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Standard Test Method for Evaluating the Potential for Galvanic Corrosion for Medical Implants¹

This standard is issued under the fixed designation F3044; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method covers conducting galvanic corrosion tests to characterize the behavior of two dissimilar metals in electrical contact that are to be used in the human body as medical implants or as component parts to medical implants. Examples of the types of devices that might be assessed include overlapping stents of different alloys, stent and stent marker combinations, orthopedic plates and screws where one or more of the screws are of a different alloy than the rest of the device, and multi-part constructs where two or more alloys are used for the various component parts. Devices which are to be partially implanted, but in long-term contact within the body (such as external fixation devices) may also be evaluated using this method.

1.2 This test method covers the selection of specimens, specimen preparation, test environment, method of exposure, and method for evaluating the results to characterize the behavior of galvanic couples in an electrolyte.

1.3 Devices and device components are intended to be tested in their finished condition, as would be implanted (that is, the metallurgical and surface condition of the sample should be in or as close as possible to the same condition as in the finished device).

1.4 This test method does not address other types of corrosion and degradation damage that may occur in a device such as fretting, crevices, or the effect of any galvanically induced potentials on stress corrosion and corrosion fatigue. Surface modifications, such as from scratches (possibly introduced during implantation) or effects of welding (during manufacture), are also not addressed. These mechanisms are outside of the scope of this test method.

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 Warning—Mercury has been designated by many regulatory agencies as a hazardous substance that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Use caution when handling mercury and mercury-containing products. See the applicable product Safety Data Sheet (SDS) for additional information. The potential exists that selling mercury or mercury-containing products, or both, is prohibited by local or national law. Users must determine legality of sales in their location.

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

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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NOTE 1—Additional information on galvanic corrosion testing and examples of the conduct and evaluation of galvanic corrosion tests in electrolytes are given in Ref. given.(1).²

1.8 *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:³

D1193 Specification for Reagent Water

F2129 Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices

G1 Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens

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)⁴

G16 Guide for Applying Statistics to Analysis of Corrosion Data

G31 Guide for Laboratory Immersion Corrosion Testing of Metals

G46 Guide for Examination and Evaluation of Pitting Corrosion

G59 Test Method for Conducting Potentiodynamic Polarization Resistance Measurements

G71 Guide for Conducting and Evaluating Galvanic Corrosion Tests in Electrolytes

G82 Guide for Development and Use of a Galvanic Series for Predicting Galvanic Corrosion Performance

G102 Practice for Calculation of Corrosion Rates and Related Information from Electrochemical Measurements

G215 Guide for Electrode Potential Measurement

3. Significance and Use

3.1 Implantable medical devices can be made of dissimilar metals or come into electrical contact with dissimilar metals leading to the potential for galvanic corrosion, which may result in the release of corrosion products with harmful biological consequences or a compromise of structural integrity of the device. Therefore, it is important to determine the susceptibility of these types of devices to galvanic corrosion.

3.2 Use of this test method is intended to provide information on the possible galvanic component of corrosion of two dissimilar metals in contact with one another. The dissimilar metals in contact may be on the same implantable medical device or as component parts of individual medical implant devices.

3.3 This test method has been designed to accommodate a wide variety of device shapes and sizes encountered by allowing the use of a variety of holding devices.

3.4 This standard is presented as a test method for conducting galvanic corrosion tests in a simulated physiological environment. Adherence to this test method should aid in avoiding some of the inherent difficulties in such testing. Other standards such as Guide **G71** are general and, while they provide valuable background information, do not provide the necessary details or specificity for testing medical device implants.

4. Apparatus

4.1 *Potentiostat*, verified in accordance with Reference Test Method **G5**. Other means of verifying the accuracy and reliability of the potentiostat may be used, so long as this is adequately documented. For this test method, the potentiostat should be a high impedance instrument configured as a zero resistance ammeter (ZRA). Alternatively, a setup consisting of a dedicated ZRA, an electrometer and a two-channel recorder for recording the galvanic current and galvanic potential with time can be used. The currents measured during the test are likely in the nA range (or lower). The instrument used should be capable of reliably measuring such currents.

² The boldface number in parentheses refers to the reference provided at the end of the document. Marek, M., "Corrosion Testing of Implantable Medical Devices," *Handbook of Materials for Medical Devices*, Vol 23, ASM International, 2012.

³ 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.

⁴ The last approved version of this historical standard is referenced on www.astm.org.

4.2 *The Tested Samples*, prepared as individual electrodes of the galvanic couple. The configuration of each electrode and holder will depend on the type of specimen being tested, as described in 5.2. The sample holder can be of various configurations, provided it allows for good electrical connection to the sample, provides a method of electrical connection outside of the test cell, ensures that the sample sits fully below the liquid level line in the test cell, does not come into physical contact with any other element of the cell or apparatus, and allows for masking of the sample at the point of connection.

4.3 *Reference Electrode*—A standard reference electrode should be used in the test. Examples of standard electrodes are provided in Guide G215, along with a table showing conversions between electrodes. The reference electrode used in the test shall be identified along with the conversion used, if necessary. Individual electrochemical potentials such as E_b and E_r should be reported relative to the saturated calomel electrode (SCE), as described in Reference Test Method (SCE). (For example, G5, is the preferred reference electrode. If another standard electrode is used (for example, Ag/AgCl), data should be adjusted so that it is reported with respect to SCE; the saturated Ag/AgCl electrode is 45 mV electronegative to SCE. A reading of 0 mV versus Sat'd. Ag/AgCl would be equivalent to a reading of -45 mV versus SCE. Therefore, to convert potentials measured using a Sat'd. Ag/AgCl electrode to the SCE scale, 45 mV should be subtracted.) When plotting curves, potentials may be plotted using raw data, that is, to the scale of the reference electrode used during the test and this shall be clearly shown on the axis label (for example, "Potential versus SCE (mV)" or "Potential versus Sat'd. Ag/AgCl (mV)").

NOTE 2—Due to the presence of mercury in the saturated calomel electrode (SCE) and the increasing regulation around the use of equipment and materials that contain mercury, there may be decreased availability of SCE as a reference electrode. When choosing a reference electrode for testing, users of this standard should also take note of any regulations relevant to their region.

4.4 *Salt Bridge*, such as a Luggin probe, may be used between the working and reference electrode, such as the type shown in Reference Test Method G5.

4.5 *Suitable Polarization Cell*, with a volume of at least ~~500 cm³~~ 500 cm³, equivalent to or similar to that recommended in Reference Test Method G5. The volume of the cell may be greater than ~~500 cm³~~ 500 cm³ if needed to accommodate a larger sample.

4.6 *Water Bath*, or other heating appliance capable of maintaining the test solution temperature at $37 \pm 1^\circ\text{C}$. Note that use of a hot plate to heat and/or agitate the solution (for example, using a magnetic stir bar) can cause excessive noise and interfere with the electrochemical data.

4.7 *Gas Bubbler*, to provide aeration and agitation, capable of delivering aeration at a rate of ~~150 cm³/min~~ 150 cm³/min.

4.8 *Thermometer*, with an accuracy for measurement within $\pm 1^\circ\text{C}$.

4.9 *pH meter*, with an accuracy for measurement within ± 0.1 .

4.10 An example of a typical test cell set-up is provided in Fig. X2.1.

5. Test Specimens

5.1 *Material*—Unless otherwise justified, all samples selected for testing should be taken from finished product that has been subjected to all normal manufacturing processes and is considered acceptable for clinical use. Cosmetic rejects or other nonclinical samples may be used if the cause for rejection would not affect the galvanic corrosion behavior of the device, but the metallurgical and surface condition of the sample should be in or as close as possible to the same condition as the finished device. Sterilization or other manufacturing processes may be omitted if it can be demonstrated that these processes have no effect on the galvanic corrosion behavior of the device.

NOTE 3—Loading or deployment of samples, as it would occur *in vivo*, should be simulated as closely as is reasonably possible, since these actions can potentially affect the overall corrosion behavior of the material. Because anode and cathode must be separated for testing, it is understood that this step may not be possible.

5.2 *Selection of Anode and Cathode:*

5.2.1 It is preferable to evaluate the components before the test is initiated to determine which one would likely be the anode and which would be the cathode. For example, in a device containing two alloys, such as a stent with markers, one material will be the anode and the other will be the cathode.

5.2.2 Published galvanic series are available to help with the determination of anode/cathode (see Guide G82, for instance.) However, it should be remembered that these series are published for specific electrolytes, which may or may not accurately represent the test electrolyte or *in vivo* conditions. Alternatively, the open circuit potential (OCP) can be measured for each material in the chosen electrolyte, in order to establish their relative positions electrochemically. The material with the less noble value of the OCP will likely be the anode.

NOTE 4—Open circuit potential, for the purpose of determining anodic or cathodic condition, should be measured after a minimum of 1 h in contact with the solution. The samples used for this measurement should not then be used in the galvanic test.

5.2.3 Where a choice exists as to the relative sizes of the anode and cathode (for example, if the device comes in several sizes and the anode-to-cathode surface area ratio is different for different sizes), it should be remembered that the most aggressive galvanic couple occurs with a smaller anode relative to a larger cathode.

5.2.4 In the case where three or more alloys are to be tested for their galvanic corrosion behavior, the single most active component (anode) should be tested against a combination of the other components. If more than one component of a multi-component device is suspected of being prone to galvanic corrosion, each can be tested against the rest of the components joined together. Joining requires mounting components together in electrical contact with one another, as a single electrode (or electrode bundle). This may be accomplished by joining the electrical connections to the components outside the cell or by joining components that are to be exposed together inside the cell. The latter may require spot welding or other techniques. It is important to mask off any areas that are spot welded or otherwise altered from their original form during connection and mounting, so that these areas do not become part of the test. Materials suitable for use in masking should be impermeable to water and capable of isolating the area masked off, without contributing unwanted crevice effects.

5.2.5 The anode and cathode should be separated for testing. In some devices, particularly those containing complex, multi-alloy component parts that may be fused or brazed together, separation of anode and cathode may be difficult or impossible. In these cases, it is acceptable to mask off various areas of the part, leaving only the desired material(s) exposed.

5.2.6 Where possible, as much of the device as possible should be tested while maintaining the ratio of surface areas between anode and cathode. It is understood that small area(s) of the device will likely be masked off due to fixturing requirements.

5.3 *Surface Area Calculation:*

5.3.1 The relative surface area ratio of anode material to cathode material in the test samples should be maintained (that is, mimic the actual device) during the test. A worst case ratio may be used, but this should be based on a ratio that can actually occur in the device based on device tolerances, size variations, or differences in intended usage (see 5.2.3). An artificial worst case (for example, choosing a ratio that does not occur or is artificially high), is not recommended.

5.3.2 The surface area of the entire anode and entire cathode should be calculated from drawings or measurements. The area where the material is connected to the testing apparatus, which is masked, should be subtracted. In the case of stents containing multiple markers, the total exposed surface area of the markers should be used.

5.3.2.1 Ideally, decoupling the anode and cathode can be accomplished such that entire sub-component parts may be tested. In this case, the surface area ratio of anode to cathode should naturally be preserved. In some cases, however, it may not be practicable to decouple the materials of interest while preserving the components. In these cases, a test specimen may be used to simulate the total area of the material of interest. For example, if a stent with multiple markers is to be tested, a single piece of the marker material (such as a strip, tube, or sheet that is in as close as possible to the same metallurgical condition as the markers themselves) with area equal to the total surface area of the exposed marker material in the device may be tested against a single bare stent with markers removed or masked.

5.4 *Number of Specimens—Specimens*—As a minimum, duplicate and preferably triplicate specimens should be tested to determine the variability in the galvanic corrosion behavior. The effect of the number of replications on the application of the results is set forth in Guide G16.

6. Test Environment

6.1 The test solution should be chosen to approximate the intended *in vivo* environment.

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

6.3 The water should be distilled or deionized (DI) and should conform to the purity requirements of Specification **D1193**, Type IV reagent water.

6.4 Unless otherwise specified, phosphate buffered saline (PBS) should be used as the standard test solution. A variety of simulated physiological solutions are listed in Test Method **F2129**, Appendix X2.

6.5 The pH of the electrolyte should be adjusted if necessary based on the nature of the solution by the addition of Na₂HPO₄ (base) or NaH₂PO₄ (acid), as needed. Several pH controlling methods are provided in Appendix X2 of Test Method **F2129**.

6.6 The test should be conducted in an aerated environment (for example, using forced bubbling of laboratory air).

7. Procedure

NOTE 5—Specimens should be handled carefully so as not to contaminate or alter them. For examples, gloves should be worn to protect samples from contamination from oils from your hands.

7.1 Examine the samples in the stereomicroscope, as received, in order to assess their condition prior to testing. The purpose of the microscopy is to document the general characteristics of the device, but not to fully characterize it.

7.2 Select the anode and cathode in accordance with 5.2. Mount the test samples on suitable holders and mask off the connection points. Samples should be fully immersed for testing. Any portion of the sample not immersed or any conductive part of the mounting apparatus should be masked off to minimize unwanted effects.

7.3 Calculate separately the total surface area of the anode and of the cathode exposed to the solution in accordance with 5.3.

7.4 Prepare enough test solution to immerse the device and auxiliary electrodes as well as sufficient additional solution 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, use 500 mL of electrolyte in a clean polarization cell. Measure and record the pH of the solution before and after the test.

7.5 Place the reference electrode (a salt bridge may be used), thermometer, and gas diffuser in the test chamber and bring the temperature of the test solution to $37 \pm 1^\circ\text{C}$. Bubble air through the test cell to aerate and agitate the electrolyte. (A rate of approximately $150\text{--}150\text{ cm}^3/\text{min}$ has been shown to be adequate.) Begin bubbling at least 30 min prior to the start of the test. Continue bubbling throughout the test. (A lower flow rate may be used during the exposure period, providing it allows for adequate mixing of the fluid.)

7.6 Gently immerse the electrodes or joined electrode bundles in the test solution. The electrodes should be as close as practical to minimize the IR potential drop between them, but at a sufficient distance to prevent metallic contact.

7.7 Briefly record the open circuit potentials of the anode and cathode specimens (uncoupled) prior to initiating the galvanic corrosion test. Each measurement should be taken for less than 1 min and the galvanic corrosion test should be initiated as quickly as is reasonably possible after this step. This step is intended to capture the instantaneous uncoupled open circuit potentials of the specimens, as an indication of the galvanic driving force prior to coupling.

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.

7.8 Connect the electrodes to the instruments electrically. It is preferable to connect the electrode or joined electrode bundle believed to be the cathode to the auxiliary (counter) electrode terminal of the potentiostat, or to the ground or neutral terminal of a dedicated ZRA. The electrode believed to be the anode should then be connected to the working electrode terminal of the potentiostat connected as ZRA, or to the live input terminal of a dedicated ZRA. This connection should result in a positive galvanic current reading. If a negative current reading is obtained, it signifies that the roles of the electrodes are reversed from the original assumptions (that is, what was believed to be the cathode is actually the anode in the galvanic pair). Sections 8.1 and 8.1.1 outline treatment of data and calculation of corrosion rate, regardless of whether a positive or negative current is obtained.

7.8.1 While using a potentiostat connected as ZRA allows a connection to the working and auxiliary (counter) electrodes before the galvanic corrosion test is initiated, some dedicated ZRAs short circuit the electrodes in a galvanic testing mode as soon as the electrodes are connected to the terminals. When using a dedicated ZRA, connect the electrode to the live input terminal only to initiate the galvanic corrosion test after recording the open circuit potentials in accordance with 7.8.

7.9 Initiate the galvanic corrosion test and maintain the conditions for a minimum of 24 h while recording the galvanic potential and current. Measurements may be made at intervals of 1 min or shorter. If ~~equilibrium~~ a steady-state has not been achieved within 24 h, the test may be continued for as long as deemed necessary to reach ~~equilibrium~~ a steady-state.

NOTE 6—If the data shows that the current density has been consistently dropping during the course of test, the galvanic current at the conclusion of the test would provide an upper bound to the calculated steady state galvanic corrosion rate.

7.10 At the end of the test, disconnect the specimens. Remove the samples from the solution, rinse with DI water, and allow to dry.

8. Evaluation of Test Specimens

8.1 *Measurements During Exposure*—Data recorded during exposures may include the open circuit potentials, galvanic current measurements and the galvanic potential. It is preferred that potentials are measured relative to a verified ~~SCE~~ standard reference electrode as recommended in Guide G215 and following the conventions recommended in Practice G3. ~~If another standard electrode is used (for example, Ag/AgCl),~~ For consistency with historical reporting, data should be adjusted so that it is reported with respect to SCE. SCE as described in Guide G215 and in 4.3. Current data can be converted into a theoretical corrosion rate based on Faraday's law (see Practice G102). For this calculation, the current and potential data collected during the last 15 min of the test should be averaged. Averaging the final 15 min of data provides reasonable steady-state values of current and mixed potential at the end of the test.

8.1.1 In some galvanic couples, the roles of the samples reverse and the measured current is negative. This means that the sample connected as the working electrode is actually the cathode, and not the anode as was originally assumed during test setup. In this case, calculation of corrosion rate can still be performed as proposed in 8.1; however the surface area and material properties (such as, density and equivalent weight) of the sample connected as the cathode should actually be used, as the negative current implies that this sample is actually the anode. Use the absolute value of the measured current in the calculation.

8.1.2 Some noise and transient spikes in current or potential, or both, may occur during measurement. These may be caused by external sources (for example, an energy spike in the building or electromagnetic noise from other equipment), the test set-up (for example, an air bubble on the reference electrode or use of a hot plate), or could be real (the sample experienced a period of transient corrosion activity). External interference should be eliminated as much as possible. When unusual noise or transient spikes are identified (source known or unknown), the effect of such observations on the integrity of test results should be evaluated.

8.2 *Evaluation After Removal*: Examine the samples in the stereomicroscope or SEM, or both, to assess the condition of the surfaces and make note of any significant changes (for example, discoloration, general corrosion, change in surface appearance, or pitting) that occurred during the test.

8.3 Where replicate couples are exposed, statistical analysis of the data, as set forth in Guide G16, may be applied to generate confidence intervals for predictive purposes.

9. Report

9.1 The report should include detailed descriptions of the exposed specimens including wetted areas, pertinent data on exposure conditions (including the sample geometry), the post-testing microscopic observations, and results of the corrosion evaluation.

9.2 Data for the exposed specimens should include physical dimensions, chemical composition, metallurgical history, surface preparation, and after-exposure cleaning methods.

9.3 Details of exposure conditions should include electrolyte composition, pH, and length of exposure.

9.4 The report should contain the electrochemical data, such as initial uncoupled open circuit potential (OCP) of the anode and cathode, galvanic potential, and current density, plotted as a function of time over the course of the test. Potentials should be reported with respect to SCE. If converting from an alternative reference scale (for example, Ag/AgCl_{SAT KCl}), reports shall include the conversion factor used. The steady-state (final) galvanic potential and current (each calculated as an average of the final 15 min of data, as outlined in 8.1) should also be reported as numeric values in the report.

9.5 The results of the tests should be expressed as corrosion rate in penetration per unit time (for example, mils per year) or loss in thickness or mass during the exposure period. The calculated corrosion current density of the galvanic pair may also be compared with the corrosion current density of the uncoupled anode as described in X1.1, assuming it is determined under similar environmental conditions (that is, aerated, same electrolyte, similar testing time, etc.). If localized corrosion is observed, it should be noted in the report.

9.6 A generic description of any changes in the appearance or corrosion observed on the specimen should be reported. Photographic documentation may be appropriate.

10. Precision and Bias

10.1 An interlaboratory study of this test method is being conducted and a complete precision statement is expected to be available on or before 2023.

11. Keywords

11.1 corrosion; dissimilar metals; medical device testing; galvanic corrosion; galvanic couple; zero resistance ammeter

APPENDIXES

XI. RATIONALE

X1.1 Multi-component implants may suffer enhanced corrosion due to galvanic interaction if the components that are in contact are made of electrochemically dissimilar metals. Such components are likely to have different individual open circuit electrode potentials. At those potentials the oxidation (anodic) and reduction (cathodic) currents on each electrode are balanced. When short-circuited by contact in the electrolyte, however, the potentials of the dissimilar electrodes change toward a common galvanic potential, disturbing the equality of the currents, although the overall balance for the whole system remains, that is, the sum of all anodic current equals the sum of the absolute values of the cathodic currents. Some component electrodes are polarized (have their potential shifted) to higher potentials and they become anodes as the anodic current on each exceeds the absolute value of the cathodic current, and the driving force for their corrosion increases. The driving force corrosion for electrode or electrodes polarized to lower potentials decreases and they become cathodes as the absolute values of the cathodic currents exceeds the values of the anodic currents.

X1.1.1 The galvanic current measured in this test method and flowing to the anode is the difference between the absolute values of the anodic (oxidation) and cathodic (reduction) currents on that electrode. It is also equal to the difference between the absolute values of the sum of anodic (oxidation) and cathodic (reaction) currents on the other electrode or set of joined electrodes short-circuited with the anode through the ZRA. If the potential shift of the anode is high enough to make the cathodic current on