under test, including the name of the manufacturer, the lot number, and supposed chemical composition when known, known; and

7.1.3 The calculated thermal life index (t*TI*$_f$) and its relative standard deviation (σ*TI*/*TI*) or relative thermal index (*RTI* values.) and its relative standard deviation (σ*RTI*/*RTI*) along with the identified thermal endurance.

7.1.3.1 *Example—TI* (60 000 hr) = 453 ± 6 K (180 ± 6°C)

7.1.4 The specific dated version of this practice that is used.

8. Precision and Bias[4]

8.1 The precision and bias of these calculations depend on the precision and bias of the kinetic data used in them. To provide an example of the precision expected, thermal life index was calculated by the procedure in this practice using data for poly(tetrafluoroethylene) from the interlaboratory study conducted to develop the precision and bias statement for Test Method E1641. Extreme values of thermal life were calculated using an arbitrarily chosen value for temperature of 600 K and the extreme values of *E* corresponding to the 95 % confidence level from that interlaboratory study. The resulting calculated extreme values were 9 years and 3700 years for this material.

9. Keywords

9.1 Arrhenius activation energy; Arrhenius pre-exponential factor; kinetic parameters; relative thermal index; thermal decomposition; thermal endurance; thermal life; thermogravimetric analysis

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E37-1024. Contact ASTM Customer Service at service@astm.org.