Designation: F2118 – 10

StandardTest Method for Constant Amplitude of Force Controlled Fatigue Testing of Acrylic Bone Cement Materials[1]

This standard is issued under the fixed designation F2118; 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 test method describes test procedures for evaluating the constant amplitude, uniaxial, tension-compression uniform fatigue performance of acrylic bone cement materials.

1.2 This test method is relevant to orthopedic bone cements based on acrylic resins, as specified in Specification F451 and ISO 16402. The procedures in this test method may or may not apply to other surgical cement materials.

1.3 It is not the intention of this test method to define levels of performance of these materials. It is not the intention of this test method to directly simulate the clinical use of these materials, but rather to allow for comparison between acrylic bone cements to evaluate fatigue behavior under specified conditions.

1.4 A rationale is given in Appendix X2.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E466 Practice for Conducting Force Controlled Constant Amplitude Axial Fatigue Tests of Metallic Materials

E467 Practice for Verification of Constant Amplitude Dynamic Forces in an Axial Fatigue Testing System

E1823 Terminology Relating to Fatigue and Fracture Testing

F451 Specification for Acrylic Bone Cement

2.2 *ISO Standard:*

ISO 16402 Flexural Fatigue Testing of Acrylic Resin Cements Used in Orthopedics[3]

3. Terminology

3.1 Unless otherwise given, the definitions for fatigue terminology given in Terminology E1823 will be used.

3.2 *Definitions:*

3.2.1 *mean fatigue life at N cycles*—the average number of cycles to failure at the specified load level. For the purposes of this test method, the fatigue life will be determined at 5 million load cycles. A rationale for this is provided in X2.4.

3.2.2 *median fatigue life at a given stress level*—the number of cycles to failure at which 50 % of the tested samples failed at the specified stress level.

3.2.3 *runout*—a predetermined number of cycles at which the testing on a particular specimen will be stopped, and no further testing on that specimen will be performed. For the purposes of this test method, the runout will be 5 million load cycles.

3.2.4 *specimen failure*—the condition at which the specimen completely breaks or is damaged to such an extent that the load frame is no longer able to apply the intended stress within the required limits.

3.2.5 *stress level*—the value of stress at which a series of duplicate tests are performed. For the purposes of this test method, the stress level is reported as the maximum stress applied to the specimen.

4. Summary of Test Method

4.1 Uniform cylindrical reduced gage section test specimens are manufactured from acrylic bone cement and mounted in a uniaxial fatigue frame. The specimen is subjected to fully reversed tensile and compressive loading in a sinusoidal cyclic manner at a specified frequency in phosphate buffered saline (PBS). The fatigue loading is continued until the specimen fails or a predetermined number of cycles (run-out limit) is reached.

[1] This test method is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

Current edition approved Dec. 1, 2010. Published January 2011. Originally approved in 2001. Last previous edition approved in 2009 as F2118 – 03 (2009).

[2] 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.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

5. Significance and Use

5.1 This test method describes a uniaxial, constant amplitude, fully reversed fatigue test to characterize the fatigue performance of a uniform cylindrical waisted specimen manufactured from acrylic bone cement.

5.2 This test method considers two approaches to evaluating the fatigue performance of bone cement:

5.2.1 Testing is conducted at three stress levels to characterize the general fatigue behavior of a cement over a range of stresses. The stress level and resultant cycles to failure of the specimens can plotted on an *S-N* diagram.

5.2.2 Another approach is to determine the fatigue life of a particular cement. The fatigue life for orthopaedic bone cement is to be determined up to 5 million (5×10^6) cycles.

5.3 This test method does not define or suggest required levels of performance of bone cement. This fatigue test method is not intended to represent the clinical use of orthopaedic bone cement, but rather to characterize the material using standard and well-established methods. The user is cautioned to consider the appropriateness of this test method in view of the material being tested and its potential application.

5.4 It is widely reported that multiple clinical factors affect the fatigue performance of orthopaedic bone cement; however, the actual mechanisms involves multiple factors. Clinical factors which may affect the performance of bone cement include: temperature and humidity, mixing method, time of application, surgical technique, bone preparation, implant design, anatomical site, and patient factors, among others. This test method does not specifically address all of these clinical factors. The test method can be used to compare different acrylic bone cement formulations and products and different mixing methods and environments (that is, mixing temperature, vacuum, centrifugation, and so forth).

6. Apparatus

6.1 *Uniaxial Load Frame*—A testing machine capable of applying cyclic sinusoidal tensile and compressive loads.

6.1.1 The crossheads of the load frame shall be aligned such that the alignment meets the requirements of section 8.2 of Practice E466. The alignment should be checked at both the maximum tensile and minimum compressive load to be applied during the course of a test program.

6.2 *Cycle Counter*—A device capable of counting the number of loading cycles applied to a specimen during the course of a fatigue test.

6.3 *Load Cell*—A load cell capable of measuring dynamic tensile and compressive loads in accordance with Practice E467.

6.4 *Limit*—A device capable of detecting when a test parameter (for example, load magnitude, actuator displacement, DC error, and so forth) reaches a limiting value, at which time the test is stopped and the current cycle count recorded.

6.5 *Environmental Chamber*—A chamber designed to immerse the fatigue specimen completely in a solution. The chamber should have provisions for maintaining a constant temperature to an accuracy of ±2°C.

7. Test Specimen

7.1 Test specimens shall be fabricated from cement that is representative of the final product with regard to materials, manufacturing processes, sterilization, and packaging. Certain sterilization methods have been shown to have an effect on fatigue performance (for example, gamma sterilization of the powder). Any deviations of the test cement from the clinically used product must be reported.

7.2 Cylindrical reduced gage section test specimens with a straight 5-mm diameter by 10-mm-long gage section shall be used. The diameter of the specimen ends shall be substantially greater than the gage diameter to ensure that fracture occurs in the gage section. A smooth surface of the test specimen in the radius or taper between the specimen ends and gage section is essential to reduce variation in reported fatigue life. Suggested specimen dimensions are provided in Fig. 1.

8. Specimen Preparation

8.1 *Cement Mixing:*

8.1.1 Store the liquid and powder portions of the cement according to the manufacturer's instructions before mixing.

8.1.2 Allow the mixing equipment to equilibrate to room temperature before mixing. Record the room temperature at the onset of mixing.

8.1.3 Mix the powder and liquid components according to the manufacturer's instructions and begin recording the time using a stopwatch when the liquid and powder are initially mixed. Report any deviations from the manufacturer's storage and mixing recommendations.

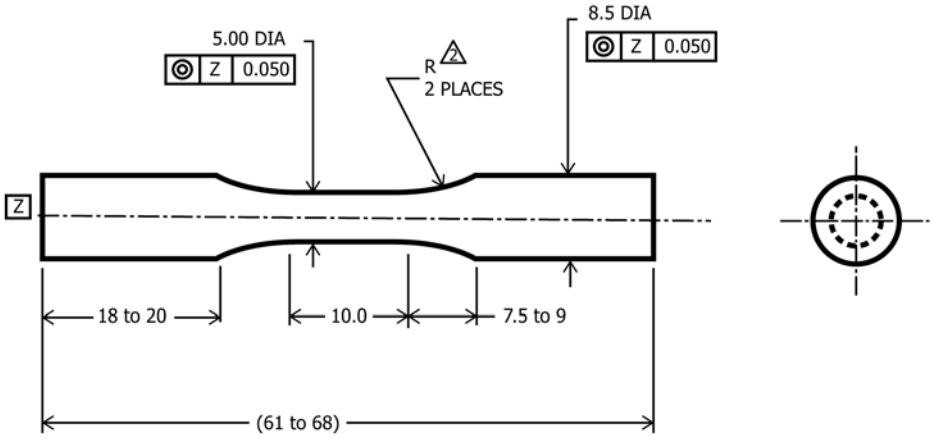
8.1.4 Report the mixing method and any equipment used. The method used for mixing the cement may affect its fatigue behavior. See X2.13 for further information.

8.2 *Specimen Fabrication*—The cylindrical reduced gage section test specimens are fabricated using the following method:

8.2.1 *Direct Molding:*

8.2.1.1 Inject the mixed cement into a specimen mold during the dough phase as determined by Specification F451 (manufactured from silicone material, see Appendix X3 (suggested specimen molding method)) with an internal cavity which has the same dimensions as the final cement test specimen. Record the method of cement insertion into the mold (that is, syringe injected). A 150 mL syringe with an inner diameter of 38 mm and a nozzle tip diameter of 10 mm should be considered for use. The mold should be placed on a flat surface. The cement injection should be performed from top to bottom in direction allowing the cement to flow down axially to the bottom . The bottom of the mold is placed on a flat surface as the bone cement is being injected into the mold unixaially from the top down. If air is entrapped and leads to resistance to injection, the mold should be rocked back and forth to release trapped air from the bottom of the mold. This will allow for air to escape from the bottom of the mold. (See X3.6 for standard operating procedure for making bone cement specimens.)

8.2.1.2 Place the mold in a container of phosphate buffered saline (PBS). The PBS solution should be maintained at 37 ±



1. All dimensions in mm
2. Radius to blend smoothly with gage section
3. Tolerances: X. = ± 1.0, X.X = ± 0.5, X.XX = ± 0.1

FIG. 1 Specimen Dimensions

2°C. After at least 1 h in the PBS bath, the specimens may be removed from the mold. Appendix X3 describes a suggested procedure for molding cement specimens.

8.3 *Specimen Examination:*

8.3.1 Visually examine specimens for surface defects. Surface defects in the gage or transition sections (radii) shall be rejected from testing and discarded. A surface defect is defined as a surface discontinuity greater than 250 mm in major diameter. All specimens should be photographed to document surface finish prior to testing. In addition, the specimens' straightness should be compared to the metal positive blank to ensure that the specimen is will not product bending moments during the unaxial fatigue testing. Straightness can be assessed by rolling the specimens and determining if there is a visible wobble as compared to the straight metallic blank used to make the mold. Specimens with surface defects or deemed not to be straight shall be rejected from testing and discarded. The total number of specimens rejected divided by the total number of specimens manufactured (rejection rate) shall be reported. A rationale for these rejection criteria is provided in X2.11.

8.4 *Specimen Finishing*—If necessary, lightly polish the gage length of the specimens with 600-grit abrasive paper in the longitudinal direction until the surface is free of machining and/or mold marks.

8.5 *Specimen Measurement*—Measure the diameter of the specimens at a minimum of three places along the gage length of each specimen. The average of these measurements shall be used as the specimen's gage diameter for calculation of the required load.

8.6 *Specimen Conditioning:*

8.6.1 Place the test specimens in PBS which is maintained at a temperature of 37 ± 2°C.

8.6.2 Maintain the specimens in the PBS solution for a minimum of 7 days. The cement specimens shall be maintained in the PBS solution for 7 to 60 days. The specimens shall be continually immersed in the test solution so that they do not dry out. Distilled water shall be added to the soaking chamber during the soaking period to make up for evaporation loss. Each specimen should be soaked up to the time immediately before its being mounted on the load frame. See X2.5 for further information.

9. Fatigue Test Procedures

9.1 Mount one specimen at a time in a test frame test such that a uniaxial load is applied. Collets, Jacob's chucks, or pressurized grips should be used to firmly grip the specimen at each end. Ensure the longitudinal centerline of the test specimen is aligned with test machine loading axis such that bending moments are minimized. Testing of multiple specimens on the same fixture in parallel or series shall not be performed as this complicated and changes the stress state in the individual specimens when cracks initiate and propagate through the specimen occurs, effectively changing the modulus of each individual specimen being tested.

9.2 Mount an environmental chamber on the load frame and fill with fresh PBS solution immediately after the specimen is mounted to keep the specimen from drying out. The chamber should be filled to a level such that the entire specimen is immersed. Distilled water shall be added to the test chamber during the course of a test to make up for any evaporation loss. The temperature controller should be programmed and activated to heat the test solution to 37°C, and then maintain that temperature within ±2°C. Fatigue testing should not begin until at least ½ h after the solution temperature has reached 37°C to ensure equilibration.

9.3 Program the test frame controller to apply a fully reversed sinusoidal cyclic waveform at a constant frequency. When testing at frequencies above 5 Hz, the user should verify that, for the formulation being tested, the chosen frequency has a negligible effect on the test results. See X2.6 for further information.

9.4 Program the test frame controller to apply the desired maximum stress level and a stress ratio of $R = -1$, indicating fully reversed loading. A rationale for using fully reversed loading is provided in X2.10. The load shall be calculated by multiplying the desired stress by the specimen's cross-section area, based on each specimen's gage diameter as determined in 8.5.

9.4.1 Report the stress level to the nearest 0.5 MPa.

9.4.2 When developing an *S-N* curve (see 10.1), it is recommended that testing be conducted at the following maximum stress levels: 15, 12.5, and 10 MPa. Other stress levels may also be appropriate for orthopedic applications such the hip and knee. However, stress levels of 5, 7, and 9 MPa should be considered for spinal applications in vertebroplasty and kyphoplasty. See X2.7 for a rationale regarding the selection of the recommended stress levels.

9.5 *Number of Specimens*—When developing an *S-N* curve, a minimum of 15 specimens shall be tested at each stress level. The desired statistical power of the comparison and the variability to be expected from the cement formulation(s) being investigated should be considered when determining the appropriate sample size; while this may require more than 15 specimens per bone cement formulation at each stress level, 15 is the recommended minimum number to test. See X2.12 for further information.

9.6 Set the cycle counter and limit settings of the test frame controller to record the cumulative number of cycles applied to the test specimen and the appropriate test limits values to indicate specimen failure or deviations from the intended load system performance.

9.7 After the solution has reached the temperature requirements in 9.2, activate the test frame controller to begin the test.

9.8 Testing shall continue until specimen failure or the run-out limit is reached.

10. Calculation and Interpretation of Results

10.1 The maximum stress and the cycles to failure for each specimen should be recorded and plotted on an Stress Level versus number of cycles diagram, which is a plot of the number of cycles to failure on the *x*-axis at each of the stress levels examined on the *y*-axis. The techniques used to measure mean fatigue lives, as well as to compare statistical differences between sample groups, and calculate fatigue life are described in 10.2 – 10.6.

10.2 *Mean Fatigue Life*—For each stress level, the mean fatigue life and standard deviation about the mean shall be determined assuming a log-normal distribution; that is, assuming that the log-transformed number of cycles to failure is approximately normally distributed (**1**).[4] The mean log fatigue life is determined as follows. A sample size of N specimens is tested, and the total number of cycles to failure for each (denoted N_i) is recorded. Next take the natural log of the number of cycles: $X_i = \ln(N_i)$. The mean log number of cycles to failure is obtained via the sample mean:

$$\bar{X}_{\log} = \sum_{i=1}^{N} \frac{X_i}{N} \tag{1}$$

where:

N = total number of specimens in the sample group,
N_i = number of cycles to failure of ith specimen,
X_i = log-transformed number of cycles to failure of ith specimen: $X_i = \ln(N_i)$, and
$\bar{X}_{\log}$ = mean log fatigue life.

10.2.1 Using a similar approach, the sample standard deviation of the log fatigue life ($S_{X_{\log}}$) is determined.

$$S_{\bar{X}_{\log}} = \sqrt{\sum_{i=1}^{N} \frac{\left(X_i - \bar{X}_{\log}\right)^2}{N-1}} \tag{2}$$

10.2.2 These are expressed in more familiar terms, as cycles to failure, by calculating the following:

$$\text{Mean fatigue life} = e^{\bar{X}_{\log}} \tag{3}$$

10.2.3 A 95 % lower and upper bound for the mean number of cycles to failure can be obtained using the following formulas (using the delta method, see X4.1):

$$e^{\bar{X}_{\log}} \pm -1.96 * \left(e^{\bar{X}_{\log}} S_{\bar{X}_{\log}}\right) \tag{4}$$

10.3 *Parametric Statistical Comparisons*—Statistical differences between specimen groups may be determined by commonly used methods such as a two-sample independent *t*-test to compare two groups, or analysis of variance (ANOVA) to compare more than two groups. This comparison is performed at each stress level using published methods (**2**) which are available through many commercial statistical software packages. The use of these tests requires several assumptions; the two most relevant are normality and equal variances. That is, these tests assume that the number of cycles to failure in each bone cement at each stress level is approximately normally distributed, and that the variance of these normal distributions is the same for all of the bone cements. These are relatively strong assumptions, which may not be upheld. It is therefore recommended that these assumptions be assessed. Tests to assess normality include the Lillie for test and the Shapiro-Wilk test (**3**). However, these tests are based on large samples approximations, and having a sample size on the order of 15–30 observations per group may not be sufficient to guarantee reliable performance.

10.3.1 Often, the decision as to whether to analyze data on an untransformed or log-scale is based on a test for normality; the most common of these is the Shapiro-Wilk test (**4**). Based on a small simulation study using the results of the "round-robin" experiment, we found that the test rejects samples from a true normal distribution approximately 7.5 % of the time (out of an expected 5 %). If the data is assumed to arise from a gamma distribution (a highly skewed distribution which appears to be a reasonable fit to this data), the untransformed data is not rejected approximately 27 % of the time. This implies that reliance on the Shapiro-Wilk test may lead to incorrect application of statistical tests assuming normality; this is likely if a relatively small number of specimens are tested (N=15).

10.3.2 It is often recommended that a parametric analysis be performed using the log-transformed data—this assumes that

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

the number of cycles to failure follows a log-normal distribution. If this is the case, then analyzing on the log scale would be expected to improve the normality of the data; the number of cycles is highly skewed with all values being non-negative, and some having extremely high values. Taking the log of the number of cycles is believed to make the resulting data more approximately normally distributed. In addition, calculating the mean based on the log scale reduces the effect of extremely large or small values (for example, outliers) on the sample mean. The disadvantage of analyzing on the log scale is that the units are in terms of log cycles rather than cycles. However, the transformed value can be back-transformed to the original scale (and an approximate 95 % confidence interval can be estimated via the delta method as shown in 10.2).

10.4 *Non-parametric Statistical Comparisons*—In situations in which the parametric statistical tests are not appropriate (for example, the number of cycles is not approximately normally distributed, or the variances of the different bone cements are unequal), non-parametric statistical methods are suggested for use in determining statistical differences between sample groups. Non-parametric tests are based upon the median, rather than the mean, and are therefore more robust because they are less influenced by the highly skewed nature of the data. In addition, as these tests are based on ranks, rather than upon the actual observation values, the results are the same regardless of whether or not the data are log-transformed. The Mann-Whitney U test (equivalent to the Wilcoxon rank sum test) is recommended for comparing two groups, and the Kruskal-Wallis test is recommended for comparing three or more groups. This comparison is performed at each stress level using published methods **(2)** which are available through many commercial statistical software packages.

10.5 *Recommendations for Analysis*—In light of these discussions, as well as an examination of the "round-robin" data and the observation that the number of cycles to failure must be non-negative and may be highly skewed (Appendix X5), an assumption of normality is somewhat tenuous. For the number of samples suggested here (n=15 per bone cement) it is recommended that non-parametric tests, which are more robust to non-normal data, be used for statistical inference and to compare different types of bone cement.

10.6 A brief description of the fracture characteristics; results of post-test photography or scanning electron microscopy or both; identification of fatigue mechanism; and the relative degree of transgranular and intergranular cracking would be highly beneficial. In addition, all fractured specimens will be examined visually for pores and failure occurring outside the gauge area.

11. Report

11.1 The test report shall include the following:

11.1.1 Manufacturer and brand of bone cement.

11.1.2 Product catalog number, lot number, and expiration date. If the cement is not in its final packing or sterilized, then the manufacturing date should be provided and noted that the bone cement components were not sterilized.

11.1.3 Composition of bone cement polymer powder and liquid.

11.1.4 Deviations from clinically used product (if applicable).

11.1.5 Description of cement storage, temperature of room during bone cement mixing and relative humidity, mixing method (that is, report duration of mixing, wait time (if applicable), determination of dough time, application time, and hardening time), and any deviations from the manufacturer's recommendations.

11.1.5.1 If vacuum mixing is used, the information and parameters described in 8.1.3 shall be reported.

11.1.6 Description of specimen fabrication method.

11.1.7 Description of specimen examination procedures, rejection rate, rejection criteria and rationale for the rejection criteria.

11.1.8 Duration of preconditioning, provided either for each specimen, or expressed as an average and range of duration.

11.1.9 Cyclic frequency.

11.1.10 A summary of the maximum cyclic stress and cycles to failure for each specimen tested.

11.1.11 A summary for each sample group describing at each stress level the following parameters:

11.1.11.1 Mean fatigue life, along with the standard deviation and 95 % confidence interval as presented in 10.2.

11.1.12 A description of the failure mode and failure location for each specimen that failed. Scanning electron microscopy (SEM) is suggested to identify the failure mode.

11.1.13 The mean fatigue life at each load level. A description of the analytical or statistical techniques used for determining the fatigue life should be included.

11.1.14 Any deviations from the specified test method.

12. Keywords

12.1 acrylic bone cement; fatigue; fatigue life

APPENDIXES

(Nonmandatory Information)

X1. FORMULAS

X1.1 Formulas are presented following the notation of Hollander and Wolfe (**5**).

X1.2 *Formula for Wilcoxon Rank Sum Test*:

X1.2.1 The Wilcoxon rank sum test (which is equivalent to the Mann-Whitney test) is a non-parametric analog of the two-sample *t*-test.

X1.2.2 This test assumes that there are two independent groups, and the question of interest is whether the medians of the two groups are equal. To implement the test, refer to the m observations from the first group as X and the n observations from the second group as Y. Order all of the observations from smallest to largest, and assign ranks to each observation. Denote the rank of all of the values from the second group as S_1, ..., S_n.

X1.2.3 Calculate the sum of the ranks of the observations in the second group:

$$W = \sum_{j=1}^{n} S_j$$

X1.2.4 To test for equivalence of medians in a 2-sided test, calculate the test statistic:

$$W^* = \frac{W - [n(m+n+1)/2]}{\sqrt{mn(m+n+1)/12}}$$

and refer W^* to a standard normal distribution; that is, reject the null hypothesis of equal medians if $|W^*| \geq z_{1-\alpha/2}$, where $z_{1-\alpha/2}$ is the 1 – α/2th percentile from the standard normal distribution.

X1.2.5 If there are ties in the ranks, assign the average rank to each of the tied values, and adjust the test statistic W^* as follows:

$$W^* = \frac{W - [n(m+n+1)/2]}{\sqrt{\frac{mn(N+1)}{12} - \left\{ \frac{mn}{12N(N-1)} \sum_{j=1}^{g} (t_j - 1)t_j(t_j+1) \right\}}}$$

where g represents the number of tied groups (thus, if there are no ties, g=N and the formula simplifies to the first form).

X1.3 *Formula for Kruskal-Wallis Test*:

X1.3.1 The Kruskal-Wallis test is an extension of the Wilcoxon rank sum test to more than two independent groups; it is a non-parametric analog of the 1-way ANOVA.

X1.3.2 To implement this test, first order all N of the observations from all of the k groups from smallest to largest. Denote as r_{ij} the rank of observation X_{ij}, and the number of samples in the jth group as n_j. Calculate:

$$R_j = \sum_{i=1}^{n_j} r_{ij} \quad \text{and} \quad R_j = \frac{R_j}{n_j}$$

X1.3.3 The test statistic for the Kruskal-Wallis test is then calculated as:

$$H = \left(\frac{12}{N(N+1)} \sum_{j=1}^{k} \frac{R_j^2}{n_j} \right) - 3(N+1)$$

X1.3.4 Compare H to a χ_{k-1}^2 (chi-square with k-1 degrees of freedom) distribution, and reject the null hypothesis of equality of medians across groups if $H \geq \chi_{k-1,\alpha}^2$, where $\chi_{k-1,1-\alpha}^2$ is the 1 – αth percentile from a chi-square distribution with k-1 degrees of freedom.

X1.3.5 If there are ties present in the data, calculate the modified test statistic:

$$H' = \frac{H}{1 - \left(\sum_{j=1}^{g} (t_j - 1)/(N^3 - N) \right)}$$

where g represents the number of tied groups (thus, if there are no ties, g=N and the formula simplifies to the first form).

X2. RATIONALE

X2.1 This test method is intended to provide the user with standard and well-established procedures for evaluating the fatigue properties of bone cement materials. Specimen parameters, test procedures, data analysis techniques, and reporting requirements are provided.

X2.2 The test method does not specify the mixing conditions to use for the preparation of the test specimens. Considerable research is currently being performed on bone cement and the committee did not want to unnecessarily limit the conditions or parameters that are being investigated by excluding them from the standard.

X2.3 It is important to realize that this test method is intended to characterize the bone cement material—not the bone cement which is used *in vivo*. Some consideration has been given to the parameters which the cement encounters during *in vivo* use (37°C temperature and PBS solution); however, it is not practical to try and completely simulate the clinical use of bone cement. The results obtained from this test method characterize the bone cement material for a specified set of conditions, but they may not necessarily reflect the