



Designation: D7309 – 11

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

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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 and health practices and determine the applicability of regulatory limitations prior to use.

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

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

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

E1591 Guide for Obtaining Data for Deterministic Fire Models

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms relating to plastics, refer to Terminology D883.

3.1.2 For definitions of terms relating to fire, refer to Terminology E176.

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.

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.

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

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

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

3.2.7 *heat release temperature*, n —the specimen temperature at which the specific heat release rate is a maximum during controlled thermal decomposition.

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	= volumetric flow rate of the combustion stream at ambient temperature and pressure measured at the terminal flow meter, cm ³ /s
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
$\Delta[O_2]$	= the change in the concentration (volume fraction) of O ₂ in the gas stream due to combustion measured at the oxygen sensor at time t , cm ³ /cm ³
$Q(t)$	= specific heat release rate at time t , W/g
Q_{max}	= maximum specific heat release rate, W/g
Q_{max}^o	= maximum specific combustion rate, W/g
ρ	= density of oxygen at ambient conditions, g/cm ³
t	= time synchronized to temperature, $x - \tau$, s
T_{max}	= heat release temperature, K
T_{max}^o	= combustion temperature, 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
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 consumed to calculate the amount, rate, and temperature of heat released by combustion of a solid specimen during controlled heating.

4.2 *Controlled Thermal Decomposition, Method A*—In this procedure the specimen is subjected to controlled heating in an oxygen-free/anaerobic environment, that is, controlled thermal decomposition. The gases released by the specimen during controlled thermal decomposition are swept from the specimen chamber by a non-oxidizing/inert purge gas (typically nitrogen), subsequently mixed with excess oxygen, and completely oxidized in a high temperature combustion furnace. The volumetric flow rate and volumetric oxygen concentration of the gas stream exiting the combustion furnace are continuously measured during the test to calculate the rate of heat release by means of oxygen consumption. In Method A the heat of combustion of the volatile component of the specimen (specimen gases) is measured but not the heat of combustion of any solid residue. Table X1.1 of Appendix X1 shows data for η_c , h_c , Y_p , and T_{max} for 14 different commercial plastics tested in triplicate ($n = 3$).

4.3 *Controlled Thermal Oxidative Decomposition, Method B*—In this procedure the specimen is subjected to controlled heating in an oxidizing/aerobic environment, that is, controlled thermal oxidative decomposition. The specimen gases evolved during the controlled heating program are swept from the specimen chamber by the oxidizing purge gas (for example, dry air) and mixed with additional oxygen, if necessary, prior to entering a high temperature combustion furnace where the gases are completely oxidized. The volumetric flow rate and volumetric oxygen concentration of the

gas stream exiting the combustion furnace are continuously measured during the test to calculate the specific combustion rate by means of oxygen consumption. In Method B the net calorific value of the specimen gases and solid residue are measured during the test.

5. Significance and Use

5.1 This laboratory test method measures thermal combustion properties of materials (1-5).³

5.2 The test uses controlled thermal decomposition of specimens and thermal oxidation of the specimen gases as they are released from the specimen to simulate the condensed and gas phase processes of flaming combustion, respectively, in a small-scale laboratory test (1-7).

5.3 The thermal combustion properties measured in the test are related to flammability characteristics of the material (4-7).

5.4 The amount of heat released in flaming combustion per unit mass of material is the fire load and the potential fire load (complete combustion) is estimated in Method A as h_c .

5.5 The net calorific value of the material (see Test Method D5865) is determined directly using Method B as h_c^o without the need to know the atomic composition of the specimen to correct for the latent heat of evaporation of the water produced by combustion, or to perform titrations to correct for the heat of solution of acid gases. See Table X1.2 for comparison of Microscale Combustion Calorimetry (MCC) data with Test Method D5865.

5.6 The heat release temperature T_{max} of Method A approximates the surface temperature at piloted ignition in accordance with Ref. (5-7) for purposes of fire modeling (See Guide E1591).

5.7 The heat release capacity η_c (J/g-K) is a flammability parameter measured in Method A that is unique to this test method.

6. Limitations

6.1 The heat release capacity (η_c) is independent of the form, mass, and heating rate of the specimen as long as the specimen temperature is uniform at all times during the test (1-5).

6.2 Test results obtained from small (milligram) samples by this method do not include physical behavior such as melting, dripping, swelling, shrinking, delamination, and char/barrier formation that can influence the results of large (decagram/kilogram) samples in flame and fire tests.

6.3 Test results obtained from small (milligram) samples by this method do not include extrinsic factors such as thickness, sample orientation, external heat flux, ignition source, boundary conditions, and ventilation rate that influence the results of large (decagram/kilogram) samples in flame and fire tests.

6.4 The specific combustion rate and combustion temperature of Method B are not generally reproducible because

sample geometry can affect the rate of surface oxidation and gas phase ignition can occur in the sample chamber at appropriate fuel/oxygen ratios. Reproducibility of Q_{max}^o and T_{max}^o are improved by using low oxygen concentration in the purge gas, small samples, and low heating rates in this test.

7. Apparatus

7.1 The equipment used in this test method shall be capable of displaying changes in combustion heat release rate as a function of specimen temperature during controlled heating and shall have the capability of subjecting the specimen to different atmospheres of oxygen concentration at ambient pressure.

7.2 Commercial thermogravimetric analyzers, pyrolysis probes, and electrically-heated ceramic tubes in thermal contact with a combustor, or attached gas analyzers, or both, have been found suitable. Detailed apparatus design criteria are given in Annex A1.

NOTE 2—In typical materials tests a material is exposed to a particular set of test conditions and the material's response to those particular test conditions is measured and reported as the test result. In these tests changing the test conditions has an effect on the result of the test. In this test, the heat release capacity (η_c) is independent of the test parameters as it is a material property and not a response of a material to a particular set of conditions. Thus, changing the test condition (within certain constraints) will have no effect on the test result. As such, the apparatus required to perform this test shall operate to provide test parameters that remain within certain constraints for each section of the device, for example, specimen chamber, mixing section, combustor. The diameter, length and shape of each section will have no effect on the test result provided the section meets the performance given in Annex A1.

7.3 Figure 1 illustrates the basic components of an apparatus, (1, 5, 8-11), satisfactory for this test method which include:

7.4 A *specimen chamber* (sample chamber) that is capable of holding and heating a small (milligram sized) specimen in a continuous flow of purge gas.

7.5 *Temperature controller*, capable of executing a temperature program that changes the specimen chamber temperature between ambient and 1123 K at a rate that is constant to within 5 % of the nominal value in the range 0.2-2 K/s.

7.6 A *means of purging the specimen chamber environment* with a constant flow of inert (for example, nitrogen) or reactive (nitrogen/oxygen mixture) gas at a rate of 50-100 cm³/min with an accuracy of ± 1 %.

7.7 A *temperature sensor*, to provide an indication of the specimen temperature to ± 0.5 K.

7.8 A *mixing chamber*, where the specimen and purge gases are mixed with sufficient oxygen to effect complete oxidation of the specimen gases in the combustion chamber.

7.9 A *means of introducing oxygen into the mixing section* at a constant flow rate of 0-50 cm³/min, such that the concentration of oxygen is between 20-50 % (± 0.1 %) by volume entering the combustion chamber.

7.10 A *combustor* (combustion chamber) capable of maintaining a constant temperature in the range of 1073-1273K (800-1000°C). Typically, the residence time in the combustor

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