Designation: ~~F451 – 16~~ F451 – 21

Standard Specification for Acrylic Bone Cement[1]

This standard is issued under the fixed designation F451; 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 self-curing resins used primarily for the fixation of internal orthopedic prostheses. The mixture may be used in either the ~~predough~~pre-dough or dough stage in accordance with the manufacturer's recommendations.

1.2 Units of ~~premeasured~~pre-measured powder and liquid are supplied in a form suitable for mixing. The mixture then sets in place.

1.3 While a variety of copolymers and comonomers may be incorporated, the composition of the set cement shall contain poly(methacrylic acid esters) as its main ingredient.

1.4 This specification covers compositional, physical performance, and biocompatibility as well as packaging requirements. The biocompatibility of acrylic bone cement as it has been traditionally formulated and used has been reported in the literature (**1**, **2**).[2]

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D638 Test Method for Tensile Properties of Plastics
D695 Test Method for Compressive Properties of Rigid Plastics
D1193 Specification for Reagent Water
D3835 Test Method for Determination of Properties of Polymeric Materials by Means of a Capillary Rheometer
D5296 Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size-Exclusion Chromatography

[1] This specification is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.11 on Polymeric Materials.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[3] 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.

D5630 Test Method for Ash Content in Plastics
E169 Practices for General Techniques of Ultraviolet-Visible Quantitative Analysis
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers
F619 Practice for Extraction of Materials Used in Medical Devices
F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices
F749 Practice for Evaluating Material Extracts by Intracutaneous Injection in the Rabbit
F756 Practice for Assessment of Hemolytic Properties of Materials
F763 Practice for Short-Term Screening of Implant Materials
F813 Practice for Direct Contact Cell Culture Evaluation of Materials for Medical Devices
F895 Test Method for Agar Diffusion Cell Culture Screening for Cytotoxicity
F981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Insertion into Bone

2.2 *ANSI/ADA Standard:*[4]
No. 15 Specification for Acrylic Resin Teeth

2.3 *ISO Standards:*[5]
ISO 5833 Implants for Surgery—Acrylic Resin Cements
ISO 80000-9 Quantities and Units—Part 9: Physical Chemistry and Molecular Physics

2.4 *NIST Document:*[6]
Special Publication 811

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *doughing time*—the time after commencement of mixing at which the mixture ceases to adhere to a standard probe (see ~~7.5~~7.6).

3.1.1.1 *Discussion*—
"Doughing time" and "dough time" are interchangeable in this standard.

3.1.2 *exothermic or maximum temperature*—the maximum temperature of the mixture due to self-curing in a standard mold (see ~~7.6~~7.7).

3.1.3 *extrusion*—the rate of flow of the material through a standard orifice under load (see ~~7.8.1~~7.9.1).

3.1.4 *intrusion*—the distance of flow of the mixture into a standard mold under load (see ~~7.8.3~~7.9.3).

3.1.5 *setting time*—the time after commencement of mixing at which the temperature of the curing mass equals the average of the maximum and ambient temperatures (see ~~7.7~~7.8).

3.1.5.1 *Discussion*—
"Setting time" and "set time" are interchangeable in this standard.

3.1.6 *unit*—one package or vial of ~~premeasured~~pre-measured powder component and one package or vial of ~~premeasured~~pre-measured liquid component.

4. Physical Requirements

4.1 ~~*Liquid:*~~ *Liquid*—The liquid component includes the monomer, inhibitors, accelerants, and, if applicable, colorants.

4.1.1 *Appearance*—The liquid shall be free of extraneous particulate matter or obvious visual contaminants in its container.

4.1.2 *Stability*—After being heated for 48 h at 60 ± ~~2°C,~~2 °C, the viscosity of the liquid shall not increase by more than 10 % of its original value (see ~~7.3~~7.4).

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.
[5] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.
[6] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

4.1.3 *Sterility*—The liquid, as poured from its container, shall pass the tests described in "Sterility Tests—Liquid and Ointments" (~~7.4~~7.5) (**3**).

4.2 ~~*Powder:*~~ *Powder*—The powder component includes the polymer particles, initiator agents, the radio-opaque agent, and if applicable, other additives such as antibiotics and colorants.

4.2.1 *Appearance*—The powder shall be pourable and free of extraneous materials, such as dirt or lint (7.2.2).

4.2.2 *Sterility*—The powder, as poured from its package, shall pass the tests described in "Sterility Tests—Solids" (~~7.4~~7.5) (**2**).

4.3 *Powder-Liquid* ~~*Mixture:*~~ *Mixture*—

~~4.3.1 If the mixture is to be used in its predough stage, the material shall conform to the properties given in Table 1.~~ The material shall conform to the properties given in Table 1.

~~4.3.2 If the mixture is to be used in its dough stage, the material shall conform to the properties given in Table 1.~~

~~4.3.3 If the mixture can be used in either its predough or dough stages, separate units must be tested for compliance with 4.3.1 and 4.3.2.~~

4.4 *Cured* ~~*Polymer*~~—*Cement*—The material after setting shall conform to the properties given in Table 2.

TABLE 2 Requirements for Cured Polymer After Setting

Property	Requirement
~~Compressive Strength, min., MPa~~	~~70~~
Compressive Strength, minimum, MPa	70

5. Weights and Permissible Variations

5.1 Weight and volume measurements shall be made on the respective powder and liquid components of five units (see ~~3.1~~9.2.2). These units may be subsequently utilized in any of the nonsterile tests of this specification.

5.2 The weights, or volume of the powder and liquid components, or both, shall not deviate by more than 5 % from those stated on the package (9.2.2), of each of five units.

6. Sampling

6.1 Units of powder and liquid shall be procured to provide sufficient material for all the tests of this specification. The units shall be obtained from regular retail distribution channels. Provided no repeat tests are required, this will amount to between seven and ten units.

TABLE 1 Requirements for Powder Liquid Mixture

Property	~~Extrusion, Viscosity Tests~~	~~Dough Usage, Intrusion Tests~~
~~Max Dough Time, min.~~	~~5.0~~	~~5.0~~
~~Setting Time Range, min.~~	~~5 to 15~~	~~5 to 15~~
~~Temperature, max., °C~~	~~90~~	~~90~~
~~Intrusion, min., mm~~	~~...~~	~~2.0~~

TABLE 1 Requirements for Powder Liquid Mixture

Property	Required Values or Ranges
Max Dough Time, minutes	5.0
Setting Time Range, minutes	5 to 15
Temperature, maximum, °C	90
Intrusion, minimum, mm	2.0

6.2 It will only be necessary to maintain sterility in tests described in ~~7.4~~7.5. All other tests described in this specification need not be conducted under sterile conditions.

7. Test Methods and Sample Size

7.1 Maintain all equipment, mixing surfaces, and materials at 23 ± ~~2°C~~ 1 °C for at least 2 h prior to testing and conduct all tests at 23 ± ~~2°C~~1 °C and 50 ± 10 % relative humidity unless otherwise specified.

7.2 *Inspection*—Use visual inspection in determining compliance to the requirements outlined in 4.1.1, 4.2.1, 8.1, and 8.2.

7.2.1 The liquid component of two separate units shall comply with the requirements of 4.1.1 and 8.1.

7.2.2 The powder component of two separate units shall comply with the requirements of 4.2.1 and 8.1.

7.3 *Radiopacifier Content in Powder Component*—The radiopacifier content in the powder component shall be assessed by net ash testing according to Test Method D5630, Procedure B. The radiopacifier content shall not vary from the nominal content by more than 10 %.

7.4 *Liquid Component ~~Viscosity~~—Viscosity Stability*—Record the viscosity change of two separate units (4.1.2) before and after the heating exposure by timing the flow of the liquid level between the 0 and 5 mL marks of a 10 mL measuring pipet. Calculate the percent change as follows:

$$\% \text{ Change} = \frac{t_a - t_b}{t_b} \times 100 \quad (1)$$

$$\% \text{ Change} = \frac{t_a - t_b}{t_b} \times 100 \quad (1)$$

where:

t_b = flow time before heating, and
~~t_a = flow time after heating exposure (4.1.2) of 60 ± 2°C for 48 h in the dark in a closed container.~~
t_a = flow time after heating exposure (4.1.2) of 60 ± 2 °C for 48 h in the dark in a closed container.

7.4.1 An alternative method for viscosity may be used if it can be demonstrated to yield similar results. Both shall comply to the less than 10 % change specified (4.1.2).

7.5 The components of the two units shall be tested for sterility in accordance with the test methods described in U.S. Pharmacopoeia, "Sterility Tests" (**3**).

7.6 *Doughing Time:*

7.6.1 *Environment*—All equipment, mixing surfaces, and material (unit size) shall be maintained at 23 ± ~~1°C~~ 1 °C for at least 2 h prior to testing and tests shall be conducted at 23 ± ~~1°C.~~1 °C. The relative humidity shall be 50 ± 10 %.

7.6.2 Mix all the powder and liquid of a single unit together as directed by the manufacturer's instructions (see 8.2). Start a ~~stop watch~~ stopwatch at the onset of combining the liquid ~~to~~and the powder and read all subsequent times from this ~~stop watch.~~ stopwatch. Approximately 1.5 min after the onset of mixing, gently probe the mixture with a non-powdered surgically gloved (latex) finger. Take visual notice as to the formation of fibers between the surface of the mix and the finger as it leaves the surface. Repeat this process from that time on at 15 s intervals with a clean portion of the glove until the gloved finger separates cleanly. Denote the time at which this is first observed as the doughing time. Mix the mixture between determinations to expose fresh material for each probing.

7.6.3 Determine the average doughing time from two separate units.

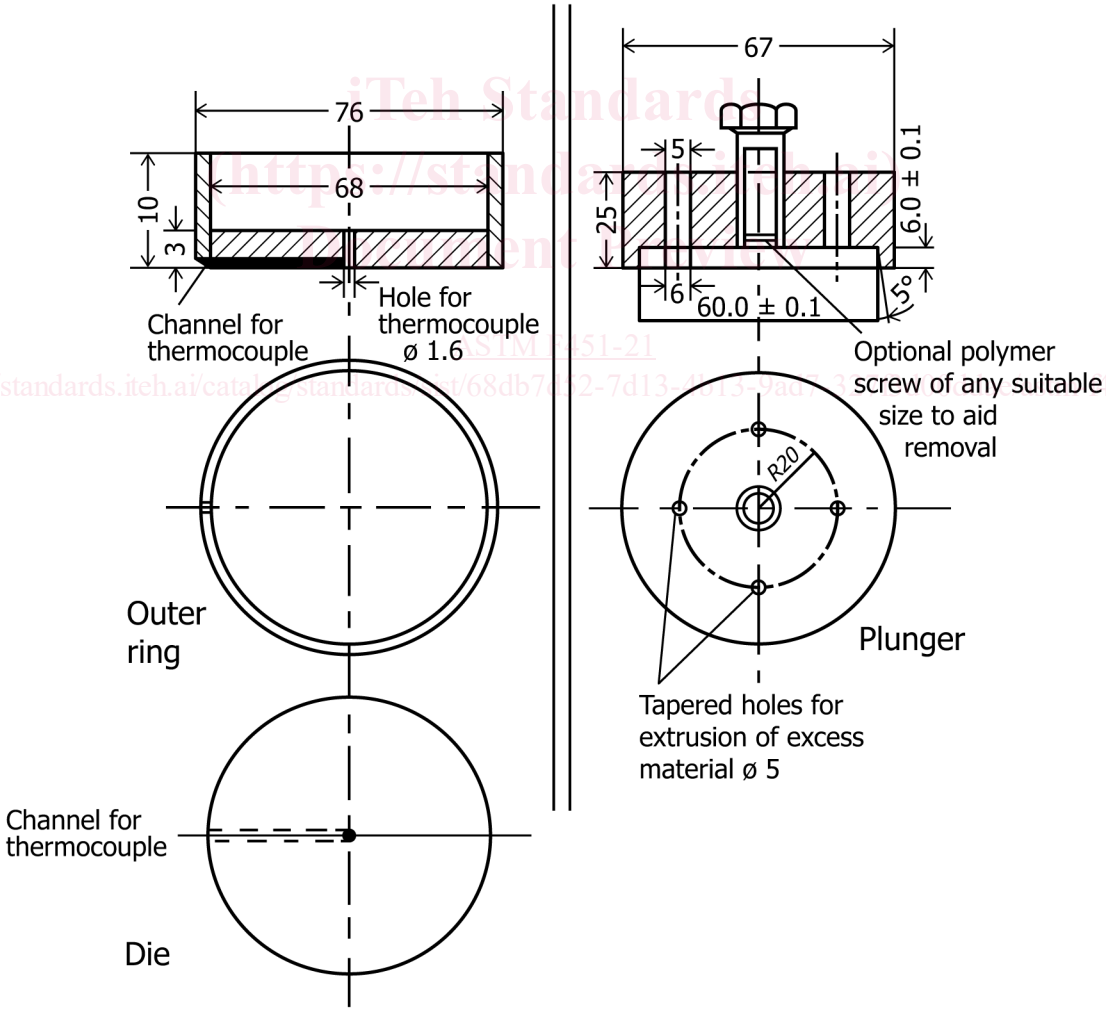
7.6.4 The two values found shall agree within 30 s of each ~~other,~~other; otherwise repeat the test on two additional units. Report the average of all four tests and the range of values.

7.6.5 Report the doughing time to the nearest 15 s as the average of all determinations. Maximum and minimum values of doughing times measured shall not differ by more than ±1½ min from the average.

7.6.6 Report the brand of non-powdered surgical glove used for dough time determinations. It is necessary that the type of glove be described in detail, including manufacturer, when the dough time is reported.

7.7 *Exothermic Temperature*—Within 1 min after doughing time, gently pack approximately 25 g of the dough described in ~~7.5~~7.6 into the mold described in Fig. 1. This mold shall be made of polytetrafluoroethylene (PTFE), poly(ethyleneterephthalate), polyoxymethylene, ~~high density~~ high-density polyethylene, or ultra-high molecular weight polyethylene (UHMWPE) and be equipped with a No. 24 gage wire thermocouple, or similar device, positioned with its junction in the center of the mold at a height of 3.0 mm in the internal cavity. Immediately seat the plunger with a C-clamp or suitable press to produce the 6.0 mm specimen height. Upon producing plunger seating, remove the excess material and the C-clamp or press for the remainder of the procedure. Continuously record the temperature with respect to time from the onset of mixing the liquid and the powder until cooling is ~~observed,~~ observed (see Fig. 2~~.~~). Report the maximum temperature recorded to the nearest ~~1°C.~~1 °C. This should not exceed the value given in Table 1.

7.7.1 The average maximum temperature shall be the calculated average of two separate maximum temperature determinations reported to the nearest ~~1°C.~~1 °C.



NOTE 1—Dimensions in millimetres and ±0.2 unless otherwise specified. Material for all components: Polytetrafluoroethylene, poly(ethyleneterephthalate), polyoxymethylene, ~~high density~~ high-density polyethylene, or ultra-high molecular weight polyethylene (UHMWPE).

FIG. 1 Exothermic Heat Mold

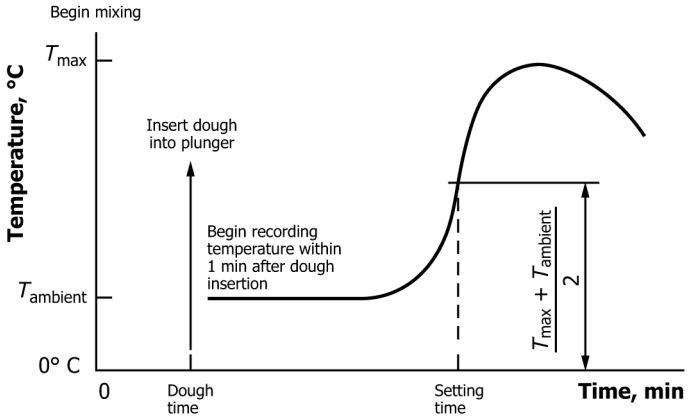


FIG. 2 Continuous Temperature Record

7.7.2 If the difference between the maximum temperature for the two determinations is greater than ~~5.0°C,~~5.0 °C, repeat the test on two additional units and report the average of all four runs to the nearest ~~1°C.~~1 °C. Individual maximum and minimum values for maximum temperature shall not differ by more than ~~±4°C~~±4 °C of the average value of all determinations.

7.8 *Setting Time*—From the continuous ~~time versus temperature~~ time-versus-temperature recording of ~~7.6~~7.7, the setting time (T_{set}) is the time when the temperature of the polymerizing mass is as follows:

$$(T_{max} + T_{amb})/2 \quad (2)$$

where:

T_{max} = maximum temperature, °C, and
~~T_{amb} = ambient temperature of 23 ± 1°C.~~
T_{amb} = ambient temperature of 23 ± 1 °C.

7.8.1 Report the setting time to the nearest 5 s.

7.8.2 Make two separate determinations of the setting time.

7.8.3 The two values should agree within 1 ~~min~~minute of each ~~other,~~other; otherwise repeat the test on two additional units and report the average of all runs.

7.8.4 Report the setting time to the nearest 15 s as the average of all determinations.

7.9 *Flow Properties and Viscosity Determination*—The manufacturer must specify whether the cement may be used in its pre-dough or dough state, or both. The determination of its usage dictates which of the following tests the cement should comply with. If the mixture is to be utilized in the pre-dough stage, use the extrusion viscosity test (~~7.8.1~~7.9.1 and/or ~~7.8.2~~7.9.2) and Table 1. If the mixture is to be utilized in the dough stage, use the intrusion test (~~7.8.3~~7.9.3) and Table 1. If the mixture is to be used as a dual usage cement, then both the extrusion (~~7.8.1~~7.9.1 and/or ~~7.8.2~~7.9.2) and intrusion (~~7.8.3~~7.9.3) tests ~~must~~shall be performed.

7.9.1 *Extrusion, Capillary Viscosity:*

7.9.1.1 *Apparatus:*

(1) Capillary Rheometer—Any capillary rheometer ~~is satisfactory~~ in which acrylic bone cement can be forced from a reservoir through a capillary die and in which temperature, applied force, output rate, and barrel and die dimensions can be controlled and measured ~~accurately.~~ accurately is satisfactory. Equipment that provides a constant shear rate has been shown to be equally useful. The capillary die of the rheometer shall have a smooth, straight bore that is held within ±0.0076 mm (±0.0003 in.) in diameter

and shall be held to within ±0.025 mm (±0.001 in.) in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity.

(2) Due to the extreme sensitivity of flow data to the capillary dimensions, it is important that the capillary dimensions are measured with precision and reported. The ~~length to diameter~~ length-to-diameter ratio shall normally be between 20 and 40. Larger ratios and ratios less than that suggested require applying large corrections to the data (**4, 5**). In addition, the ratio of the reservoir diameter to capillary diameter should be between 3 and 15. See Test Method D3835 for further details of capillary rheometers.

7.9.1.2 *Calibration*—Perform the test with a certified standard viscosity fluid approximating that expected for bone cement (50 N·s/m^2 to 500 N·s/m^2). Determine the viscosity of the standard fluid and the percent error from its specified value. Report this error along with the viscosity of the tested cements.

7.9.1.3 *Corrections*—Bone cement is a non-Newtonian ~~fluid,~~fluid; the data may be reported as corrected data. For example, true shear rates, corrected for non-Newtonian flow behavior, and true shear stress, corrected for end effects or kinetic energy losses, may be calculated. In such cases, the exact details of the mode of correction ~~must~~shall be reported. Some correction factors which may apply are:

(1) Piston friction,
(2) Plunger back flow,
(3) Cement compressibility,
(4) Barrel back pressure,
(5) Capillary entrance effects (Bagley correction) (**6**), and
(6) Rabinowitsch shear rate correction (**7**).

7.9.1.4 *Procedure:*

(1) Select conditions of temperature and shear stress or shear rate in accordance with expected usage so that the flow rate will fall within the desired limits.

(2) Inspect the rheometer and clean it if necessary. Ensure that previous cleaning procedures and usage have not changed the dimensions or caused scratches or defects in the capillary or apparatus. Make the necessary measurements on the apparatus for future calculations. Prepare the apparatus for running the test.

(3) Mix one or more complete unit(s) of powder and liquid in the recommended manner. Start a stopwatch at the onset of mixing and read all subsequent times from this watch. After complete mixing, transfer the cement to the thermally equilibrated reservoir and eject any entrapped air or excess bone cement.

(4) Start the apparatus at a time not greater than 2½ min from the start of mixing and continue operating until the estimated dough time or the viscosity exceeds 500 N·s/m^2.

(5) Disassemble the apparatus quickly before the cement sets and clean the apparatus of all remaining cement.

7.9.1.5 *Calculations:*

(1) Perform the calculation for viscosity of the cement at time intervals of 15 s from the start to finish of test run. Use the following equations:

$$\text{Shear Stress, Pa} = \frac{Pr}{2L} = \frac{Fr}{2\pi R^2 L} \tag{3}$$

$$\text{Shear Rate, s}^{-1} = \frac{4Q}{\pi r^3} = \frac{4V}{\pi r^3 t} \tag{4}$$

$$\text{Viscosity, Pa·s} = \frac{P\pi r^4}{8LQ} = \frac{Fr^4 t}{8R^2 LV} \tag{5}$$

where:

~~*P* = pressure by ram, Pa,~~
~~*F* = force on ram, *N*,~~
~~*r* = radius of capillary, m,~~
~~*R* = radius of barrel, m,~~
~~*L* = length of capillary, m,~~
~~*Q* = flow rate, m^3/s,~~
~~*V* = volume extruded, m^3, and~~
~~*t* = extrusion time, s.~~

P = pressure by ram in Pa,
F = force on ram in *N*,

r = radius of capillary in m,
R = radius of barrel in m,
L = length of capillary in m,
Q = flow rate in m^3/s,
V = volume extruded in m^3, and
t = extrusion time in s.

(2) These equations yield true shear rate and true viscosity for Newtonian fluids only; for non-Newtonian fluids, such as bone cement, the apparent shear rate and viscosity are obtained.

7.9.1.6 *Report*—The report of the flow properties of the cement shall include:

(1) Description of the rheometer used.

(2) Temperature at which the data were obtained.

(3) The capillary diameter and length to diameter ratio of the capillary.

(4) The shear rate at which the test was performed.

(5) Viscosity versus observation time for three runs.

(6) Statement as to whether any correction factors (~~7.8.1.3~~7.9.1.3) were applied.

7.9.2 *Extrusion, Rotational Shear Viscosity:*

7.9.2.1 *Apparatus—Rotational Shear Rheometer*—Any parallel plate rotational shear rheometer that can use 4 cm diameter plates, a 1000 µm gap, and maintain a temperature of 23 ± ~~0.5ºC~~0.5 °C is satisfactory.

7.9.2.2 *Calibration*—Calibrate the rheometer according to the manufacturer's specifications.

7.9.2.3 *Procedure:*

(1) Mount a parallel plate geometry on the top fixture. A disposable plate system may be used. A stainless steel 4 cm diameter plate is recommended. A removable bottom plate can be added to facilitate sample removal.

(2) Control the temperature of the rheometer so that at least one of the plates is at 23 ± 0.5 ~~ºC.~~°C.

(3) Move the plates apart to allow sample loading.

(4) Mix the cement according to the manufacturer's specifications. Start a laboratory timer from the start of mixing.

(5) After the requisite mixing procedure is complete, place a sufficient quantity of bone cement between the plates so as to completely fill the gap between the plates and no bubbles in excess of 1 mm are visible. Reduce the gap height between the plates to 1000 µm. Scrape away excess cement.

(6) Start a steady shear experiment at 0.5 s^{+1}—^{1}, monitoring the shear viscosity as a function of time at a sampling rate of 0.5 Hz or better. Note the elapsed time from the start of mixing to when the first data point is obtained.

(7) Collect data until the viscosity reaches 1000 Pa·s. Stop the test and remove the cement before it completely hardens.

(8) It is recommended that three runs are conducted per cement formulation.

7.9.2.4 *Report*—The report of the flow properties of the cement shall include:

(1) Description of the rheometer used.

(2) The shear viscosity as a function of time from the start of mixing. The reported instrument time points will need to be shifted by the elapsed time measured in ~~7.8.2.3~~7.9.2.3(*6*).

7.9.3 *Intrusion:*

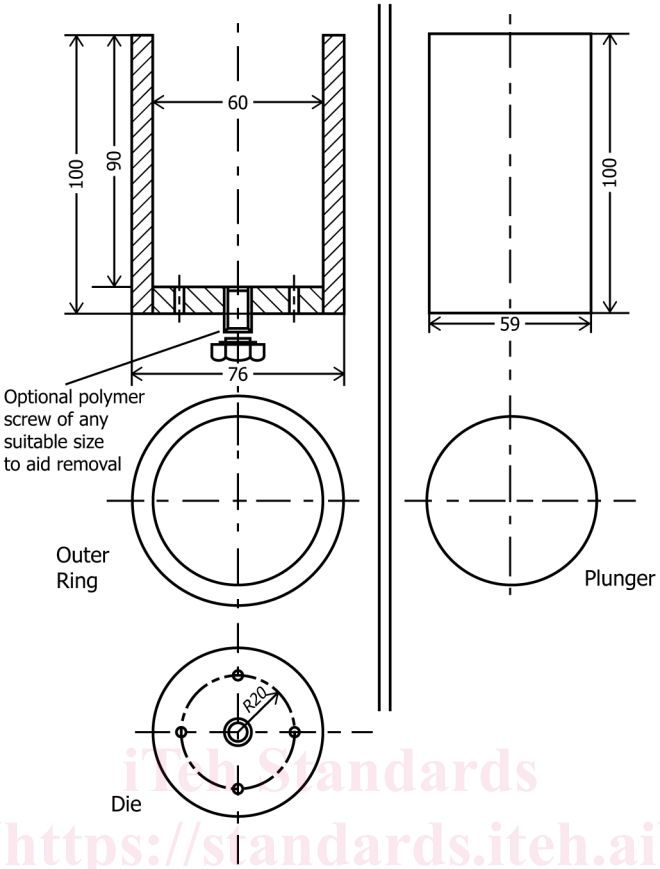
7.9.3.1 The mold necessary for this test shall be made of polytetrafluoroethylene (PTFE), poly(ethyleneterephthalate), polyoxymethylene, ~~high density~~ high-density polyethylene, or ultra-high molecular weight polyethylene (UHMWPE) and is shown in Fig. 3.

7.9.3.2 Follow the procedure outlined in ISO 5833, section D.4.3 for intrusion testing.

7.9.3.3 Following the set, remove the specimen and measure the average height of the intrusion into all four of the 1.0-mm diameter holes of the die to the nearest 0.5 mm.

7.9.3.4 Run this test once. If the requirement is not met, it must be met ~~so~~ in a repeat test.

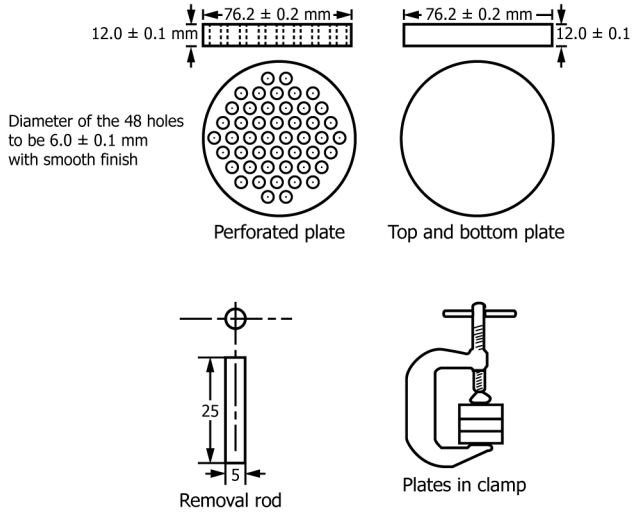
7.10 *Compressive Strength*—The test specimens shall be cylinders 12 mm high and 6 mm in diameter. The ends of the specimens shall be flat and smooth and shall be parallel to each other and at right angles to the long axis of the cylinder. An apparatus found



NOTE 1—Dimensions in millimetres; ~~4~~four holes in bottom to be 1.00 ± 0.05. Tolerance on all other dimensions ±0.2. Material for all components: ~~Polytetrafluoroethylene,~~polytetrafluoroethylene, poly(ethyleneterephthalate), polyoxymethylene, ~~high-density~~ high-density polyethylene, or ultra-high molecular weight polyethylene (UHMWPE).

FIG. 3 Intrusion Mold

convenient for forming these test cylinders is shown in Fig. 4. An apparatus containing additional or fewer holes may be used as



NOTE 1—Material for ~~Perforated Plate: Stainless Steel, Aluminum, Polytetrafluoroethylene, high-density~~ perforated plate: stainless steel, aluminum, polytetrafluoroethylene, high-density polyethylene, or ultra-high molecular weight polyethylene (UHMWPE).

FIG. 4 Compression Specimens Mold

long as adequate spacing between the holes is maintained. A mold release agent or silicone spray may be sparingly applied to facilitate specimen removal.

7.10.1 Place the specimen mold on a flat glass or smooth metal plate and slightly overfill using one unit of mixed cement of standard proportions at the commencement of dough time. Press a second flat glass or smooth metal plate on top of the mold. Hold the mold and plates firmly together with a small C-clamp. Then, 1 h later, surface the ends of the cylinder plane at right angles to the axis. The ends of the specimens may be ground flat to the axis by use of a small amount of 240-mesh silicon carbide powder and water. Draw the molds containing the specimens back and forth across the plate coated with the abrasive and water. After surfacing, remove the specimens from the mold. The specimens should be visually examined for surface defects. A surface defect is defined as a surface discontinuity greater than 500 microns in major diameter. Acceptable specimens for testing shall appear to be uniform and meet the dimensional requirements of ~~7.9~~7.10. A minimum of five specimens shall be selected from the remaining acceptable specimens and tested. Report the results of all specimens tested.

7.10.2 The time lapse between the start of mixing and the measurement of the compressive strength testing shall be 24 ± 2 h. Storage of the specimens before testing shall be at 23 ± ~~2 °C~~ 2 °C and 50 ± 10 % relative humidity. Run specimens on any universal testing machine equipped to record load versus deformation. Employ a deformation ~~cross head~~ cross-head speed of 20 to 25.4 mm/min. Test the specimens without use of any type of pad between the specimen and the platens of the machine. The failure load shall be the load at the 2.0 % offset (2.0 % proof stress), upper yield point, or at fracture, whichever occurs first (Fig. 5).

7.10.2.1 The load at 2.0 % offset is the load at the intersection of the load deformation curve and a straight line parallel to the Hookean portion of the curve ~~(See~~(see Fig. X1.1 in Test Method D695) but offset along the deformation axis by 2.0 % of the ~~test's~~ test specimen's gauge length (specimen's height).

7.10.2.2 Calculate the compressive strength as the failure load divided by the calculated cross-sectional area.

7.10.2.3 Report the compressive strength of the material as the average of the compression strengths of the specimens tested in ~~7.9.2~~7.10.2 to the nearest 1 MPa (145 psi). A minimum of five specimens is required.

7.11 *Molar Mass by Gel Permeation Chromatography (GPC):*

7.11.1 The molar mass (see Note 1) distribution and molar mass averages (number-average molar mass, weight-average molar mass, and z-average molar mass) of the powder will be determined by gel permeation chromatography with reference to Test Method D5296. Tetrahydrofuran (THF) will be used to dissolve the powder according to Test Method D5296. If the powder contains radiopacifier, the mass of powder used to make the solution should be increased to account for the radiopacifier. The solution should be filtered as suggested in Test Method D5296 to remove any radiopacifier.

NOTE 1—The term molecular weight (abbreviated MW) is obsolete and should be replaced by the SI (Système Internationale) equivalent of either relative molecular mass (M_r), which reflects the dimensionless ratio of the mass of a single molecule to an atomic mass unit (see ISO 80000-9), or molar mass (M), which refers to the mass of a mole of a substance and is typically expressed as grams/mole. For polymers and other macromolecules, use of the

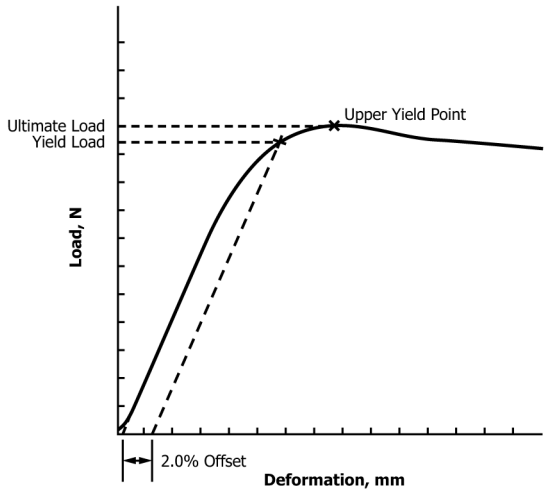


FIG. 5 Failure Load Criteria

symbols M_w,M_n, and M_z continue, referring to mass-average molar mass, number-average molar mass, and z-average molar mass, respectively. For more information regarding proper utilization of SI units, see NIST Special Publication SP811.

7.11.2 Poly(methyl methacrylate) molar mass standards should be used to calibrate the GPC system. Poly(styrene) standards may be used for the purpose of comparison to historical results, but it should be understood that the results will be relative and will not represent an absolute determination of the polymer's molar mass distribution.

7.11.3 Three aliquots of powder shall be tested for each bone cement.

7.11.4 This method may also be used on cured bone cement to determine the molar mass distribution of the cured material.

7.11.5 The number-average, weight-average, and z-average molar mass, along with the molar mass distribution relative to either polystyrene or polymethyl methacrylate standards shall be reported for each aliquot of powder. Report the average and standard deviation of the number-average, weight-average, and z-average molar mass for the three samples.

7.12 *Leachable Monomer:*

7.12.1 The residual monomer during curing and post-curing may be determined using the protocols described in Annex A5 and Annex A6.

7.13 *Stabilizer Concentration:*

7.13.1 If a quinone-based stabilizer is used in the liquid portion, the amount of hydroquinone or monomethyl ether hydroquinone may be determined using the protocols described in Annex A3 or Annex A4. Alternative protocols may be used if they can be shown to have the required sensitivity.

7.14 *Benzoyl Peroxide Concentration:*

7.14.1 If benzoyl peroxide is used as an initiator, the amount of benzoyl peroxide in the powder portion may be determined using the protocol described in Annex A1.

7.15 *N,N-dimethyl-p-toluidine Concentration:*

7.15.1 If N,N-dimethyl-p-toluidine is used as a reaction accelerator in the liquid portion, its concentration may be determined by high-performance liquid chromatography (HPLC) or similar assays (**8**).

7.16 *Stability Testing*—The shelf life stability of bone cement powder-liquid systems shall be evaluated using the test methods listed in Table 3.

7.17 *Precision and Bias*—Since 1976, the original Specification F451 methodologies have reportedly been routinely utilized by

TABLE 3 Requirements for Stability Testing

Test Type	Test Description	Test Material
~~Viscosity~~	~~7.3~~	~~Liquid Component~~
Viscosity	7.4	Liquid Component
Residual Peroxide	Annex A1, Annex A2, or equivalent	Powder Component
~~Dough Time~~	~~7.5~~	~~Curing Cement~~
Dough Time	7.6	Curing Cement
~~Set Time~~	~~7.7~~	~~Curing Cement~~
Set Time	7.8	Curing Cement
~~Compressive Strength~~	~~7.9~~	~~Cured Cement~~
Compressive Strength	7.10	Cured Cement
Tensile Strength	D638	Cured Cement
~~Leachable Monomer~~	~~Annex A2, or equivalent~~	~~Cured and Curing Cement~~
Leachable Monomer	Annex A5, Annex A6, or equivalent	Cured and Curing Cement

the various manufacturers. With the exception of the viscosity method of ~~7.8.1~~7.9.1, which is based on another accepted ASTM document (Test Method D3835), each test methodology in Section 7 contains its own statement of reporting acceptable levels of performance, reproducibility, and precision. Therefore, no interlaboratory studies have been performed by ~~the~~ Committee F04.

8. Packaging

8.1 Materials shall be supplied in properly sealed containers made of materials that shall not contaminate or permit contamination of the contents. The containers shall be packaged so as to prevent damage or leakage during shipping and storage. Materials must be packaged to permit sterile transfer of contents to the surgical sterile field.

8.2 The contents shall be easily accessible, easy to open, and convenient to mix in the operating room. Entire package contents (both powder and liquid) must be mixed ~~to achieve recommended proportions.~~per the manufacturer's instructions for use.

9. Labeling

9.1 Labeling on these cements must be in conformance with the Federal Food, Drug, and Cosmetic Act, Code of Federal Regulations, and other pertinent laws and regulations.

9.2 The following minimal information ~~must~~shall appear on the container ~~label.~~label:

9.2.1 It shall be clearly stated or color coded, or both, if the mixture is intended for usage in the pre-dough, dough, or dual usage state.

9.2.2 The weight or volume, or both, of the liquid and powder components ~~must~~shall be stated.

9.2.3 Constituents of the powder and liquid shall be clearly stated in terms of weight or volume percent. This information shall include the generic names of polymers, copolymers, chemical initiators, stabilizers, cross-linking agents, and any other ingredients, such as radiopacify agents, gels, fillers, or antibiotics.

9.2.4 A statement that the contents are sterile and that the sterility shall be guaranteed only if the containers are undamaged. Sterilization of the final polymerized cement is not applicable for this *in-situ* polymerization system. Sterilization has been conducted on both the starting liquid and powder components.

9.2.5 The following warning shall appear on the label: (*a*) Flammable liquid; (*b*) Store below ~~25°C,~~25 °C, and (*c*) Protect from light.

9.2.6 A statement to the effect that federal law restricts this device for sale by or on the order of a physician should be displayed.

9.2.7 The manufacturer and distributor shall be identified.

9.2.8 Each individual component of the package unit ~~must~~shall be clearly identified as to batch or lot number.

9.3 The following information shall appear on the product insert labeling accompanying each package.

9.3.1 Adequate and accurate instruction shall be given for handling the components and preparing the cement. Instructions shall include a directive to mix all of the powder with all the liquid of a single unit. Procedures required to mix the materials, along with recommended mixing utensils, shall be given.

9.3.2 Proper technique for administration and recommended procedures for using the cement, including any special precautions, shall be indicated.

9.3.3 Toxic, hazardous, or irritating characteristics associated with the handling and use of the components and cement shall be indicated.

9.3.4 A ~~statement shall be included~~ declaration that states that high temperatures of either the ambient surroundings or material will cause shorter doughing and setting times, while low temperatures of either the ambient surroundings or material will increase doughing and setting ~~times.~~ times shall be included. In addition, if the instructions for use (IFU) have an allowable temperature