Designation: E 148 – 66 (Reapproved 1981)

Standard Specification for
APPARATUS FOR MICRODETERMINATION OF NITROGEN BY DUMAS METHOD[1]

This standard is issued under the fixed designation E 148; 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 specification covers apparatus used for the microdetermination of nitrogen by the Dumas method, including carbon-dioxide supply, gas-control unit, combustion unit, and measuring apparatus.

NOTE 1—Many of these specifications were originally developed by the Committee on Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society.[2]

NOTE 2—The values stated in U.S. customary units are to be regarded as the standard. The metric equivalents of U.S. customary units may be approximate.

2. Carbon-Dioxide Supply

2.1 Apparatus for the supply of carbon dioxide may be one of two types, as follows:

2.1.1 *Kipp Generator*, 2000-ml capacity, as shown in Fig. 1.

2.1.2 *Dry Ice Unit:*

2.1.2.1 Dewar flask, minimum capacity, 1.5 liters, and

2.1.2.2 Valve for carbon dioxide pressure regulator as shown in Fig. 2.

2.2 Dry ice is used in the Dewar flask, mercury in the capillary portion, and a filter paper disk in the cap.[3]

NOTE 3—Any suitable source of pure carbon-dioxide gas may be used.

3. Gas-Control Unit

3.1 The gas-control unit shall consist of a gasometer as shown in Fig. 3, and a 250-ml capacity leveling bulb.

4. Combustion Unit

4.1 The combustion unit shall consist of a long furnace, a sample furnace, a combustion tube, a combustion-tube closure, and a connection tube.

4.1.1 *Long Furnace:*

4.1.1.1 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 1/4 in. (6 mm). The heating well in the furnace shall accommodate combustion tubes up to 13-mm outside diameter. Electric heating elements shall be easily replaceable. The furnace shall be mounted firmly on a substantial support.

4.1.1.2 The furnace shall be capable of continuous operation at temperatures up to 800 C,[4] as measured inside the combustion tube in the middle of the furnace. The temperature drop from the center to points 1 in. (25 mm) and 1 3/4 in. (45 mm) from either end shall not exceed 15 percent and 7 percent, respectively, based on the temperature in the middle of the furnace.[5] Means shall be provided for varying the temperature.

4.1.1.3 Provision shall be made for rapid

[1] This specification is under the jurisdiction of ASTM Committee E-41 on Laboratory Apparatus and is the direct responsibility of Subcommittee E 41.07 on Microchemical Apparatus.

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[2] Committee for the Standardization of Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society, "1949 Report on Recommended Specifications for Microchemical Apparatus," *Analytical Chemistry*, ANCHA, Vol 21, 1949, p. 1555.

[3] For directions regarding use, see Niederl, J. B., and Niederl, V., *Micromethods of Quantitative Organic Analysis*, John Wiley & Sons, Inc., New York, N. Y., 2nd edition, 1942, p. 97, and Steyermark, A., *Quantitative Organic Microanalysis*, Blakiston Co., Philadelphia, Pa., 1951, pp. 76–77.

[4] Established in cooperative tests by the Association of Official Agricultural Chemists, C. L. Ogg, *Journal, Assoc. Official Agricultural Chemists*, JOACA, Vol 36, 1953, p. 344; *Ibid*, Vol 37, 1954, p. 450.

[5] Established by tests made on several types of commercially available electric furnaces.

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

4.1.1.4 The furnace shall be equipped with some device for continuous or provisional indication of the temperature at the middle of the furnace.

4.1.2 *Sample Furnace:*

4.1.2.1 The sample furnace shall have an overall length not less than $2^1/_2$ in. (65 mm) nor more than 4 in. (102 mm) with wall thickness at the ends not to exceed $^1/_4$ in. (6 mm). The heating well in the furnace shall accommodate combustion tubes up to 13-mm outside diameter. Electric heating elements shall be easily replaceable. The furnace shall be mounted firmly on a substantial support.

4.1.2.2 The furnace shall be capable of continuous operation at temperatures up to 800 C,[4] as measured inside the combustion tube in the middle of the furnace. The temperature drop from the center to points $^3/_4$ in. (19 mm) from either end shall not exceed 17 percent of the temperature in the middle of the furnace.[5] Means shall be provided for varying the temperature.

4.1.2.3 Sections 4.1.1.3 and 4.1.1.4 also apply to the sample furnace.

4.1.2.4 If the furnace is moved mechanically, it shall travel a distance of $6^1/_4$ in. (159 mm) min, 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 $^1/_8$ to $^5/_8$ in. (3 to 16 mm) per min. An automatic control shall be provided to stop the travel of the sample furnace when it reaches the long furnace.

4.1.3 *Combustion Tubes*—The combustion tubes shall conform to one of two types, as follows:

4.1.3.1 Combustion tube with tip as shown in Fig. 4.

4.1.3.2 Combustion tube with tip and sidearm as shown in Fig. 5.

NOTE 4—The ends (and sidearm) of the combustion tube may be equipped with interchangeable ground joints, provided the maximum outside diameter of the tip end does not exceed 13 mm.

4.1.4 *Combustion Tube Closure*[6]—For combustion tubes with sidearm a solid rubber stopper as shown in Fig. 6 shall be used; for those without, a one-hole rubber stopper, as shown in Fig. 7, shall be used.

4.1.5 *Connectin Tube*—When the one-hole stopper is used as a combustion tube closure, a Z-tube as shown in Fig. 8 shall be used.

5. Measuring Unit

5.1 The measuring unit shall consist of a flow-control device and a nitrometer.

5.1.1 *Flow Control Device*—Either of the following items may be used:

5.1.1.1 Precision stopcock as shown in Fig. 9, or

5.1.1.2 All-metal needle valve as shown in Fig. 10.

NOTE 5—An interchangeable ground joint may be attached to the constricted end of the precision stopcock or to each end of the needle valve.

5.1.2 *Nitrometer*, Pregl type with leveling bulbs (either type bulb may be used), as shown in Fig. 11. The volume contained by the nitrometer at 20 C shall be certified by the manufacturer to the nearest 0.001 ml at 6 points. The following procedure, in use by the National Bureau of Standards, is recommended.[7]

5.1.2.1 Calibrate the nitrometer, without the chamber, at the 0.1, 0.3, 0.5, 0.8, 1.1, and 1.5-ml marks. After cleaning and drying, lubricate the stopcock with a minimum of petroleum jelly. Tare the instrument and mount it in the inverted position with the stopcock closed. Suspend a thermometer beside it and place a transparent screen around the assembly. Pour mercury into a small glass reservoir provided with a stopcock and having a long slender delivery tube. Insert the delivery tube into the tube of the nitrometer down to the stopcock for the initial filling at the 0.1-ml mark.

[6] Committee for the Standardization of Microchemical Apparatus, Division of Analytical Chemistry, Am. Chemical Soc., *Analytical Chemistry*, ANCHA, Vol 22, 1950, p. 1228.

[7] Hughes, J. C., National Bureau of Standards, Washington, D. C., private communication to Steyermark, A., July 8, 1959; Mulligan, G. C., National Bureau of Standards, Washington, D. C. private communication to Steyermark, A., December 27, 1948.