



Designation: E 191 – 64 (Reapproved 1999)^{ε1}

Standard Specification for Apparatus For Microdetermination of Carbon and Hydrogen in Organic and Organo-Metallic Compounds¹

This standard is issued under the fixed designation E 191; 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.

^{ε1} NOTE—Keywords were added editorially in March 2000.

1. Scope

1.1 This specification covers apparatus and basic equipment for the determination of carbon and hydrogen in organic and organometallic compounds along the lines of the conventional method of Pregl, but with modifications more in line with modern practice. Owing to the diversity of apparatus by which correct results can be obtained, this specification is intended to indicate what is acceptable rather than what is mandatory.

NOTE 1—Specifications for several items subsequently listed were developed by the Committee for the Standardization of Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society.²

1.2 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

2. Referenced Documents

2.1 ASTM Standards:

E 124 Specification for Weighing and Drying Apparatus for Microchemical Analysis³

E 148 Specification for Apparatus for Microdetermination of Nitrogen by Dumas Method⁴

3. Oxygen Supply

3.1 Any cylinder or other suitable source of oxygen free of hydrogen and organic impurities may be used. Oxygen prepared from liquid air is satisfactory.

¹ This specification is under the jurisdiction of ASTM Committee E-41 on Laboratory Apparatus and is the direct responsibility of Subcommittee E41.01 on Apparatus.

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² Committee for the Standardization of Microchemical Apparatus. Division of Analytical Chemistry, American Chemistry Society, 1949 Report on Recommended Specifications for Microchemical Apparatus, Carbon-Hydrogen, Dumas Nitrogen, Sulfur, and Halogen, *Analytical Chemistry* Vol 21, p. 1555 (1949).

³ *Annual Book of ASTM Standards*, Vol 14.04.

⁴ Discontinued, see 1987 *Annual Book of ASTM Standards*, Vol 14.02.

4. Pressure Regulator

4.1 The pressure regulator, shown in Fig. 1 and Fig. 2,⁵ consisting of a bell and inlet tube submerged in a suitable confining liquid, shall be used to supply air or oxygen, or both, at constant pressure to the combustion system. Substitution of a fine gas-control valve and flowmeter for the pressure regulator is optional.

5. Drying and Purifying Tube²

5.1 The conventional bubble-container U-tube shown in Fig. 3 shall be used for drying and purifying the entering air or oxygen, or both.

6. Flowmeter

6.1 The rate of gas flow shall be determined by the bubble counter of the bubble-counter U-tube, or by a more accurate flow-meter, such as the floating ball-in-column.⁶

7. Combustion Unit

7.1 The combustion unit shall consist of a long furnace, a sample furnace, a constant-temperature heating mortar, a combustion tube, and a combustion-tube closure.

7.1.1 Long Furnace:

7.1.1.1 The long furnace shall have a maximum over-all length of 8 in. (203 mm) with the wall thickness at the ends not to exceed ¼ in. (6 mm). The furnace shall accommodate combustion tubes up to 13 mm in outside diameter. Electric heating elements shall be easily replaceable. The furnace shall be mounted firmly on a substantial support.

7.1.1.2 The furnace shall be capable of continuous operation at temperatures up to 900°C as measured inside the combustion tube at the middle of the furnace. The temperature drop from the center to points 1 in. (25 mm) and 1 ¾ in. (45 mm) from

⁵ Furter, M. F., and Steyermark, A., "Pressure Regulator for Use in Microdetermination of Carbon and Hydrogen," *Analytical Chemistry*, Vol 20, 1948, p. 257.

⁶ Kuck, J. A., and Altieri, P. L., "Spherical Ground-Glass Joints vs. Rubber Tubing Connectors on the C and H Absorption Train," *Mikrochimica Acta*, 1956, p. 1556.

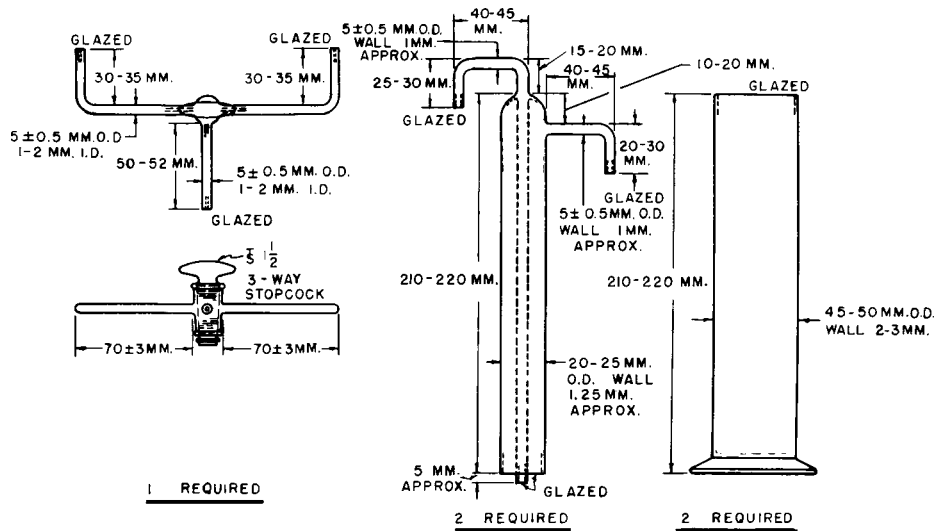


FIG. 1 Pressure Regulator, Glass Parts

either end shall not exceed 15 and 7 %, respectively, based upon the temperature in the middle of the furnace.⁷ Means shall be provided for varying the temperature.

7.1.1.3 The furnace shall be equipped with some device for indicating the temperature at the middle of the furnace.

7.1.2 *Sample Furnace, Traveling Type (Note 2) :*

7.1.2.1 The sample furnace shall have an over-all length not less than 2½ in. (64 mm) nor more than 4 in. (102 mm) with wall thickness at the ends not to exceed ¼ in. (6 mm). The furnace shall accommodate combustion tubes up to 13 mm in outside diameter. Electric heating elements shall be easily replaceable. The furnace shall be mounted firmly on a substantial support.

7.1.2.2 The sample furnace shall be capable of continuous operation up to 900°C as measured inside the combustion tube at the middle of the furnace. The temperature drop from the center to points ¾ in. (19 mm) from either end shall not exceed 17 % of the temperature at the middle of the furnace.⁶ Means shall be provided for varying the temperature.

7.1.2.3 The sample furnace shall be designed so that it may be either moved away from the combustion tube or turned off and cooled rapidly. In the latter case, the time for cooling to room temperature and reheating to operating temperature shall not exceed 30 min.

7.1.2.4 The sample furnace shall be equipped with some device for indicating the temperature at the middle of the furnace.

7.1.2.5 If the furnace is moved mechanically, it shall travel a minimum distance of 6¼ in. (159 mm) with provision for

manual setting at any point. Whether the furnace has only one or more than one speed of movement over this distance, the rates of travel shall be within the limits of ⅛ to ⅝ in. (3 to 16 mm)/min. An automatic control shall be provided to stop the travel of the sample furnace when it reaches the long furnace.

NOTE 2—The stationary multiple-section type of sample furnace may be used instead of the traveling type, which differs from the traveling type as follows: It consists of not less than three sections with a maximum length of 7 in. (178 mm) and a maximum wall thickness of ¼ in. (6 mm) at the ends of any section. Each section shall be provided with its individual adjustable time switch, which can be set to go on or off automatically for any time from 0 to 30 min. Provision shall be made for at least two sequences of heating of the various sections. Each section, when operated alone, shall be capable of continuous operation at temperatures up to 900°C as measured inside the combustion tube at the middle of the section.

7.1.3 *Heating Mortar*—Any type of heating mortar² may be used provided it is capable of maintaining constant temperature to within ±1°C in the range from 175 to 190°C. If electric, the elements shall be easily replaceable. It shall accommodate combustion tubes of 13 mm in outside diameter. Means shall be provided for heat transfer to the inlet capillaries of the water absorption tube to prevent condensation of water.

7.1.4 *Combustion Tube*—The combustion tube shall have a tip and side arm. Depending upon the length of furnace assembly used, the tube shall be either as specified in Fig. 4 of Specification E 148 with a body length of 520 to 530 mm,² or of similar construction with a body length of 620 to 630 mm.

7.1.5 *Combustion Tube Closure*—A solid stopper of rubber or suitable substitute, such as cork, neoprene, etc., with dimensions as specified in Fig. 5 of Specification E 148, shall be used.

⁷ Several types of commercially available electric furnaces meet these requirements.

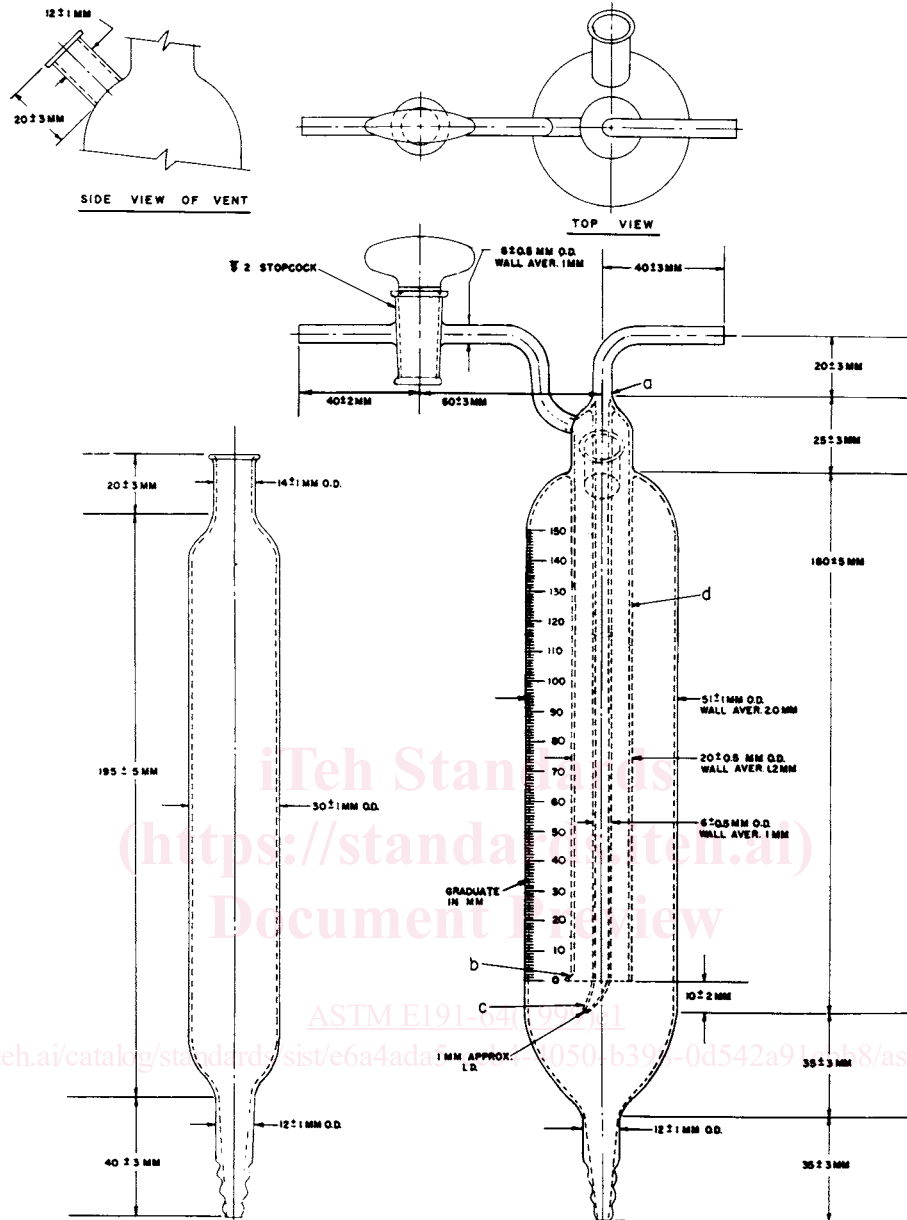


FIG. 2 Pressure Regulator, Details of Construction

8. Absorption Tubes²

8.1 Absorption tubes, preferably of soda-lime glass, shall be used. Two suitable sizes are shown in Fig. 4.

9. Guard Tube²

9.1 The guard tube shall be as shown in Fig. 5.

10. Mariotte Bottle²

10.1 A Mariotte bottle, as shown in Fig. 6, shall be used with the pressure regulator described in Section 4.

11. Weighing Accessories

11.1 Weighing accessories needed in the microdetermination of carbon and hydrogen are described in Specification E 124.

12. Keywords

12.1 carbon; compounds; hydrogen; microdetermination