Designation: E1877 – 15

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 describes the determination of thermal endurance, thermal index, and relative thermal index for 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.

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

E2958 Test Methods for Kinetic Parameters by Factor Jump/Modulated 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*—the temperature corresponding to a selected time-to-failure when compared with that of a control with proven thermal endurance characteristics.

3.1.4.1 *Discussion*—The *TI* and *RTI* are considered to be the maximum temperature below which the material resists changes in its properties over a selected period of time. In the absence of comparison data for a control material, a thermal endurance (time-to-failure) of 60 000 h has been arbitrarily selected for measuring *TI* and *RTI*.

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

4. Summary of Practice

4.1 The Arrhenius activation energy obtained from other Test Methods (such as Test Methods E1641 and E2958, etc.) is used to construct the thermal endurance curve of an organic material from which an estimate of lifetime at 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

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[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.

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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.

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 are used to calculate thermal endurance, estimated thermal life and failure temperature.

6.1.1 The following definitions apply to 6.1 – 6.4:

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

NOTE 1—E may be obtained from another methods (such as Test Methods E1641 and E2958, etc.).

6.1.1.2 R = universal gas constant (= 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 TI = thermal index (K),

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

6.1.1.6 α = constant conversion failure criterion,

6.1.1.7 t_f = estimated thermal endurance (thermal life) for a constant conversion (α) taken as the failure criterion (min),

6.1.1.8 T_c = failure temperature taken as temperature for the point of constant conversion for β (K) obtained from Test Method E2550,

6.1.1.9 RTI = Relative Thermal Index (K),

6.1.1.10 σ = standard deviation in activation energy (J/mol) obtained from Test Methods E1641 and E2958, 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.

TABLE 1 Numerical Integration Constants (1, 2)[3]

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) **(3)**.[3]

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

6.2.5 Determine the relative standard deviation ($\sigma TI/TI$) using Eq 2.

$$\sigma TI/TI \approx 1.2\sigma E/E \quad (2)$$

6.2.6 Report the thermal index (TI) and its relative standard deviation ($\sigma TI/TI$) along with the thermal endurance (t_f).

6.3 *Method B – Thermal Endurance Curve:*

[3] The boldface numbers in parentheses refer to a list of references at the end of this standard.

6.3.1 Arbitrarily select two or three temperatures in the region of interest and calculate the corresponding logarithm of the thermal endurance ($\log[t_f]$) values at each temperature using Eq 3.

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

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 thermal endurance (t_f) may be estimated using Eq 4.

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

6.4 *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.

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

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

$$\sigma RTI/RTI = 1.4\sigma E/E \tag{6}$$

7. Report

7.1 Report the following information:

7.1.1 The value, standard deviation (or relative standard deviation), and 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; and

7.1.3 The calculated thermal index (*TI*) and its relative standard deviation ($\sigma TI/TI$) or relative thermal index (*RTI*) and its relative standard deviation ($\sigma RTI/RTI$) 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 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.

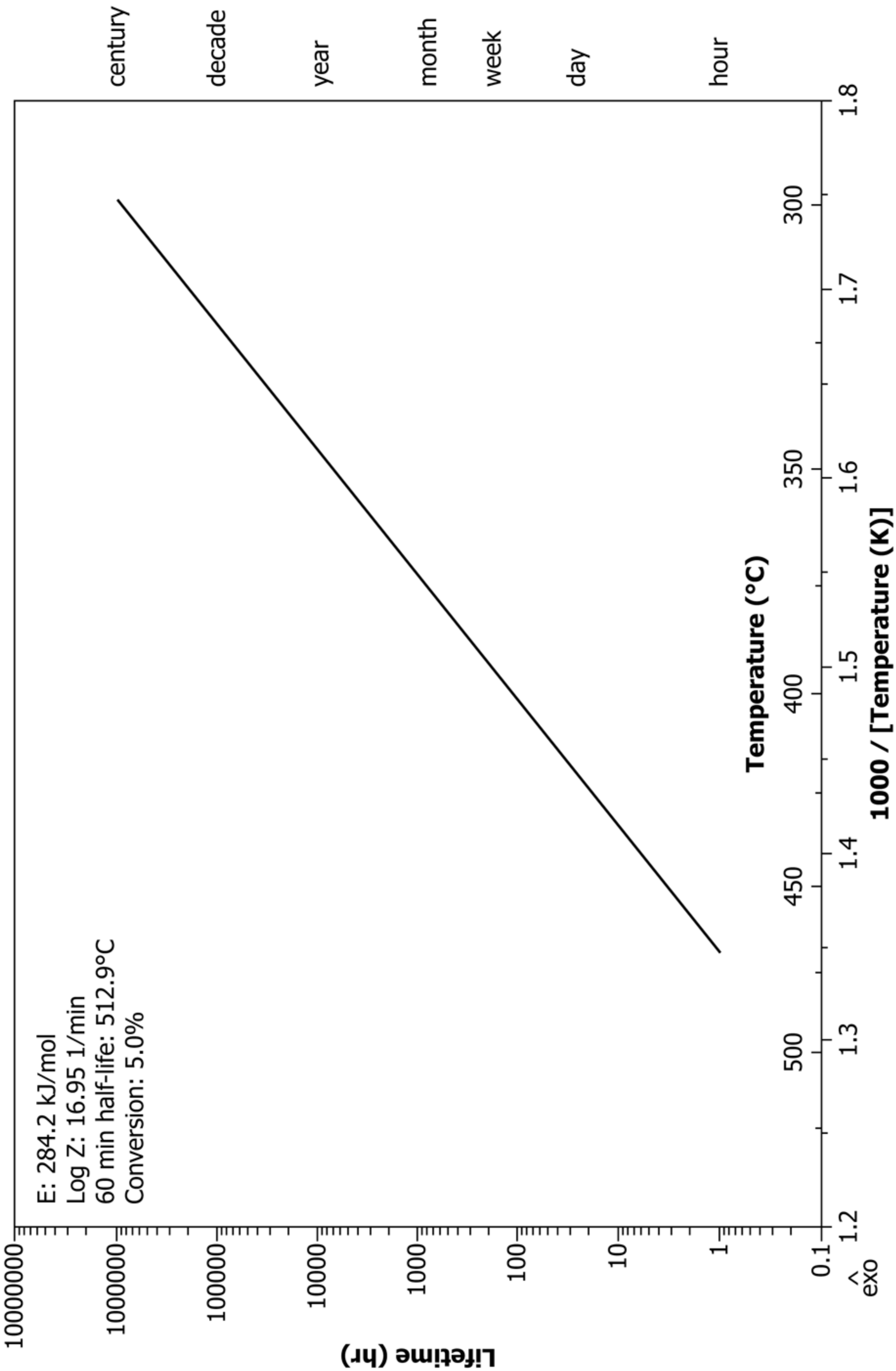

FIG. 1 Thermal Endurance Curve

APPENDIX

(Nonmandatory Information)

X1. EXAMPLE CALCULATIONS

X1.1 Example Calculations for the Values Determined in This Standard

X1.1.1 Example data obtained from Test Method E1641 includes:

X1.1.1.1 E = 320 kJ/mol = 320 000 J/mol

X1.1.1.2 σE = 24 kJ/mol = 24 000 J/mol

X1.1.1.3 R = 8.31451 J/(mol K)

X1.1.1.4 β = 5.0 K/min

X1.1.2 Example data obtained from Test Method E2550 includes:

X1.1.2.1 T_c = 783 K

X1.1.2.2 σT_c = 6 K

X1.1.3 Arbitrarily selected:

X1.1.3.1 t_f = 60 000 hr = 3 600 000 min = 6.8 yr

X1.1.3.2 T_r = 683 K

X1.1.3.3 t_r = 100 000 hr = 6 000 000 min = 11 yr

X1.2 Example Calculations for Thermal Index (*TI*)

X1.2.1 Determine the value for *E*/*RT* from values in X1.1.1.1, X1.1.1.3, and X1.2.1:

$$E/RT = (320\ 000\ \text{J/mol})/[8.31451\ \text{J/(mol K)} \times 783\ \text{K}]$$
$$= 49.1532$$

X1.2.2 Using the value of *E*/*RT* from X1.2.1, determine the value for a by interpolation in Table 1:

$$a = 24.7471$$

X1.2.3 Substitute values from X1.1.1.1, X1.1.1.3, X1.1.1.4, X1.1.3.1, and X1.2.2 into Eq 1:

$$\begin{aligned}
TI &= E/(2.303\ R\ \{[\log[t_f] - \log[E/(R\ \beta)]] + a\}) \\
&= \{320\ 000\ \text{J/mol}/(2.303 \times 8.314\ \text{J/(mol K)})\} \\
&\quad /\{\log[3.6 \times 10^6\ \text{min}] \\
&\quad - \log[320\ 000\ \text{J/mol}/(8.31451\ \text{J/(mol K)}) \times 5\ \text{K/min}] \\
&\quad - 24.7471 \\
&= \{16\ 712\ \text{K}\}/\{6.5563 - \log[7697.39/\text{min}] - 24.7471\} \\
&= 16\ 712\ \text{K}/\{6.5563 - 3.8863 - 24.7471\} \\
&= 16\ 712\ \text{K}/27.4171 \\
TI &= 609.5\ \text{K} = 336.3\ °\text{C}
\end{aligned}$$

X1.3 Example Calculation for the Imprecision in Thermal Index

X1.3.1 Substituting values from X1.1.1.2 and X1.1.1.3 into Eq 2:

$$\begin{aligned}
\sigma TI &= 1.2\ \sigma E/E \\
&= 1.2 \times 24\ 000\ \text{J/mol}/320\ 000\ \text{J/mol} \\
&= 0.090
\end{aligned}$$

X1.4 Example Calculation for Thermal Endurance

X1.4.1 Substituting the values from X1.1.1.1, X1.1.1.3, X1.1.1.4, X1.1.3.2, and X1.2.2 into Eq 3:

$$\begin{aligned}
\log[t_f] &= E/[2.303\ R\ T] + \log[E/R\ \beta] - a \\
&= 320\ 000\ \text{J/mol}/(2.303 \times 683\ \text{K}) \\
&\quad + \log[320\ 000\ \text{J/mol}/8.31451\ \text{J/(mol K)}] \\
&\quad - 24.7471 \times 5\ \text{K/min} \\
&= 24.4680 + \log[7697.39] - 24.7471 \\
&= 24.4680 + 3.8863 - 24.7471 \\
\log[t_f] &= 3.6072 \\
t_f &= 4048\ \text{min} \times (\text{hr}/60\ \text{min}) = 67.46\ \text{hr}
\end{aligned}$$

X1.5 Example Calculation of the Imprecision in Thermal Endurance (t_f)

X1.5.1 Substituting value from X1.1.1.1, X1.1.1.2, X1.1.1.3, X1.1.3.2, and X1.2.2 into Eq 4:

$$\begin{aligned}
\sigma t_f/t_f &= (1 + 0.052\ E/R\ T) \times \sigma E/E \\
&= [(1 + 0.052 \times 320\ 000\ \text{J/mol}) \\
&\quad /(8.31451\ \text{J/mol K} \times 683\ \text{K}) \\
&\quad \times 24\ 000\ \text{J/mol}/320\ 000\ \text{J/mol} \\
&= (1 + 2.930) \times 0.075 \\
&= 3.930 \times 0.075 \\
&= 0.29
\end{aligned}$$

X1.6 Example Calculation of Relative Thermal Index

X1.6.1 Substituting values from X1.1.1.1, X1.1.1.3, X1.1.3.1, X1.1.3.2, and X1.1.3.3 into Eq 5:

$$\begin{aligned}
RTI &= E/R\{[\ln[t_f] - \ln[t_r] + E/RT_r]\} \\
&= 320\ 00\ \text{J/mol}/8.31451\text{J/molK}\{\ln[3\ 600\ 000\ \text{min}] \\
&\quad - \ln[6\ 000\ 000\ \text{min}] \\
&\quad + 320\ 000\ \text{J/mol K} \\
&\quad /(8.31451\ \text{J/mol K} \times 683\ \text{K}) \\
&= 38\ 487\ \text{K}/(15.0964 - 15.6073 + 56.3706) \\
&= 38\ 487\ \text{K}/55.8597 \\
&= 689\ \text{K}
\end{aligned}$$

X1.7 Example Calculation of the Standard Deviation of Relative Thermal Index

X1.7.1 Substituting values from X1.1.1.1 and X1.1.1.2 into Eq 6:

$$\begin{aligned}
\sigma RTI/RTI &= 1.4 \times 24\ 000\ \text{J/mol}/320\ 000\ \text{J/mol} \\
&= 0.105
\end{aligned}$$

REFERENCES

(1) Toop, D. J., "Theory of Life Testing and Use of Thermogravimetric Analysis to Predict the Thermal Life of Wire Enamels," *IEEE Transactions on Electrical Insulation*, Vol EI-6, No. 1, 1971, pp. 2–14.

(2) Flynn, J. H., "The Isoconversional Method for Determination of Energy of Activation at Constant Rates – Corrections for the Doyle Approximation," *Journal of Thermal Analysis*, Vol 27, 1983, pp. 95–102.

(3) Krizanovsky, L., and Mentlik, V., "The Use of Thermal Analysis to Predict the Thermal Life of Organic Electrical Insulating Materials," *Journal of Thermal Analysis*, Vol 13, 1978, pp. 571–580.