INTERNATIONAL

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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.

[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.

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.~~lifetime or time-to-failure.

4. Summary of Practice

4.1 The Arrhenius activation energy obtained from other Test Methods (such as Test ~~Method~~Methods E1641~~, Refs~~ and E2958~~(1, 2),~~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 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 ~~Method~~Methods E1641 and E2958, ~~Ref~~ etc.).~~(1, 2), etc.)~~

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

6.1.1.3 β = ~~Heating~~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~~constant conversion failure criterion,

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

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

6.1.1.9 RTI = Relative Thermal Index ~~(K).~~(K),

6.1.1.10 ~~σE~~σ = standard deviation in activation energy (J/mol) obtained from Test ~~Method~~Methods E1641 and E2958, ~~Ref~~ etc.,~~(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~~thermal index (K),

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

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

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

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

6.1.1.16 T_r = ~~Reference~~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.

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~~3**).[3]

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

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

TABLE 1 Numerical Integration Constants (~~3~~1, ~~4~~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

6.2.5 Determine the relative standard deviation ($\sigma TI/TI$) 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 ($\sigma TI/TI$) along with the thermal endurance (t_f).

6.3 *Method B – Thermal Endurance Curve:*

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.

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

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

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 ~~time-to-failure~~ thermal endurance (t_f) may be estimated using Eq 4.

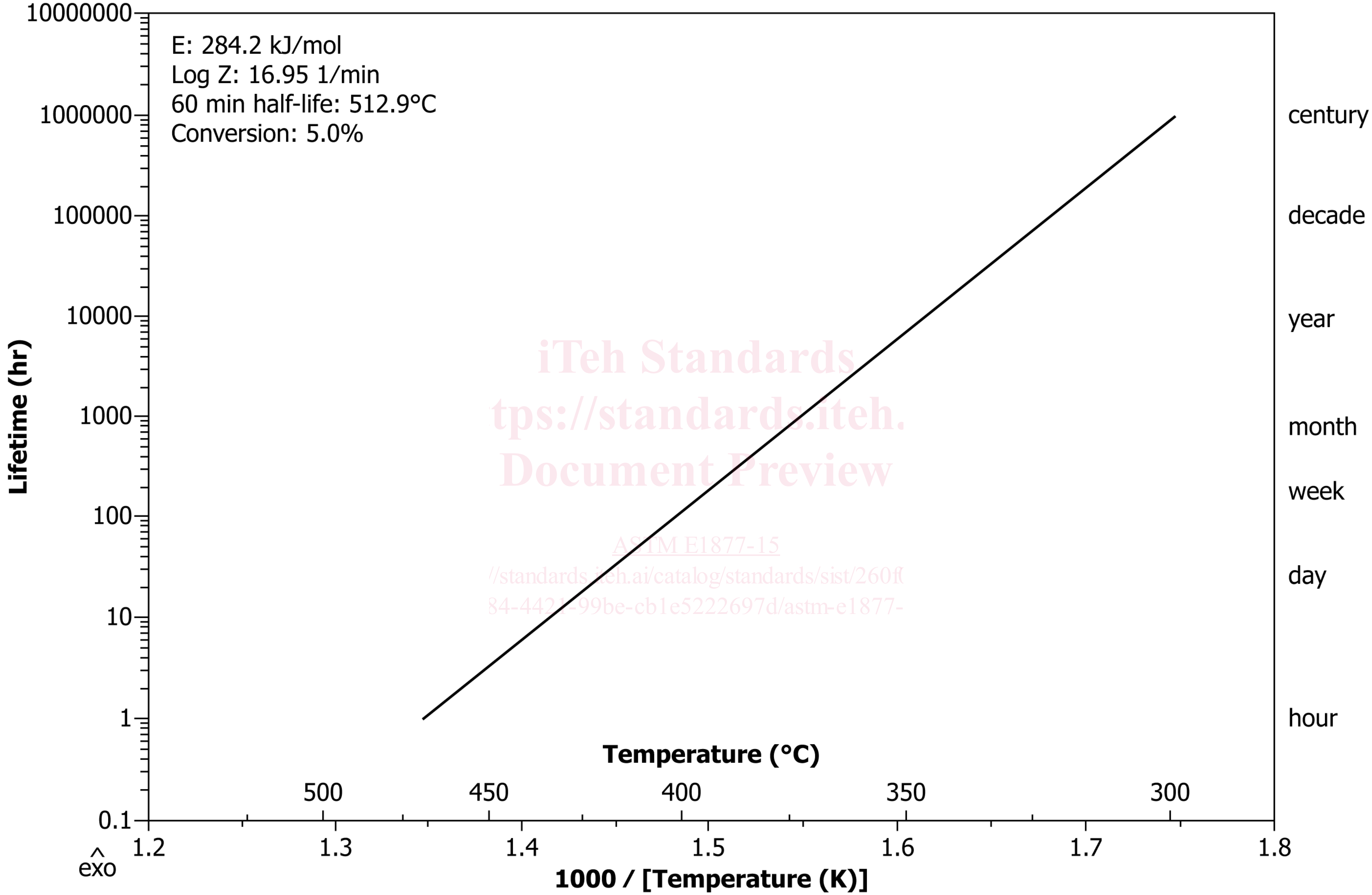

FIG. 1 Thermal Endurance Curve