



Designation: F 746 – 87 (Reapproved 1999)

Standard Test Method for Pitting or Crevice Corrosion of Metallic Surgical Implant Materials¹

This standard is issued under the fixed designation F 746; 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 covers the determination of resistance to either pitting or crevice corrosion of metals and alloys from which surgical implants will be produced. It is a modified version of an established test² and is used as a screening test to rank surgical implant alloys in order of their resistance to localized corrosion.

1.2 This test method applies only to passive metals and alloys. Nonpassive alloys (other than noble alloys) are susceptible to general corrosion and are not normally suitable for implant use.

1.3 The test method is intended for use as a laboratory screening test of metals and alloys which undergo pitting or crevice corrosion, or both.

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

1.5 *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:

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing³

G 5 Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements³

G 15 Terminology Relating to Corrosion and Corrosion Testing³

3. Summary of Test Method

3.1 A cylindrical specimen fitted with an inert tapered collar is immersed in a saline electrolyte at 37°C for 1 h to establish

a corrosion potential. Pitting (or crevice corrosion) is then stimulated by potentiostatically polarizing the specimen to a potential much more noble than the corrosion potential. Stimulation of pitting (or crevice corrosion) will be marked by a large and generally increasing polarizing current.

3.2 Immediately after the stimulation step, the potential is decreased as rapidly as possible to one of several preselected potentials at, or more noble than, the corrosion potential. If the alloy is susceptible to pitting (or crevice corrosion) at the preselected potential, the polarizing current will remain at relatively high values and will fluctuate or increase with time. A post-test examination of the metal specimen establishes whether localized corrosion has occurred by pitting of the exposed surface or by preferential attack at the crevice formed by the tapered collar, or both.

3.3 If the pit (or crevice) surface repassivates at the preselected potential and localized corrosion is halted, the polarizing current will drop to values typical for passive surfaces and the current will decrease continuously. The parameter of interest, the critical potential for pitting (or crevice corrosion), is defined as the highest (most noble) preselected potential at which pit (or crevice) surfaces repassivate after the stimulation step.

4. Significance and Use

4.1 This method is designed solely for determining comparative laboratory indices of performance. The results may be used for ranking alloys in order of increasing resistance to pitting and crevice corrosion under the specific conditions of this method. It should be noted that the method is intentionally designed to reach conditions that are sufficiently severe to cause breakdown of at least one alloy (Type 316 L stainless steel) currently considered acceptable for surgical implant use, and that those alloys which suffer pitting or crevice corrosion during the more severe portions of the test do not necessarily suffer localized corrosion when placed within the human body as a surgical implant.

5. Apparatus

5.1 The following required equipment is described in Practice G 5:

5.1.1 *Standard Polarization Cell*, of 1000 cm³.

¹ This test method is under the jurisdiction of ASTM Committee F-4 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

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² Syrett, B. C., *Corrosion*, Vol 33, 1977, p. 221.

³ *Annual Book of ASTM Standards*, Vol 03.02.

5.1.2 *Electrode Holders*, for auxiliary and working electrodes.

5.1.3 *Potentiostat*, calibrated in accordance with Practice G 5.

5.1.4 *Potential-Measuring Instrument*.

5.1.5 *Current-Measuring Instrument*.

5.1.6 *Anodic Polarization Circuit*.

5.1.7 *Platinum Auxiliary Electrodes*.

5.1.8 *Saturated Calomel Electrode (SCE)*.

5.1.9 *Salt Bridge Probe*.

5.2 A cylindrical working electrode is fabricated from the test material by machining, grinding, and suggested final polishing with 600-grit metallographic paper. It is suggested that the part of the cylindrical specimen that is exposed to the test solution have a length of 20.00 ± 1.00 mm (0.787 ± 0.039 in.) and a diameter of 6.35 ± 0.03 mm (0.250 ± 0.001 in.) (see Fig. 1).

5.3 A crevice is created by fitting the cylindrical specimen with a tapered collar, machined from commercial purity polytetrafluoroethylene (PTFE). The collar should have an outer diameter of 12.70 ± 0.05 mm (0.500 ± 0.002 in.) and a thickness of 3.18 ± 0.20 mm (0.125 ± 0.008 in.). The inside diameter of the tapered collar should range from 0.38 mm (0.015 in.) smaller than the diameter of the specimen to 0.38 mm (0.015 in.) larger. To be consistent with the dimensions suggested in 5.2, the inside diameter should taper from 5.97 ± 0.05 mm (0.235 ± 0.002 in.) to 6.73 ± 0.05 mm (0.265 ± 0.002 in.). See Fig. 1 for drawing of the tapered collar. The relatively fine tolerances are needed to ensure a reproducible fit and crevice.

5.4 In Practice G 5, the method of specimen attachment is to drill and tap the specimen to receive a threaded stainless steel connection rod. A 4-40 thread is used, typically. However, because many surgical implant alloys are not easily drilled, external threads may also be machined, ground, or cast, as illustrated in Fig. 1. A small stainless steel adapter is fitted on to these threads and the adapter then accepts the connection rod.

5.5 Determine the total exposed surface area of the specimen before placement of the PTFE collar, A_T ; determine the area on the internal surface of the collar (the creviced area), A_C ; and determine the exposed surface area of the specimen after placement of the collar, A_S (where: $A_S = A_T - A_C$). Dimensions should be measured to the nearest 0.1 mm.

5.5.1 *Example*—Using the dimensions suggested previously for the specimen diameter ($d = 6.35$ mm), the specimen length ($l = 20.00$ mm), and the collar thickness ($t = 3.18$ mm),

$$A_T = \pi dl + \frac{\pi d^2}{4} = 431 \text{ mm}^2 \quad (1)$$

$$A_C = \pi dt = 63 \text{ mm}^2 \quad (2)$$

$$A_S = A_T - A_C = 386 \text{ mm}^2 \quad (3)$$

6. Reagents

6.1 *Electrolyte*—Dissolve 9 g of reagent grade sodium chloride (NaCl) in distilled water to make 1000 cm³ of solution.

6.1.1 After transferring the appropriate amount of electrolyte to the test cell (7.5), the pH is to be measured both before and after the test (see rationale in X1.6).

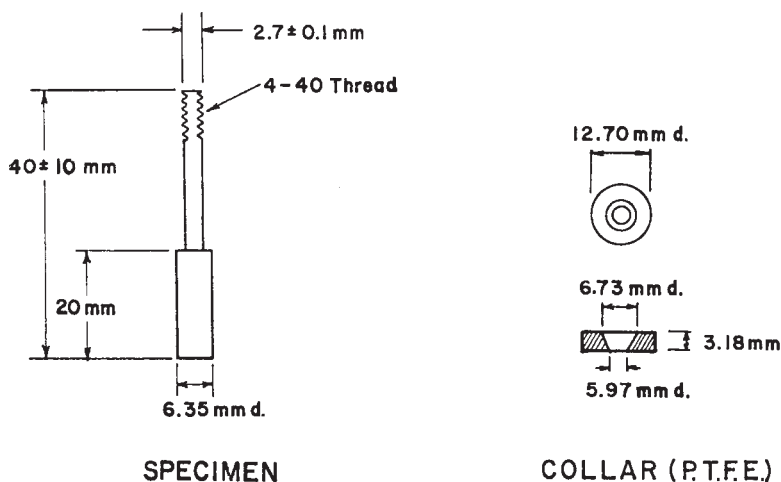
7. Preparation of Specimens and Conditioning

7.1 Prepare the test specimen surface within 1 h of the start of the experiment by the method described in Practice G 5.

7.2 Using a suitable mechanical jig, force-fit the PTFE collar onto the cylindrical specimen so that the base of the collar is up 10 ± 2 mm (0.393 ± 0.079 in.) from the bottom of the specimen (see Fig. 2). Care should be taken to avoid scratching the metal surface.

NOTE 1—Once the collar is removed from the specimen, it should not be reused.

7.3 Mount the specimen on the holder and on the electrode rod as described in Practice G 5.



NOTE 1—Unless shown, dimensional tolerances are given in text.

FIG. 1 Dimensions of Specimen and Collar

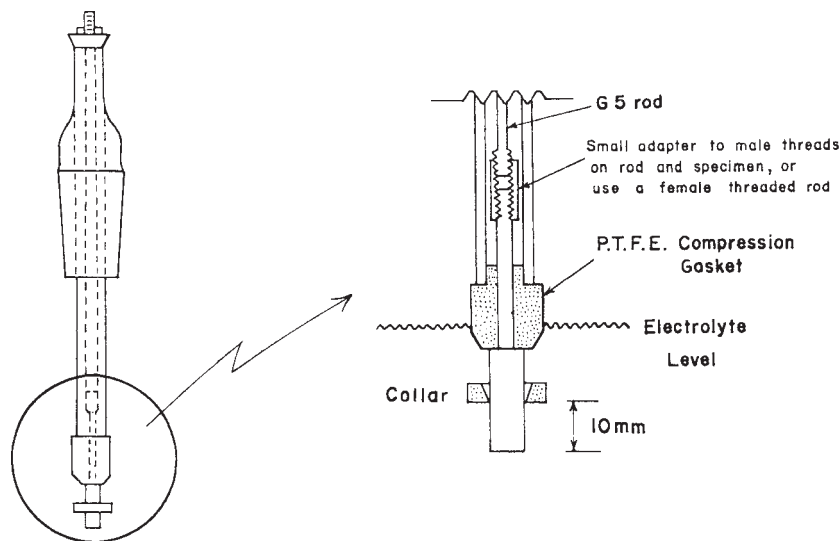


FIG. 2 Assembly into G5 Electrode Holder

7.4 Ultrasonically degrease the electrode assembly in either acetone, toluene, or boiling benzene (with caution, under hood), rinse in distilled water, and dry.

7.5 Transfer 500 mL of electrolyte solution to a clean polarization cell. Bring the temperature of the solution to $37 \pm 1^\circ\text{C}$ by immersing the test cell in a controlled temperature water bath or by other suitable means.

7.6 Place the platinum auxiliary electrodes, salt bridge probe and other components in the test cell and temporarily close the center opening with a stopper. Fill the salt-bridge with the electrolyte.

NOTE 2—The levels of the solution in the reference and the polarization cells should be the same to avoid siphoning. If this is not possible, a solution-wet (not greased) stopcock can be used in the salt-bridge to eliminate siphoning.

7.7 Transfer the specimen electrode assembly to the test cell and adjust the submerged salt bridge probe tip so it is about 2 mm (0.08 in.) from the center of the bottom portion of the specimen (below the collar).

8. Procedure

8.1 Continuously record the corrosion potential of the working electrode (specimen) with respect to the saturated calomel electrode for 1 h, starting immediately after immersing the specimen. The potential observed upon immersion in the electrolyte shall be called the initial corrosion potential. The potential at the end of the 1 h shall be known as the final corrosion potential, E_1 .

8.2 After the 1-h period, the potential should be potentiostatically shifted to +0.8 V (SCE) to stimulate pitting (or crevice corrosion).

NOTE 3—In the stimulation step, the change in potential either from E_1 or from one of the preselected potentials to +0.8 V (SCE) should be essentially instantaneous. Such instantaneous changes are facilitated by use of a two-channel potentiostat in which the new control voltage can be selected on the channel not in use. However, if a single channel potentiostat is used, it should be switched temporarily to the standby mode (no impressed current) while the set-potential control is being adjusted to a setting of +0.8 V (SCE); after so adjusting, the potentiostat should be

switched from the standby mode to the operate mode to allow stimulation of localized corrosion. After stimulation, the single-channel potentiostat must remain in the operate mode during the shift to the preselected potential, and the latter shift should be performed manually as rapidly as possible. Manual shifting of the potential may also be necessary after the stimulation step when using a two-channel potentiostat if the switch from +0.8 V (SCE) to the preselected potential would result in a potential transient to values more active than the preselected potential. Such transients could lead to repassivation and to the incorrect assumption that the repassivation occurred at the preselected potential.

8.3 Record the current using a strip chart recorder with a minimum chart speed of 60 mm/min and a maximum current scale of 0 to 3 mA. The current will be recorded at +0.8 V (SCE) for a period that depends upon the reaction (see Fig. 3).

8.3.1 If localized corrosion is not stimulated in the initial 20 s, the polarizing currents will remain very small or decrease rapidly with time. Proceed to 8.4.

8.3.2 Stimulation of localized corrosion will be marked either by polarization currents that generally increase with time or by current densities that exceed $500 \mu\text{A}/\text{cm}^2$ (for the suggested specimen size this would be equivalent to a current of approximately 2 mA).

8.3.2.1 If the current generally increases with time, after 20 s proceed to 8.5.

8.3.2.2 If at any time a current density of $500 \mu\text{A}/\text{cm}^2$ is exceeded, proceed immediately to 8.5. In some instances, upon shifting to +0.8 V (SCE), the current density will almost instantaneously exceed $500 \mu\text{A}/\text{cm}^2$. In such cases, proceed directly to 8.5 without pause.

8.4 If localized corrosion is not stimulated within the initial 20 s, continue at +0.8 V (SCE) for an additional 15 min; the chart speed may be reduced to a minimum of 5 mm/min after the initial 20 s. If localized corrosion is eventually stimulated, proceed to 8.5. If localized corrosion cannot be stimulated even in 15 min, the test is terminated, and the material is considered to have a very high resistance to localized corrosion in the test environment. Report the critical potential as $> +0.8 \text{ V (SCE)}$.

8.5 If localized corrosion is stimulated at +0.8 V (SCE), the potential is then returned as rapidly as possible (see Note 3) to