Designation: F2129 – 17b

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 F2129; 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 that may be evaluated by this test method include, but are not limited to, vascular stents, ureteral stents (Specification F1828), 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 Reference Test Method G5 and Test Method G61 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 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.*

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:*[2]

D1193 Specification for Reagent Water

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

F1828 Specification for Ureteral Stents

G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing

G5 Reference Test Method for Making Potentiodynamic Anodic Polarization Measurements

G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)[3]

G61 Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys

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 G15).

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 G3. The potential scan is continued until a predetermined potential

[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] 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] The last approved version of this historical standard is referenced on www.astm.org.

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 G3. The potential scan direction is then 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 G15. An increase in the resistance to pitting corrosion is associated with an increase in E_b.

3.2.2 E_r = *Rest Potential*—the potential of the working electrode relative to the reference electrode measured under virtual open-circuit conditions (working electrode is not polarized).

3.2.3 E_{zc} = *Zero Current Potential*—the potential at which the current reaches a minimum during the forward scan.

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

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

3.2.6 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 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.

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

3.2.8 i_t = *Threshold Current Density (mA/cm^2)*—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 rest potential (E_r) is recorded for 1 h or, alternatively, until the rest potential stabilizes to a rate of change less than 3 mV/min. The potentiodynamic scan is then started at E_r and scanned in the positive or noble (forward) direction. The scan is reversed after either the vertex potential (E_v) is reached or 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 reaches E_r. 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).

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. During the selection process of a material for use as an implantable device, testing the corrosion resistance of the material is an essential step; however, 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 necessarily be 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,* calibrated in accordance with Reference Test Method G5.

6.2 *Working Electrode,* to be used as the test specimen, as described in Section 9. 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.

6.3 *Reference Electrode*—A saturated calomel electrode (SCE), as described in Reference Test Method G5, 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 Reference Test Method G5.

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 Reference Test Method G5.

6.5.2 Alternatively, high-purity graphite auxiliary electrodes may be used in accordance with Reference Test Method G5. Care should be taken to ensure 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 sufficient volume to allow the solution to cover the sample and the counter

electrode, and to prevent changes in pH during testing. Furthermore, the cell needs to be appropriately sealed to avoid oxygen access and include a secondary bubbler for the release of exhaust gas without the back diffusion of oxygen. The test cell must be able to hold a minimum of 500 ml.

6.7 *Water Bath,* or other heating appliance capable of maintaining the test solution temperature at 37 ± 1°C (see X1.5).

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 test method. 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 Reference Test Method G5 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. 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.

7.2.4 Alternatively, one may choose to coat the portion of the specimen out of the solution and the connection to the specimen holder with a suitable coating. The surfaces out of solution will tend to have test solution condensed on them and this may lead to undesirable results.

7.3 One method for holding stents or cylindrical devices is shown in Appendix X3.

8. Reagents

8.1 Reagent grade chemicals shall be used for this test method when they are commercially available (for example, some components in bile solutions are not available in reagent grade). Such reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society.[4]

8.1.1 The water shall be distilled or deionized conforming to the purity requirements of Specification D1193, Type IV reagent water.

8.1.2 Unless otherwise specified, phosphate buffered saline (PBS) should be used as the standard test solution. A representative PBS formulation is given in Appendix X2, along with the formulations of two simulated bile solutions for testing implantable medical devices intended for use in the biliary system, the formulations of two artificial urine solutions for testing implantable indwelling materials intended for use in the urinary tract, and the compositions of two other commonly used physiological solutions.

8.1.3 The pH of the electrolyte should be adjusted as needed based on the nature of the solution (e.g., for PBS, adjust the pH to a value of 7.4 ± 0.2 by the addition of NaH_2PO_4 (acid) or Na_2HPO_4 (base)). When the electrolyte is deaerated, its pH may change significantly if it is not sufficiently buffered. Several pH controlling methods are provided in Appendix X2.

8.1.4 Nitrogen gas with a minimum purity of 99.99 % should be used for purging the test solution of oxygen.

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.

9.1.1 Test specimens used for design parameter studies can be prepared as detailed in Reference Test Method G5 for working electrodes, with the requirement that the metallurgical and surface conditions of the specimens are the same as the intended implantable medical 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 density (current per surface area) generated by the specimen during the test.

10.2 Prepare enough test solution to immerse the device and auxiliary electrodes and so to avoid any appreciable change in the solution corrosivity during the test through exhaustion of the corrosive constituents or by accumulation of corrosion products that may affect further corrosion. At a minimum, transfer 500 mL of electrolyte to a clean polarization cell. Measure and record the pH of the solution before and after each test.

10.3 Place the auxiliary electrodes, salt bridge probe, thermometer, and gas purge diffuser in the test chamber and bring the temperature of the test solution to 37 ± 1°C.

10.4 Purge the solution for a minimum of 30 min with nitrogen gas at a flow rate of 150 cm^3/min.

10.5 Gently immerse the test specimen in the test solution and connect it to a potentiostat. Continue the nitrogen purge throughout the test.

[4] *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.

10.6 Record E_r for 1 h or, alternatively, until the rest potential stabilizes to a rate of change less than 3 mV/min.

10.7 At the end of the E_r recording period, start the potentiodynamic scan in the positive or noble (forward) direction, as defined in Practice G3. The scanning program should be set with the following parameters:

10.7.1 Starting or initial potential (E_i) at E_r.

10.7.2 A scan rate of either 0.167 mV/s or 1 mV/s should be used. Note that the scan rate may affect the breakdown potential of the device and the shape of the passive region of the polarization curve. Comparisons should not be made between test results using different scan rates, even if all other experimental parameters are held constant.

10.7.3 A current density threshold two decades greater than the current density recorded at breakdown can be used to reverse the voltage scan.

10.7.3.1 Alternatively, a minimum reversing or vertex potential (E_v) of 800 mV (SCE) may be used to control the potentiostat (see X1.6).

10.7.4 The final potential (E_f) is set to E_r. The reverse scan may be manually stopped at potentials above E_r in cases in which a protection potential (E_p) is observed as a drop in current density below that of the passive current density or when no hysteresis loop is formed once the scan is reversed (E_v), indicating repassivation (Fig. 1a), no protection potential (Fig. 1b), or oxygen evolution (Fig. 1c).

10.8 If control specimens are used, they shall be tested using the same method as the investigated devices.

11. Report

11.1 The report should contain a detailed description of the test specimen, including metallurgical and surface conditioning.

11.1.1 When specimens are not finished devices, for example, surrogates, the sample preparation should be described in detail.

11.2 A description of the test conditions should also be reported, including the following:

11.2.1 The volume of the test cell;

11.2.2 The approximate volume of solution used;

11.2.3 If performed, a description of the preconditioning/simulated use, together with the rationale for the choice of the preconditioning;

11.2.4 A description of the method used to determine the total estimated exposed surface area of the specimen.

11.3 The following results should be presented in the report (see Fig. 1):

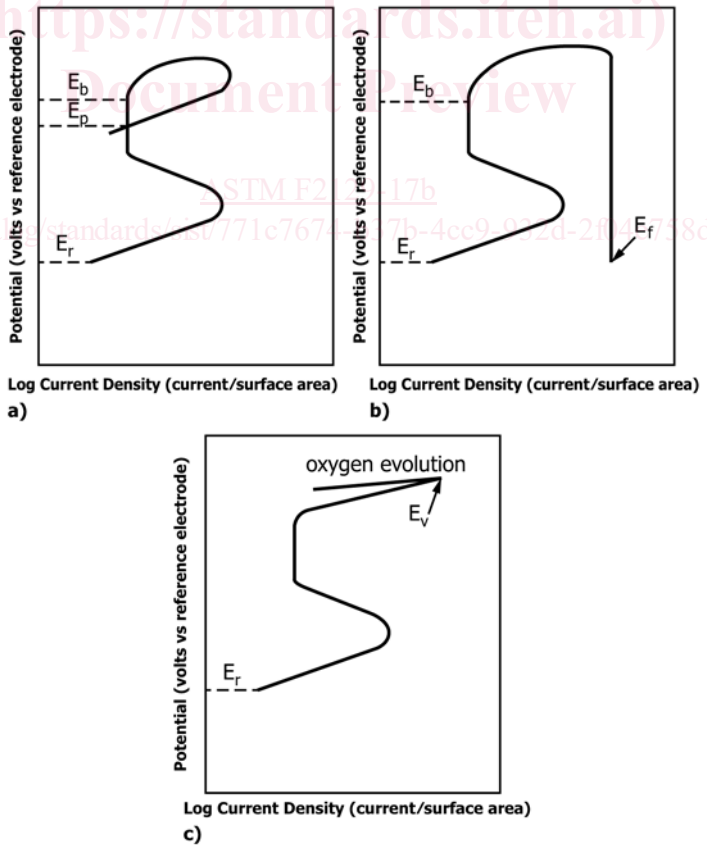

FIG. 1 Schematic of Cyclic Potentiodynamic Curves Illustrating Corrosion Parameters: (*a*) Material That Exhibits a Protection Potential (E_r, E_b, and E_p), (*b*) Material That Does Not Exhibit a Protection Potential (E_r, E_b, and E_f), and (*c*) Material That Exhibits Oxygen Evolution at Its Surface (E_r and E_v).