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Standard Test Method for Heat of Ablation¹

This standard is issued under the fixed designation E 458; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers determination of the heat of ablation of materials subjected to thermal environments requiring the use of ablation as an energy dissipation process. Three concepts of the property parameter are described and defined: cold wall, effective, and thermochemical heat of ablation.

1.2 *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

E 285 Test Method for Oxyacetylene Ablation Testing of Thermal Insulation Materials

E341 Practice for Measuring Plasma Arc Gas Enthalpy by Energy Balance²

E377 Practice for Internal Temperature Measurements in Low Conductivity Materials²

E422 Test Method for Measuring Heat Flux Using a Water-Cooled Calorimeter²

E457 Test Method for Measuring Heat-Transfer Rate Using a Thermal Capacitance (Slug) Calorimeter²

E459 Test Method for Measuring Heat-Transfer Rate Using a Thin-Skin Calorimeter²

E470 Method for Measuring Gas Enthalpy Using Calorimetric Probes

E471 Test Method for Obtaining Char Density Profile of Ablative Materials by Machining and Weighing²

E511 Test Method for Measuring Heat Flux Using a Copper-Constantan Circular Foil, Heat-Flux Gage²

E 422 Test Method for Measuring Heat Flux Using a Water-Cooled Calorimeter

E 457 Test Method for Measuring Heat-Transfer Rate Using a Thermal Capacitance (Slug) Calorimeter

E 459 Test Method for Measuring Heat Transfer Rate Using a Thin-Skin Calorimeter

E 511 Test Method for Measuring Heat Flux Using a Copper-Constantan Circular Foil, Heat-Flux Transducer

E 617 Specification for Laboratory Weights and Precision Mass Standards

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

3.1.1 *heat of ablation*—a property parameter that indicates the ability of a material to provide heat protection when used as a sacrificial thermal protection device. The property parameter is a function of both the material and the environment to which it is subjected. In general, it is defined as the incident heat dissipated by the ablative material per unit of mass removed, or

$$Q^* = q/m \quad (1)$$

where:

Q^* = heat of ablation, kJ/kg,

q = incident heat transfer rate, kW/m², and

m = total mass transfer rate, kg/m²·s.

~~3.2~~

3.1.2 The heat of ablation may be represented in three different ways depending on the investigator's requirements:

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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* Vol 15.03 volume information, refer to the standard's Document Summary page on the ASTM website.

3.3

3.1.3 cold-wall heat of ablation—The most commonly and easily determined value is the cold-wall heat of ablation, and is defined as the incident cold-wall heat dissipated per unit mass of material ablated, as follows:

$$Q^* = q_{cw}/m \quad (2)$$

$$Q^*_{cw} = q_{cw}/m \quad (2)$$

where:

Q^*_{cw} = cold-wall heat of ablation, kJ/kg,

q = heat transfer rate from the test environment to a cold wall, kW/m², and

$\frac{hwq_{cw}}{m}$ = total mass transfer rate, kg/m²·s.

The temperature of the cold-wall reference for the cold-wall heat transfer rate is usually considered to be room temperature or close enough such that the hot-wall correction given in Eq 78 is less than 5 % of the cold-wall heat transfer rate.

3.1.4 effective heat of ablation—The effective heat of ablation is defined as the incident hot-wall heat dissipated per unit mass ablated, as follows:

$$Q^*_{eff} = q_{hw}/m \quad (3)$$

$$Q^*_{eff} = q_{hw}/m \quad (3)$$

where:

Q^*_{eff} = effective heat of ablation, kJ/kg,

q_{hw} = heat transfer rate from the test environment to a nonablating wall at the surface temperature of the material under test, kW/m², and

m = total mass transfer rate, kg/m²·s.

3.5

3.1.5 thermochemical heat of ablation—The thermochemical heat of ablation is defined as the incident hot-wall heat dissipated per unit mass ablated, corrected for reradiation heat rejection processes and material eroded by mechanical removal, as follows:—The derivation of the *thermochemical heat of ablation* originated with the simplistic surface energy equation employed in the early 60s to describe the effects of surface ablation, that is:

$$Q^*_{tc} = (q_{hw} - q_{rr} - q_{mech})m_{tc} \quad (4)$$

$$q_{hw} - q_{rr} = q_{cond} + q_{abl} + q_{block} \quad (4)$$

$m_{tc} = m - m_{mech}$

$$q_{mech} = m_{mech}(h_m) \quad (6)$$

q_{rr}

where:

Q^*_{tc} = thermochemical heat of ablation, kJ/kg, energy re-radiated from the heated surface, kW/m²,

q_{rr} = reradiative heat transfer rate, kW/m², net energy conducted into the solid during steady-state ablation = $mc_p(T_w - T_o)$, kW/m²,

m_{tc} = mass transfer rate due to thermochemical processes, kg/m²·s, energy absorbed by surface ablation which, in simple terms, can be

represented by $m\Delta H_v$, kW/m²·s,

m = mass-transfer rate due to mechanical processes, kg/m²·s, energy dissipated (blockage) by transpiration of ablation products into the boundary

layer, which, in simple terms, can be represented by $m\eta(h_r - h_w)$, kW/m²·s,

q = heat-transfer rate due to energy carried away with mechanically removed material, kW/m², and absolute surface

temperature of ablating material, K,

$h_m c_p$ = enthalpy of mechanically removed material, kJ/kg, specific heat at constant pressure of ablating material, kJ/kg·K,

T_o = initial surface temperature of ablating material, K,

ΔH_v = an effective heat of vaporization, kJ/kg,

η = a transpiration coefficient,

h_r = gas recovery enthalpy, kJ/kg, and

h_w = the wall enthalpy, kJ/kg.

Mechanical removal of material takes place in the more severe test environments where relatively high aerodynamic shear or particle impingement is present. The effects of mechanical removal and theories relating to the mechanism of this process are presented in Refs (1-5). If the effects of mechanical removal of material cannot be determined or are deemed unimportant, the term q_{mech} in Eq 4 goes to zero. The investigator should, however, be aware of the existence of this phenomenon and also note whether this effect was considered when reporting data.

3.6 The three heat of ablation values described in 3.2 require two basic determinations: the heat-transfer rate and the mass-transfer rate. These two quantities then assume various forms depending on the particular heat of ablation value being

determined. Using the definitions above, Eq 4 can be rewritten as:

$$q_{lw} - q_{rr} = mc_p(T_w - T_o) + m\Delta H_v + m\eta(h_r - h_w) \quad (5)$$

where it should be apparent that the definition of the *thermochemical heat of ablation* is obtained by dividing Eq 4 by m , where it is understood that m is a steady-state ablation rate. The result is:

$$Q_{tc}^* = (q_{lw} - q_{rr})/m = c_p(T_w - T_o) + \Delta H_v + \eta(h_r - h_w) \quad (6)$$

As seen from Eq 6, definition of the *thermochemical heat of ablation* requires an ability to measure the cold-wall heat flux, an ability to define the recovery enthalpy, an ability to measure the surface temperature, knowledge of the total hemispherical emittance (at the temperature and state of the ablating surface), and the ability to determine the steady-state mass loss rate. Assuming these parameters can be measured (or estimated), the right hand side of Eq 6 implies that the *thermochemical heat of ablation* is a linear function of the enthalpy difference across the boundary layer, that is, $(h_r - h_w)$. Consequently, a plot of Q_{tc}^* (determined from several tests at different conditions) versus $(h_r - h_w)$ should allow a linear fit of the data where the slope of the fit is interpreted as η , the transpiration coefficient, and the y -intercept is interpreted as $c_p\Delta T + \Delta H_v$. If the specific heat of the material is known, the curve fit allows the effective heat of vaporization to be empirically derived.

3.2 The three heat of ablation values described in 3.1.2 require two basic determinations: the heat transfer rate and the mass transfer rate. These two quantities then assume various forms depending on the particular heat of ablation value being determined.

4. Significance and Use

4.1 The heat of ablation provides a measure of the ability of a material to serve as a heat protection element in a severe thermal environment. The property is a function of both the material and the environment to which it is subjected. It is therefore required that laboratory measurements of heat of ablation simulate the service environment as closely as possible. Some of the parameters affecting the property are pressure, gas composition, heat transfer rate, mode of heat transfer, and gas enthalpy. As laboratory duplication of all parameters is usually difficult, the user of the data should consider the differences between the service and the test environments. Screening tests of various materials under simulated use conditions may be quite valuable even if all the service environmental parameters are not available. These tests are useful in material selection studies, materials development work, and many other areas.

5. Determination of Heat Transfer Rate Significance and Use

4.1 *General*—The heat of ablation provides a measure of the ability of a material to serve as a heat protection element in a severe thermal environment. The parameter is a function of both the material and the environment to which it is subjected. It is therefore required that laboratory measurements of heat of ablation simulate the service environment as closely as possible. Some of the parameters affecting the heat of ablation are pressure, gas composition, heat transfer rate, mode of heat transfer, and gas enthalpy. As laboratory duplication of all parameters is usually difficult, the user of the data should consider the differences between the service and the test environments. Screening tests of various materials under simulated use conditions may be quite valuable even if all the service environmental parameters are not available. These tests are useful in material selection studies, materials development work, and many other areas.

4.2 *Steady-State Conditions*—The nature of the definition of heat of ablation requires steady-state conditions. Variances from steady-state may be required in certain circumstances; however, it must be realized that transient phenomena make the values obtained functions of the test duration and therefore make material comparisons difficult.

4.2.1 *Temperature Requirements*—In a steady-state condition, the temperature propagation into the material will move at the same velocity as the gas-ablation surface interface. A constant distance is maintained between the ablation surface and the isotherm representing the temperature front. Under steady-state ablation the mass loss and length change are linearly related.

$$mt = \rho_o \delta_L + (\rho_o - \rho_c) \delta_c \quad (7)$$

where:

- t = test time, s,
- ρ_o = virgin material density, kg/m³,
- δ_L = change in length or ablation depth, m,
- ρ_c = char density, kg/m³, and
- δ_c = char depth, m.

This relationship may be used to verify the existence of steady-state ablation in the tests of charring ablators.

4.2.2 *Exposure Time Requirements*—The exposure time required to achieve steady-state may be determined experimentally by the use of multiple models by plotting the total mass loss as a function of the exposure time. The point at which the curve departs significantly from linearity is the minimum exposure time required for steady-state ablation to be established. Cases exist, however, in the area of very high heating rates and high shear where this type of test for steady-state may not be possible.

5. Determination of Heat Transfer Rate

5.1 Cold-Wall Heat Transfer Rate :

5.1.1 Determine the cold-wall heat transfer rate to a specimen by using a calorimeter. These instruments are available commercially in several different types, some of which can be readily fabricated by the investigator. Selection of a specific type

is based on the test configuration and the methods used, and should take into consideration such parameters as instrument response time, test duration, and heat transfer rate (61³).

5.1.1.1 The calorimeters discussed in 5.1.1 measure a “cold-wall” heat-transfer rate because the calorimeter surface temperature is much less than the ablation temperature. The value thus obtained is used directly in computing the cold-wall heat of ablation.

5.1.2 Install the calorimeter in a calorimeter body that duplicates the test model in size and configuration. This is done in order to eliminate geometric parameters from the heat-transfer rate measurement and to ensure that the quantity measured is representative of the heat-transfer rate to the test model. If the particular test run does not allow an independent heat-transfer rate measurement, as in some nozzle liner and pipe flow tests, mount the calorimeter as near as possible to the location of the mass-loss measurements. Take care to ensure that the nonablating calorimeter does not affect the flow over the area under test. In axisymmetric flow fields, measurements of mass loss and heat-transfer rate in the same plane, yet diametrically opposed, should be valid.

5.2 Computation of Effective and Thermochemical Heats of Ablation:

5.2.1 In order to compute the effective and thermochemical heats of ablation, correct the cold-wall heat-transfer rate for the effect of the temperature difference on the heat transfer. This correction factor is a function of the ratio of the enthalpy potentials across the boundary layer for the hot and cold wall as follows:

$$q_{hw} = q_{cw} [(h_e - h_{hw}) / (h_e - h_{cw})] \quad (8)$$

$$q_{hw} / q_{cw} = [(h_e - h_{hw}) / (h_e - h_{cw})] \quad (8)$$

where:

h_e = gas recovery enthalpy at the boundary layer edge, kJ/kg,

h_{hw} = gas enthalpy at the surface temperature of the test model, kJ/kg, and

h_{cw} = gas enthalpy at a cold wall, kJ/kg.

5.2.2 This correction is based upon laminar flow in air and subject to the restrictions imposed in Ref (72). Additional corrections may be required regarding the effect of temperature on the transport properties of the test gas. The form and use of these corrections should be determined by the investigator for each individual situation.

5.3 Gas Enthalpy Determination :

5.3.1 The enthalpy at the boundary layer edge may be determined in several ways: energy balance, enthalpy probe, spectroscopy, etc. Details of the methods may be found elsewhere (8-113-6). Take care to evaluate the radial variation of enthalpy in the nozzle. Also, in low-density flows, consider the effect of nonequilibrium on the evaluation. Determination of the gas enthalpy at the ablator surface and the calorimeter surface requires pressure and surface temperature measurements. The hot-wall temperatures are generally measured by optical methods such as pyrometers, radiometers, etc. Other methods such as infrared spectrometers and monochromators have been used (127,138). Effects of the optical properties of the boundary layer of an ablating surface make accurate determinations of surface temperature difficult.

5.3.2 Determine the wall enthalpy from the assumed state of the gas flow (equilibrium, frozen, or nonequilibrium), if the pressure and the wall temperature are known. It is further assumed that the wall enthalpy is the enthalpy of the freestream gas, without ablation products, at the wall temperature. Make the wall static pressure measurements with an ordinary pitot arrangement designed for the flow regime of interest and by using the appropriate transducers.

5.4 Reradiation Correction:

5.4.1 Calculate the heat-transfer rate due to reradiation from the surface of the ablating material from the following equation:

$$q_{rr} = \epsilon \sigma T_s^4 \quad (9)$$

$$q_{rr} = \sigma \epsilon T_w^4 \quad (9)$$

where:

σ = Stefan-Boltzmann constant, Stefan-Boltzmann constant, and,

T_s = absolute surface temperature of ablating material, K, and

ϵ = thermal emittance of the ablating surface.

5.4.2 Eq 89 assumes radiation through a transparent medium to a blackbody at absolute zero. Consider the validity of this assumption for each case and if the optical properties of the boundary layer are known and are deemed significant, or the absolute zero blackbody sink assumption is violated, consider these effects in the use of Eq 8-9.

5.5 Mechanical Removal Correction :

5.5.1 Determine the heat-transfer rate due to the mechanical removal of material from the ablating surface from the mass-loss rate due to mechanical processes and the enthalpy of the material removed as follows:

$$q_{mech} = m_{mech} h_m \quad (10)$$

$$q_{mech} = m_{mech} h_m \quad (10)$$

³ Discontinued, see 1982 *Annual Book of ASTM Standards*, Part 41.

³ The boldface numbers in parentheses refer to the references listed at the end of the standard.

5.5.2 Approximate the enthalpy of the material removed by the product of the specific heat of the mechanically removed material, and the surface temperature (**1-59-13**).

6. Determination of Mass- Transfer Rate

6.1 The determination of the heat of ablation requires the measurement of the mass- transfer rate of the material under test. This may be accomplished in several ways depending on the type of material under test. The heat of ablation value can be affected by the choice of method.

6.1.1 Ablation Depth Method:

6.1.1.1 The simplest method of measurement of mass-loss rate is the change in length or ablation depth. Make a pretest and post-test measurement of the length and calculate the mass-loss rate from the following relationship:

$$m = \rho_o(\delta L/t) \quad (11)$$

where:

ρ_o = virgin material density, kg/m³,

δL = change in length or ablation depth, m, and

t = test time, s.

($\delta L/t$)

6.1.1.2 Determine the change in length with the time of a model under test, by using motion picture techniques. Note that observation of the front surface alone does not, however, verify the existence of steady state ablation. Take care, however, to provide appropriate reference marks for measuring the length change from the film. Timing marks on the film are also required to accurately determine the time parameter. Avoid using framing speed as a reference, as it generally does not provide the required accuracy.

6.1.1.3 Use the length change measurement of mass-loss rate for non-charring ablators, subliming materials, or with charring ablators under steady state ablation conditions (see Section 74) and only with materials that do not swell or grow in length.

6.1.2 Direct Weighing Method:

6.1.2.1 A second method of determining mass- transfer rate is by the use of a pretest and post-test mass measurement. This procedure yields the mass transfer rate directly. A disadvantage of this method is that the mass- transfer rate obtained is averaged over the entire test model heated area. The heat- transfer rate is generally varying over the surface and therefore leads to errors in heat of ablation. The mass- transfer rate is also averaged over the insertion period which includes the early part of the period when the ablation process is transient and after the specimen has been removed where some mass loss occurs. The experimenter should be guided by Section 74.1 in determining the magnitude of these effects.

6.1.2.2 In cases where the mass loss is low, the errors incurred in mass loss measurements could become large. It is therefore recommended that a significant mass loss be realized to reduce measurement errors. The problem is one of a small difference of two large numbers.

6.1.3 Core Sample Method:

6.1.3.1 Accomplish direct measurement of the mass loss by coring the model after testing by using standard core drills. The core size is determined by the individual experiment; however, core diameters of 5.0 to 10.0 mm should be adequate. Coring the model at the location of the heat- transfer rate measurement makes the mass- transfer rate representative of the measured environment. Obtain the mass- transfer rate from the core sample as follows:

$$m = (\rho_o V_o - w_f)/(t A_c) \quad (12)$$

$$m = (\rho_o V_o - w_f)/(t A_c) \quad (12)$$

where:

V_o = original calculated volume of core, m³,

w_f = final mass of core, kg, and

A_c = cross-sectional area of core, m².

6.1.3.2 Calculate the original core volume using the measured diameter of the core after removal from the test model. The core drill dimensions should not be used due to drilling inaccuracies.

6.1.4 *Shrouded Core Method*—A second core sample method used in measuring ablation properties of materials involves the use of a model that includes a core and model shroud of the same material where the core has been prepared prior to testing. This method is described in detail in Ref:Ref (**513**). This type of test model offers the advantages of ease of installation of thermal instrumentation, and direct pretest mass and dimensional measurements of the core. Calculate the heat of ablation in the same manner for the drilled core sample (6.1.3.2).

6.1.5 Core sample methods are useful with charring ablators, or materials that swell or grow on heating. The resulting core sample is also useful in observing the composition and formation of the char layer.

7. Steady-State Conditions

7.1 *General*—The nature of the definition of heat of ablation requires steady-state conditions. Variances from steady-state may be required in certain circumstances; however, it must be realized that transient phenomena make the values obtained functions

of the test duration and therefore make material comparisons difficult.

7.2 Temperature Requirements:

7.2.1 In a steady-state condition, the temperature propagation into the material will move at the same velocity as the gas-ablation surface interface. A constant distance is maintained between the ablation surface and the isotherm representing the temperature front. Under steady-state ablation the mass loss and length change are linearly related:

$$mt = \rho_o \Delta L + (\rho_o - \rho_c) \delta_c \quad (12)$$

where:

ρ_c = char density, kg/m³, and

δ_c = char depth, m.

7.2.2 This relationship may be used to verify the existence of steady-state ablation in the tests of charring ablators.

7.3 **Exposure Time Requirements**—The exposure time required to achieve steady-state may be determined experimentally by the use of multiple models by plotting the total mass loss as a function of the exposure time. The point at which the curve departs significantly from linearity is the minimum exposure time required for steady-state ablation to be established. Cases exist, however, in the area of very high heating rates and high shear where this type of test for steady-state may not be possible.

8.

7. Apparatus

8.1

7.1 **Environmental**—The primary apparatus required is a means of providing the required thermal environment. Several devices have been used to accomplish this task including arc powered plasma jets, oxy-acetylene torch heaters (see Test Method E 285) liquid and solid propellant rocket exhausts, radiant heating lamps, etc. Each type of test facility has certain advantages and capability limitations and the type used will depend on the required test environment. The test facility used should be thoroughly described as part of the test report.

8.2

7.2 **Instrumentation**—The measurement apparatus such as calorimeters, enthalpy probes, temperature measuring devices, and instrumentation for enthalpy and pressure measurement of the test environment have been described in other ASTM standards (see Section 2). **Related Materials at the end of this standard**. A description of all primary instrumentation should be included in the test report.

9.8. Test Specimen or Model

9.1 **The 8.1** The test specimen size and shape will depend on the apparatus used, the desired results, service conditions, and type of test. Details of the specimen should be included as part of the test report.

10.9. Procedure

10.1 **The 9.1** The exact procedure followed will depend on the heat source used, test specimen configuration and test objectives. A sample procedure is presented in the following paragraphs.

10.1.1 **Weigh 9.1.1** Weigh and measure the test specimen. Do not include any supporting devices that are easily removed, both before and after test, in the mass measurement, in order to reduce the tare. For hygroscopic materials or chars, or both, the mass measurements before and after the test should be made under conditions of equal humidity (**513**). The important linear dimension is that parallel to the expected recession. Take both measurements such that the final length and mass change will be accurate to 2 %.

10.1.2 **Energize 9.1.2** Energize the heat source and bring it to the required test condition. Verify the test condition by measuring the cold-wall heat-transfer rate, surface and pitot pressure, and gas enthalpy. Expose the diagnostic probes for sufficient length of time, so as to allow full instrument response and the equilibration of any transients induced by the probe insertion process.

10.1.2.1 **After 9.1.2.1** After verification of the test condition, insert the material specimen in the test environment and expose it for a predetermined test time or until the test objectives have been accomplished. Insertion and retraction times should be short with respect to the test duration, of the order of 5 % or less of the total test time.

10.1.2.2 **In 9.1.2.2** In certain char forming materials, take care so that the retraction dynamics do not disturb the fragile char. In the case of fragile char, it is sometimes desirable to terminate the test by extinguishing the heat source rather than by retracting the test specimen.

10.1.2.3 **During 9.1.2.3** During the test period, take measurements of the specimen surface temperature for calculation of Q_{eff}^* and Q_{ic}^* , internal temperatures, and any other measurements required by the test objective.

10.1.2.4 **After 9.1.2.4** After the test specimen is allowed to cool for handling, take post-test mass and length measurements. Take care in order to preserve the char structure. Take a still photograph of the specimen and include it in the report.

10.1.2.5 **Evaluate 9.1.2.5** Evaluate variations from this test procedure as to their conformance to the steady-state concepts.

11.10. Calculation

11.0.1 Calculate the mass loss in two ways, length change and mass loss. Mass loss may be a gross mass loss from the entire