



Designation: D7309 – 21b

Standard Test Method for Determining Flammability Characteristics of Plastics and Other Solid Materials Using Microscale Combustion Calorimetry¹

This standard is issued under the fixed designation D7309; 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 test method, which is similar to thermal analysis techniques, establishes a procedure for determining flammability characteristics of combustible materials such as plastics.

1.2 The test is conducted in a laboratory environment using controlled heating of milligram specimens and complete thermal oxidation of the specimen gases.

1.3 Specimens of known mass are thermally decomposed in an oxygen-free (anaerobic) or oxidizing (aerobic) environment at a constant heating rate between 0.2 and 2 K/s.

1.4 The heat released by the specimen is determined from the mass of oxygen consumed to completely oxidize (combust) the specimen gases.

1.5 The rate of heat released by combustion of the specimen gases produced during controlled thermal or thermoxidative decomposition of the specimen is computed from the rate of oxygen consumption.

1.6 The specimen temperatures over which combustion heat is released are measured.

1.7 The mass of specimen remaining after the test is measured and used to compute the residual mass fraction.

1.8 The specimen shall be a material or composite material in any form (fiber, film, powder, pellet, droplet). This test method has been developed to facilitate material development and research.

1.9 *This standard is used to measure and describe the response of materials, products, or assemblies to heat and flame under controlled conditions, but does not by itself incorporate all factors required for fire hazard or fire risk assessment of the materials, products, or assemblies under actual fire conditions.*

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

NOTE 1—There is no known ISO equivalent to this test method.

1.11 *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

D883 Terminology Relating to Plastics

D5865 Test Method for Gross Calorific Value of Coal and Coke

E176 Terminology of Fire Standards

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E967 Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers

E1591 Guide for Obtaining Data for Fire Growth Models

E2935 Practice for Evaluating Equivalence of Two Testing Processes

2.2 *ISO Standard:*

ISO 13943 Fire Safety—Vocabulary³

3. Terminology

3.1 *Definitions:*

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal 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 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

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

3.1.1 Terms used in this standard are defined in accordance with Terminology **D883**, unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology **E456**. For terms relating to fire, the terms in this standard are defined in accordance with Terminology **E176** and ISO 13943. In case of conflict, the definitions given in Terminology **E176** shall prevail.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *combustion residue, n*—the non-volatile chemical species remaining after controlled thermal oxidative decomposition of a specimen.

3.2.2 *combustion temperature, n*—the specimen temperature at which the specific combustion rate is a maximum during controlled thermal oxidative decomposition.

3.2.3 *controlled heating, n*—a controlled temperature program used to effect thermal decomposition or oxidative thermal decomposition in which the temperature of the specimen is uniform throughout and increases with time at a constant rate.

3.2.4 *controlled thermal (or thermal oxidative) decomposition, n*—thermal (oxidative) decomposition under controlled heating.

3.2.5 *fire growth capacity, n*—a parameter derived from the temperature dependence of the chemical processes responsible for ignition and burning of combustible materials.

3.2.6 *heat release capacity, n*—the maximum specific heat release rate during a controlled thermal decomposition divided by the heating rate in the test.

3.2.7 *heating rate, n*—the constant rate of temperature rise of the specimen during the controlled temperature program.

3.2.8 *maximum specific combustion rate, n*—the maximum value of the specific combustion rate recorded during the test.

3.2.9 *maximum specific heat release rate, n*—the maximum value of the specific heat release rate recorded during the test.

3.2.10 *net calorific value, n*—the net heat of complete combustion of the specimen measured during controlled thermal oxidative decomposition per unit initial specimen mass.

3.2.11 *oxidative thermal decomposition, n*—a process of extensive chemical species change caused by heat and oxygen (thermal oxidation, oxidative pyrolysis).

3.2.12 *pyrolysis residue, n*—the fraction of the initial specimen mass remaining after controlled anaerobic thermal decomposition.

3.2.13 *specific combustion rate, n*—the rate at which combustion heat is released per unit initial mass of specimen during controlled thermal oxidative decomposition.

3.2.14 *specific heat of combustion of specimen gases, n*—net calorific value of gases.

3.2.15 *specific heat release rate, n*—the rate at which combustion heat is released per unit initial mass of specimen during controlled thermal decomposition.

3.2.16 *specific heat release, n*—the net heat of complete combustion of the volatiles liberated during controlled thermal decomposition per unit initial specimen mass.

3.2.17 *specimen gases, n*—the volatile chemical species liberated during controlled thermal (oxidative) decomposition of a specimen.

3.3 Symbols:

β	= heating rate, K/s
E	= 13.1 kJ/g-O ₂ is the average heat released by complete combustion of organic compounds per unit mass of oxygen consumed
F_0	= volumetric flow rate of the combustion stream at ambient temperature and pressure measured at the terminal flow meter prior to the start of the test, cm ³ /s
F	= volumetric flow rate of the combustion stream at ambient temperature and pressure measured at the terminal flow meter during the test, cm ³ /s
FGC	= fire growth capacity of sample, J/g-K
h_c	= specific heat release of sample, J/g
h_c^o	= net calorific value of sample, J/g
$h_{c,gas}$	= specific heat of combustion of specimen gases, J/g
η_c	= heat release capacity, J/g-K
m_o	= initial specimen mass, g
m_c	= residual specimen mass after oxidative pyrolysis, g
m_p	= residual specimen mass after the anaerobic pyrolysis, g
$Q(t)$	= specific heat release rate at time t , W/g
Q_{max_0}	= maximum specific heat release rate, W/g
Q_{max}	= maximum specific combustion rate, W/g
ρ_{O_2}	= density of oxygen at ambient conditions, g/cm ³
T	= time synchronized to temperature, $x - \tau$, s
T_0	= standard room temperature, 298K.
$T_{5\%}$	= temperature at 5 % of h_c measured at heating rate, $\beta = 1$ K/s, K
$T_{95\%}$	= temperature at 95 % of h_c measured at heating rate, $\beta = 1$ K/s, K
T_{max_0}	= heat release temperature at Q_{max_0} , K
T_{max}	= combustion temperature at Q_{max} , K
τ	= transit time of the gas stream between the specimen location and the oxygen analyzer, s
x	= time at which the oxygen analyzer signal is recorded, s
$X_{O_2}^o$	= volume (mole) fraction of oxygen in the combustion stream measured at the oxygen sensor prior to the start of the test, cm ³ /cm ³
X_{O_2}	= volume (mole) fraction of oxygen measured at the oxygen sensor during the test at time, t , cm ³ /cm ³
Y_c	= combustion residue, g/g
Y_p	= pyrolysis residue, g/g

4. Summary of Test Method

4.1 This test method provides two procedures for determining flammability characteristics of materials in a laboratory test using controlled heating (controlled temperature programming) and oxygen consumption calorimetry. This test measures flammability characteristics using a controlled temperature program to force the release of specimen gases, thermal oxidation of the specimen gases (and optionally the specimen residue) in excess oxygen, and measurement of the oxygen