Designation: F2129 − 17

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

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.*

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G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)[3]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

---

[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 Jan. 1, 2017. Published March 2017. Originally approved in 2001. Last previous edition approved in 2015 as F2129 – 15. DOI: 10.1520/F2129-17.

[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.

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.

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:

---

[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.

10.7.1 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.

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

11.3.1 The final rest potential (E_r) and the rest potential recording time;

11.3.2 The breakdown potential (E_b);

11.3.3 The protection potential (E_p). In the absence of repassivation, the final potential (E_f) shall be reported instead of E_p. If no hysteresis loop is formed, the vertex potential (E_v) shall be reported instead of E_b and E_p.

11.3.4 All potentials should be reported relative to the SCE. If a reference electrode other than an SCE was used, it shall be reported along with the conversion factor used to convert data to the SCE scale.

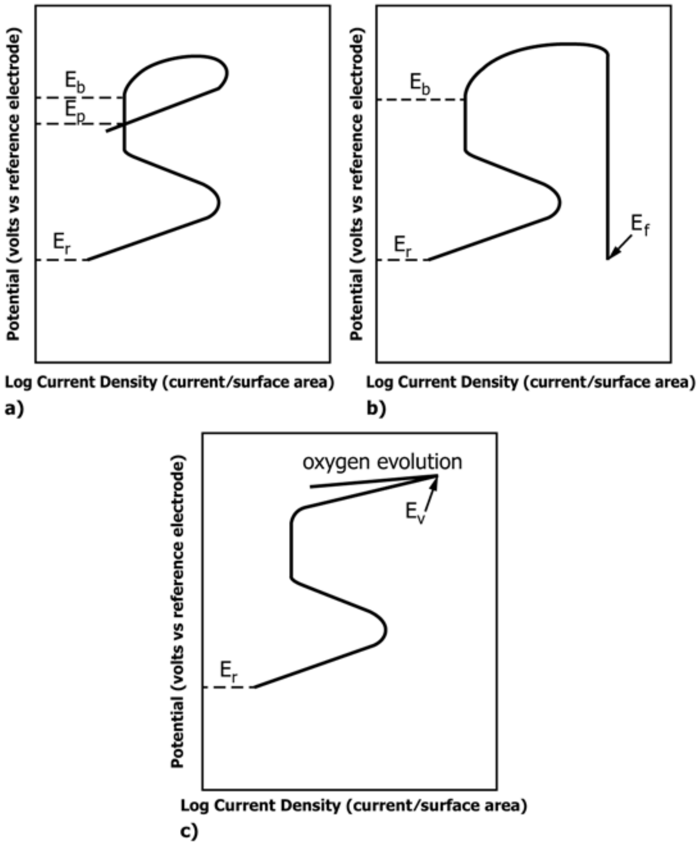

FIG. 1 Schematic of Cyclic Potentiodynamic Curves Illustrating Corrosion Parameters:
(*a*) Material That Exhibits a Protection Potential (E_r, E_b, and E_p),
(*b*) Material That Does Not Exhibit a Protection Potential (E_r, E_b, and E_f), and
(*c*) Material That Exhibits Oxygen Evolution at Its Surface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

11.4 The pH of the solution before and after each test should be reported.

11.5 A copy of the cyclic polarization curve should be provided in the report.

11.6 A generic description of the appearance of any corrosion observed on the specimen should be described. Photographic documentation may be appropriate.

12. Precision and Bias

12.1 An interlaboratory study was conducted in accordance with Practice E691 in twelve laboratories with four different materials. Each laboratory tested eight samples per material. The details of this study are provided in an ASTM Research Report. The results are summarized in Tables 1-4, which provide the repeatability and reproducibility statistics for each output parameter from the test. The terms repeatability limit and reproducibility limit are used as specified in Practices E177 and E691. As defined in Practice E691, repeatability is concerned with the variability between independent test results within one laboratory under tightly controlled conditions. Reproducibility is concerned with the variability between independent test results in different laboratories. No measurement bias is possible with this test method since there is no accepted reference material. No precision statement is possible for the repassivation potential, E_p, for 316LVM stainless steel or 455 stainless steel since there was insufficient data to generate the statistics. Neither of these materials exhibited repassivation in the majority of the experiments.

13. Keywords

13.1 corrosion; cyclic polarization; medical device testing; pitting potential; protection potential; rest potential

TABLE 1 Precision of Rest Potential E_r (mV)

Material	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	–7	33	64	93	178
455 SS	–30	38	67	105	187
Nitinol A	–519	35	49	98	137
Nitinol B	–482	21	49	60	138

TABLE 2 Precision of Breakdown Potential E_b (mV)

Material	Grand Mean	Repeatability StandardDeviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	679	161	190	451	531
455 SS	269	36	40	100	113
Nitinol A	160	82	108	230	302
Nitinol B	180	54	94	152	263

TABLE 3 Precision of Repassivation Potential E_p (mV)

Material	Grand Mean	Repeatability StandardDeviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	...	...	...	...	...
455 SS	...	...	...	...	...
Nitinol A	–171	57	108	160	302
Nitinol B	–126	38	58	107	162

TABLE 4 Precision of Breakdown Potential minus Rest Potential: $E_b — E_r$ (mV)

Material	Grand Mean	Repeatability Standard Deviation	Reproducibility Standard Deviation	95 % Repeatability Limit	95 % Reproducibility Limit
316 SS	674	154	176	432	494
455 SS	298	47	69	132	192
Nitinol A	679	83	110	232	309
Nitinol B	662	57	92	159	257

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 This test method is a modification to Reference Test Methods G5 and Test Method G61, to provide information regarding the corrosion susceptibility of small, finished medical devices in physiologic solutions. It is based on the original work of Pourbaix et al. (**1**),[5] Wilde and Williams (**2**) and Wilde (**3**), who showed that susceptibility to pitting was indicated by the breakdown potential (E_b) and susceptibility to crevice corrosion by the protection potential (E_p). These concepts were applied to orthopedic implant materials by Cahoon et al. (**4**). The critical data point is the potential above which pits nucleate and grow, that is, E_b. The higher the E_b, the more resistant the metal is to pitting corrosion. Once the direction of the potential scan is reversed, and the potential begins to drop, a measure of how quickly the pits will heal is attained. If E_p is high, that is, minimal hysteresis, then the metal is said to be very resistant to crevice corrosion. If there is some hysteresis, as in Fig. 1, then the metal may be susceptible to crevice corrosion; however, for materials or devices exhibiting a value of E_b above the physiological range of potentials, the presence of hysteresis during the reverse scan does not necessarily indicate susceptibility to crevice corrosion under normal physiological conditions. If the metal does not repassivate until a potential below E_r is reached, then it is very susceptible to crevice corrosion.

X1.2 While all currently used metallic biomaterials have well characterized corrosion properties, many device manufacturing processes may alter the cyclic polarization characteristics of finished implant devices. Furthermore, complex-shaped devices with corners, recesses, and other design irregularities may have a significant effect on localized current densities. It is of concern that finished device testing may create fluctuating current densities that cannot be normalized over the complex-shaped surface areas. In such cases, careful examination of test specimens after testing is necessary. For some devices, cyclic polarization may not provide useful information.

X1.3 Deaerating the solution with nitrogen gas before and during the test will lower the concentration of dissolved oxygen in the solution. This condition is necessary for the determination of the critical potentials E_b and E_p, if their actual values are close to or lower than the rest potential in the presence of oxygen. Since the current measured during anodic polarization (the applied anodic current) is the difference between the anodic and cathodic currents, cathodic reduction of dissolved oxygen may cause an error in the measurement of the anodic current density (that is, a greater cathodic current will cause a smaller difference between the anodic and cathodic currents). Consequently, this may result in artificially higher values of E_b or E_p. Lowering the oxygen concentration moves the potential at which the oxidation and reduction currents are equal to a lower value. This allows determination of true values of E_b or E_p at potentials at which the oxygen reduction current in the aerated solution would be significant.

---

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

X1.4 Since the absolute potential range that an implant should be able to withstand *in vivo* has not been established, absolute potential values such as the breakdown potential (E_b) cannot ensure that a device has sufficient resistance to corrosion; thus, if possible, it is recommended that tests be performed on reference specimens, under the same conditions, for comparison. If used, the reference should consist of a device that is similar to the investigated device, 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 or condition.

X1.5 Corrosion cell setup and the methods of heating should be carefully chosen to avoid creating electromagnetic noise, which can create an offset bias in the system. It has been observed in laboratory experiments that this type of electrical bias can generate potential shifts in excess of 100 mV. A method of testing for this is to monitor the rest potential of a test sample with the heating system on, and then turn it off and monitor the system for any changes. Higher noise environments are suspected of reducing breakdowns.

X1.6 It is acknowledged that for the temperature and pressure conditions of the test cell in this test method the Nernst equation predicts oxygen evolution at potentials slightly above 0.5 V (SCE). However, exceeding this potential does not equate to an immediate increase in current as a result of the generation of oxygen. In practice, even though oxygen evolution is thermodynamically favorable, the kinetics of the reaction is typically slow (the exchange current density is very low).

X1.6.1 The rationale for using 800 mV (SCE) or greater for the reversing potential is to allow for a "safety margin" over potentials that could reasonably be expected to exist in the human body while stopping short of the anodic breakdown of water. Stable specimens that do not break down may begin to see significant increases in current above 800 mV due to the breakdown of water and evolution of oxygen. Proceeding beyond this point leads to an experimental condition that is not physiologically relevant. However, as one may wish to evaluate new materials to higher values, this reversing potential is considered a minimum value.

X1.7 The open-circuit potential may vary over a long time period. The rest potential recording period is utilized to allow the specimen to stabilize to some degree in the test solution. A 1 h rest period has historically been used to achieve such relative stabilization. An alternative is to initiate the potentiodynamic scan when the rate of potential change becomes small, such as less than 3 mV/min.

X1.8 The protection potential, E_p, needs to be interpreted with caution due to the fact that it's value can be affected by factors in this test. Wilde (**2**) showed that the extent of pit propagation in a cyclic potentiodynamic polarization (CPP) test alters the protection potential value one achieves in the experiment. Dunn (**5**) summarized the data from multiple authors that have since demonstrated the same phenomena on a variety of stainless steels, including 316L, 304, 18-8, 317L and 430, as well as nickel based alloy 825 in chloride containing environments. The challenge for CPP testing in generating values for the protection potential is pitting corrosion is inherently stochastic (**6**) and leads to a range of breakdown potentials for any sample population. This, in combination with a fixed scan rate, creates significant differences in the net charge passed while pitting corrosion is occurring in the experiment and changes the repassivation behavior recorded in the experiment.

X2. COMPOSITION OF DIFFERENT PHYSIOLOGICAL ENVIRONMENTS

X2.1 Table X2.1 presents the composition of three different body fluids (**7**).

X2.2 Table X2.2 presents the comparison of blood plasma composition with saliva and bile (**8**).

TABLE X2.1 Composition of Selected Components of Three Body Fluids[A]

Component	Interstitial Fluid, mg/L	Synovial Fluid, mg/L	Serum, mg/L
Sodium	3280	3 127	3 265
Potassium	156	156	156
Calcium	100	60	100
Magnesium	24	-	24
Chloride	4042	3 811	3 581
Bicarbonate	1892	1 880	1 648
Phosphate	96	96	96
Sulfate	48	48	48
Organic acids	245	-	210
Protein	4144	15 000	66 300

[A] Based on data from *Documenta Geigy Scientific Tables*, L. Diem and C. Lentner, Eds., 7th ed., Ciba-Geigy.

TABLE X2.2 Composition of Blood Plasma, Saliva, and Bile

Component	Blood Plasma, mg/L	Saliva, mg/L	Bile, mg/L
pH	7.35–7.45	5.8–7.1	7.8
Sodium	3128–3335	240–920	3082–3588
Potassium	140–220	560–1640	156–252
Chloride	3430–3710	525–1085	2905–3850
Bicarbonate	1403–1708	122–793	2318

X2.3 For reference purposes, the composition of different artificial physiological solutions used as electrolytes for corrosion testing is reported in Table X2.3.

X2.4 Since corrosion behavior of metals is often strongly affected by the pH of the electrolyte, it is important to ensure when using one of the solutions simulating blood or interstitial fluid, that the test is performed at the physiological pH value of 7.4. When simulated test solutions are prepared in the laboratory according to the compositions in Table X2.3, and the pH is adjusted to 7.4, deaeration causes a pH increase of about one to one and a half pH units, as a result of the displacement of

TABLE X2.3 Composition of Simulated Physiological Solutions at a pH of 7.4

	Phosphate Buffered Saline[A] g/L	Ringer's,[B] g/L	Hanks,[C] g/L
NaCl	8.0	8.6	8.0
$CaCl_2$		0.33	0.14
KCl	0.2	0.3	0.4
$MgCl_2$ $6H_2O$			0.10
$MgSO_4$ $7H_2O$			0.10
$NaHCO_3$			0.35
Na_2HPO_4	1.15		
$Na_2HPO_4.12H_2O$			0.12
KH_2PO_4	0.2		0.06
Phenol red			0.02
Glucose			1.00

[A] Sigma-Aldrich Co., 2002

[B] The Pharmacopeia of the United States, Twenty-Sixth Revision, and the National Formulary, Twenty-First Editions.

[C] J.H. Hanks and R.E. Wallace, *Proc. Soc. Exp. Biol. Med.* 71, 196, (1949).

carbon dioxide from the solution. To maintain the pH at 7.4 during a test, one of the following methods may be used: (*a*) pH adjustment after deaeration, using appropriate measures to avoid oxygen access; (*b*) use of a suitable buffer; however, for simulated physiological solutions other than the phosphate buffered saline recommended in Table X2.3 (which is adequately buffered with Na_2HPO_4 so that the pH does not change significantly with bubbling nitrogen over six hours) evidence must be provided or available that the buffer does not affect the corrosion behavior or parameters; (*c*) saturation of the electrolyte with a gas mixture containing CO_2 in conjunction with the appropriate amount of $NaHCO_3$ in the electrolyte. A $NaHCO_3$ concentration of about 1.45 g/L in Hanks solution or 1.35 g/L in Ringer's solution, together with a mixture of 5 % CO_2 in nitrogen provide effective buffering at a pH of 7.4, as well as bicarbonate and CO_2 concentrations close to physiological values.

X2.5 *Simulated Bile Solutions*:

X2.5.1 When testing implantable medical devices for use in the biliary system, two different simulated bile solutions are the following: (*1*) Ox bile—1000 mL distilled water and 100 g unfractionated dried bovine bile; heat at 37°C and stir until the bile is in solution; pH of 6.5 desired; and (*2*) Human simulated bile[6]—1000 mL lactated Ringer's irrigation, 25.3 g cholic acid, 15.2 g chenodeoxycholic acid, 7.6 g deoxycholic acid, 9.5 g glycine, 2.5 g lithocholic acid, and 5.0 g sodium hydroxide pellets; heat at 37°C and stir for at least 15 min; add small amounts of sodium hydroxide pellets (in addition to the amount listed in the primary mix) as needed to completely dissolve the acids; add a few drops of nitric acid and let stir until the precipitate that forms completely dissolves; pH of 8.5 ± 0.2 desired (repeat adding nitric acid until the desired pH is obtained).

X2.5.2 Investigation has shown that the composition of bile is dynamic and modulated through a complex series of feedback mechanisms. An evaluation of the literature showed that no single pH could be utilized for testing. Rather, measured pH values range from 6.5 to 8.5 (**9**, **10**). The two simulated bile solutions listed in this test method encompass these values.

X2.6 *Artificial Urine Formulations*:

X2.6.1 *Formulation Number 1* (**11**)*:*

X2.6.1.1 Components per litre of solution:

NaCl	6.17 g
NaH_2PO_4	4.59 g
$Na_3Citrate$	0.944 g
$MgSO_4$	0.463 g
Na_2SO_4	2.408 g
KCl	4.75 g
$CaCl_2$	0.638 g
$Na_2Oxalate$	0.043 g
Distilled water	bring to 1 L volumetrically

Note X2.1—Add the above salts to a 1000 mL volumetric flask, then add the distilled water for a total volume of 1000 mL.

Note X2.2—Adjust pH to 5.5 to 6.5 range with a 1 *N* solution of NH_4OH or 1 *N* H_4Cl.

X2.6.2 *Formulation Number 2* (**12**)*:*

X2.6.2.1 Components per litre of solution:

Urea	25.0 g
NaCl	9.0 g
Disodium hydrogen orthophosphate, anhydrous	2.5 g
Potassium dihydrogen orthophosphate, anhydrous	2.5 g
NH_4CL	3.0 g
Creatinine	2.0 g
Sodium sulfite, hydrated	3.0 g
Distilled water	bring to 1 L volumetrically

---

[6] Based on Guidant Corporation internal test solution for simulated human bile, Guidant Corporation, Vascular Intervention Group, Santa Clara, CA, 2003.

X3. METHOD FOR MOUNTING STENTS OR CYLINDRICAL DEVICES

X3.1 A fixture for holding stents (**13**) or alternative methods can be used to create an electrical connection.

X3.2 The fixture consists of a cylindrical mandrel of the shape shown in Fig. X3.1.

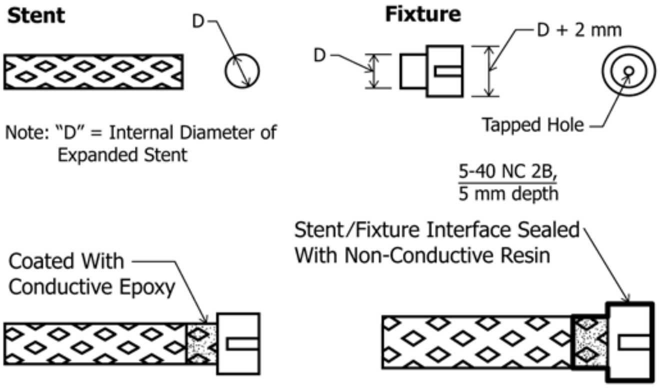

FIG. X3.1 Diagram for Assembly of Stent-Holding Fixture

X3.3 The larger diameter end of the mandrel has a recessed thread that will accommodate a standard electrode holder described in Reference Test Method G5. The smaller diameter end of the mandrel is machined to the maximum internal diameter of the stent to be mounted on it.

X3.4 The stent is stress fitted over the smaller end of the cylindrical mandrel.

X3.5 A conductive epoxy is then 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 onto an electrode holder in accordance with Reference Test Method G5.

X3.6 The surface area of the specimen shall be calculated based on the surface area of the stent in contact with the test solution.

REFERENCES

(1) Pourbaix, M., Klimzack-Mathieiu, L., Merterns, C., Meunier, J., Vanluegen-Haghe, C., de Munck, L., Laureys, J., Neelemans, L., Warzee, M., "Potentiokinetic and Corrosimetric Investigations of the Corrosion Behavior of Alloy Steels," *Corr Sci*3: 239-259, 1963.

(2) Wilde, B.E., Williams, E., "The Relevance of Accelerated Electrochemical Pitting Tests to the Long-Term Pitting and Crevice Corrosion Behavior of Stainless Steels in Marine Environments," *J Electrochem Soc*188: 1057 -1062, 1971.

(3) Wilde, B.E., "A Critical Appraisal of Some Popular Laboratory Electrochemical Tests for Predicting the Localized Corrosion Resistance of Stainless Alloys in Sea Water," *Corrosion*28: 283-291, 1972.

(4) Cahoon, J.R., Bandyopadhya, R., Tennese L., "The Concept of Protection Potential Applied to the Corrosion of Metallic Orthopedic Implants," *J Biomed Mater Res*9: 259–264, 1975.

(5) Dunn, D.S., Cragnolino, G.A., Sridhar N., "An Electrochemical Approach to Predicting Long-Term Localized Corrosion of Corrosion-Resistant High-Level Waste Container Materials," *Corrosion*, 56 (2000) 90.

(6) Shibata, T., "Statistical and Stochastic Approaches to Localized Corrosion," *Corrosion Science*, 52 (1996) 813.

(7) Kuhn, A.T., Neufeld, P., Rae, T., "Synthetic Environments for the Testing of Metallic Biomaterials," *The Use of Synthetic Environments for Corrosion Testing, ASTM STP 970,* P.E. Francis and T.S. Lee, Eds., American Society for Testing and Materials, 1988.

(8) Burke, S.R., *The Composition and Function of Body Fluids*, 3rd ed., C.V. Mosby Co., 1980.

(9) Mountcastle, V.B., *Medical Physiology Volume II*, 14th ed., C.V. Mosby Co., 1980, p. 1311.

(10) Ganong, W.F., *Review of Medical Physiology*, 16th ed., Appleton and Lange, 1993, pp. 456-458.

(11) Burns, J.R., and Finlayson, B., "Proposal for a Standard Reference Artificial Urine in *in vitro* Urolithiasis Experiments," *Investigative Urology*, 18, 1980, pp. 167-169.

(12) British Standard 1695, *Urological Catheters, Part 2: Specification for Sterile, Single-Use Urethral Catheters of the Tiemann, Whistle-Tip, 3-Way, and Haematuria Types*, Section D.2.4, BSI, September 1990.

(13) Venugopalan, R., "Corrosion Testing of Stents: A Novel Fixture to Hold Entire Device in Deployed Form and Finish," *J Biomed Mat Res (Appl Biomater)*48: 829–832, 1999.