



Designation: E147 – 61 (Reapproved 2006)

# Standard Specification for Apparatus for Microdetermination of Nitrogen by Kjeldahl Method<sup>1</sup>

This standard is issued under the fixed designation E147; 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 digestion and distillation apparatus used for the microdetermination of nitrogen by the Kjeldahl method, and modifications thereof at atmospheric pressure.

NOTE 1—This specification is based on those originally developed by the Committee on Microchemical Apparatus, Division of Analytical Chemistry, American Chemical Society.<sup>2</sup>

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>3</sup>

E124 Specification for Weighing and Drying Apparatus for Microchemical Analysis

## 3. Tolerances

3.1 The following commercial length tolerance shall be adhered to, unless otherwise specified:

Up to 10 mm	±1 mm
10 to 100 mm	±2 mm
100 to 200 mm	±3 mm
200 mm	±4 mm

## 4. Digestion Flasks

4.1 Digestion flasks of three types shall conform to the requirements prescribed in Table 1 and Fig. 1, Fig. 2, and Fig. 3. The flasks shall be made of shock-resistant glass with linear coefficient of expansion not greater than  $33.5 \times 10^{-7}$  cm/cm<sup>°</sup>C.

NOTE 2—The weighing tubes described in Specification E124 Figs. 12–14 are recommended for introducing solid samples into the micro Kjeldahl digestion flasks. Combustion boats, Fig. 1 and Fig. 2 of

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee E41 on Laboratory Apparatus and is the direct responsibility of Subcommittee E41.01 Apparatus.

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<sup>2</sup> Committee on Microchemical Apparatus, "Recommended Specification for Microchemical Apparatus, Micro Kjeldahl Nitrogen," *Analytical Chemistry*, Vol 23, 1951, p. 523.

<sup>3</sup> 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.

TABLE 1 Digestion Flasks

Capacity, mL	Figure	Use
10	1	with digests of 1 mL or less
30	2	with digests of 1 to 4 mL
30 (Soltys)	3	with digests of 1 to 4 mL when danger of spattering is great.

Specification E124, are recommended for use with waxy and nonvolatile liquid samples.

## 5. Digestion Rack

5.1 The micro Kjeldahl digestion rack shall be portable and shall consist of flask heaters (either gas or electric), a flask support, and a fume duct. The rack shall be supplied with a heat deflector mounted under the burners to prevent overheating of the bench top.

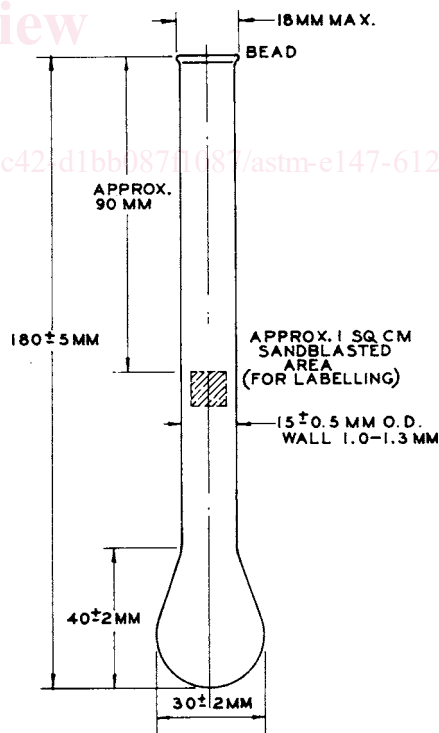


FIG. 1 10-mL Kjeldahl Flask

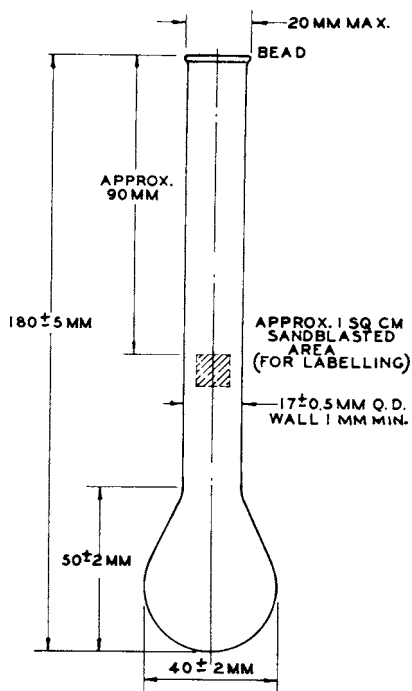


FIG. 2 30-mL Kjeldahl Flask

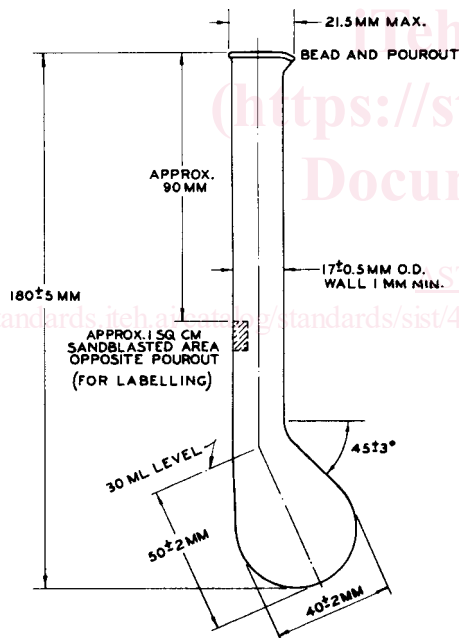


FIG. 3 30-mL (Soltys) Kjeldahl Flask

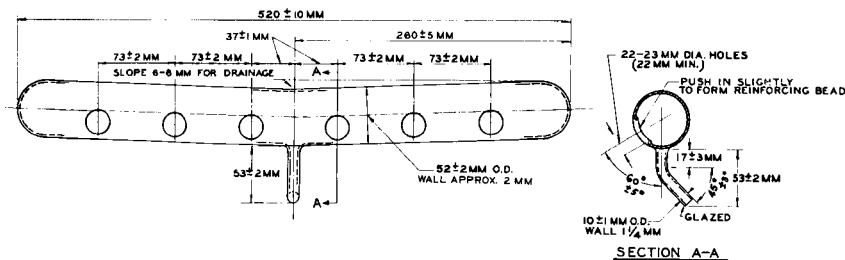


FIG. 4 Manifold for Micro Kjeldahl Digestion Rack

5.1.1 *Heaters*—The source of heat may be either electric or gas. In operation, each heater shall supply sufficient heat to a 30-mL flask to cause 15 mL of water at 25°C to come to a rolling boil in not less than 2 or more than 3 min. A device shall be provided for regulating the amount of heat supplied to the flask so that these conditions can be met. The amount of heat supplied to the flasks also shall be so adjustable that low-temperature digestions are possible. Switches and controls shall be conveniently located and shall remain cool enough to handle even after long periods of operation.

NOTE 3—Boiling chips shall be added when testing the heaters to minimize superheating, and the burners and rack shall be at equilibrium temperature.

5.1.2 *Flask Support*—The support on which the bulbs of the flasks rest shall shield the necks of the flasks from excessive heating during digestion and shall be provided with circular openings not exceeding 26 mm in diameter, centered over each heater.

5.1.3 *Fume Duct*—The fume duct shall provide means for adequately removing the fumes evolved during the digestion and shall support the necks of the flasks at an angle greater than 35° to the horizontal. Fume ducts meeting these specifications are shown in Fig. 4 and Fig. 5. The fume duct illustrated in Fig. 4 is supported and held in place by suitable clamps mounted at either end of the digestion rack. The fume duct in Fig. 5 rests upon a circular support plate and is held in place by springs from the plate to the four glass hooks. The concave center permits clearance for the nut which fastens the support plate to the center support post.

## 6. One-Piece Distillation Apparatus

6.1 The one-piece model distillation apparatus shall conform to the requirements of Fig. 6. The steam-generator vessel illustrated in Fig. 7 and the immersion heater in Fig. 8 are recommended for use with this apparatus, although any other suitable combination may be substituted.

6.2 The steam-generator vessel, Fig. 7, is a wide-mouthed commercially available resin reaction kettle. The interchangeable cover, with flat-ground rim and four ground-glass tubulations, is held in place with a suitable clamp. The distillation unit is connected to the center tubulation. Two of the outer three tubulations are used for the leads of the immersion-heater assembly, Fig. 8. The remaining tubulation is used for adding water to the kettle.

6.3 The rate of steam generation may be controlled by a 7.5-A variable transformer. The immersion heater shall be of the type that does not burn out when not immersed.

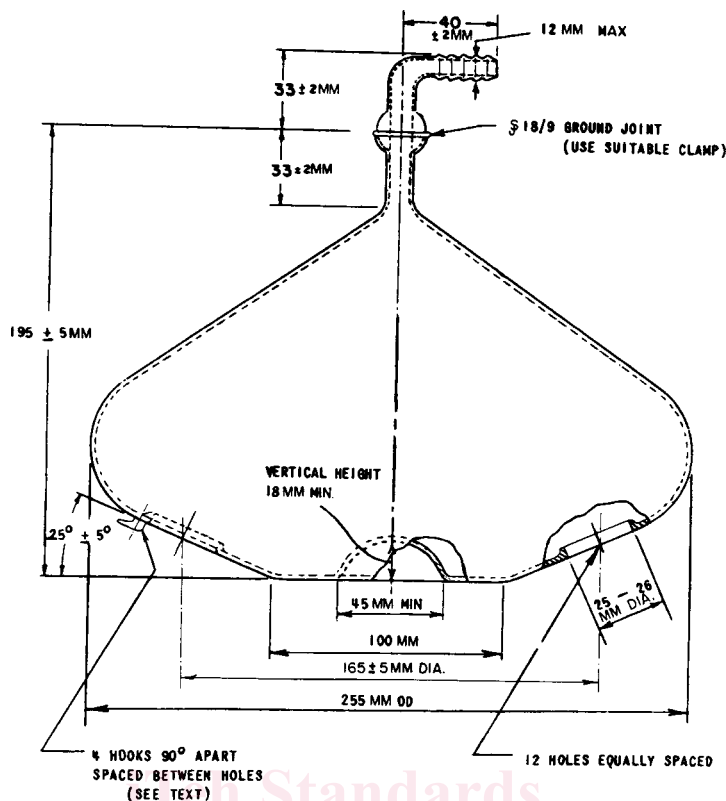


FIG. 5 Manifold for Micro Kjeldahl Digestion Rack (Circular Type)

NOTE 4—Several methods of cleaning can be used. The following method has been found satisfactory, particularly when boiling chips are employed in the digestion: Turn off the steam generator while both stopcocks are closed. The reduced pressure in the generator, brought about by cooling, causes the alkaline mixture in the distillation flask to be sucked into the outer jacket. Open both stopcocks to drain off the liquid. Then, while steam is being generated and with both stopcocks closed, fill the sample inlet funnel with water and immerse the tip of the condenser in about 100 mL of water contained in a beaker. Stop the steam generation, and open the stopcock of the funnel slowly, closing it before all the water has drained into the apparatus. Reduced pressure causes water to be sucked from the beaker into the apparatus, thereby washing it. Open the

stopcock in the drainage tube to empty the liquid that has collected in the outer jacket. If necessary, start the steam generator again, and repeat the rinsing procedure.

## 7. Pregl Type (Parnas-Wagner) Distillation Apparatus

7.1 The Pregl (Parnas-Wagner) type apparatus shall conform to the requirements of Fig. 9, Fig. 10, and Fig. 11. The steam-generator vessel illustrated in Fig. 7 and the immersion heater in Fig. 8 are recommended for use with this apparatus, although any other suitable combination may be substituted.