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Standard Specification for
PTFE MOLDING AND EXTRUSION MATERIALS[1]

This standard is issued under the fixed designation D 1457; 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 molding and extrusion resins of PTFE (polytetrafluoroethylene) that have never been melted after preforming or molding and are normally processed by methods similar to those used in powder metallurgy or ceramics, or by ram extrusion or extrusion with a volatile aid. These PTFE resins are homopolymers of tetrafluoroethylene or copolymers containing not more than 1 % by weight of other fluoromonomers. The usual methods of plastics processing generally are not applicable to these materials because of their viscoelastic properties at processing temperatures. The materials included herein do not include mixtures of PTFE resin with additives such as colors, fillers, plasticizers, any fabricated articles, or reprocessed or reground resin. The methods and properties included are those required to identify the various types of resins. Additional procedures are provided in the Appendixes for further characterization of the resins.

1.2 The values stated in SI units are to be regarded as the standard.

2. Applicable Documents

2.1 *ASTM Standards:*

D618 Methods of Conditioning Plastics and Electrical Insulating Materials for Testing[2]
D638 Test Method for Tensile Properties of Plastics[2]
D792 Test Methods for Specific Gravity and Density of Plastics by Displacement[2]
D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[2]
D 1895 Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials[3]
D 1898 Recommended Practice for Sampling of Plastics[3]
D 3297 Practice for Molding and Machining Tolerances for PTFE Resin Parts[4]
D 3892 Practice for Packaging/Packing of Plastics[4]
E 11 Specification for Wire-Cloth Sieves for Testing Purposes[5]
E 177 Recommended Practice for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material[5]
E 380 Standard for Metric Practice[6]

2.2 *Military Standard:*[7]

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes

3. Types

3.1 This specification covers the following six types of PTFE resin generally used for compression molding or extrusion, or both:

3.1.1 *Type I*—Granular resin used for general-purpose molding and extrusion.

3.1.2 *Type III*—Resin produced from a coagulated dispersion and normally used with a volatile processing aid. Type III resins are divided into four grades by such characteristics

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[2] *1983 Annual Book of ASTM Standards*, Vol 08.01.

[3] *1983 Annual Book of ASTM Standards*, Vol 08.02.

[4] *1983 Annual Book of ASTM Standards*, Vol 08.03.

[5] *1983 Annual Book of ASTM Standards*, Vol 14.02.

[6] *1983 Annual Book of ASTM Standards*, Complete in Vol 14.02 only. Excerpts in all other parts.

[7] Available from Naval Publications and Forms Center, 5801 Tabor Ave., Philadelphia, Pa. 19103. Attn: NPFC 105.

as bulk density, agglomerate size, melting peak temperature, standard specific gravity, tensile properties, etc. Each grade is divided into three classes to indicate performance in the test for extrusion pressure described in 12.8.

3.1.3 *Type IV*—Finely divided resin with an average particle size less than 100 μm.

NOTE 1—See Tables 1 and 2 for division of types by grades and classes and footnotes to Table 1 for former classifications. Type II was deleted from this specification in an earlier edition.

3.1.4 *Type V*—A finely divided granular resin typically used in applications requiring improved resistance to creep and stress relaxation in end use.

3.1.5 *Type VI*—Free-flowing resins. Generally made by treatment of finely divided resin to produce free-flowing agglomerates. Type VI resins are divided into two grades according to bulk density level.

3.1.6 *Type VII*—Presintered. Resin that has been treated thermally at or above the melting point of the resin at atmospheric pressure without having been previously molded or preformed.

4. General Requirements

4.1 The resin shall be of uniform composition and shall contain no additives or foreign material.

4.2 The color of the resin as shipped by the seller shall be white.

5. Detail Requirements

5.1 The resins covered by this specification shall conform to the requirements prescribed in Tables 1 and 2, when tested by the procedures specified herein. Table 1 lists tests to be carried out on resins. Table 2 lists tests requiring a specimen molded as described in Section 9.

6. Test Specimens

6.1 Test specimens shall be cut from disks or billets molded in accordance with the procedures given in Section 9.

7. Sampling

7.1 Unless otherwise agreed upon between the seller and the purchaser, the resin shall be sampled in accordance with Sections 9 to 12 of Recommended Practice D 1898. Adequate statistical sampling prior to packaging shall be considered an acceptable alternative.

7.2 A lot of resin shall consist of one continuous production run or a blend of two or more production runs. The producer shall take (and test) sufficient within-lot samples to assure adequate in-process quality control and continuing conformance to the lot requirements of this specification.

8. Number of Tests

8.1 Unless otherwise agreed upon in writing by the purchaser and seller, routine lot inspection tests shall consist of those specified in Table 1, except melting endotherms, plus measurement of specific gravity (see Table 2). Periodic tests shall consist of all the tests specified in Tables 1 and 2 and shall be made at least once per year.

8.2 The tests listed in Tables 1 and 2, as they apply, are sufficient to establish conformity of a material to this specification. When the number of test specimens is not stated in the test method, single determinations may be made. If more than single determinations on separate portions of the same sample are made, the results shall be averaged. The single or average result shall conform to the requirements prescribed in this specification.

9. Sample Preparation

9.1 *Test Disks*:

9.1.1 The die shown in Fig. 1 shall be used for the molding of test disks (Note 2). Place flat aluminum disks, 0.08 to 0.38 mm (3 to 15 mils) thick and 76 mm (3 in.) in diameter, on both sides of the resin when molding Type III resins.

NOTE 2: **Caution**—Although PTFE resin can be used continuously at temperatures of 260°C (500°F) or intermittently up to 327°C (620°F), it can evolve small quantities of gaseous products when heated above 204°C (400°F). Some of these gases are harmful. Consequently, exhaust ventilation must be used whenever the resins are heated above this temperature. Since a burning cigarette would exceed 204°C (400°F), those working with PTFE resins should ensure that tobacco is not contaminated.

9.1.2 Screen 14.5 g (for tensile properties) or 7.25 g (for electrical properties discussed in Appendix X1.7) of PTFE resin through a No. 10 hand sieve into the mold. Adjust lower plug height so that the resin can be leveled by drawing a straightedge in contact with and across the top of the mold cavity. Insert mold in suitable hydraulic press and apply pressure gradually (Note 3) until a total of 13.8 MPa (2000 psi) for Type III resin or 34.5 MPa (5000 psi) for all other types is attained. After this point has been reached, hold the pressure on the disk for 3 min. Remove the disk from the die. A wax pencil may be used to write sample identification on the disk at this time.

NOTE 3—As a guide, increasing the pressure at a rate of 3.45 MPa (500 psi)/min is suggested until the desired maximum is attained.

9.1.3 Place the sintering oven in a laboratory hood (or equip it with an adequate exhaust system) and sinter preforms according to Table 3, Procedure B for Types I, III, IV, and VI, and Procedure C for Type V.

NOTE 4—Although the rate of pressure application is not critical, the cooling cycle is most important and the conditions cited in this procedure must be followed very closely. If they are not followed, the crystallinity of the disks and the resulting physical properties will be markedly changed. Therefore, the use of a cam-controlled, automatically timed oven is recommended for the most precise sintering cycle control so that the hood window may be left down during the entire sintering procedure, the latter being an important safety consideration.

9.2 *Test Specimens for Standard Specific Gravity and Thermal Instability Index*:

9.2.1 A cylindrical preforming mold,[8] 28.6 mm (1⅛ in.) internal diameter by at least 76.2 mm (3 in.) deep, is used to prepare the preforms. Clearance should be sufficient to ensure escape of entrapped air during pressing. Place flat aluminum foil disks, normally 0.013 mm (0.005 in.) thick and 28.6 mm (1⅛ in.) diameter, on both sides of the resin, when molding Type III resins. The test resin should be near ambient temperature prior to molding.

NOTE 5—For maximum precision, the weighing and preforming operations should be carried out in a constant-temperature/humidity room at 23 ± 1°C (73.4 ± 1.8°F) and 50 ± 2 % relative humidity. The method should not be run below 22°C (71.6°F) due to the crystalline transition which leads to possible cracks in sintered specimens and differences in specimen density. Practice D 3297 provides additional details.

9.2.2 Weigh out 12.0 ± 0.1 g and place in preforming mold. Nonfree-flowing resins shall be screened through a No. 10 sieve. Compacted resins can be broken up by hand shaking cold resin in a half-filled sealed glass container. Condition the resin in the sealed glass container in a freezer or dry-ice chest. After breaking up resin lumps, allow the sealed container to equilibrate to near ambient temperature. Then screen and weigh the 12.0 ± 0.1 g sample. Insert the mold in a suitable hydraulic press and apply pressure gradually (Note 3) *until a total load of* 13.8 MPa (2000 psi) or 34.5 MPa (5000 psi) is attained. Hold the pressure on the preform for 2 min. Remove the preform from the mold. A wax pencil may be used to write the sample identification on the preform at this time.

9.2.3 Sinter the preforms by Procedure A or B for Type III, Procedure B for Types I, IV, and VI, and Procedure C for Type V given in Table 3.

NOTE 6—Improved precision in the standard specific gravity test results has been obtained with sintering Procedure A2, the use of an upright, cylindrical oven, and an aluminum sintering rack. The cylindrical oven has an inside diameter of 140 mm (5.5 in.) and a depth of 203 mm (8 in.) plus additional depth to accommodate a 50.8 mm (2 in.) cover and is equipped with adequate band heaters and controls to accomplish the sintering of specimens according to Procedure A2, Table 3. The rack, as shown in Fig. 2, allows preforms to be placed symmetrically in the center region of the oven. Place six preforms on each of the middle oven rack shelves. (If six or less preforms are to be sintered, place them on the middle rack, filling in with "dummy" specimens as needed.) Place "dummy" specimens on the top and bottom shelves. Specimens must be spaced evenly in a circle on each shelf, with none of them touching. An oven load must be no less than 18 pieces including the additional "dummy" pieces. "Dummies" are used to complete the load as necessary. ("Dummies" are defined as normal 12-g specimens that have previously been through the sintering cycle. "Dummies" must only be used for an additional two or three thermal cycles, due to eventual loss of thermal stability and physical form.)

9.2.4 Remove flash from each specimen, so that no air bubbles will cling to the edges when it is immersed in the solution for weighing during the standard specific gravity and instability index tests. It is recommended for this section and during testing that cotton gloves be worn while handling test specimens.

9.3 *Test Billets*:

9.3.1 Test specimens cut or skived from billets may be used as an alternative to the test disks described in 9.1 and 9.2 for Types I, IV, V, and VI resins.

9.3.2 Mold test billets in a die similar to Fig. 3, having an inside diameter of 57 mm (2.25 in.) and of sufficient height to contain the sample. Clearance should be sufficient to insure escape of entrapped air during pressing. A 254-mm (10-in.) length will produce a billet ap-

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[8] A "Carver Test Cylinder" has been found satisfactory for this purpose. It is available from standard laboratory equipment suppliers.

proximately 76 mm (3 in.) long from a resin charge of 400 ± 50 g. The billet length may be varied in accordance with the amount of testing to be done.

9.3.2.1 Adjust the lower plug height so that the resin level will not come within 13 mm (0.5 in.) of the top of the cavity. Add the resin to the mold, insert the top plug, and apply hand pressure making certain that the potential preform is centered in the mold. Remove the support ring, and place the mold in a hydraulic press. Do not allow the two end plugs to "bottom" on the mold shell.

9.3.2.2 Apply an initial load to the mold of 3.45 MPa (500 psi) ± 10 % and hold for 1 to 2 min. Increase the loading smoothly to the final performing pressure in 3 to 5 min. The final pressure attained should be as recommended by the manufacturer for the particular material, otherwise 34.5 MPa (5000 psi) for Type I and 17.2 MPa (2500 psi) for Types IV, V, and VI should be used. Hold under maximum pressure for 2 to 5 min. Release the pressure by gradually "cracking" the pressure release valve without an apparent movement of the press platens. Remove the top pusher and force the preform vertically out of the mold shell using a continuous, smooth movement.

NOTE 7—Care must be taken in removing the preform from the die. If a vertical force is not maintained, cracking of the preform may occur.

9.3.3 Insert the preform in a sintering can (Fig. 4) and place the assembly in an oven. Sinter according to procedures in Table 3. Use Procedure D for Types I, IV, and VI and Procedure E for Type V resins.

NOTE 8—The sintering can may be eliminated if an oven is used which provides equivalent uniformity of temperature about the specimen being heated.

9.4 *Sectioning and Skiving the Test Billet*:

9.4.1 Divide the test billet into sections by making transverse cuts by machining, or by a suitable alternate procedure, according to Fig. 5. The rough cuts between Sections I and II may be made with a saw, but the Faces C and D must be prepared by machining.

9.4.2 Prepare five test specimens for the determination of tensile strength and elongation from 0.8 mm (1⁄32 in.) thick slices machined from Section II, Face C, and machine a slice of suitable thickness for standard specific gravity measurements as described in 12.5.1. Care should be taken to avoid wedge-shape cuts. Use the remainder of Section II to prepare tape specimens by skiving 0.13 mm (5 mils). Discard the initial five revolutions of skived tape before taking the test sample. The tape may be used for the determination of tensile strength and elongation, as an alternative to machined disks. If electrical properties, discussed in the Appendix, are to be determined on tape, Sections II and III must be left together in order that a tape of sufficient width is obtained to allow the cutting of a 2-in. diameter electrical test specimen.

10. Conditioning Test Specimens

10.1 For tests of standard specific gravity and tensile properties the molded test specimens shall be conditioning in accordance with Procedure A of Methods D 618, for a period of at least 4 h prior to test. The other tests require no conditioning of the molded test specimens.

11. Test Conditions

11.1 Tests shall be conducted at the Standard Laboratory Temperature of 23 ± 2°C (73.4 ± 3.6°F), unless otherwise specified in the testing methods or in this specification. Since this resin does not absorb water, the maintenance of constant humidity during testing is not important.

12. Test Methods

12.1 *Melting Characteristics by Thermal Analysis*—An initial melting peak temperature (Fig. 6) greater than 5°C (41°F) above subsequent melting peak temperatures is indicative that the resin has not been melted previously. A resin that has been melted is not compatible with the scope of this specification except for Type VII. The peak temperature obtained on the second and any subsequent meltings is defined as the melting endotherm and occurs at about 327°C (620.6°F) for PTFE resins. If peak temperatures are difficult to discern from the curves, that is, if the peaks are rounded rather than pointed, straight lines should be drawn tangent to the sides of the peak. Where these lines intersect beyond the peak should be taken as the peak temperature. Multiple peaks during the initial melting test are not uncommon. Where more than one peak occurs, the presence of any peak <5°C (<41°F) above the peak temperature obtained during first melting test may indicate the presence of some previously melted material.

12.2 *Bulk Density*:

12.2.1 Bulk density gives some indication, on a volumetric basis, of how the resin may perform during feeding of molding and extrusion equipment. PTFE resins have a tendency to compact during shipment and storage, and even though the material is broken up by screening or some other means, original "as produced" results may not be duplicated. Because of this tendency to pack under small amounts of compression or shear, Test Method D 1895 is not applicable to these resins. The procedure given in the following paragraphs must be used to measure this property.

12.2.2 *Definition*—The *bulk density* of the resin is defined as the weight in grams of a volume of 1000 mL of resin measured as described in this specification.

12.2.3 *Apparatus*:

12.2.3.1 *Funnel*—A funnel arrangement as shown in Fig. 7.

12.2.3.2 *Feeder*[9]—A feeder with a No. 8 wire screen placed over approximately the top two-thirds of the trough. The funnel shall be mounted permanently in the feeder outlet.

12.2.3.3 *Controller*[10]:

12.2.3.4 *Volumetric Cup and Cup Stand (Fig. 8)*—The volumetric cup shall be calibrated initially to 250 mL by filling with distilled water, placing a plane glass plate on top, drying the outside of the cup, and weighing. The net weight shall be 250 ± 0.5 g. The top and bottom faces of the volumetric cup and the cup stand shall be machined plane and parallel.

12.2.3.5 *Leveling Device*—The leveler (Fig. 9) shall be affixed permanently to the table and adjusted so that the saw-tooth edge of the leveler blade passes within 0.8 mm (1⁄32 in.) of the top of the volumetric cup.

12.2.3.6 *Work Surface*—The work surface for holding the volumetric cup and leveler shall be essentially free from vibration. The feeder, therefore, must be mounted on an adjoining table or wall bracket.

12.2.3.7 *Balance*—The balance having an extended beam shall have a capacity of 500 g and a sensitivity of 0.1 g or equivalent.

12.2.4 Place the clean, dry volumetric cup on the extended beam of the balance and adjust the tare to zero. Select about 500 mL of the resin to be tested, place it on the feeder screen and vibrate all of the resin through screen and back into the sample container twice to break up any lumps. This screening step is not necessary for Type III resins. Put the cup in the cup stand and place the assembly such that the distance of free polymer fall from the feeder outlet to the top rim of the cup shall be 31.8 ± 3.2 mm (1½ ± ⅛ in.). Increased fall causes packing in the cup and higher bulk density values. Set the controller so that the cup is filled in 20 to 30 s (usually about 70 on the controller). Pour the sample on the vibrating screen and fill the cup so that the resin forms a mound and overflows. Let the resin settle for about 15 s and then gently push the cup and its stand beneath the leveler. Exercise care to avoid agitation of the resin and cup before leveling. Weigh the resin to the nearest 0.1 g.

12.2.5 *Calculation*—Calculate the bulk density as follows:

$$\text{Grams of resin} \times 4 = \text{bulk density (grams per litre)}$$

12.2.6 *Precision*—The precision on a single determination shall be between ± 5 and 6 g/L (two σ limits).

12.3 *Particle Size*:

12.3.1 The fabrication of PTFE resins either by molding or extrusion is affected significantly by particle or agglomerate size and size distribution. (See Appendix for further details on particle characteristics.)

12.3.2 *Wet-Sieve Method*.

12.3.2.1 *Principle of Method*—The average particle or agglomerate size of PTFE resins is determined by fractionation of the material with a series of sieves. Fractionation is facilitated by spraying with perchloroethylene (Note 9) which breaks up lumps and prevents clogging of the sieve openings. This method is recommended for Types I, IV, V, VI, and VII resins, but not for Type III.

NOTE 9: **Caution**—Perchloroethylene is under investigation by government agencies and industry for its carcinogenic effects. Protective nitrile or butyl gloves should be worn to prevent skin contact and adequate ventilation provided to remove the vapors.

12.3.2.2 *Apparatus*:

12.3.2.2.1 *Balance*, capable of weighing to ± 0.1 g.

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[9] A "Vibra-Flow" Feeder, Type FO, Style 1687, Available from Syntron Electric Co., 1938 Black Street, Homer City, Pa., has been found satisfactory for this purpose.

[10] A "Syntron" controller, Type FC05, Style 4283, available from Syntron Electric Co., 1938 Black Street, Homer City, Pa., has been found satisfactory for this purpose.

12.3.2.2.2 *Sieves*—U.S. Standard Sieve Series, 203-mm (8-in.) diameter conforming to Specification E 11, except that sieves shall be selected from Table 4.

12.3.2.2.3 *Ventilated Hood.*

12.3.2.2.4 *Beakers*—Six tared, 150-mL beakers.

NOTE 10—As an alternative, sieves may be tared, dried, and weighed on a balance to avoid transferring of fractionated samples to the tared beakers.

12.3.2.2.5 *Apparatus for Sieving and Spraying*—A suggested arrangement of an apparatus for recirculating perchloroethylene is shown in Fig. 10 (*a*). This must be located in a ventilated hood or adequately ventilated area.

12.3.2.3 *Reagents*—Perchloroethylene, 20 L (5 gal). Other liquids may be used; however, their applicability and hazards associated with their use must be thoroughly investigated.

12.3.2.4 *Procedure:*

12.3.2.4.1 Select the appropriate sample size and combination of sieves from Table 4 for the type of resin under test. Adjust the flow rate of the perchloroethylene to 6 ± 0.5 L/min.

12.3.2.4.2 Place the weighed resin on the top sieve and spray it with perchloroethylene for 1 ± 0.2 min. The shower-head shall be about level with the top of the sieve and be moved in a circular fashion. Take care to break up all of the lumps and to wash the material from the sides of the sieve.

12.3.2.4.3 Remove the top sieve and place it in the hood to dry.

12.3.2.4.4 Repeat the procedure specified in 12.3.2.4.2 and 12.3.2.4.3 until all the sieves have been sprayed. It is not necessary to spray the No. 325 sieve since this serves only to remove the resins from the perchloroethylene. The resin retained by this sieve may be discarded. Air-dry the sieves in the hood for 30 min or longer, or oven-dry at 90°C (194°F) for 15 min and then cool to room temperature. Remove the resin from each sieve by tapping on a piece of paper as shown in Fig. 10 (*b*). Pour each fraction into a tared beaker and weigh to ± 0.1 g (See Note 10).

12.3.2.4.5 Record the weight of resin on each sieve.

12.3.2.4.6 Clean the sieve by inverting it over the No. 325 sieve and spraying with perchloroethylene. Take care to prevent the resin from getting into the perchloroethylene.

12.3.2.5 *Calculation:*

12.3.2.5.1 Calculate the net percentage of resin on each sieve as follows:

$$\text{Net percent on sieve } Y = F \times \text{weight of resin in grams on sieve } Y,$$

where:

F = 2 for 50-g sample, and

F = 10 for 10-g sample.

12.3.2.5.2 Calculate the cumulative percentage of resin on each sieve as follows:

Cumulative percent on sieve Y = sum of net percentages on sieve, Y and sieves having numbers smaller than Y.

NOTE 11—*Example*—Cumulative percent on No. 35 sieve = net percent on No. 18 + net percent on No. 25 + net percent on No. 35 sieves.

12.3.2.5.3 Plot the cumulative percent versus the sieve opening size (or sieve number) on log probability paper as shown in the sample plot (Fig. 11). The sieve numbers and sieve opening sizes in microns are indicated below the figure. Draw the best straight line through the points and read the particle size at the 50 % cumulative percentage point ($d50$).

12.3.2.5.4 Calculate the average particle diameter, $\bar{d}$, as follows:

$$\bar{d} = d50$$

12.3.2.6 *Precision*—Because the resin particles have complex shapes, and because on each sieve there is a distribution of particle sizes, the values for particle size and particle size distribution obtained will be only relative numbers. The 95 % confidence limits based on a limited series of tests are ± 2.8 % for the average particle size.

12.3.3 *Dry-Sieve Method:*

12.3.3.1 *Principle of Method*—The average agglomerate size of PTFE coagulated dispersion powders is determined by fractionation of the resin with a series of sieves. Fractionation is accomplished by mechanically shaking the material in the assembly of sieves for a predetermined period. This method is recommended for Type III resins.

12.3.3.2 *Apparatus*:

12.3.3.2.1 *Balance,* capable of weighing to ± 0.1 g.

12.3.3.2.2 *Sieves*—U.S. Standard Sieve Series (Table 4).

12.3.3.2.3 *Sieve Shaker*—A mechanical sieve shaking device capable of imparting uniform rotary and tapping action.

12.3.3.2.4 *Freezer*—Any commercial ice

freezer. (A dry-ice chest may be used.)

12.3.3.3 *Procedure*:

12.3.3.3.1 Select the appropriate sample size and combination of sieves from Table 4 for the type of resin under test. Place the sample in an aluminum pan. Cool the pan and contents to less than 10°C (50°F). The dew-point temperature in the screening room must be less than the temperature in the cooling chamber. Obtain the tare weight on the selected sieves. Put conditioned sample on top sieve of the assembly and shake in the sieve shaker for 10 ± 0.5 min. Determine the weight of resin retained on each sieve.

12.3.3.4 *Calculation:*

12.3.3.4.1 Calculate the same as in the wet-sieve method; see 12.3.2.5.

12.3.3.5 *Precision*—The test precision is ± 3.2 % (two σ limits) for the combination of 25 + 35 + 45 − sieve fractions for a resin where this combination of sieves retains, on the average, 78 % of the sample.

12.4 *Water Content:*

12.4.1 *Principle of Method*—A sample of PTFE resin of known weight is dried in a vacuum oven in a tared aluminum weighing dish. When the resin is dry, it is removed from the oven, placed in a desiccator, allowed to cool, and then reweighed. From the weight lost during drying, the moisture content is determined and reported as "percent moisture."

12.4.2 *Apparatus*:

12.4.2.1 *Vacuum Oven.*

12.4.2.2 *Aluminum Weighing Dishes,* with lids.

12.4.3 *Procedure* (Note 13)—Wash the aluminum weighing dish with water and rinse with acetone. When the acetone has evaporated from the dish, dry it thoroughly in any oven at 50 to 80°C (122 to 176°F), then store in a desiccator until ready for use. Obtain the tare weight, *B*, of the aluminum weighing dish, plus lid, to the nearest 0.0001 g. Place 35 to 40 g of PTFE resin in the tared aluminum weighing dish and record the weight, *A*, to the nearest 0.0001 g (Note 14). Dry to constant weight in a vacuum oven (660 mm (25 in.) Hg) at 150°C (302°F), with the dish lid removed. Remove the dish from the oven, replace the lid on the weighing dish, and allow to cool in the desiccator for at least 30 min. Reweigh the dish (plus the resin and lid), *C*, and calculate the weight loss.

NOTE 12—If volatiles, other than moisture, are suspected, use the alternative method described in 12.4.4.

NOTE 13—From each group of samples, one sample is selected, and duplicate moisture determinations are run on it. If the difference between the duplicate results exceeds 0.01 %, the entire group of samples must be run over.

NOTE 14—When a group of samples is run at the same time, it is good practice to place the lids from the weighing dishes directly under their corresponding dishes while the samples are drying in the oven. This eliminates the possibility of introducing errors in the tare weights. Also, overnight drying in a circulating air oven may be used if the data can be shown to be equivalent to those obtained with the above procedure.

12.4.3.1 *Calculation*—Calculate the percentage of moisture as follows:

$$\text{Moisture, \%} = [(A - C)/(A - B)] \times 100$$

where:

A = weight of resin, dish, and lid, g,
B = weight of dish and lid, g and
C = weight of resin, dish, and lid after drying, g.

12.4.3.2 *Precision*—The precision of this test is ± 0.0063 % (two σ limits).

12.4.4 *Alternative Method For Determination of Water By Karl Fischer Reagent*[11]—Weigh 35 ± 1 g of resin into a glass-stoppered flask containing about 50 mL of pretitrated methanol. Shake to mix with a swirling motion for a few minutes. Titrate with standardized Karl Fischer reagent[12] to a visual or electrometric end point.

12.5 *Specific Gravity:*

12.5.1 *Standard Specific Gravity (SSG):*

12.5.1.1 Determine the standard specific gravity of specimens prepared by one of the procedures given in 9.2 or 9.3.

12.5.1.1.1 If specimens from 9.2 (12 g) are to be tested, use them "as is."

12.5.1.1.2 If specimens from 9.3 (from billets) are to be tested, use the center portion of the sintered billet (Section II of Fig. 5). From it, cut an approximately cubical shape which weighs at least 10 g.

12.5.1.2 Make SSG determinations in accord-

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[11] Details of this method can be found in J. Mitchell, Jr. and D. M. Smith's *Aquametry*, 2nd Edition, published by Interscience Publishers, Inc., New, York, N.Y. 1977.

[12] Karl Fischer Reagent (Catalog No. So-K-3) is available from the Fischer Scientific Co., Pittsburgh, Pa.

[13] Examples of suitable wetting agents are "Glim" detergent, B. J. Babbitt, Inc.; "Joy" detergent, Proctor and Gamble, Inc; and "Triton" X-100, Rohm and Haas Co.

ance with the procedures described in Test Methods D 792, Annex A1. Add two drops of a wetting agent[13] to the water in order to reduce the surface tension and ensure complete wetting of the specimen.

12.5.2 *Specific Gravity by Density Gradient Technique*—When the better precision of the SSG method is not required, the density gradient test described in Test Method D 1505 may be used.

12.6 *Thermal Instability Index*:

12.6.1 The thermal instability index (TII) test measures the decrease in molecular weight of the resin after it has been heated for a prolonged period of time. When this test is to be run, duplicate specimens are prepared from the same sample or billet.

12.6.1.1 If 12-g specimens are used, determine SSG (12.5.1.1 and 12.5.1.2) on one specimen and extended specific gravity (ESG) on the other. Sinter the ESG specimen according to Procedure F for Types I, III, IV, and VI, and Procedure G for Type V, Table 3. Determine the ESG as in 12.5.2.

12.6.1.2 If billet specimens are used, use Section III as shown in Fig. 5 as the ESG specimen. Sinter according to Procedures in Table 3. Use Procedure H for Types I, IV, and VI, and Procedure I for Type V resins. Determine ESG as in 12.5.1.2.

12.6.2 *Calculation*—Calculate the thermal instability index (TII) as follows:

$$\text{TII} = (\text{ESG} - \text{SSG}) \times 1000$$

12.7 *Tensile Properties*:

12.7.1 *Procedure*—Cut five tension specimens from the 14.5-g disk prepared in 9.1, or from a billet, as prepared in Section 9 and cut or skived as in 9.4.1, with the microtensile die described in Fig. 14[14]. Determine the tensile properties in accordance with the procedures described in Test Method D 638, except that the initial jaw separation shall be 22.2 ± 0.13 mm (0.875 ± 0.005 in.) and the speed of testing shall be 50 mm (2 in.)/min. Clamp the specimens with essentially equal lengths in each jaw. Determine elongation from the chart, expressed as a percentage of the initial jaw separation.

12.7.2 *Precision*—The single-operator, single-machine precision of tensile strength is 1.09 MPa (158 psi) (1s) and of elongation is 10.9 % (1s %), as defined in Recommended Practice E 177.

12.8 *Extrusion Pressure of Type III PTFE Resins:*

12.8.1 *Definition*—Extrusion pressure is the pressure that is observed during the extrusion of a lubricated Type III resin at one of the reduction ratio values established to represent processing under a low, intermediate or high reduction ratio condition (see 12.8.2.2 and Table 1).

12.8.2 *Apparatus* (Equivalent apparatus may be substituted.)

12.8.2.1 *Paste Extruder* (Fig. 12):

12.8.2.1.1 One paste extruder which may be used is a vertically disposed, breech-loading extruder with a 31.8-mm (1.25 in.) inside diameter extrusion cylinder. The barrel length is approximately 305 mm (12 in.), which is not critical so long as it will hold enough lubricated resin to extrude for about five min. The ram is 31.6 mm (1.245 in.) outside diameter, with a ring groove near its end to hold an O-ring that makes a tight seal between the ram and extruder cylinder. The extruder is equipped with devices for sensing and recording pressure on the face of the ram. The range of the pressure transducer in the ram face is greater than 69 MPa (10 000 psi). Temperature-controlling equipment maintains the extruder at 30 ± 1°C. A hydraulic system drives the ram at a speed of about 18 mm/min (0.7 in./min) to give an output rate of 19 g/min on a dry-resin basis (about 23.5 g/min of lubricated resin). The extruder-die assembly slides on tracks from under the ram to allow easy access for loading and cleaning the cylinder.

12.8.2.1.2 An alternative breech-loaded paste extruder may be used which has a detachable die assembly that drops from the cylinder. The die assembly is dropped, a preformed charge of resin inserted up into the cylinder and the die assembly reattached.

12.8.2.2 *Extrusion Dies* (Fig. 13)—Interchangeable extrusion dies, each having 30° included angles, give the desired reduction ratios, as follows:

Reduction Ratio	Die Orifice, (Inside Diameter), mm (in.)	Land Length, mm (in.)
100:1	3.18 (0.125)	25.35 (0.998)
400:1	1.59 (0.0625)	4.78 (0.188)
1600:1	0.79 (0.0312)	0.38 (0.015)

NOTE 15—Reduction ratio in this specification is

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[14] The steel rule type of die has been found satisfactory.

the ratio of the cross-sectional area of the preform to the cross-sectional area of the die. This must not be confused with another definition wherein reduction ratio is the ratio of the cross-sectional area of the extruder cylinder to the cross-sectional area of the sintered extrudate.

12.8.2.3 *Miscellaneous Equipment*—Equipment is provided for weighing, blending, conditioning (at 30°C), and preforming resin as well as extruder cleaning.

12.8.3 *Procedure:*

12.8.3.1 Screen the dry resin through a 4-mesh sieve onto a clean, dry, lint-free sheet of paper.

12.8.3.2 Transfer 200 ± 0.5 g of the screened resin to a clean, dry glass jar about 92 mm (3.625 in.) in diameter (approximately 1-L capacity) having an airtight closure, or into a V-blender of laboratory size.

12.8.3.3 Add 47.6 ± 0.1 g of kerosine-type hydrocarbon lubricant having a relatively low volatility at room temperature,[15] to the resin in the jar or blender. Care should be taken to prevent the lubricant from wetting the walls of the jar, as this hampers the mixing action. The screw-type closure should be taped in place when a jar is used to prevent any loss of lubricant. Shake the jar briefly to minimize the wetting of the jar wall with liquid.

12.8.3.4 Place the jar on rubber-coated mill rolls and roll at 30 rpm for 25 ± 5 min or blend in the V-blender for 15 ± 5 min. If a V-blender has been used, drop the resin from it into a jar of approximately 1-L capacity and seal jar.

12.8.3.5 After blending, place the jar with its contents, in a controlled atmosphere at 30 ± 1°C, to age for a minimum of 2 h. A water bath has been found to be satisfactory. This enables the lubricant to diffuse to the interior of individual particles and surfaces not reached during the rolling process.

12.8.3.6 Place the proper extrusion die for the desired reduction ratio at the bottom of the extruder cylinder if using the paste extruder of 12.8.2.1.1.

12.8.3.7 *Resin Preform:*

12.8.3.7.1 To preform the resin for the paste extruder of 12.8.2.1.1, slide the extruder-die assembly forward, mount a 31.8 mm (1.25 in.) inside diameter extension tube about 610 mm (24 in.) in length at the breech end of the extruder cylinder. Quickly pour the lubricated resin through a funnel into the extension and force the resin into the extruder cylinder with a tamping rod. Apply the force with hard pressure and a very slow, even stroke.

12.8.3.7.2 To preform the resin for the paste extruder of 12.8.2.1.2, mount a 31.8 mm (1.25 in.) inside diameter preforming tube about 610 mm (24 in.) in length with its cross section resting against a flat, smooth surface. Quickly pour the lubricated resin through a funnel into the tube and force the resin down in the tube. The force may be applied with a hydraulically controlled tamping device to compact the resin with a slow, even stroke to a minimum of 690 kPa (100 psi) on the resin. Remove the preform from the preforming tube, insert the preform up into the cylinder of the extruder and attach the die assembly.

12.8.3.8 Use the fast speed drive to run the ram down into the cylinder cavity. When the first bit of beading emerges from the orifice, stop the descent of the ram.

12.8.3.9 Immediately change to slow drive, start the pressure-recording system and extrude the lubricated resin at a rate of 19.0 ± 1.0 g/min (lubricant-free basis).

12.8.3.10 Record the pressure developed on the face of the ram in contact with the resin in the cylinder as a function of time. The extrusion pressure is the average pressure required to extrude the sample as measured between the third and fourth minutes of the extrusion, provided it gives a continuous, smooth extrudate. This has been called the steady-state extrusion pressure.

12.8.4 *Precision:*

12.8.4.1 The test precision is to be determined by round-robin testing.

13. Inspection

13.1 Inspection of the resin shall be to determine that the material meets the requirements of this specification.

14. Retest and Rejection

14.1 If any resin fails to meet the requirements of this specification, it may be retested to establish conformity.

15. Packaging and Marking

15.1 *Packaging*—The resin shall be packaged in standard commercial containers so constructed as to ensure acceptance by common or

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[15] Varsol® 1, available from Humble Oil and Refining Co., has been found satisfactory for this purpose.

other carriers for safe transportation to the point of delivery, unless otherwise specified in the contract or order.

15.2 *Marking*—Shipping containers shall be marked with the name of the resin, type, and quantity contained therein.

15.3 All packing, packaging, and marking provisions of Practice D 3892 shall apply to this specification.

QUALITY ASSURANCE PROVISIONS FOR GOVERNMENT/MILITARY PROCUREMENT

These requirements apply *only* to Federal/Military procurement, not domestic sales or transfers.

S1. Sampling for inspection and testing shall be carried out in accordance with the recommendations of Recommended Practice D 1898.

S2. Selection of Acceptable Quality Level (AQL) and of Inspection Level (IL) shall be made, with consideration of the specific use requirements. This is discussed in Sections 7 and 8 of the above document, with reference to Military Standard MIL-STD-105.

S3. In the absence of contrary requirements the following values shall apply:

	IL	AQL
Defects of appearance and workmanship	II	2.5
Defects of preparation for delivery	S-2	2.5
Testing (products)	S-1	1.5
Testing (resin, unfabricated)	S-1[A]	...

[A] Samples shall be drawn from the required number of units, and pooled for preparation of molded samples for mechanical properties evaluation.

TABLE 1 Detail Requirements for Tests on Resins

Type[A]	Grade	Class	Bulk Density, g/L	Particle Size, Average Diameter, μm	Water Content, max, %	Melting Peak Temperature		Extrusion Pressure	
						Initial °C	Second °C	MPa	psi
I[B]	...	...	675 ± 50	375 ± 75	0.04	[N]	327 ± 10	NA[J]	NA[J]
III	1[C]	A	475 ± 100	500 ± 150	0.04	[N]	327 ± 10	9.7 ± 4.2[K]	1410 ± 610[K]
		B	475 ± 100	500 ± 150	0.04	[N]	327 ± 10	24.1 ± 10.3[L]	3500 ± 1500[L]
		C	475 ± 100	500 ± 150	0.04	[N]	327 ± 10	55.2 ± 20.7[M]	8000 ± 3000[M]
	2[D]	A	475 ± 100	500 ± 150	0.04	[N]	327 ± 10	9.7 ± 4.2[K]	1410 ± 610[K]
		B	475 ± 100	500 ± 150	0.04	[N]	327 ± 10	24.1 ± 10.3[L]	3500 ± 1500[L]
		C	475 ± 100	500 ±150	0.04	[N]	327 ± 10	55.2 ± 20.7[M]	8000 ± 3000[M]
	3[E]	A	475 ± 100	425 ± 150	0.04	[N]	327 ± 10	9.7 ± 4.2[K]	1410 ± 610[K]
		B	475 ± 100	425 ± 150	0.04	[N]	327 ± 10	24.1 ± 10.3[L]	3500 ± 1500[L]
		C	475 ± 100	425 ± 150	0.04	[N]	327 ± 10	55.2 ± 20.7[M]	8000 ± 3000[M]
	4	A	475 ± 100	650 ± 200	0.04	[N]	327 ± 10	9.7 ± 4.2[K]	1410 ± 610[K]
		B	475 ± 100	650 ± 200	0.04	[N]	327 ± 10	24.1 ± 10.3[L]	3500 ± 1500[L]
		C	475 ± 100	650 ± 200	0.04	[N]	327 ± 10	55.2 ± 20.7[M]	8000 ± 3000[M]
IV[F]	...	...	...	<100	0.04	[N]	327 ± 10	NA[J]	NA[J]
V[G]	...	...	350 ± 75	<100	0.04	[N]	327 ± 10	NA[J]	NA[J]
VI	1[H]	...	650 ± 150	525 ± 200	0.04	[N]	327 ± 10	NA[J]	NA[J]
	2[I]	...	>800	...	0.04	[N]	327 ± 10	NA[J]	NA[J]
VII	...	...	635 ± 100	500 ± 200	0.04	327 ± 10	327 ± 10	NA[J]	NA[J]

[A] Former Type I, Classes 1 through 5, Type II, Type IV, Classes 5, 6, and 9, Type V, Class 2 have been deleted because they are no longer listed as commercial products.

[B] Formerly Type I, Class 6.

[C] Formerly Type III, Class 1.

[D] Formerly Type III, Class 2.

[E] Formerly Type III, Class 3.

[F] Formerly Type IV, Classes 1 through 4.

[G] Formerly Type V, Class 1.

[H] Formerly Type IV, Class 7.

[I] Formerly Type IV, Class 8.

[J] Not applicable.

[K] Tested at a reduction ratio of 100 to 1.

[L] Tested at a reduction ratio of 400 to 1.

[M] Tested at a reduction ratio of 1600 to 1. (Reduction ratio is the ratio of the cross-sectional area of the preform to the cross-sectional area of the die.)

[N] > 5° above second melting peak temperature.

TABLE 2 Detail Requirements for Tests on Molded Specimens

Type	Grade	Class	Thermal Instability Index, max	Standard Specific Gravity		Tensile Strength, min		Elongation, min, %
				min	max	MPa	psi	
I	...	...	50	2.13	2.18	17.2	2500	200
III	1	A	50	2.19	2.24	18.6	2700	400
		B	50	2.19	2.24	18.6	2700	400
		C	50	2.19	2.24	18.6	2700	400
	2	A	50	2.14	2.20	20.7	3000	200
		B	50	2.14	2.20	20.7	3000	200
		C	50	2.14	2.20	20.7	3000	200
	3	A	50	2.17	2.23	20.7	3000	200
		B	50	2.17	2.23	20.7	3000	200
		C	50	2.17	2.23	20.7	3000	200
	4	A	50	2.14	2.23	18.6	2700	200
		B	50	2.14	2.23	18.6	2700	200
		C	50	2.14	2.23	18.6	2700	200
IV	...	...	50	2.13	2.19	27.6	4000	300
V	...	...	50	2.16	2.22	20.7	3000	300
VI	1	...	50	2.13	2.19	25.5	3700	275
	2	...	50	2.13	2.19	27.6	4000	300
VII	...	...	NA[A]	NA[A]	NA[A]	NA[A]	NA[A]	NA[A]

[A] Not applicable by molding techniques included in this specification.

TABLE 3 Sintering Procedures

	A[A]	B	C	D	E	F	G	H	I
Initial temperature, °C (°F)	380 ± 6 (716 ± 10)	290 (554)	290 (554)	238 (460)	238 (460)	290 (554)	238 (460)	238 (460)	238 (460)
Rate of heating, °C/h (°F/h)	NA[B]	120 ± 10 (216 ± 18)	120 ± 10 (216 ± 18)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	120 ± 10 (216 ± 18)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)
Hold temperature, °C (°F)	380 ± 6 (716 ± 10)	380 ± 6 (716 ± 10)	357 ± 8 (675 ± 15)	371 ± 6 (700 ± 10)	363 ± 6 (685 ± 10)	380 ± 6 (716 ± 10)	357 ± 8 (675 ± 15)	380 ± 6 (716 ± 10)	371 ± 6 (700 ± 10)
Hold time, min	30 + 2, −0	30 + 2, −0	30 + 2, −0	240 ± 15	240 ± 15	360 ± 5	240 ± 15	960 ± 15	120 ± 5
Cooling rate, °C/h (°F/h)	72 ± 5 (132 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)	60 ± 5 (108 ± 9)
Final or second hold temperature, °C (°F)	300 ± 6 (572 ± 10)	294 ± 6 (561 ± 10)	294 ± 6 (561 ± 10)	238 ± 6 (460 ± 10)	238 ± 6 (460 ± 10)	294 ± 6 (561 ± 10)	238 ± 6 (460 ± 10)	238 ± 6 (460 ± 10)	238 ± 6 (460 ± 10)
Second hold time, min	NA[B]	24 + 0.5, −0	24 + 0.5, −0	NA[B]	NA[B]	24 + 0.5, −0	NA[B]	NA[B]	NA[B]
Period to room temperature[C], min, h	½	½	½	6	6	½	6	6	6

[A] Procedure A has been specified in previous issues of Specification D 1457 for Type III resins. For improved precision Procedure B should be used.

[B] Not applicable.

[C] After the specimens have cooled, store them at ambient temperature (ideally 23 ± 1°C) and remote from any laboratory heat source (that is ovens, etc.)

TABLE 4 Sieving Requirements[A]

Sieve Number	Type				
	III	IV[B]	V[B]	VI	VII
14 (1.41 mm)	X			X	
18 (1.00 mm)	X			X	X
25 (710 μm)	X	X	X	X	X
35 (500 μm)	X			X	X
45 (350 μm)	X			X	X
60 (250 μm)	X			X	
80 (177 μm)	X			X	X
120 (125 μm)					X
170 (88 μm)		X	X		
200 (74 μm)		X	X		
230 (63 μm)		X	X		
270 (53 μm)		X	X		
325 (44 μm)		X	X		
400 (37 μm)		X	X		X
Sample Size, g					
10 ± 0.1		X	X		
50 ± 0.1	X			X	X

[A] It is suggested that the sieves and sample size checked in a "Type" column be used when performing the sieve analysis on that particular type. However, other configurations of sieves may be used to obtain equivalent results.

[B] A discussion of the particle characteristics of finely divided resins is found in the Appendix.

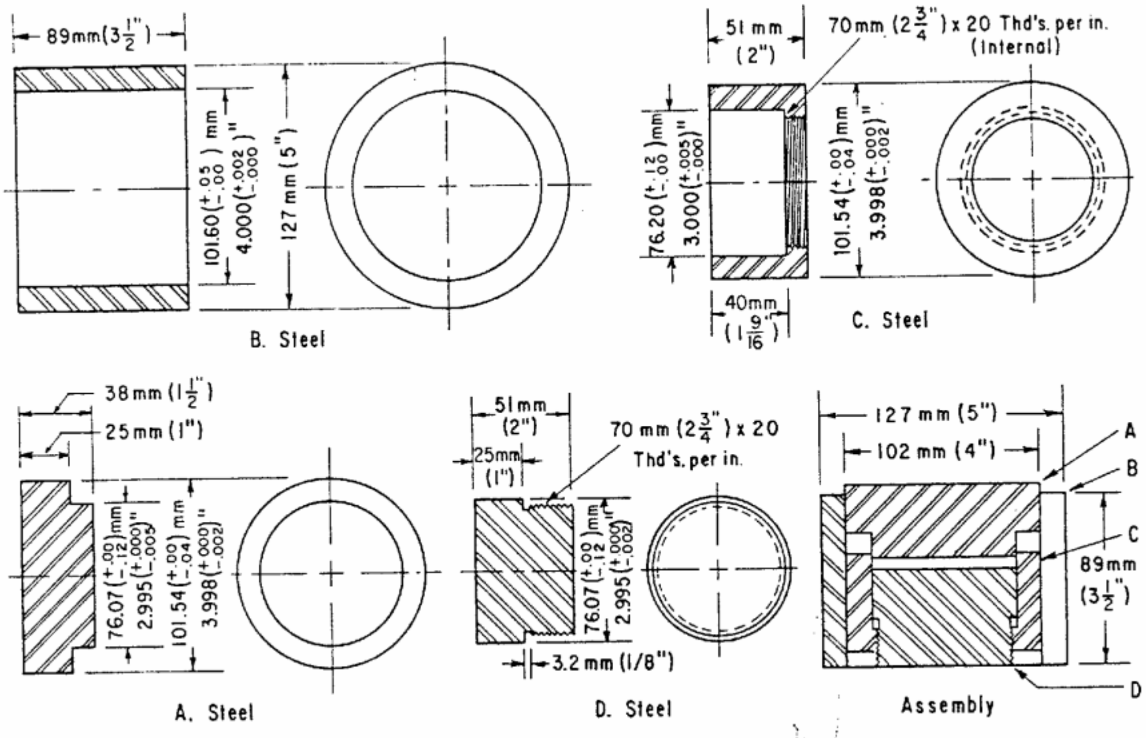
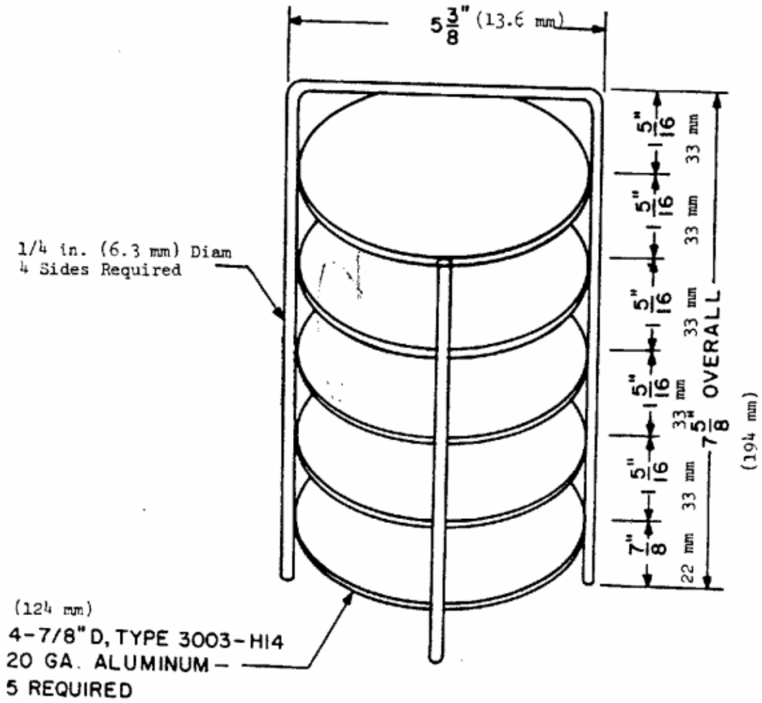


FIG. 1 Assembly and Details of Die for Molding Test Specimens



NOTE: ALUMINUM PLATES
TACK WELDED TO RODS

FIG. 2 Conditionary Rank for SSG Samples

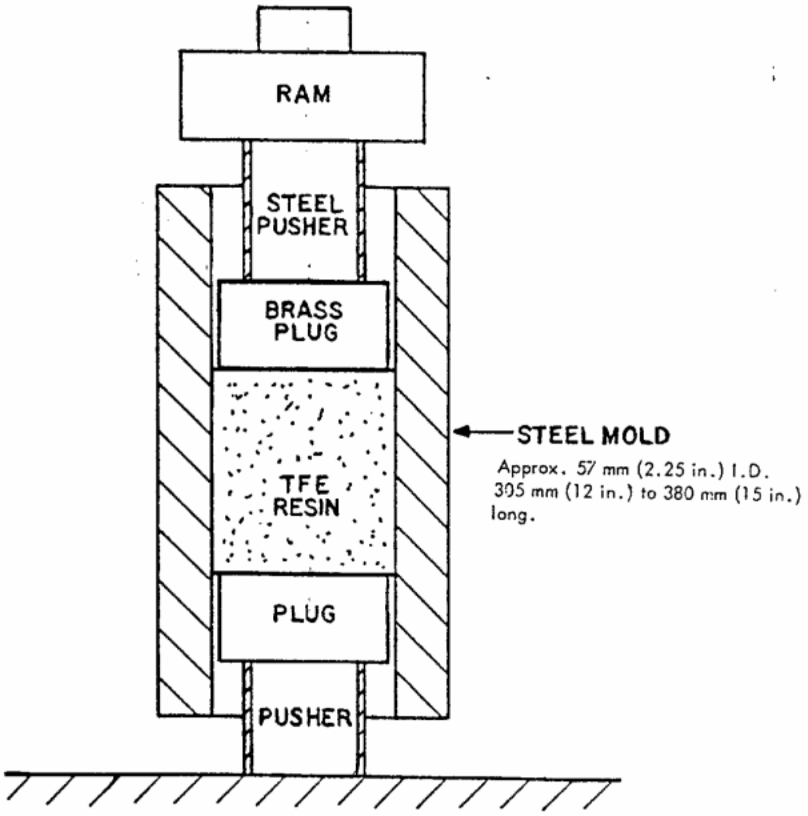


FIG. 3 Preforming of PTFE Test Billet

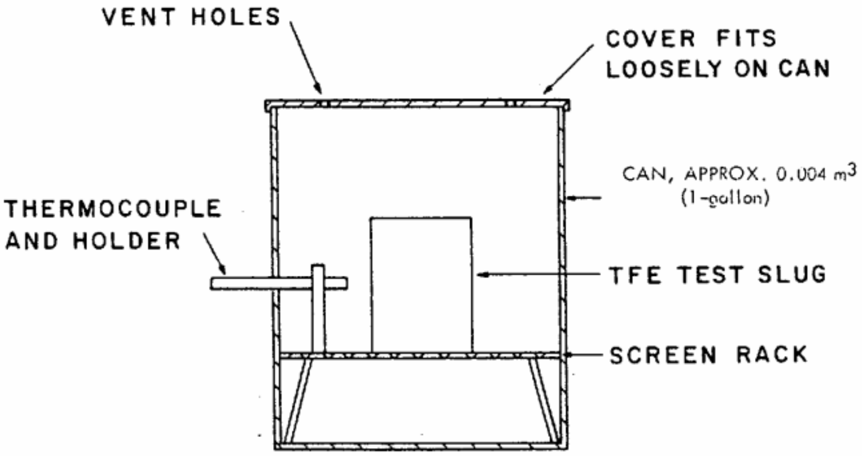


FIG. 4 Sintering Can

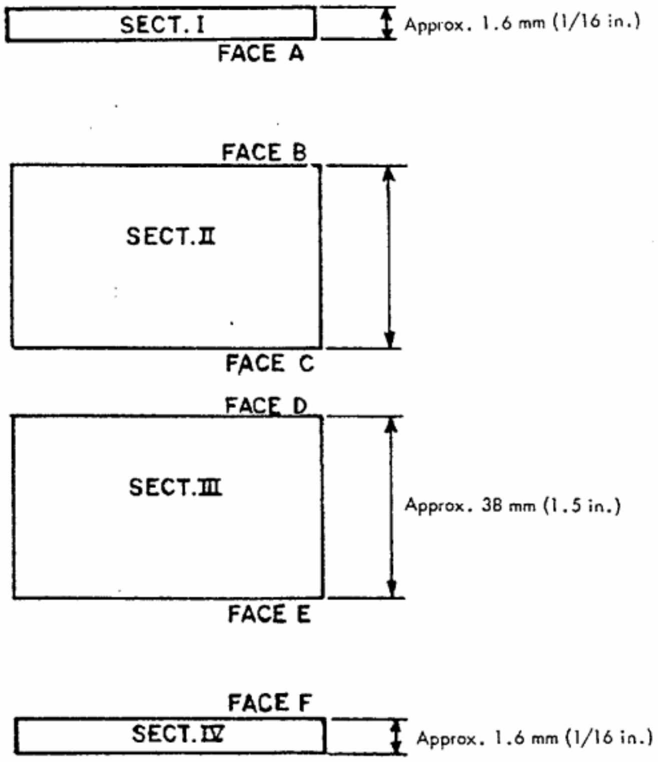


FIG. 5 Sectioned PTFE Test Billet

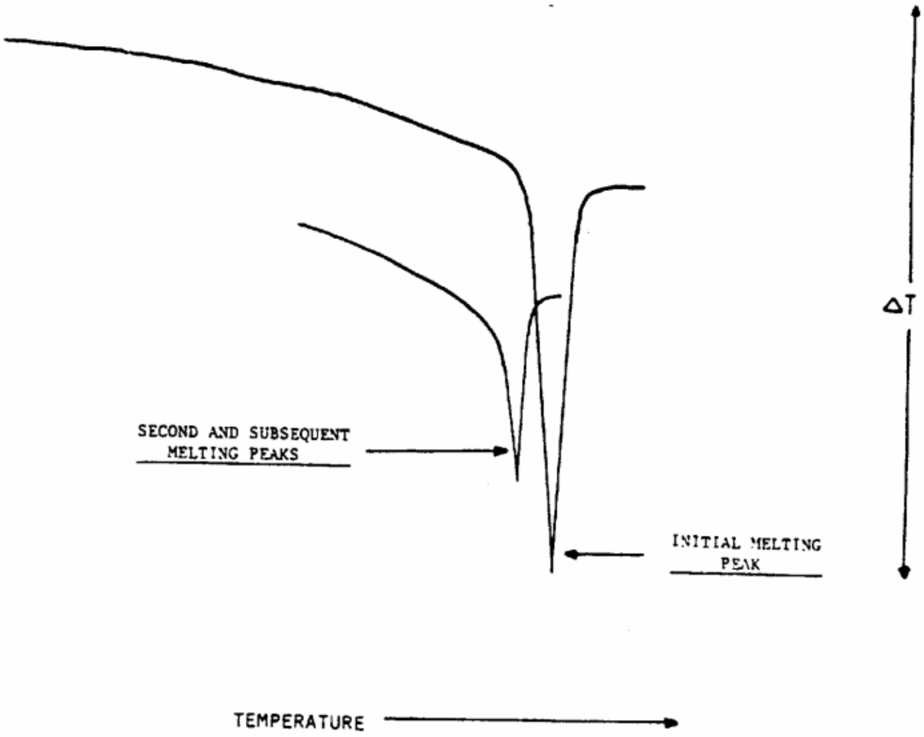


FIG. 6 Melting Characteristics by Thermal Analysis

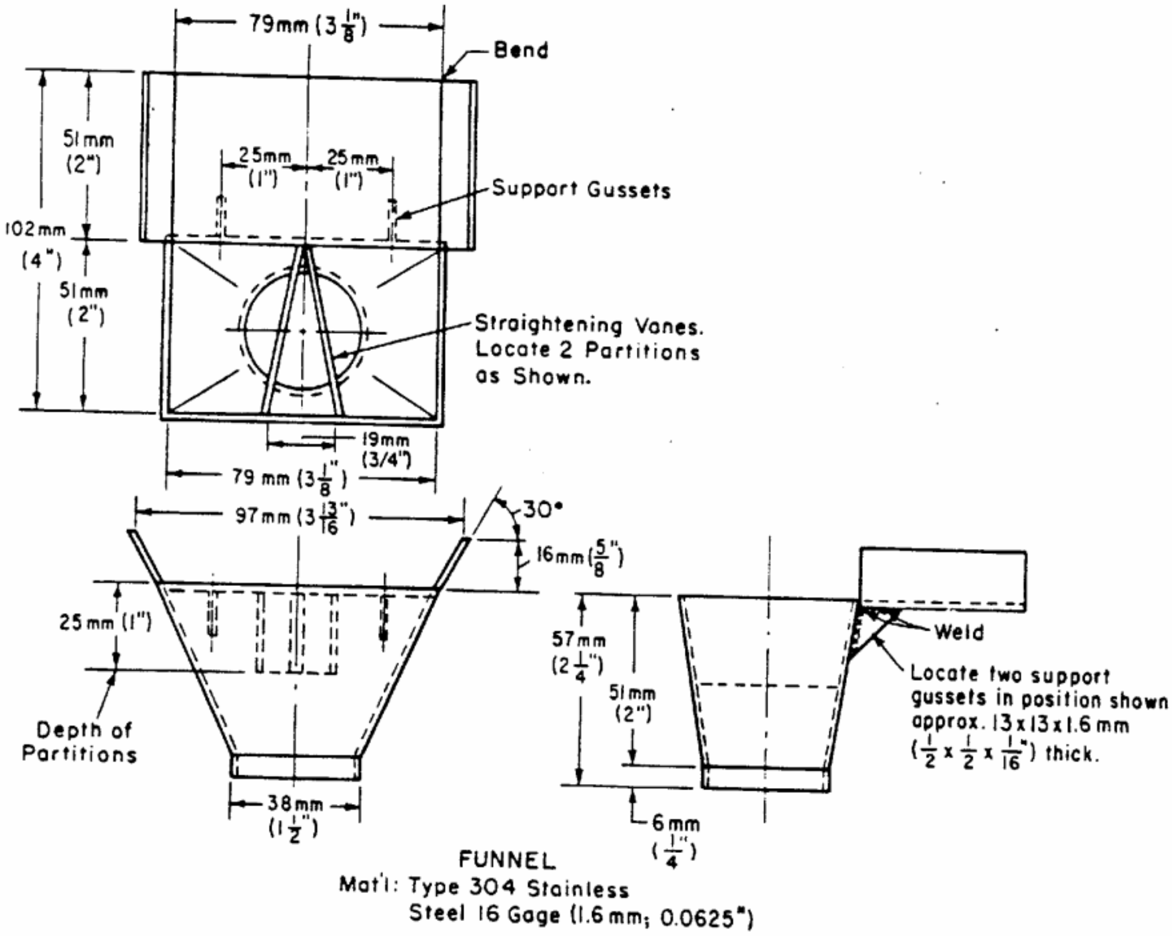


FIG. 7 Details of Funnel for Bulk Density Test

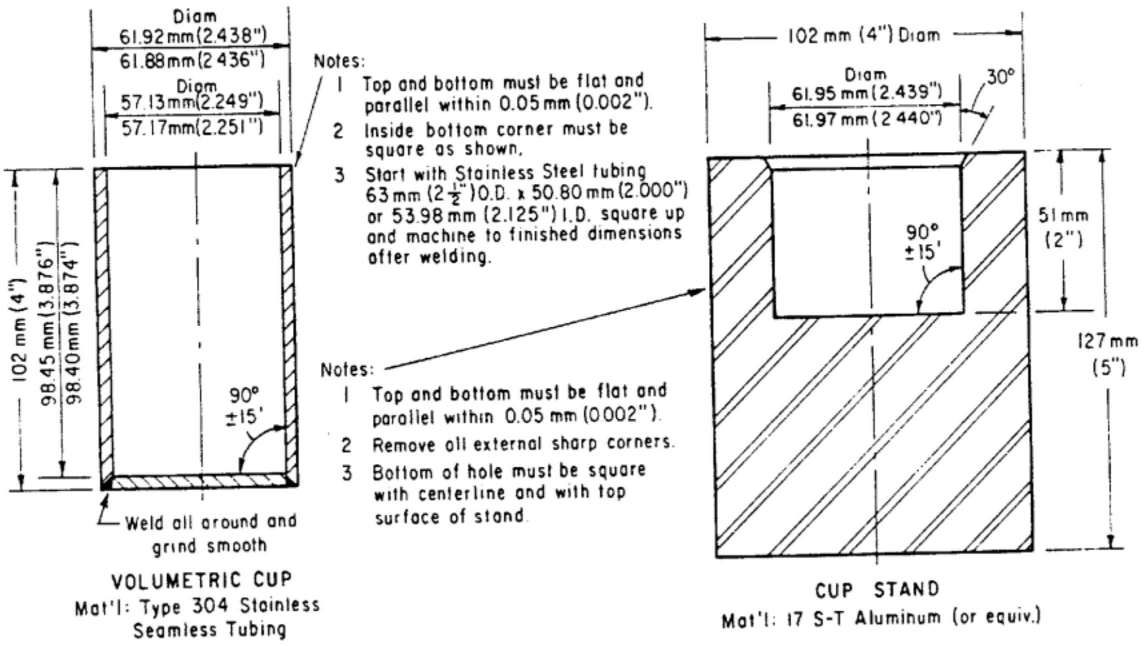


FIG. 8 Volumetric Cup and Cup Stand for Bulk Density Test

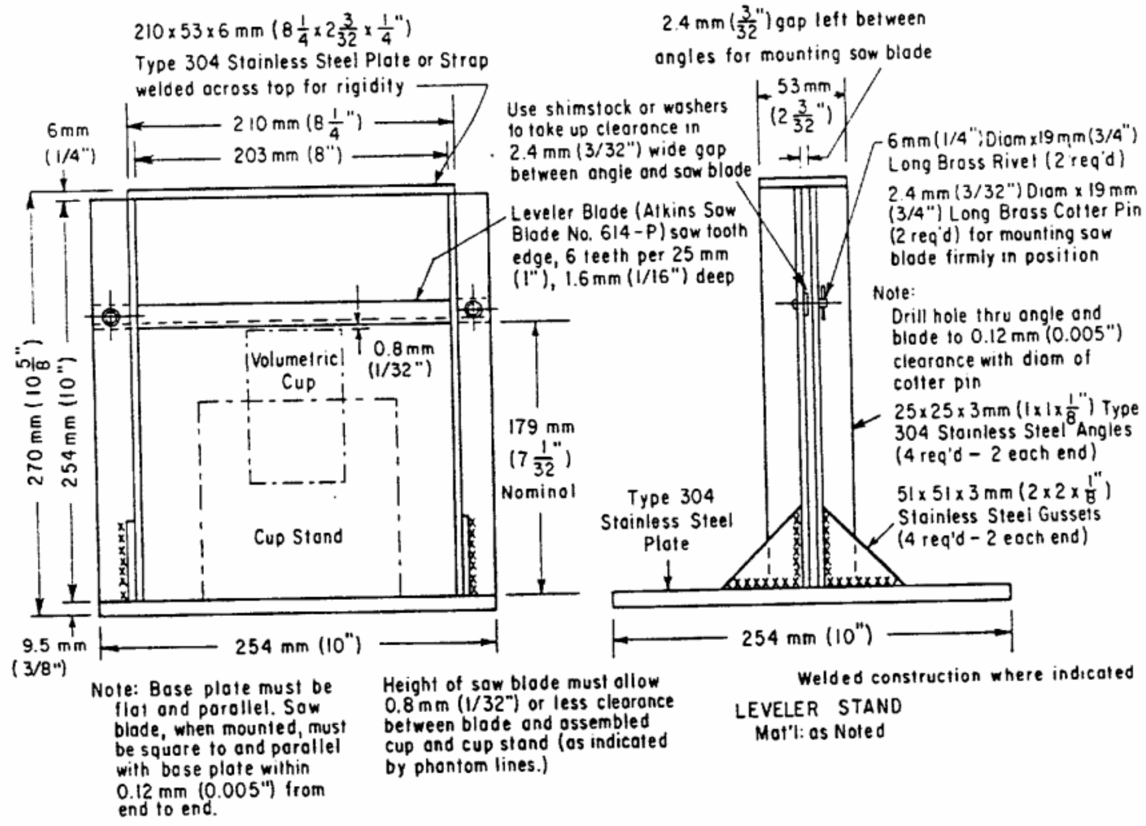
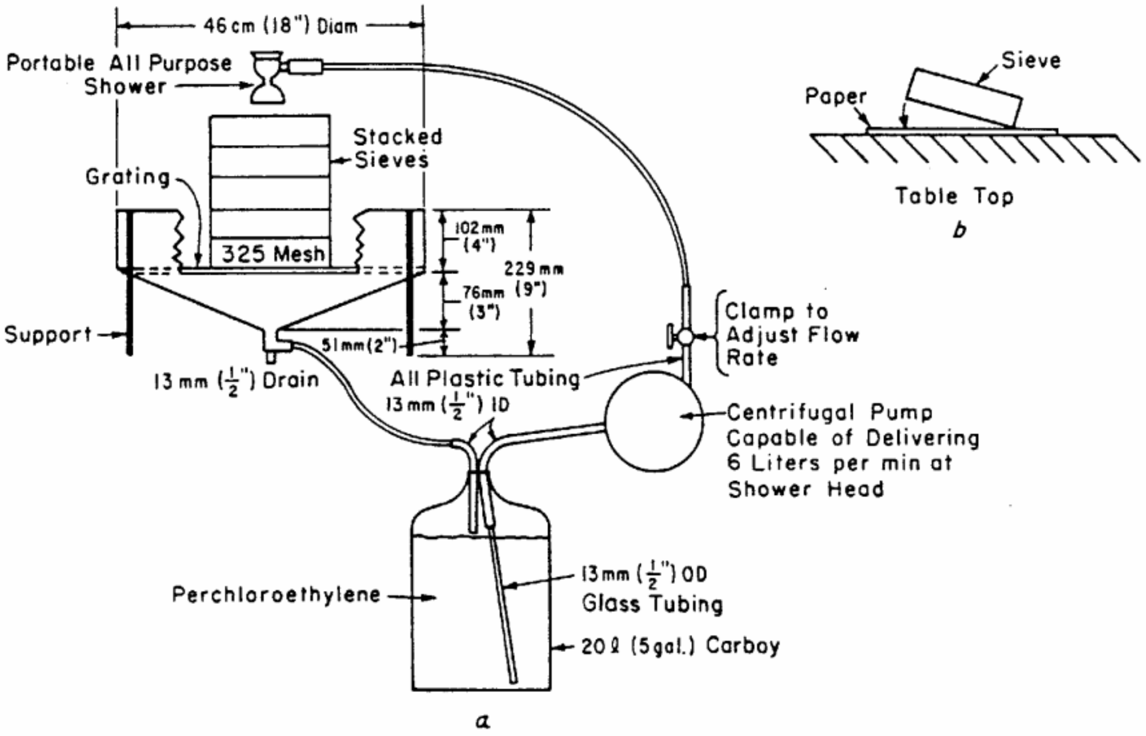
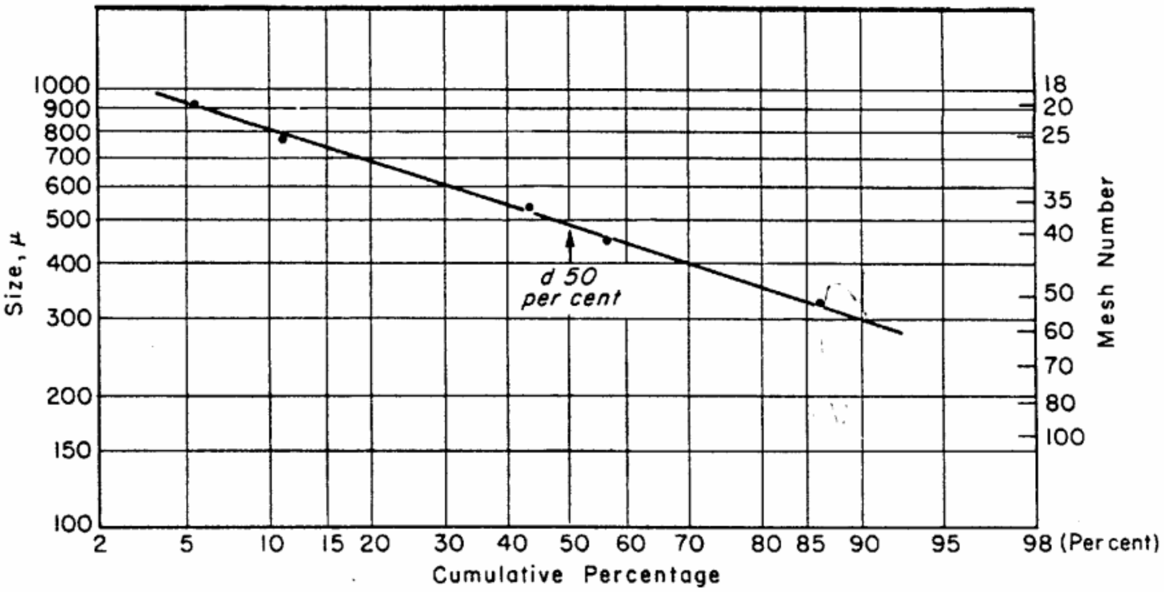


FIG. 9 Leveler Stand for Bulk Density Test



(*a*) Sieve and Spray Arrangement (*b*) Removing Powder from Sieve.

FIG. 10 Apparatus for Particle Size Test



Sieve No.	Sieve Opening, µm	Sieve No.	Sieve Opening, µm
18	1000	80	177
25	710	120	125
35	500	170	88
45	350	230	62
60	250	325	44

FIG. 11 Sample Plot of Cumulative Percent Versus Sieve Opening Size for Determination of Particle Size

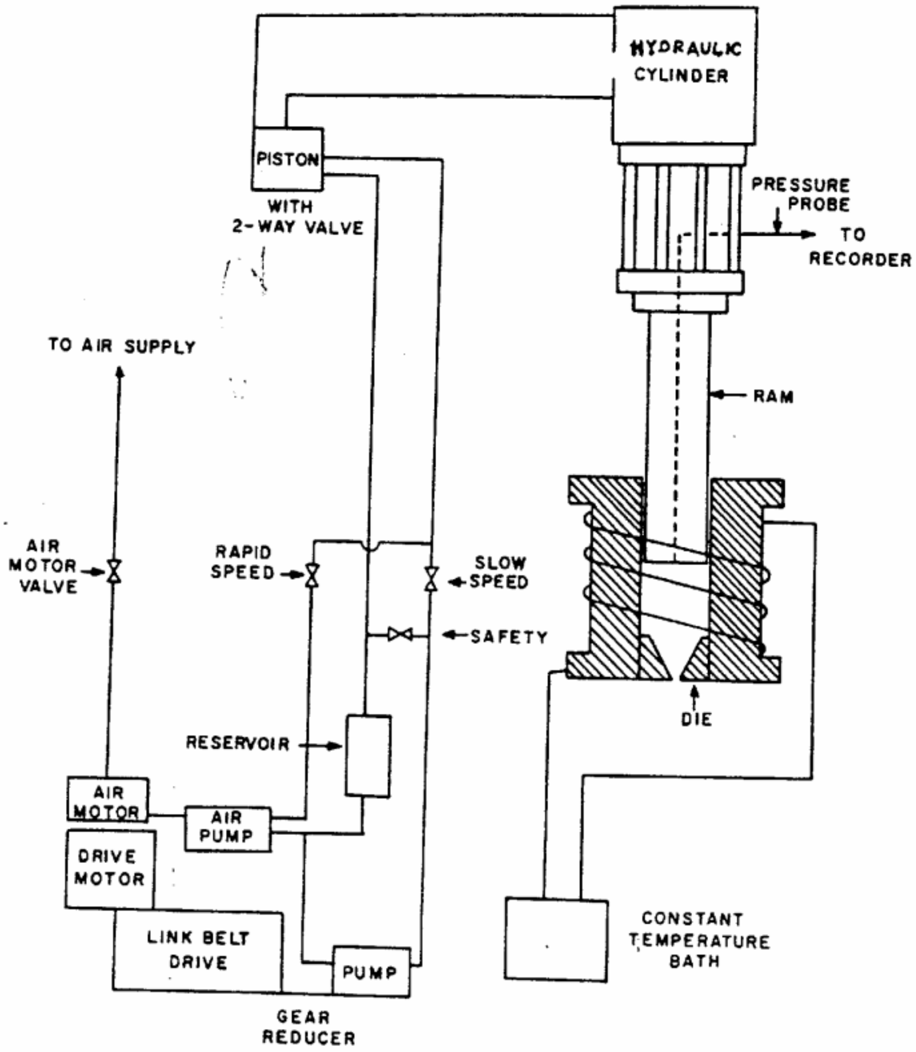


FIG. 12 Paste Extruder and Accessories

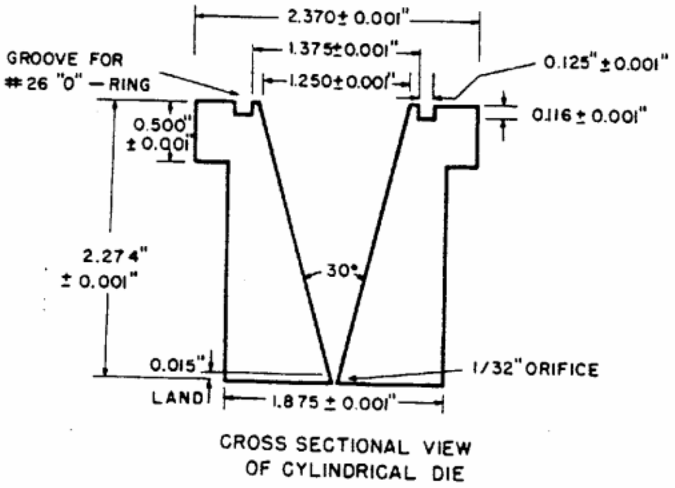
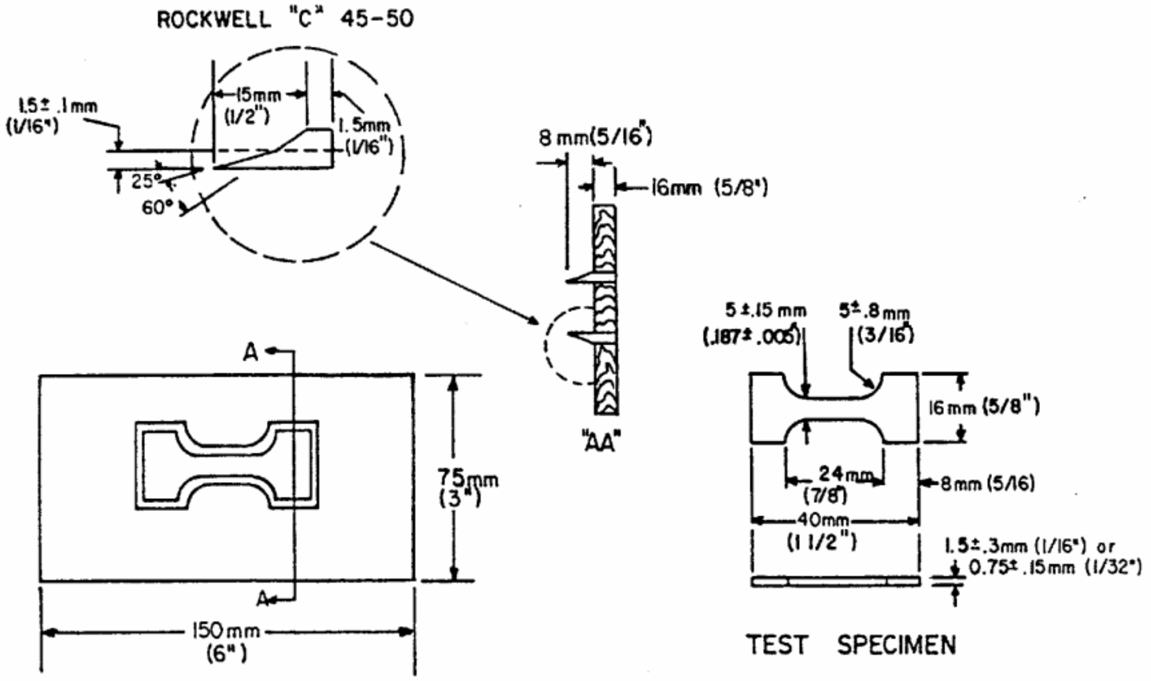


FIG. 13 Extruder Die Assembly



STEEL RULE DIE

(INSIDE DIMENSIONS FOR DIE ARE THE SAME AS TEST SPECIMEN)

DIE TO BE SHARPENED ON INSIDE EDGE ONLY! (AS SHOWN IN "AA")

NOT TO SCALE

FIG. 14 Microtensile Die

APPENDIX

(Nonmandatory Information)

XI. ADDITIONAL USEFUL TESTS

X1.1 Scope

X1.1.1 In addition to their use for specification purposes, the tests described in this specification have utility for characterizing PTFE resins. Other useful properties of PTFE can be measured by adding a few details to the specification tests. Others, taken from earlier edition of this specification still have utility. The purpose of this Appendix is to provide the details needed to determine these additional characteristics. The scope is summarized in Table X1.1.

X1.2 Applicable Documents

X1.2.1 *ASTM Standards:*

D 150 Test Methods for A-C Loss Characteristics and Permittivity (Dielectric Constant) of Solid Electrical Insulating Materials[2]
D 2990 Test Methods for Tensile, Compressive, and Flexural Creep and Creep-Rupture of Plastics[3]
D 3293 Specification for PTFE Resin Molded Sheet[4]
D 3294 Specification for PTFE Resin Molded Basic Shape[4]
D 3308 Specification for PTFE Resin Skived Tape[4]
D 3369 Specification for PTFE Resin Cast Film[4]

X1.3 Dimensional Changes During Molding (Shrinkage and Growth)

X1.3.1 Measure the inside diameter (ID) to ± 0.00254 mm (0.0001 in.) of the die used to make the preform in 9.1, 9.2, or 9.3. Measure the diameter and height at the preform. After the piece has been sintered and before using it for other testing, remeasure the diameter and height.

X1.3.2 *Calculations*:

$$\text{Percent mold shrinkage} = [(\text{diameter of sintered piece}/\text{ID of die}) - 1]\ 100$$

$$\text{Percent preform shrinkage} = [(\text{diameter of sintered piece}/\text{diameter of preform}) - 1]\ 100$$

$$\text{Percent growth} = [(\text{height of sintered piece}/\text{height of preform}) - 1]\ 100$$

Positive values reflect an increase in the dimension during sintering. Negative values reflect a decrease in the dimension during sintering.

X1.3.3 *Precision*—Data on precision are being assembled.

X1.4 Size and Distribution of Size of Particles or Agglomerates in PTFE Resins

X1.4.1 *Average Size of Fine-Particle Size PTFE*—Wet-sieve analysis, while having disadvantages, can be used to measure the average size of Type IV and Type V PTFE resins. The procedure of 12.3.2.5 should be followed using the set of sieves listed in Table 4 and a sample size of 10.0 g.

X1.4.2 *Material Retained on No. 230 (63-μm) Sieve*:

X1.4.2.1 *Scope*—A wet sieving is performed with the apparatus used for the determination of particle size, except that only two sieves are employed. This method is applicable to ultrafine resins such as Type IV resins.

X1.4.2.2 *Principle of Method*—The resin is sieved on a No. 230 (63-μm) sieve by spraying with perchloroethylene which breaks up agglomerates and prevents clogging of the sieve openings (See Note 10).

X1.4.2.3 *Apparatus*—Same as in 12.3.2, except that the following sieves are used: U.S. Standard Sieves, No. 20 (850-μm), No. 230 (63-μm), and No. 325 (45-μm).

X1.4.2.4 *Procedure*:

X1.4.2.4.1 Weigh 10 ± 0.01 g of resins. Assemble the sieves as shown in Fig. 10(*a*) with the No. 20 on top of the No. 230. Adjust the flow rate of the perchloroethylene to 6 ± 0.5 L/min.

X1.4.2.4.2 Place the weighed sample on the No. 20 sieve and spray with perchloroethylene for exactly 1 min using a timer. This step assists in breaking up agglomerates. Move the shower head in a circular fashion, taking care to break up all the agglomerates and to wash the material from the sides of the sieve.

X1.4.2.4.3 Remove the No. 20 sieve and spray the No. 230 sieve for exactly 6 min, using a timer. Wash the material to the side of the sieve during the last minute.

X1.4.2.4.4 Dry the sieve and retained resin in an oven for 20 min or longer at 80 to 120°C. The No. 20 sieve does not require drying.

X1.4.2.4.5 Remove the material from the No. 230 sieve by inverting on a piece of filter paper and tapping to free dry polymer. A stiff brush may be used to help free all the material from the sieve. Pour the dried resin into a tared weighing dish and weigh to ± 0.01 g. Alternatively, determine the tare mass of the resin retained on the sieve from the difference between the gross mass after sieving and the tare mass of the sieve before sieving. A balance with a sensitivity of about 0.01 g is required for good precision.

X1.4.2.4.6 Calculate the percentage retained on the No. 230 sieve as follows:

$$\text{Amount retained, }\% = (\text{weight retained}/\text{sample weight}) \times 100$$

X1.4.3 *Distribution of Particle or Agglomerate Sizes in PTFE Resin*:

X1.4.3.1 *Procedure*—Using the graph plotted in accordance with 12.3.2.5.3 or 12.3.3.4, draw the "best" smooth curve through the data points and read the values for the sizes at cumulative percentages of 16 and 84. These values, identified as $d16$ and $d84$, are, respectively, the size of the resin at the average diameter ($\bar{d}$) plus 1 sigma and $\bar{d}$ minus 1 sigma. Calculate a distribution factor (DF) and skewness (SKEW) as follows:

$$\mathrm{DF} = d16/d50$$

$$\mathrm{SKEW} = \mathrm{DF}/(d50/d84)$$

X1.4.3.2 *Precision*—Because the resin particles have complex shapes, and because on each sieve there is a distribution of particle sizes, the values for particle size and particle-size distribution obtained will be only relative numbers. The 95 % confidence limits based on a limited series of tests are ± 2.8 % for the average particle size and ± 6 % for the particle size distribution function.

X1.5 Yield Behavior and Tangent Modulus at Rupture

X1.5.1 Most of the PTFE resins covered in this standard do not show a yield stress as defined in Test Method D 638. Rather than the stress having a zero slope, the rate of increase of stress with strain decreases and then increases again. An approximate yield stress can be reported as the stress at the intersection of the two lines that best represent the initial "linear" part of the stress strain curve and the second "linear" part of the curve.

X1.5.2 *Tangent Modulus at Rupture*—The shapes of tensile stress-strain curves for PTFE resins are highly dependent on the crystallinity of the test specimen. Values for tensile strength and elongation at break do not reflect these shapes clearly. The value of the tangent to the recorded stress-strain curve measured as the best straight line from the point of rupture back along the curve is a convenient measure of the relative crystallinity of the test specimen. High values for the tangent modulus at rupture (>7.6 MPa) (1200 psi)) indicate relatively low crystalline contents. As the crystallinity increases, the tangent modulus at rupture decreases until it approaches zero at high levels of crystallinity.

X1.6 Heats of Fusion and Crystallization

X1.6.1 If the melting characteristics of the PTFE resin, as determined by Section 12, are determined by Differential Scanning Calorimetry (DSC) rather than in DTA mode, additional quantitative information can be obtained on the nature of the resin.

X1.6.2 Following the procedures for determining heats of fusion (ΔHF) and heat of crystallization (ΔHc) by DSC, measure and report ΔHf for the initial and second endotherms and ΔHc for the exotherm that is observed during controlled cooling between the two heating steps. These heats of transition, especially ΔHc, provide additional characterization of crystalline content and relative molecular weight of PTFE resins.

X1.7 Electrical Properties

X1.7.1 Determine dielectric constant and dissipation factor in accordance with Test Method D 150. Determine dielectric breakdown voltage and dielectric strength in accordance with Test Method D 150. Typical property values for dielectric constant and dissipation factor are listed in Table X1.2. Standards for dielectric strength of sheet, basic shapes, skived tape, and film are described in Specifications D 3293, D 3294, D 3308, and D 3369, respectively.

X1.8 Tensile Creep

X1.8.1 Determine the tensile creep of Type V materials on Type II tensile bars die cut or machined from the sheets produced in X1.8.2. Make measurements in accordance with Test Method D 2990. Conditions of test shall be 5.52 MPa (800 psi) stress at the Standard Laboratory Temperature of 23 ± 2°C (73.4 ± 3°F) for a test duration of a minimum of 100 h. Typical values for moldings of Type V resins would be a maximum of 4.0 % tensile creep strain after 100 h.

X1.8.2 Mold test sheets for Type V resins for tensile creep requirements in a picture frame mold having inside dimensions of 203 mm (8.0 in.) square and of sufficient height to contain the sample. A frame 102 mm (4 in.) in height has been found adequate when using 25-mm (1-in.) thick pusher plugs to produce a sheet approximately 3 mm (⅛ in.) in thickness from a resin charge of 300 g. Take care to level the resin charge in the mold. The molded sheet thickness shall be 3 mm (⅛ in.).

X1.8.3 Sinter the preform according to Procedure E of Table 3.

TABLE X1.1

Specification Test Reference	Specification Property Measured	Additional Information Available
12.5.1	Standard specific gravity	Dimensional change during molding (shrinkage and growth)
12.1	Melting temperature (by DSC)	Heat of fusion and crystallization
12.3.2.5 and 12.3.3	Average size of the resin	(*a*) Average size of resins smaller than covered by the standard (*b*) Percent coarse particles (percent > 63 μm) in small particle size resins
12.7	Tensile strength and elongation at break	Yield stress and tangent modulus at rupture Electrical properties: Dielectric constant Dissipation factor Dielectric breakdown voltage Dielectric strength Tensile creep strain

TABLE X1.2 Typical Electrical Properties from Tests on Molded Specimens

	Type III	Type IV	Type V
Dielectric constant, max, 10^3 Hz	2.1	2.1	2.1
Dissipation factor, max, 10^3 Hz	0.0003	0.0003	0.0003

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