INTERNATIONAL

Designation: ~~E1877 – 17~~ E1877 – 21

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 for thermal decomposition measured by thermogravimetry.

1.2 This practice is generally applicable to materials with a well-defined thermal decomposition ~~profile,~~ profile upon heating, 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.~~

1.4 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3045 Practice for Heat Aging of Plastics Without Load
E1641 Test Method for Decomposition Kinetics by Thermogravimetry Using the Ozawa/Flynn/Wall Method
E2550 Test Method for Thermal Stability by Thermogravimetry

[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.

***A Summary of Changes section appears at the end of this standard**

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~~(*Tf*),~~f)~~, *n*—the absolute temperature at which a material fails after a selected time.

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

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~~ specific time period. ~~TI and RTI.~~

3.1.4.2 *Discussion—*
In the absence of other comparison data for a control material, a thermal endurance (time-to-failure) of 20 000 h is arbitrarily selected for measuring *TI* and *RTI*.

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

3.1.5.1 *Discussion—*
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~~ practice 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~~ practice 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 are used to calculate thermal endurance, ~~estimated~~ thermal ~~life and failure temperature.~~index, and relative thermal index.

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~~other methods (such as Test Methods E1641 and E2958~~, etc.).~~).

6.1.1.2 *R* = universal gas constant ~~(= 8.31451~~(= 8.314 J/(mol K)),

6.1.1.3 β = heating rate (K/min),

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

6.1.1.4 *TI* = thermal index (K),

6.1.1.5 ~~t_f~~ tf = ~~estimated~~ thermal endurance (thermal life) ~~for a constant~~with an identified conversion (α) taken as the failure criterion ~~(min),~~(h),

6.1.1.6 ~~T_f~~ Tf_c = failure absolute temperature taken as temperature for the point of constant conversion for β ~~(K)~~(K/min) obtained from Test Methods E2550 or E2958~~,~~ (K),

6.1.1.7 *RTI* = ~~Relative Thermal Index~~relative thermal index (K),

6.1.1.8 σ*E* = 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~~is exponentially dependent on the uncertainty of activation energy value used. Care should be taken to use only the most precise values of *E*.

6.1.1.9 σlog[~~*TI*~~*tf* ~~= thermal index (K),~~] = standard deviation of the logarithm of thermal endurance with *tf* in h,

6.1.1.10 σ*TI* = standard deviation of the thermal index (K),

6.1.1.11 σ*RTI* = standard deviation of the relative thermal index (K),

6.1.1.12 σ~~t_f~~tf = standard deviation of the thermal endurance ~~(min),~~(h),

6.1.1.13 ~~t_r~~ tr = reference time value for thermal endurance ~~(min),~~(h), and

6.1.1.14 ~~T_r~~ Tr = reference value for thermal index (K).

6.2 *Method ~~1~~A – Thermal Index:*

6.2.1 ~~Using the~~ Obtain activation energy (*E*) ~~and~~ determined previously (for example, see Test Method E1641). Obtain the failure temperature (~~T_c~~ Tf_c~~), determine~~) from a thermal curve determined at heating rate (β = 300 K/h = 5 K/min) (see Test Method E1641~~the value for~~). Select the thermal endurance (~~E~~ tf~~/~~)~~RT~~ (typically ~~$_c$~~ 20 000 h).

~~6.2.2 Using the value of E/RT_c, determine the value for *TI* using Eq 1.~~

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

6.2.2 Substitute the values for E,R,t_f,tf, and β into Eq 1 to obtain the thermal index (TI) (**1, 2**).[3]

$$TI = \{E/(2.303\ R)\}/\{\log[100.4\ tf\ \beta\ R/E]+0.463\ E/R\ Tf\} \quad (1)$$

$$TI = \{E/(2.303\ R)\}/\{\log[100.4\ t_f\ \beta\ R/E]+0.463\ E/R\ T_c\} \quad (1)$$

NOTE 4—See derivation in Appendix X3.

6.2.3 Determine the thermal index relative standard deviation (σTI/TI) using Eq 2.

$$\sigma TI/TI = \sigma E/E \quad (2)$$

$$\sigma TI/TI \approx \pm 0.19\ \sigma E/E \quad (2)$$

6.2.4 Report the thermal index (TI) and its relative standard deviation (σTI/TI) along with the and thermal endurance ($t_f$$tf$).

6.3 *Method B – Thermal Endurance Curve:*

6.3.1 Arbitrarily select two or three temperatures (Tr) in the region of interest and calculate the corresponding logarithm of the thermal endurance (log[$t_f$$tf$]) values in hours at each temperature using Eq 3. with β expressed in units of K/h.

$$\log[tf\ ,\mathrm{h}] = (E/2.303\ R\ Tr) + \log[E/100.4\ R\ \beta] - 0.463\ E/RTf \quad (3)$$

$$\log[t_f] = (E/2.303\ R\ T_c) + \log[E/100.4\ R\ \beta] - 0.463\ E/RT_c \quad (3)$$

NOTE 5—β shall be in units of K h^{-1}.

6.3.2 Prepare a display of logarithm of thermal endurance on(log[tf, the ordinate h]) versus the reciprocal of absolute temperature on(Tr the abscissa]) (see Fig. 1).

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

6.3.4 The standard deviation in the logarithm of the thermal endurance ($t_f$$tf$) may be estimated using Eq 4.

$$\sigma\log[tf\ ,\mathrm{h}]/\log[tf\ ,\mathrm{h}] = \sigma E/E \quad (4)$$

$$\sigma\log[t_f]/\log[t_f] \approx \pm\sigma E/E \quad (4)$$

6.3.5 From the law of propagation of uncertainties (**2**):

$$\sigma t_f/t_f = 2.303\log[t_f]\ \sigma E/E \quad (5)$$

6.4 *Method C – Relative Thermal Index:*

6.4.1 Relative Thermal Index thermal index (RTI) for an alternative thermal endurance (tf(RTI)) may be determined from the activation energy determined by thermogravimetry (E) and the reference thermal index (TI at tf(RTI)) obtained by some other method (such as electrical or mechanical tests) using Eq 65.

$$RTI = TI/\{((2.303\ \ RTI/E)\ \log[tf\ (RTI)/tf(TI)])+1\} \quad (5)$$

$$RTI = E/R[\ln[t_f] - \ln[t_r] + E/(R\ T_r)] \quad (6)$$

NOTE 6—See derivation in Appendix X3.

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 thefor this determination;

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

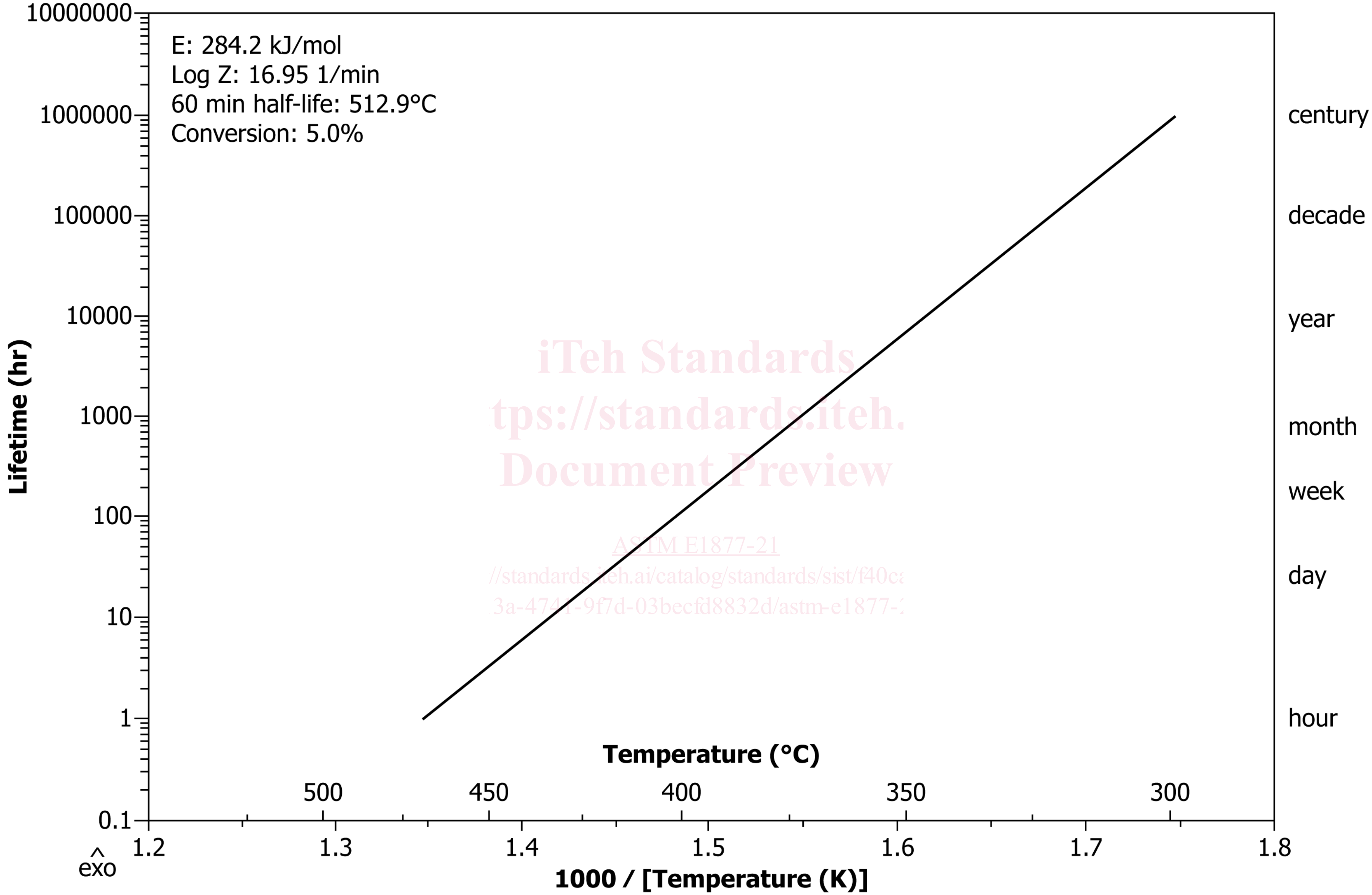

FIG. 1 Thermal Endurance Curve

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 (σ*TI*/*TI*) or relative thermal index (*RTI*) and its relative standard deviation (σ*RTI*/*RTI*) along with the identified thermal endurance.

7.1.3.1 *Example:*

$$TI(60\ 000\ \text{hr}) = 453 \pm 6\ \text{K}(180 \pm 6\ °\text{C})$$

$$TI(20\ 000\ \text{h}) = 456 \pm 6\ \text{K}(180 \pm 6\ °\text{C})$$

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

~~APPENDIX~~APPENDIXES

(Nonmandatory Information)

X1. EXAMPLE CALCULATIONS

X1.1 Example Calculations for the Values Determined in This Standard (These values are not typical. They are for illustration purposes only.)

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 σ*E* = 24 kJ/mol = 24 000 J/mol

X1.1.1.3 *R* = ~~8.314510~~8.314 J/(mol K)

X1.1.1.4 β = 5.0 K/min =300 K/h

X1.1.1.5 *Tf* = 783 K

[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.

~~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~~

X1.1.2 Arbitrarily selected:

X1.1.2.1 ~~t~~*tf$_f$(TI)* = 60 000 ~~hr = 3 600 000 min = 6.8 yr~~h

X1.1.2.2 ~~T~~*TI$_r$* = ~~683~~528 K

X1.1.2.3 ~~t~~*tf$_r$(RTI)* = ~~100 000 hr = 6 000 000 min = 11 yr~~20 000 h

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

X1.2.1 Determine the value for *E/*~~*RT*~~*RTf* from values in X1.1.1.1, X1.1.1.3, and ~~X1.1.2.1~~X1.1.1.5:

$$E/RTf = (320\ 000\ \mathrm{J/mol})/[8.314\ \mathrm{J/(mol\text{-}K)} \times 783\ \mathrm{K}] = 49.16$$

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

X1.2.2 Substitute values from X1.1.1.1, X1.1.1.3, X1.1.1.4, ~~X1.1.3.1,~~ and X1.2.1 into Eq 1:

~~$$\begin{aligned} TI &= \{E/2.303\,R\}/\{\log[100.4\ tf\ \beta\ R/E] + 0.463E/RT\} \\ &= \{320\ \ 000\ \mathrm{J\,mol^{-1}}/2.303 \times 8.314\ \mathrm{J\,mol^{-1}\,K^{-1}}\} \\ &\quad /\{\log[100.4 \times 3\,600\,00\ \mathrm{min} \times 5\ \mathrm{K\,min^{-1}} \times 8.314\ \mathrm{J\,mol^{-1}\,K^{-1}}/320\,000\ \mathrm{J\,mol^{-1}}] + 0.463 \times 49.1532\} \\ &= \{16\,713\ \mathrm{K}\}/\{\log[46\,953] + 22.758\} \\ &= \{16\,713\ \mathrm{K}\}/\{4.672 + 22.758\} \\ &= \{16\,713\ \mathrm{K}\}/\{27.430\} \\ &= 609.3\ \mathrm{K} = > 336.1°\mathrm{C} \end{aligned}$$~~

$$\begin{aligned} TI &= \{E/2.303\,R\}/\{\log[100.4\ tf\ \beta\ R/E] + 0.463E/RTf\} \\ &= \{320\ \ 000\ \mathrm{J\,mol^{-1}}/2.303 \times 8.314\ \mathrm{J\,mol^{-1}\,K^{-1}}\} \\ &\quad /\{\log[100.4 \times 3\,600\,000\ \mathrm{min} \times 5\ \mathrm{K\,min^{-1}} \times 8.314\ \mathrm{J\,mol^{-1}\,K^{-1}}/320\,000\ \mathrm{J\,mol^{-1}}] + 0.463 \times 49.16\} \\ &= \{16\,713\ \mathrm{K}\}/\{\log[46\,953] + 22.758\} \\ &= \{16\,713\ \mathrm{K}\}/\{4.672 + 22.758\} \\ &= \{16\,713\ \mathrm{K}\}/\{27.430\} \\ &= 609.3\ \mathrm{K}(=> 336.1\ °\mathrm{C}) \end{aligned}$$

X1.3 Example Calculation for the Imprecision in Thermal Index

X1.3.1 Substituting values from X1.1.1.1 and X1.1.1.2 into Eq 2: