



Designation: E711 – 23^{ε1}

Standard Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter¹

This standard is issued under the fixed designation E711; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

^{ε1} NOTE—Editorial corrections were made throughout in April 2023.

1. Scope

1.1 This test method covers the determination of the gross calorific value of a prepared analysis sample of solid forms of refuse-derived fuel (RDF) by the bomb calorimeter method.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 *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.* For specific cautionary and precautionary statements, see 6.9 and Section 8.

1.4 *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

[D1193 Specification for Reagent Water](#)

[D5681 Terminology for Waste and Waste Management](#)

[E1 Specification for ASTM Liquid-in-Glass Thermometers](#)

[E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Spe-](#)

[cialty Chemicals \(Withdrawn 2009\)](#)³

[E775 Test Methods for Total Sulfur in the Analysis Sample of Refuse-Derived Fuel](#)

[E790 Test Method for Residual Moisture in Refuse-Derived Fuel Analysis Samples](#)

[E829 Practice for Preparing Refuse-Derived Fuel \(RDF\) Laboratory Samples for Analysis](#)

3. Terminology

3.1 For definitions of general terms used in D34 Waste Management standards, refer to Terminology [D5681](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *calorific value*—the heat of combustion of a unit quantity of a substance. It may be expressed in joules per gram (J/g), British thermal units per pound (Btu/lb), or calories per gram (cal/g) when required.

3.2.1.1 *Discussion*—The unit equivalents are as follows:

1 Btu (International Table) = 1055.06 absolute joules

1 Calorie (International Table) = 4.1868 absolute joules

1 Btu/lb = 2.326 J/g

1.8 Btu/lb = 1.0 cal/g

3.2.2 *calorimeter*—describes the bomb, the vessel with stirrer, and the water in which the bomb is immersed.

3.2.3 *energy equivalent*—the energy required to raise the temperature (see 3.2.3.1) of the calorimeter system 1 °C (or 1 °F) per gram of sample. This is the number that is multiplied by the corrected temperature rise in degrees and divided by the sample weight in grams to give the gross calorific value after thermochemical corrections have been applied.

3.2.3.1 *Discussion*—Temperature change is measured in thermal units. Temperature changes may also be recorded in electromotive force, ohms, or other units when other types of temperature sensors are used. Consistent units must be used in both the standardization and actual calorific determination. Time is expressed in minutes. Weights are measured in grams.

3.2.4 *gross calorific value*—the heat produced by combustion of a unit quantity of solid fuel, at constant volume, in an

¹ This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01 on Monitoring and Characterization.

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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 The last approved version of this historical standard is referenced on www.astm.org.

oxygen bomb calorimeter under specified conditions such that all water in the products remains in liquid form.

3.2.5 *net calorific value*—a lower value calculated from the gross calorific value. It is equivalent to the heat produced by combustion of a unit quantity of solid fuel at a constant pressure of one atmosphere, under the assumption that all water in the products remains in the form of vapor.

3.2.6 *refuse-derived fuels*—solid forms of refuse-derived fuels from which appropriate analytical samples may be prepared are defined as follows in ASTM STP 832:⁴

RDF-1—Wastes used as a fuel in as-discarded form with only bulky wastes removed.

RDF-2—Wastes processed to coarse particle size with or without ferrous metal separation.

RDF-3—Combustible waste fraction processed to particle sizes, 95 % passing 2 in. square screening.

RDF-4—Combustible waste fraction processed into powder form, 95 % passing 10-mesh screening.

RDF-5—Combustible waste fraction densified (compressed) into the form of pellets, slugs, cubettes, or briquettes.

4. Summary of Test Method

4.1 Calorific value is determined in this method by burning a weighed analysis sample in an oxygen bomb calorimeter under controlled conditions. The calorific value is computed from temperature observations made before and after combustion, taking proper allowance for thermometer and thermochemical corrections. Either isothermal or adiabatic calorimeter jackets may be used.

5. Significance and Use

5.1 The calorific value, or heat of combustion, is a measure of the energy available from a fuel. Knowledge of this value is essential in assessing the commercial worth of the fuel and to provide the basis of contract between producer and user.

6. Apparatus

6.1 *Test Room*—The apparatus should be operated in a room or area free of drafts that can be kept at a reasonably uniform temperature and humidity for the time required for the determination. The apparatus should be shielded from direct sunlight and radiation from other sources. Controlled room temperature and humidity are highly desirable.

6.2 *Oxygen Bomb*, constructed of materials that are not affected by the combustion process or products sufficiently to introduce measurable heat input or alteration of end products. If the bomb is lined with platinum or gold, all openings shall be sealed to prevent combustion products from reaching the base metal. The bomb shall be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There shall be no gas leakage during a test. The bomb shall be capable of withstanding a hydrostatic pressure test to 21 MPa (3000 psig) at room temperature without stressing any part beyond its elastic limit.

6.3 *Calorimeter*, made of metal (preferably copper or brass) with a tarnish-resistant coating and with all outer surfaces highly polished. Its size shall be such that the bomb will be completely immersed in water when the calorimeter is assembled. It shall have a device for stirring the water thoroughly and at a uniform rate, but with minimum heat input. Continuous stirring for 10 min shall not raise the calorimeter temperature more than 0.01 °C (0.02 °F) starting with identical temperatures in the calorimeter, room, and jacket. The immersed portion of the stirrer shall be coupled to the outside through a material of low heat conductivity.

6.4 *Jacket*—The calorimeter shall be completely enclosed within a stirred water jacket and supported so that its sides, top, and bottom are approximately 10 mm from the jacket walls. The jacket may be arranged so as to remain at constant temperature or with provisions for rapidly adjusting the jacket temperature to equal that of the calorimeter for adiabatic operation. It shall be constructed so that any water evaporating from the jacket will not condense on the calorimeter.

6.5 *Thermometers*—Temperatures in the calorimeter and jacket shall be measured with the following thermometers or combinations thereof:

6.5.1 *Beckmann Differential Thermometer*, having a range of approximately 6 °C in 0.01 °C subdivisions reading upward and conforming to the requirements for Thermometer 115 °C, as prescribed in Specification E1. Each of these thermometers shall be tested for accuracy against a known standard at intervals no larger than 1 °C over the entire graduated scale. The maximum difference between any two test points shall not be more than 0.02 °C.

6.5.2 *Calorimetric-Type Platinum Resistance Thermometer*, 25-, tested for accuracy against a known standard.

6.5.3 *Other Thermometers*—A high precision electronic thermometer employing balanced thermistors, or a quartz thermometer may be used, provided the temperature rise indication is accurate within ± 0.003 °C per 1 °C rise.

6.6 *Sample Holder*—Samples shall be burned in an open crucible of platinum, quartz, or acceptable base-metal alloy. Base-metal alloy crucibles are acceptable if after a few preliminary firings the weight does not change significantly between tasks.

6.7 *Firing Wire* shall be 100 mm of No. 34 B&S nickel-chromium alloy wire or 100 mm of No. 34 B&S iron wire. Equivalent platinum or palladium wire may be used provided constant ignition energy is supplied or measured, and appropriate corrections made.

6.8 *Firing Circuit*—A 6 to 16 V alternating or direct current is required for ignition purposes with an ammeter or pilot light in the circuit to indicate when current is flowing. A stepdown transformer connected to an alternating current lighting circuit or batteries may be used.

6.9 **CAUTION:** The ignition circuit switch shall be of momentary double-contact type, normally open, except when held closed by the operator. The switch should be depressed only long enough to fire the bomb.

⁴ *Thesaurus on Resource Recovery Terminology*, ASTM STP 832, ASTM International, 1983, p. 72.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type III, conforming to Specification D1193.

7.3 *Benzoic Acid, Standard (C₆H₅COOH)*—Use National Bureau of Standards SRM (Standard Reference Material) benzoic acid. The crystals shall be pelletized before use. Commercially prepared pellets may be used provided they are made from National Bureau of Standards benzoic acid. The value of heat of combustion of benzoic acid, for use in the calibration calculations, shall be in accordance with the value listed in the National Bureau of Standards certificate issued with the standard.

7.4 *Methyl Orange, Methyl Red, or Methyl Purple Indicator*—May be used to titrate the acid formed in the combustion. The indicator selected shall be used consistently in both calibrations and calorific determinations.

7.5 *Oxygen*, free of combustible matter. Oxygen manufactured from liquid air, guaranteed to be greater than 99.5 % pure, will meet this requirement. Oxygen made by the electrolytic process may contain a small amount of hydrogen, rendering it unfit without purification.

7.6 *Sodium Carbonate, Standard Solution (0.34 N)*—One millilitre of this solution should be equivalent to 20.0 J in the nitric acid (HNO₃) titration. Dissolve 18.02 g of anhydrous sodium carbonate (Na₂CO₃) in water and dilute to 1 L. The Na₂CO₃ should be previously dried for 24 h at 105 °C. The buret used for the HNO₃ titration shall be of such accuracy that estimations to 0.1 mL can be made. A more dilute standard solution may be used for higher sensitivity.

8. Precautions

8.1 Due to the origins of RDF in municipal waste, precautions should be observed when conducting tests on the samples. Recommended hygienic practices include use of gloves when handling RDF and washing hands before eating or smoking.

8.2 The following precautions are recommended for safe calorimeter operation:

8.2.1 The weight of solid fuel sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

8.2.2 Bomb parts should be inspected carefully after each use. Threads on the main closure should be checked frequently for wear. The bomb should be returned to the manufacturer occasionally for inspection and possibly proof of firing.

8.2.3 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety codes. Suitable reducing valves and adaptors for 2 to 3.5 MPa (300 to 500 psig) discharge pressure are obtainable from commercial sources of compressed gas equipment. The pressure gage shall be checked periodically for accuracy.

8.2.4 During ignition of a sample, the operator shall not permit any portion of his body to extend over the calorimeter.

9. Sampling⁶

9.1 RDF products are frequently nonhomogeneous. Thus, significant care is needed to obtain a representative laboratory sample for the RDF lot to be characterized.

9.2 The sampling method for this procedure should be based on agreement between the involved parties.

9.3 The laboratory sample must be air dried and particle size reduced to pass a 0.5 mm screen as described in Practice E829.

10. Standardization

10.1 Determine the energy equivalent of the calorimeter as the average of a series of ten individual runs, made over a period of not less than three days or more than five days. To be acceptable, the standard deviation of the series shall be 6.9 kJ/°C (6.5 Btu/°C) or less (see Appendix X1, Table X1.1). For this purpose, any individual run may be discarded only if there is evidence indicating incomplete combustion. If this limit is not met, repeat the entire series until a series is obtained with a standard deviation below the acceptable limit.

10.2 The weights of the pellets of benzoic acid in each series should be regulated to yield the same temperature rise as that obtained with the various samples tested in the individual laboratories. The usual range of weight is 0.9 to 1.3 g. Make each determination in accordance with the procedure described in Section 11, and compute the corrected temperature rise, *T*, as described in 12.1. Determine the corrections for HNO₃ and firing wire as described in 12.2 and substitute into the following equation:

$$E = [(H)(g) + e_1 + e_3 + e_4] \times t \quad (1)$$

where:

E = energy equivalent, J/°C,

H = heat of combustion of benzoic acid, as stated in the National Bureau of Standards certificate, J/g,

g = weight of benzoic acid, g,

t = corrected temperature rise, °C,

*e*₁ = titration correction, J,

⁵ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁶ ASTM Subcommittee E38.01 is currently in the process of developing procedures for sampling RDF-3 and the preparation of an analysis sample. The chairman of E38.01 should be contacted for details.

e_3 = fuse wire correction, J, and
 e_4 = correction for ignition energy if measured and corrected for, J.

10.3 Standardization tests should be repeated after changing any part of the calorimeter and occasionally as a check on both calorimeter and operating technique.

11. Procedure

11.1 *Weight of Sample*—Thoroughly mix the analysis sample of solid fuel in the sample bottle, taking care that the heavies and lights (fluff) are distributed in the sample (Note 1). Carefully weigh approximately 1 g of the sample directly into the crucible in which it is to be burned or into a tared weighing scoop from which the sample is transferred to the crucible. Weigh the sample to the nearest 0.1 mg. Some form of compaction may be necessary to ensure satisfactory ignition and complete combustion.

NOTE 1—In the event segregation of the heavies and lights cannot be avoided, attempt to remove sample from the bottle in such a way that a representative sample is transferred.

11.2 *Water in Bomb*—Add 1.0 mL of water to the bomb by a pipet. Before adding this water, rinse the bomb, drain the excess water, and leave undried.

11.3 *Firing Wire*—Connect a measured length of firing wire to the ignition terminals with enough slack to allow the firing wire to maintain contact with the sample.

11.4 *Oxygen*—Charge the bomb with oxygen to a consistent pressure between 2.03 and 3.04 MPa (20 and 30 atm). This pressure must remain the same for each calibration and for each calorific determination. If, by accident, the oxygen introduced into the bomb should exceed the specified pressure, do not proceed with the combustion. Detach the filling connection and exhaust the bomb in the usual manner. Discard this sample.

11.5 *Calorimeter Water*—It is recommended that calorimeter water temperature be adjusted before weighing as follows:

11.5.1 *Isothermal Jacket Method*, 1.6 to 2.0 °C (3.0 to 3.5 °F) below jacket temperature (Note 2).

NOTE 2—Perform the residual moisture determination of the sample simultaneously using Test Method E790.

11.5.2 *Adiabatic Jacket Method*, 1.0 to 1.4 °C (2.0 to 2.5 °F) below room temperature.

NOTE 3—This initial adjustment will ensure a final temperature slightly above that of the jacket for calorimeters having an energy equivalent of approximately 10 200 J/K (2450 cal/°C). Some operators prefer a lower initial temperature so that the final temperature is slightly below that of the jacket. This procedure is acceptable provided it is used in all tests, including standardization. Use the same amount (± 0.5 g) of water in the calorimeter vessel for each test and for calibration. The amount of water (2000 g is usual) can be most satisfactorily determined by weighing the calorimeter vessel and water together on a balance. The water may be measured volumetrically if it is always measured at the same temperature. Tap water may be satisfactory for use in calorimeter bucket.

11.6 *Observations, Isothermal Jacket Method*—Assemble the calorimeter in the jacket and start the stirrer. Allow 5 min for attainment of equilibrium, then record the calorimeter temperatures (Note 4) at 1 min intervals for 5 min. Fire the

charge at the start of the sixth minute and record the time and temperature, T_a . Add to this temperature 60 % of the expected temperature rise and record the time at which the 60 % point is reached (Note 3). After the rapid rise period (about 4 to 5 min), record temperatures at 1 min intervals on the minute until the difference between successive readings has been constant for 5 min.

NOTE 4—Use a magnifier and estimate all readings (except those during the rapid rise period) to the nearest 0.002 °C (0.005 °F) when using an ASTM Bomb Calorimeter Thermometer 56C (56F). Estimate Beckmann thermometer readings to the nearest 0.001 °C.

NOTE 5—When the approximate expected rise is unknown, the time at which the temperature reaches 60 % of the total can be determined by recording temperatures at 45, 60, 75, 90, and 105 s after firing and interpolating.

11.7 *Observations, Adiabatic Jacket Method*—Assemble the calorimeter in the jacket and start the stirrer. Adjust the jacket temperature to be equal to or slightly lower than the calorimeter and run for 5 min to obtain equilibrium. Adjust the jacket temperature to match the calorimeter with ± 0.01 °C (0.02 °F) and hold for 3 min. Record the initial temperature (Note 4) and fire the charge. Adjust the jacket temperature to match that of the calorimeter during the period of rise, keeping the two temperatures as nearly equal as possible during the rapid rise, and adjusting to within ± 0.01 °C (0.02 °F) when approaching the final equilibrium temperature. Take calorimeter readings at 1 min intervals until the same temperature is observed in three successive readings. Record this as the final temperature. Do not record time intervals since they are not critical in the adiabatic method.

11.8 *Analysis of Bomb Contents*—Remove the bomb and release the pressure at a uniform rate, in such a way that the operation will require not less than 1 min. Examine the bomb interior and discard the test if unburned sample or sooty deposits are found. Carefully wash the interior of the bomb including the capsule with distilled or deionized water containing the titration indicator until the washings are free of acid. Collect the washings in a beaker and titrate the washings with standard carbonate solution. Remove and measure or weigh the combined pieces of unburned firing wire and subtract from the original length or weight to determine the wire consumed in firing. Determine the sulfur content of the sample by any of the procedures described in Test Methods E775.

12. Calculation

12.1 *Temperature Rise in Isothermal Jacket Calorimeter*—Using data obtained as prescribed in 11.6, compute the temperature rise, T , in an isothermal jacket calorimeter as follows:

$$T = T_c - T_a - r_1(b - a) - r_2(c - b) \quad (2)$$

where:

T = corrected temperature rise,

a = time of firing,

b = time (to nearest 0.1 min) when the temperature rise reaches 60 % of total,

c = time at beginning of period in which the rate of temperature change with time has become constant (after combustion),

- T_a = temperature at time of firing, corrected for thermometer error (Note 5),
- T_c = temperature at time c , corrected for thermometer error (Note 5),
- r_1 = rate (temperature units per minute) at which temperature was rising during 5 min period before firing, and
- r_2 = rate (temperature units per minute) at which temperature was rising during the 5 min period after time c . If the temperature is falling, r_2 is negative and the quantity $r_2(c - b)$ is positive.

12.2 *Temperature Rise in Adiabatic Jacket Calorimeter*—Using data obtained as prescribed in 11.7, compute the corrected temperature rise, T , as follows:

$$T = T_f - T_a \quad (3)$$

where:

- T = corrected temperature rise, °C or °F,
- T_a = initial temperature when charge was fired, corrected for thermometer error, and
- T_f = final temperature corrected for thermometer error.

12.3 *Thermochemical Corrections* (Appendix X3)—Compute the following for each test:

- e_1 = correction for the heat of formation of HNO_3 , J. Each millilitre of standard alkali is equivalent to 20.0 J.
- e_2 = correction for heat of formation of H_2SO_4 , J.
= $55.2 \times$ percent of sulfur in sample \times weight of sample, g.
- e_3 = correction for heat of combustion of firing wire, J (Note 6).
= 9.6 J/cm or 5980 J/g for No. 34 B&S gage Chromel C.
= 11.3 J/cm or 7330 J/g for No. 34 B&S iron wire.
- e_4 = correction for ignition energy of platinum or palladium if measured and corrected for.

NOTE 6—There is no correction for platinum or palladium wire, provided the ignition energy is constant.

12.4 *Calorific Value:*

12.4.1 Calculate the gross calorific value (gross heat of combustion) as follows:

$$H_s = [(T)(E) - e_1 - e_2 - e_3 - e_4] / g \quad (4)$$

where:

- H_s = gross calorific value, J/g,
- T = corrected temperature rise as calculated in 12.1 or 12.2, °C or °F, consistent with the water equivalent value,
- E = energy equivalent (see Section 10), e_1, e_2, e_3, e_4 = corrections as prescribed in 12.3, and
- g = weight of sample, g.

12.4.2 Calculate the net calorific value (net heat of combustion) as follows:

$$H_i = H_s - 23.96 (H \times 9) \quad (5)$$

where:

- H_i = net calorific value (net heat of combustion), J/g,
- H_s = gross calorific value (gross heat of combustion), J/g,
- H = total hydrogen, %, and
- 23.96 = benzoic acid ($\text{C}_6\text{H}_5\text{CO}_2\text{H}$) is the standard (that is, 23.96) used for this calculation and as calculated by a standard's reported value or as calculated by user.

13. Precision and Bias⁷

13.1 *Precision*—The standard deviations of individual determinations, in Btu/lb, are:

	Within laboratory	Between laboratories
Average HHV-1:		
6400	27.1	135.5
5200	48.8	239.6
HHV-2:		
7900	32.3	118.0
7400	38.1	227.8
HHV-3:		
9700	111.3	290.4
9500	99.2	249.2
9300	40.3	67.6

13.2 These precision estimates are based on an interlaboratory study conducted in accordance with Practice E180.

⁷ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E38-1000. Contact ASTM Customer Service at service@astm.org.

APPENDIXES

(Nonmandatory Information)

X1. CALCULATION OF STANDARD DEVIATIONS FOR CALORIMETER STANDARDIZATION

X1.1 The example given in Table X1.1 illustrates the method of calculating standard deviations for calorimeter standardizations.