Designation: F 2129 – 01

Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices[1]

This standard is issued under the fixed designation F 2129; 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 assesses the corrosion susceptibility of small, metallic, implant medical devices, or components thereof, using cyclic (forward and reverse) potentiodynamic polarization. Examples of device types, which may be evaluated by this test method include, but are not limited to, vascular stents, filters, support segments of endovascular grafts, cardiac occluders, aneurysm or ligation clips, staples, and so forth.

1.2 This test method is used to assess a device in its final form and finish, as it would be implanted. These small devices should be tested in their entirety. The upper limit on device size is dictated by the electrical current delivery capability of the test apparatus (see Section 6). It is assumed that test methods, such as Test Methods G 5 and G 61 have been used for material screening.

1.3 Because of the variety of configurations and sizes of implants, this test method provides a variety of specimen holder configurations.

1.4 This test method is intended for use on implantable devices made from metals with a relatively high resistance to corrosion.

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:*

D 1193 Specification for Reagent Water[2]

G 3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing[3]

G 5 Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements[3]

G 15 Terminology Relating to Corrosion and Corrosion Testing[3]

G 61 Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys[3]

G 102 Practice for Calculation of Corrosion Rates and Related Information from Electrochemical Measurements[3]

3. Terminology

3.1 *Definitions:*

3.1.1 *potentiostat, n*—an instrument for automatically maintaining an electrode in an electrolyte at a constant potential or controlled potentials with respect to a suitable reference electrode (see Terminology G 15).

3.1.2 *potentiodynamic cyclic polarization (forward and reverse polarization), n*—a technique in which the potential of the test specimen is controlled and the corrosion current measured by a potentiostat. The potential is scanned in the positive or noble (forward) direction as defined in Practice G 3. The potential scan is continued until a predetermined potential or current density is reached. Typically, the scan is run until the transpassive region is reached, and the specimen no longer demonstrates passivity, as defined in Practice G 3. The potential scan direction then is reversed until the specimen repassivates or the potential reaches a preset value.

3.1.3 *scan rate, n*—the rate at which the controlling voltage is changed.

3.2 *Symbols:*

3.2.1 E_b = *Breakdown or Critical Pitting Potential*—the least noble potential at which pitting or crevice corrosion or both will initiate and propagate as defined in Terminology G 15. An increase in the resistance to pitting corrosion is associated with an increase in E_b.

3.2.2 E_{corr} or *OCP*—the potential of a corroding surface in an electrolyte relative to a reference electrode measured under open-circuit conditions, as defined in Terminology G 15.

3.2.3 E_f = *Final Potential*—a preset potential at which the scan is stopped.

3.2.4 E_i = *Initial Potential*—the potential at which the potentiostat begins the controlled potentiodynamic scan.

3.2.5 E_p = *Protection Potential*—the potential at which the reverse scan intersects the forward scan at a value that is less noble than E_b. E_p cannot be determined if there is no breakdown. Whereas, pitting will occur on a pit-free surface

[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.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

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

above E_b, it will occur only in the range of potentials between E_p and E_b if the surface is already pitted. The severity of crevice corrosion susceptibility increases with increasing hysteresis of the polarization curve, the difference between E_b and E_p.

3.2.6 E_v = *Vertex Potential*—a preset potential, at which the scan direction is reversed.

3.2.7 i_{corr} = *Corrosion Current Density (mA/cm²)*—the corrosion current density is extrapolated from the anodic and cathodic Tafel regions to the OCP (in accordance with Practice G 102).

3.2.8 i_t = *Threshold Current Density (mA/cm²)*—a preset current density, at which the scan direction is reversed. Typically, the scan is reversed when a current density two decades higher than the current density at the breakdown potential (E_b) is reached.

4. Summary of Test Method

4.1 The device is placed in an appropriate deaerated simulated physiological solution and the corrosion potential (E_{corr}) is monitored for 1 h. The potentiodynamic scan is then started at an initial potential (E_i) 100 mV more negative than E_{corr}, and scanned in the positive or noble (forward) direction. The scan is reversed after the current density has reached a value approximately two decades greater than the current density measured at the breakdown potential. The reverse scan is stopped after the current has become less than that in the forward direction or the potential is 100 mV negative to E_{corr}. The data is plotted with the current density in mA/cm^2 on the *x* axis (logarithmic axis) versus the potential in mV on the *y* axis (linear axis). Appropriate reference medical devices in their final form and finish, as they would be implanted, are used as controls.

5. Significance and Use

5.1 Corrosion of implantable medical devices can have deleterious effects on the device performance or may result in the release of corrosion products with harmful biological consequences; therefore, it is important to determine the general corrosion behavior as well as the susceptibility of the devices to localized corrosion.

5.2 The forming and finishing steps used to create an implantable device may have significant effects on the corrosion resistance of the material out of which the device is fabricated. While testing the corrosion resistance of the materials is essential in the process of selecting materials to be used, it does not necessarily provide critical data regarding device performance.

5.3 To accommodate the wide variety of device shapes and sizes encountered, a variety of holding devices can be used.

5.4 Note that the method is intentionally designed to reach conditions that are sufficiently severe to cause breakdown and deterioration of the medical devices and that these conditions may not be necessarily encountered in vivo. The results of this corrosion test conducted in artificial physiological electrolytes can provide useful data for comparison of different device materials, designs, or manufacturing processes. However, note that this test method does not take into account the effects of cells, proteins, and so forth on the corrosion behavior in vivo.

6. Apparatus

6.1 *Potentiostat*, capable of maintaining an electrode potential within 1 mV of a preset value over a wide range of potentials, as described in Test Methods G 5 and G 61. The potential measuring circuit should have a high input impedance, that is, on the order of 10^{11} to 10^{14} Ω. The current measuring circuit should be capable of measuring current in the range of 1.0 to 10^5 µA.

6.2 *Working Electrode*, to be used as the test specimen. Its configuration and holder will depend on the type of specimen being tested, as described in Section 7. In all cases, the metallurgical and surface condition of a specimen simulating a device must be in the same condition as the device.

6.2.1 An appropriate reference medical device in its final form and finish, as it would be implanted, should be used as a reference or control. Appropriate reference device shall consist of a device, which is similar to the investigated device and has a history of good corrosion resistance in vivo, is used in a similar environment or location and is used to treat a similar disease. Again, as for the working electrode, the configuration and holder will depend on the type of reference specimen tested.

6.3 *Reference Electrode*—A saturated calomel electrode (SCE), as defined in Practice G 3, shall be used as a reference electrode.

6.4 *Salt Bridge*, such as a Luggin probe, shall be used between the working and reference electrode, such as the type shown in Test Method G 5.

6.5 *Auxiliary Electrodes*:

6.5.1 Two platinum auxiliary electrodes may be prepared from high-purity rod stock. The surfaces may be platinized, as per Test Method G 5.

6.5.2 Alternatively, high-purity graphite auxiliary electrodes may be used in accordance with Test Method G 5. Care should be taken to insure that they do not get contaminated during a test.

6.5.3 The auxiliary electrode surface area should be at least four times greater than the sample surface area. Use of wire-mesh platinum might be more cost-effective than platinum cylinders when testing larger specimens or whole devices.

6.6 *Suitable Polarization Cell*, with a volume of about 1000 cm^3, equivalent to or similar to that recommended in Test Method G 5.

6.7 *Water Bath*, or other heating appliance capable of maintaining the test solution temperature at 37 ± 1°C.

6.8 *Purge Gas Delivery System*, capable of delivering nitrogen gas at 150 cm^3/min.

7. Specimen Holders

7.1 There are a variety of holders that may be used in this practice. Each is designed for a specific type or class of device.

7.2 Short wire or coil specimens.

7.2.1 Specimens can be held suspended from a clamping device. For example, the threaded end of a Test Method G 5 holder can be used to hold two stainless steel nuts. The wire test specimen is clamped between these nuts and bent so as to enter the test solution.

7.2.2 The surface area of the test specimen shall be calculated based on the length of wire or coil immersed in the test solution.

7.2.3 This type of holder exposes the specimen to the air-liquid interface, which is subject to localized crevice corrosion. Test specimens should be examined carefully after testing to ensure that there is no localized corrosion at or just below the interface.

7.2.4 If specimens show evidence of localized corrosion at the air-liquid interface, then the portion of the specimen passing across this interface shall be sealed with an impervious coating.

7.3 Stents or cylindrical devices.

7.3.1 Fixture for holding stents **(1)**[4] or alternative methods can be used to create an electrical connection.

7.3.2 The fixture consists of a cylindrical mandrel of the shape shown in Fig. 1.

7.3.3 The larger diameter end of the mandrel has a recessed thread that will accommodate a standard electrode holder described in Test Method G 5. The smaller diameter end of the mandrel is machined to the maximum internal diameter of the stent to be mounted on it.

7.3.4 The stent is stress fit over the smaller end of the cylindrical mandrel.

7.3.5 A conductive epoxy then is used to bind the stress fit stent to the mandrel to obtain good electrical contact. This interface is sealed by applying a nonconductive masking agent over the interface. The whole fixture then is threaded on to an electrode holder in accordance with Test Method G 5.

7.3.6 The surface area of the specimen shall be calculated based on the surface area of the stent in contact with the test solution.

8. Reagents

8.1 Reagent grade chemicals shall be used for this test method. Such reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[5]

8.1.1 The water shall be distilled or deionized conforming to the purity requirements of Specification D 1193, Type IV reagent water.

8.1.2 The standard test solution should be prepared according to the specifications. As a reference, a list of common physiological solutions and their composition is provided in Appendix X2.

8.1.3 The pH of the electrolyte should be adjusted based on the nature of the solution by the addition of NaOH or HCl.

8.1.4 High-purity nitrogen gas for purge should be used when possible depending on the nature of the solution used. Gas purge may not be appropriate for simulated solutions that tend to foam excessively when agitated.

9. Test Specimen

9.1 Unless otherwise justified, all samples selected for testing should be taken from finished, clinical-quality product. Cosmetic rejects or other nonclinical samples may be used if the cause for rejection does not affect the corrosion behavior of the device. Sterilization may be omitted if it can be demonstrated that prior sterilization has no effect on the corrosion behavior of the device.

9.2 Surrogate devices used for design parameter studies should be prepared with the same processes and should have the same mechanical and electrochemical surface characteristics as the intended finished device.

10. Procedure

10.1 Prepare the specimen such that the portion exposed to the test solution is in the same metallurgical and surface condition as the implantable form of the medical device being studied.

10.1.1 Calculate the total surface area of the specimen exposed to the solution in order to determine the current

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

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

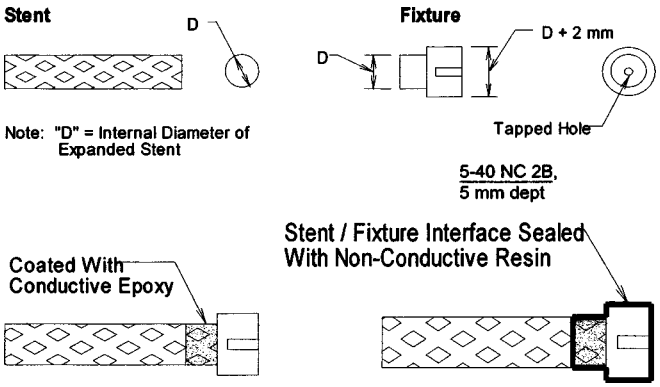

FIG. 1 Diagram for Assembly of Stent-Holding Fixture