Standard Test Method for Carbon and Hydrogen in the Analysis Sample of Refuse-Derived Fuel[1]

This standard is issued under the fixed designation E 777; 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 covers the determination of total carbon and hydrogen in a sample of refuse-derived fuel (RDF). Both carbon and hydrogen are determined in one operation. This test method yields the total percentages of carbon and hydrogen in RDF as analyzed and the results include not only the carbon and hydrogen in the organic matter, but also the carbon present in mineral carbonates and the hydrogen present in the free moisture accompanying the analysis sample as well as hydrogen present as water of hydration.

NOTE 1—It is recognized that certain technical applications of the data derived from this test procedure may justify additional corrections. These corrections could involve compensation for the carbon present as carbonates, the hydrogen of free moisture accompanying the analysis sample, and the calculated hydrogen present as water of hydration.

1.2 This test method may be applicable to any waste material from which a laboratory analysis sample can be prepared.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 1193 Specification for Reagent Water

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals

E 790 Test Method for Residual Moisture in a Refuse-Derived Fuel Analysis Sample

E 791 Test Method for Calculating Refuse-Derived Fuel Analysis Data from As-Determined to Different Bases

E 829 Practice for Preparing Refuse-Derived Fuel (RDF) Laboratory Samples for Analysis

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *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:*[3]

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-inch 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 The determination is made by burning the sample to convert all of the carbon to carbon dioxide and all of the hydrogen to water. The combustion is carried out by high purity oxygen that has been passed through a purifying train. The carbon dioxide and water are recovered in an absorption train. The combustion tube packing is used to remove any interfering substances. This test method gives the total percentages of carbon and hydrogen in the RDF as analyzed, including the carbon in carbonates and the hydrogen in any form of water.

5. Significance and Use

5.1 The standard sample is available to producers and users of RDF as a method of determining the weight percent of carbon and hydrogen in the analysis sample.

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[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.06 on Recovery and Reuse.

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

5.2 Carbon and hydrogen are part of the ultimate analysis of a fuel and can be used for calculations of combustion parameters.

6. Apparatus

6.1 *Oxygen-Purifying Train*—The high-purity oxygen is passed through water and carbon dioxide absorbers prior to use for combustion. The oxygen-purifying train consists of the following three units in order of passage of oxygen (see Fig. 1):

6.1.1 *First Water Absorber*—A container constructed so that the oxygen must pass through a column of reagent. The container shall have a capacity for at least 45 cm^3 of solid reagent, and the minimum gas travel through the reagent shall be at least 80 mm. A container of large volume and long path of oxygen travel through the reagent will be found to be advantageous where many carbon and hydrogen determinations are made.

6.1.2 *Carbon Dioxide Absorber*—If solid reagents are used for carbon dioxide absorption, the container shall be as described in 6.1.1. If a solution is used, the container shall be a Vanier bulb. It shall provide a column of reagent adequate to remove the carbon dioxide completely.

6.1.3 *Second Water Absorber*—Same as specified in 6.1.1.

6.2 *Flowmeter*, used to permit volumetric measurement of the rate of flow of oxygen during the determination. It shall be suitable for measuring flow rates within the range from 50 to 100 mL/min (standard temperature and pressure). The use of a double-stage pressure-reducing regulator with gage and needle valve is recommended to permit easy and accurate adjustment to the rate of flow.

6.3 *Combustion Unit*, consisting of three electrically heated furnace sections, individually controlled, which may be mounted on rails for easy movement. The upper part of each furnace may be hinged so that it can be opened for inspection of the combustion tube. The three furnace sections shall be as follows (see Fig. 1):

6.3.1 *Furnace Section 1*—Furnace 1 is nearest the oxygen inlet end of the combustion tube, approximately 130 mm long and used to heat the inlet end of the combustion tube and the sample. It shall be capable of rapidly attaining an operating temperature of 875 ± 25°C.

NOTE 2—Combustion tube temperature shall be measured by means of a thermocouple placed immediately adjacent to the tube near the center of the appropriate tube section.

6.3.2 *Furnace Section 2*—Furnace 2 shall be approximately 330 mm in length and used to heat that portion of the tube filled with cupric oxide. The operating temperature shall be 850 ± 20°C (see Note 2).

6.3.3 *Furnace Section 3*—Furnace 3 shall be approximately 230 mm long, and used to heat that portion of the tube filled with lead chromate or silver. The operating temperature shall be 500 ± 50°C.

6.3.4 *Combustion Tube*, made of fused quartz, or high-silica glass and having a nominal inside diameter which may vary within the limits of 19 to 22 mm and a minimum total length of 970 mm. The exit end shall be tapered down to provide a tubulated section for connection to the absorption train. The tubulated section shall have a length of 20 to 25 mm, an internal diameter of not less than 3 mm, and an external diameter of approximately 7 mm. The total length of the reduced end shall not exceed 60 mm. If a translucent fused quartz tube is used, a transparent section 190 mm long, located 250 mm from the oxygen inlet end of the tube, will be found convenient (see Fig. 2).

6.3.5 *Combustion Boat*, made of glazed porcelain, fused silica, or platinum. Boats with internal dimensions of approximately 70 by 8 by 8 mm have been found convenient.

6.4 *Absorption Train*, identical to the oxygen absorption train indicated in 5.1 to obtain system equilibrium. Therefore, the absorption train shall consist of the following units arranged as listed in the order of passage of oxygen (see Fig. 1):

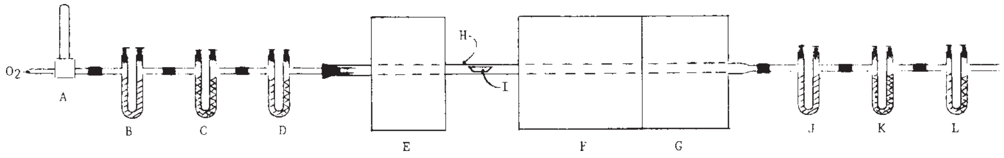
6.4.1 *First Water Absorber*, as described in 5.1.1.

6.4.2 *Carbon Dioxide Absorber*, as described in 6.1.2.

6.4.3 *Second Water Absorber*, as described in 6.1.3. The second water absorber is also known as a guard tube.

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 American



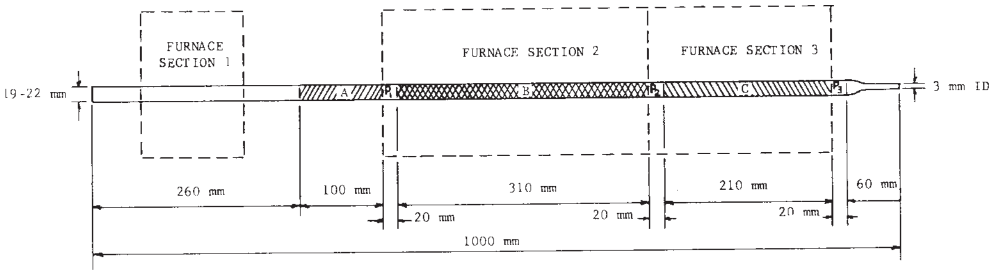
A = flowmeter (6.2)
B, C, D = oxygen purifying train (6.1)
B = first water absorber (6.1.1)
C = carbon dioxide absorber (6.1.2)
D = second water absorber (6.1.3)

E, F, G, H, I = combustion unit (6.3)
E = furnace 1 (6.3.1)
F = furnace 2 (6.3.2)
G = furnace 3 (6.3.3)
H = combustion tube (6.3.4)
I = combustion boat (6.3.5)

J, K, L = absorption train (6.4)
J = first water absorber (6.4.1)
K = carbon dioxide absorber (6.4.2)
L = guard tube (6.4.3)

NOTE 1—*C* and *K* can substitute a Vanier bulb if liquid absorbent is used.

FIG. 1 Set-Up of Apparatus



A =clear fused quartz section (optional when a translucent quartz tube is used)
B =cupric oxide filling
C =lead chromate or silver gauze filling
P_1, P_2, P_3 =oxidized copper gauze plugs

NOTE 1—When furnace sections longer than those specified in 6.3 are to be used, changes in the above dimensions shall be in accordance with provisions of Note 5.

FIG. 2 Arrangement of Tube Filling for Combustion Tube

Chemical Society, where such specifications are available.[4] 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 D 1193.

7.3 *Oxygen*, with minimum acceptable purity 99.5 %.

NOTE 3—If the blank tests for flow (see 10.3.2) indicate interfering impurities in the oxygen supply by consistent weight-gain in the absorbers, eliminate these impurities by using a preheater furnace and tube, filled with cupric oxide. Operate this preheater at 850 ± 20°C and insert in series between the supply tank of oxygen and the purification train.

7.4 *Combustion Tube Reagents*:

7.4.1 *Cupric Oxide* (CuO), wire form, dust-free.

7.4.2 *Fused Lead Chromate* ($PbCrO_4$), approximately 2.38 to 0.84 mm in size.

7.4.3 *Silver Gauze*, 99.9 % silver minimum purity, 0.84 mm, made from approximately No. 27 B&S gage wire.

7.4.4 *Copper Gauze*, 99.0 % copper minimum purity, 0.84 mm, made from approximately No. 26 B&S gage wire.

7.5 *Purification and Absorption Train Reagents*:

7.5.1 *Water Absorbent*—Anhydrous magnesium perchlorate ($Mg(ClO_4)_2$) approximately 2.38 to 0.35 mm in size.[5]

7.5.2 *Carbon Dioxide Absorbent*—If a solid reagent is used, it shall be sodium or potassium hydroxide (NaOH or KOH) impregnated in an inert carrier approximately 2.38 to 0.84 mm in size. Use of soda lime in place of the above or in admixture with them is permissible (Note 4). If a solution is used, it shall be 30 weight % potassium hydroxide (KOH).

NOTE 4—Acceptable carbon dioxide absorbing reagents using sodium or potassium hydroxide are sold under the tradenames: Ascarite, Caroxite, and Mikohbite. If soda lime is used in admixture with any of the foregoing, it should not exceed 30 weight % of the total reagent. In using Ascarite it may be necessary to add a few drops of water to this reagent to assure complete absorption of carbon dioxide.

8. Precautions

8.1 Due to the origins of RDF in municipal waste, common sense dictates that some precautions should be observed when conducting tests on the samples. Recommended hygienic practices include use of gloves when handling RDF; wearing dust masks (NIOSH-approved type), especially while milling RDF samples; conducting tests under a negative pressure hood when possible; and washing hands before eating or smoking.

9. Sampling

9.1 RDF products are frequently nonhomogeneous. For this reason significant care should be exercised to obtain a representative laboratory sample from 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 E 829. This procedure must be performed carefully to preserve the sample's representative characteristics (other than particle size) while preparing the analysis sample to be used in the procedures.

10. Preparation of Apparatus

10.1 *Combustion Tube Packing*—To ensure complete oxidation of combustion products and complete removal of interfering substances such as oxides of sulfur, the combustion tube shall be packed with cupric oxide and lead chromate or silver gauze. The arrangement and lengths of the tube fillings and separating plugs shall be as shown in Fig. 2 (see Note 5). It is recommended that the tube be placed in a vertical position (constricted end downward) for packing. When filling the tube with lead chromate, any residual reagent adhering to the walls of the empty portion of the tube must be removed. When silver gauze is used as a tube filling, the required length of filling may be prepared conveniently from three or four strips 150 to 200 mm in length, by rolling each strip into a cylindrical plug and inserting the strips end-to-end in the tube.

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[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia".

[5] Tradenames of this reagent are Anhydrone or Dehydrite.

NOTE 5—Longer furnaces with appropriate lengths of tube packing will be satisfactory.

10.2 *Purification and Absorption Trains*:

10.2.1 *Water Absorbers*—Fill a container, described in 6.1.1, with a permissible solid desiccant, as described in 7.5.1, by adding the required amount in small portions and settling each portion by gently tapping between additions. Place a glass wool plug between the reagent and absorber outlet to prevent loss of reagent dust.

10.2.2 *Carbon Dioxide Absorbers*—If a solid reagent is used for the retention of carbon dioxide, 7.5.2, fill the absorber, 6.1.2, as described in 10.2.1. Place a layer or cap of desiccant in the outlet section of the container; it shall be the same as that used in the water absorber. This layer shall have a bulk volume not less than one fourth nor more than onethird of the combined volume of both reagents.

10.2.2.1 If a liquid absorbent is used, fill the inner tube of the Vanier bulb with the same desiccant used in the water absorber. If a solid absorbent is used, place a glass wool plug in the outlet section of the container to prevent loss of reagent dust.

10.2.3 *Guard Tube*—Pack a container, as described in 5.1.1, with equal volumes of the water absorbent and a solid carbon dioxide absorbent.

10.2.4 *Connections*—To ensure a closed system from the supply tank of oxygen to the guard tube at the end of the absorption train, it is recommended that all connections be glass-to-glass or glass-to-quartz butt joints with short lengths of flexible tubing as seals. The connection between the purification train and the combustion tube may be made by means of a rubber stopper or other suitable device. All connections shall be gas tight. No lubricant shall be used for making tubing connections in the absorption train.

10.3 *Conditioning of Apparatus*.

10.3.1 *Newly Packed Combustion Tube*—Burn a sample of RDF as described in 11.4 except that the products of combustion need not be fixed in a weighed absorption train.

10.3.2 *Used Combustion Tube*—After any extended shut down (one day or more) test the combustion train under procedure conditions, but without burning a sample, for 40 min with weighed absorbers connected. A variation of not more than 0.5 mg of both water and carbon dioxide absorbers shall be considered satisfactory (see Note 3).

10.3.3 *Absorption Train*—Condition freshly packed absorbers and guard tubes by burning a sample of RDF, as described in 11.4, except that the absorber weights need not be determined.

10.3.4 Make standard checks frequently, particularly when intermittent use of the combustion train is common or when any changes have been made in the system. Burn a standard substance of certified analysis, such as benzoic acid or sucrose as furnished by the National Bureau of Standards, as described in Section 11. A variation from the theoretical of not more than 0.07 % for hydrogen nor more than 0.30 % for carbon shall be considered satisfactory.

11. Procedure

11.1 After the combustion tube and absorbers have been conditioned as prescribed in Section 10, conduct the test as follows:

11.2 *Absorption Train*—Bring the water and carbon dioxide absorbers to room temperature near the balance for 15 to 20 min, vent momentarily to the atmosphere, wipe with a chamois or lint-free cloth in the areas where handled, and weigh the water and carbon dioxide absorbers to the nearest 0.1 mg.

11.3 *Sample*—After thoroughly mixing the RDF analysis sample to provide the best possible mix of heavy fines with milled fluff, weigh approximately 0.2 g to the nearest 0.1 mg of sample into a combustion boat.

NOTE 6—The final milling stage may produce a suitably mixed analysis sample without further mixing. It is important that no unwanted segregation of particle sizes take place. True representation of the analysis sample must be contained in the 200 mg used for the analysis.

11.4 *Sample Analysis*—With furnace 2 and 3 at specified temperatures and positioned as shown in Fig. 2, perform the following operations in rapid succession in the order listed:

11.4.1 If a conventional type of sample heating furnace is used for heating furnace 1, place it so that its left-hand edge is about 100 mm from the oxygen inlet end of the combustion tube.

11.4.2 Attach the weighed absorption train to the combustion tube.

11.4.3 Push the sample boat containing the weighed sample into the combustion tube to a point within approximately 20 mm from plug P_1 (see Fig. 2).

11.4.4 Close the tube and adjust the oxygen flow to a rate of 50 to 100 mL/min (standard temperature and pressure), being the same as used in blanking (see 10.3.2).

11.4.5 Apply full heat to heating Furnace 1 to bring it to an operating temperature of 850 to 900°C as rapidly as possible. Move the heater slowly toward the boat so that it completely covers the boat and is brought into contact with furnace 2 over a period of 10 to 20 min (Note 7). Allow it to remain in this position for an additional 5 to 10 min, and then shut off the heat and return the sample heater to its original position. Continue the flow of oxygen through the system for 10 min (Note 9), close the absorbers under a positive pressure of oxygen and detach them from the train. Remove the absorbers to the vicinity of the balance, allow them to cool to room temperature for 15 to 20 min, vent momentarily to the atmosphere, wipe them with a chamois or lint-free cloth in the areas handled, and finally weigh them to the nearest 0.1 mg. While the absorbers are cooling, it is recommended that the ash remaining in the combustion boat be examined for tracers of unburned carbon which, if present, will nullify the determination.

NOTE 7—Some variation in operating technique and heater manipulation may be permitted at the discretion of the analyst, provided that it is conducive to a gradual and controlled release of volatile matter. Conditions that lead to visible burning (flame combustion) of the sample shall be avoided.

NOTE 8—Since water may condense in the cooler outlet end of the combustion tube or in the inlet arm of the water absorber, the use of an external or internal heat-conducting device (a metal heat bridge) is recommended to prevent such condensation or promote reevaporation during this flushing period.

12. Calculation

12.1 Calculate the percentage of carbon in the analysis sample as follows:

$$Carbon, \% = \frac{A}{B} \times 27.289 \quad (1)$$

where:

A = increase in weight of CO_2 absorber, g,
B = grams of sample used, and
27.289 = percentage of carbon in CO_2.

12.2 Calculate the percentage of hydrogen in the analysis sample as follows (see Note 7).

$$Hydrogen, \% = \frac{C}{B} \times 11.19 \quad (2)$$

where:

C = increase in weight of water absorber, g,
B = grams of sample used, and
11.19 = percentage of hydrogen in water.

NOTE 9—The water absorbed in the water absorber includes not only water formed as a product of combustion, but also free water (moisture) in the sample and water of hydration of materials that may be contained in RDF.

13. Report

13.1 The results of the carbon and hydrogen analysis may be reported on any of a number of bases, differing from each other in the manner by which moisture is treated.

13.2 The numerical moisture value established by Test Method E 790 shall be used for converting carbon and hydrogen data on the as-determined basis to the dry basis as in Test Method E 791.

14. Precision and Bias [6]

14.1 *Precision*:

14.1.1 The standard deviations of individual determinations, in percent absolute, are as follows:

Typical Average Value, %	Within-Laboratory, %	Between-Laboratories, %
Carbon:		
40	0.5	1.6
Hydrogen:		
5.4	0.2	0.5

14.1.2 These precision estimates are based on an interlaboratory study conducted in accordance with Practice E 180.

14.2 *Bias*—The bias of this test method has not been determined because of the lack of a recognized standard reference material.

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[6] Supporting data are available on loan from ASTM Headquarters. Request RR:E38-1000.