Designation: 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 pre-dough or dough stage in accordance with the manufacturer's recommendations.

1.2 Units of 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, health, and 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

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

[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.

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 7.6).
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 7.7).

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

3.1.4 *intrusion*—the distance of flow of the mixture into a standard mold under load (see 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 7.8).
3.1.5.1 *Discussion*—"Setting time" and "set time" are interchangeable in this standard.

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

4. Physical Requirements

4.1 *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, the viscosity of the liquid shall not increase by more than 10 % of its original value (see 7.4).
4.1.3 *Sterility*—The liquid, as poured from its container, shall pass the tests described in "Sterility Tests—Liquid and Ointments" (7.5) (**3**).

4.2 *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" (7.5) (**2**).

4.3 *Powder-Liquid Mixture*—The material shall conform to the properties given in Table 1.

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

4.4 *Cured 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, 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 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.

6.2 It will only be necessary to maintain sterility in tests described in 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 ± 1 °C for at least 2 h prior to testing and conduct all tests at 23 ± 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

[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.

B. The radiopacifier content shall not vary from the nominal content by more than 10 %.

7.4 *Liquid Component 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:

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

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.

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 for at least 2 h prior to testing and tests shall be conducted at 23 ± 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 stopwatch at the onset of combining the liquid and the powder and read all subsequent times from this 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; 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 7.6 into the mold described in Fig. 1. This mold shall be made of polytetrafluoroethylene (PTFE), poly(ethyleneterephthalate), polyoxymethylene, 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 (see Fig. 2). Report the maximum temperature recorded to the nearest 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.

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

7.8 *Setting Time*—From the continuous time-versus-temperature recording of 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.

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 minute of each 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 (7.9.1 and/or 7.9.2) and Table 1. If the mixture is to be utilized in the dough stage, use the intrusion test (7.9.3) and Table 1. If the mixture is to be used as a dual usage cement, then both the extrusion (7.9.1 and/or 7.9.2) and intrusion (7.9.3) tests shall be performed.

7.9.1 *Extrusion, Capillary Viscosity:*

7.9.1.1 *Apparatus:*

(1) Capillary Rheometer—Any capillary rheometer 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 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.