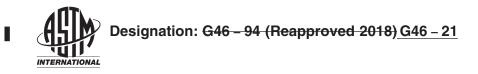
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Standard Guide for Examination and Evaluation of Pitting Corrosion¹

This standard is issued under the fixed designation G46; 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 guide covers the selection of procedures that can be used in the identification and examination of pits and in the evaluation of pitting (See Terminology examination and evaluation of pitted metals. These G15) corrosion to determine the extent of its effect.procedures include both nondestructive and destructive approaches.

1.2 The procedures covered in this guide include those that may be used in laboratory evaluations of corroded metal specimens and field examinations and inspections.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3.1 Exception—In X1.2.1, mils per year (MPY) are regarded as standard for the target corrosion rate.

1.4 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.

1.5 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

E3 Guide for Preparation of Metallographic Specimens

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

G15 Terminology Relating to Corrosion and Corrosion Testing (Withdrawn 2010)³

G16 Guide for Applying Statistics to Analysis of Corrosion Data

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

G193 Terminology and Acronyms Relating to Corrosion

¹ This guide is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

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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 standard's Document Summary page on the ASTM website.

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2.2 ISO Standard:³

ISO 25178-604:2013(E) Geometrical product specifications (GPS) — Surface texture: Areal — Part 604: Nominal characteristics of non-contact (coherence scanning interferometry) instruments

2.3 National Association of Corrosion Engineers Standard: NACE Standards:⁴

NACE RP-01-73 Collection and Identification of Corrosion Products⁵

NACE SP0775 Preparation, Installation, Analysis, and Interpretation of Corrosion Coupons in Oilfield Operations

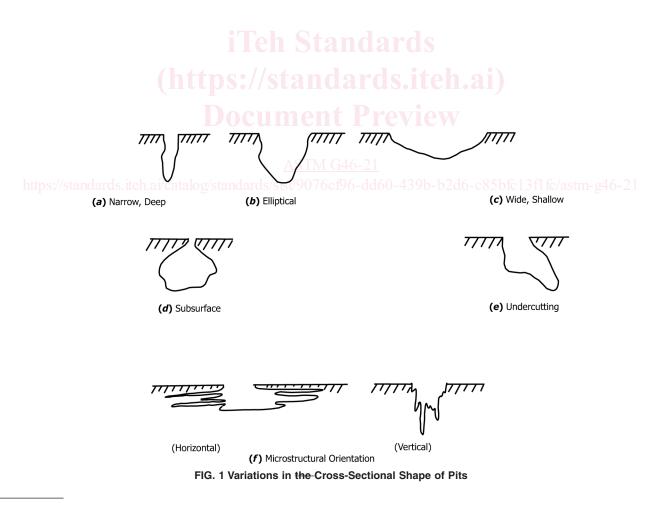
3. Terminology

3.1 Terms and acronyms used in this guide are defined in Terminology G193.

4. Significance and Use

4.1 It is important to be able to determine the extent of pitting, either in a service application where in which it is necessary to predict the remaining life in a metal structure, or in laboratory test programs that are used to select the most pitting-resistant materials for service. The purpose of the study is crucial in determining the appropriate examination and evaluation steps.

4.2 Some typical purposes of laboratory tests include, but are not limited to, evaluating performance of alloys, determining whether an alloy is resistant to the environment, evaluating how environmental conditions including corrosion inhibitor affect or prevent pitting, and evaluating whether a lot of metal is sufficiently resistant for its use in a particular application or environment.



³ The last approved version of this historical standard is referenced on www.astm.org. Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.

Available from Association for Materials Protection and Performance (AMPP), 15835 Park Ten Pl., Houston, TX 77084, http://www.ampp.org. ⁵ Insert in *Materials Protection and Performance*, Vol 12, June 1973, p. 65.

4.3 Some typical purposes of field studies include, but are not limited to, determining if pits are likely to grow and cause leak or release of process fluid, and assisting a determination of whether to replace or repair damage from pits (remaining life assessment).

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5. Identification and Examination of Pits

5.1 *Preliminary Visual Inspection*—An initial visual examination of the corroded metal surface is usually conducted in an as-received condition before any cleaning or destructive inspection.

5.1.1 It is important to distinguish between as-received, precorroded surfaces, post-hydrotest surfaces, and other surface conditioning, such as nitriding and nano coatings.

5.1.2 It is often advisable to photograph the corroded surface so that it can be compared with the clean surface after the removal of corrosion products.

5.1.3 The composition of the corrosion products may be of value in determining the cause of corrosion, especially if the specimen has been exposed to an unknown environment. Where analysis of corrosion products is desired, follow recommended procedures for the removal of particulate corrosion products (for example, NACE RP-01-73⁶) and preserve them for future identification.

5.1.4 Examine the corroded surface to determine the extent of corrosion and the apparent location of pits as well as identify areas of interest for further examination.

5.1.4.1 It is often advisable to perform a more detailed examination through a microscope using low-magnification (20×) to photograph the corroded surface at this point, so that it can be compared with the clean surface after the removal of corrosion products.

5.1.4.2 This preliminary visual inspection is typically performed under ambient light, with or without the use of a low-power magnifying glass or additional light source.

5.2 Cleaning/Pit Exposure:

5.2.1 Exposing the pits fully using recommended cleaning procedures to remove the corrosion products (see Practice G1).

5.2.1.1 Avoid solutions that attack the base metal excessively. c196-dd60-439b-b2d6-c85bfc13f1fc/astm-g46-21

5.2.1.2 Scrubbing with a stiff, nonmetallic bristle brush will often enlarge the pit openings sufficiently by removal of corrosion products or undercut metal, or both, making the pits easier to evaluate;

5.2.1.3 It may be advisable during cleaning to probe the pits with a pointed tool to determine the extent of undercutting, tunneling, or other subsurface corrosion (Fig. 1).

5.3 <u>Post-Cleaning Visual Inspection—Inspection:</u> A visual examination of the corroded metal surface is usually beneficial, and this is done under ordinary light, with or without the use of a low-power magnifying glass, to determine the extent of corrosion and the apparent location of pits. It is often advisable to photograph the corroded surface at this point so that it can be compared with the clean surface after the removal of corrosion products.

4.1.1 If the metal specimen has been exposed to an unknown environment, the composition of the corrosion products may be of value in determining the cause of corrosion. Follow recommended procedures in the removal of particulate corrosion products and reserve them for future identification (see NACE RP-01-73).

4.1.2 To expose the pits fully, use recommended cleaning procedures to remove the corrosion products and avoid solutions that attack the base metal excessively (see Practice G1). It may be advisable during cleaning to probe the pits with a pointed tool to determine the extent of undercutting or subsurface corrosion (Fig. 1). However, scrubbing with a stiff bristle brush will often enlarge the pit openings sufficiently by removal of corrosion products, or undercut metal to make the pits easier to evaluate.

⁶NACE has been changed to AMPP, which may impact how this standard is labeled in the future.

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5.3.1 Examine the cleaned metal surface under ordinary light to determine the approximate size and distribution of pits. Follow this procedure by a more detailed examination through a microscope using low magnification (20×).extent of corrosion and the apparent location of pits as well as to identify areas of interest for further examination.

5.3.2 Determine and note the size, shape (1, 2),⁷ aspect ratio (diameter/depth) (3), uniformity, and density of pits (corroded area/total surface area) (1, 2), as needed. Pit size is often defined as the diameter of the pit mouth for hemispherical pits or equivalent diameter, $[2\times\operatorname{sqrt}(\operatorname{area}/\pi)]$, or at times it can refer to the depth, length, or width of the pit. It is important to record which parameter is being measured when reporting the pit size.

5.3.2.1 Pits may have various sizes and shapes, be distributed in a uniform or nonuniform manner, and be arranged in a dense or sparse pattern. All of these traits may be relevant to the evaluation of the corrosion process.

5.3.2.2 The diverse nature of internal and external standards and specifications for evaluating pitting corrosion may mean that the level of importance on each of the above criterion may be different for each document.

5.3.3 Determine the size, shape, and density of pits. Evaluation of pit density or the number of pits per given area can be made easier by the use of a plastic grid. Place the grid, containing 3 mm to 6 mm squares, on the surface. Count and record the number of pits in each square and move across the grid in a systematic manner until the desired surface area has been covered. Obtain the average from all the measurements from each square for a final measurement value.

4.1.4.1 Pits may have various sizes and shapes. A visual examination of the metal surface may show a round, elongated, or irregular opening, but it seldom provides an accurate indication of corrosion beneath the surface. Thus, it is often necessary to cross section the pit to see its actual shape and to determine its true depth. Several variations in the cross-sectioned shape of pits are shown in Fig. 1.

5.3.3.1 It is a tedious job to determine pit density by counting pits through a microscope eyepiece, but the task can be made easier by the use of a plastic grid. Place the grid, containing 3 to 6-mm squares, on the metal surface. Count and record the number of pits in each square, and move across the grid in a systematic manner until all the surface has been covered. This approach minimizes eyestrain because the eyes can be taken from the field of view without fear of losing the area of interest.

5.3.3.2 Pit density will be affected if pit clusters, interconnected pits, or the occurrence of pits within pits are treated as one or multiple pits. In some cases, the fraction of the total area covered by pits can be considered as a parameter more relevant than pit density. $\underline{ASTM G40-21}$

https://standards.iteh.ai/catalog/standards/sist/9076cf96-dd60-439b-b2d6-c85bfc13f1fc/astm-g46-21

5.3.4 *Metallographic Examination*—Select and cut out a representative portion of the metal surface containing the pits and prepare a metallographic specimen in accordance with the recommended procedures given Evaluation of pit density can also be accomplished using available software that can post-process electronic images of the corroded surface. Electronic images can be set to a contrast threshold to delineate the corrosion pits from the noncorroded specimen surface. The number of pits can be counted and divided by the actual area of specimen in the electronic image (4in Methods-).E3. Examine microscopically to determine whether there is a relation between pits and inclusions or microstructure, or whether the cavities are true pits or might have resulted from metal dropout caused by intergranular corrosion, dealloying, and so forth.

5.3.5 Other available software commonly used in profilometry and topography measurements with built-in function of pit measurement can also be used to determine the pit density.

5.4 *Metallographic Examination*—A visual examination of the metal surface may show a round, elongated, or irregular opening, but it seldom provides an accurate indication of the nature of any corrosion beneath the surface. Thus, it is often necessary to cross section the pit to see its actual shape and to determine its true depth and size. Several variations in the cross-sectioned shape of pits are shown in Fig. 1.

5.4.1 Select and cut out a representative portion of the metal surface containing the pits and prepare a metallographic specimen in accordance with the recommended procedures given in Guide E3.

5.4.2 Examine the cross section microscopically.

⁷ The boldface numbers in parentheses refer to the list of references at the end of this practice.

5.4.2.1 Determine whether there is a relation between pits and inclusions/microstructure.

5.4.2.2 Determine whether the cavities might have resulted from metal dropout caused by intergranular corrosion, dealloying, and so forth.

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(1) The diverse nature of internal and external standards for evaluating pitting corrosion may mean that there is an importance in measuring features related only to a specific form of attack. This means that there is a high level of importance in discerning the characteristics of the attack observed to prevent incorrectly weighting results. Determination and recordkeeping should follow standard requirements.

(2) High levels of magnification (200× to 500×) may be required to identify dropout of fine grains because of intergranular corrosion

5.5 *Nondestructive Inspection*—A number of techniques have been developed to assist in the detection of cracks or cavities in a metal surface without destroying the material (15). These methods are less effective for locating and defining the shape of pits than some of those previously discussed, but they merit consideration because they are often used in situ, and thus are more applicable to field applications.

5.5.1 *Radiographic*—Radiation, such as $\frac{X \text{ rays}}{X \text{ rays}}$, are passed through the object. The intensity of the emergent rays varies with the thickness of the material. Imperfections may be detected if they cause a change in the absorption of $\frac{X \text{ rays}}{X \text{ rays}}$. Detectors or films are used to provide an image of interior imperfections. The metal thickness that can be inspected is dependent on the available energy output. Pores or pits must be as large as $\frac{1}{2}$ — $\frac{6}{2}$ — $\frac{6}{2}$ of the metal thickness to be detected. This technique has only slight application to pitting detection, but it might be a useful means to compare specimens before and after corrosion to determine whether pitting has occurred and whether it is associated with previous porosity. It may also be useful to determine the extent of subsurface and undercutting pitting (Fig. 1).

5.5.2 *Electromagnetic:*

5.5.2.1 Eddy currents can be used to detect defects or irregularities in the structure of electrically conducting materials. When a specimen is exposed to a varying magnetic field, produced by connecting an alternating current to a coil, eddy currents are induced in the specimen, and they in turn produce a magnetic field of their own. Materials with defects will produce a magnetic field that is different from that of a reference material without defects, and an appropriate detection instrument is required to determine these differences. This method is typically not used on ferromagnetic materials.

5.5.2.2 The induction of a magnetic field in ferromagnetic materials is another approach that is used. Discontinuities that are transverse to the direction of the magnetic field cause a leakage field to form above the surface of the part. Ferromagnetic particles are placed on the surface to detect the leakage field and to outline the size and shape of the discontinuities. Rather small imperfections can be detected by this method. However, the method is limited by the required directionality of defects to the magnetic field, by the possible need for demagnetization of the material, and by the limited shape of parts that can be examined.

5.5.3 *Sonies:*Sonie:

5.5.3.1 In the use of ultrasonics, pulses of sound energy are transmitted through a couplant, such as oil or water, onto the metal surface where waves are generated. The reflected echoes are converted to electrical signals that can be interpreted to show the location of flaws or pits. Both contact and immersion methods are used. The test has good sensitivity and provides instantaneous information about the size and location of flaws. However, reference standards are required for comparison, and training is needed to interpret the results properly.

5.5.3.2 An alternative approach is to use acoustic emissions in detecting flaws in metals. Imperfections, such as pits, generate high-frequency emissions under thermal or mechanical stress. The frequency of emission and the number of occurrences per unit time determine the presence of defects.

5.5.4 <u>Penetrants—Penetrant</u>—Defects opening to the surface can be detected by the application of a penetrating liquid that subsequently exudes from the surface after the excess penetrant has been removed. Defects are located by spraying the surface with a developer that reacts with a dye in the penetrant, or the penetrant may contain a fluorescent material that is viewed under black light. The size of the defect is shown by the intensity of the color and the rate of bleed-out. This technique provides only an approximation of the depth and size of pits.

5.5.5 Other Profilometry and Topography Tools:

5.5.5.1 Different noncontact inspection tools (for example, laser scanner, white-light interferometer, and digital three-dimensional (3-D) microscope) are available to determine the profile of the corrosion pit without having to cross section the specimen.

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NOTE 1-To capture the true shape and size of the corrosion pit using the noncontact inspection tools, the corrosion products need to be removed.

5.5.5.2 The laser scanner uses sticker targets placed on the surface of the specimen as reference for the 3-D reconstruction. As the sample is being scanned, the laser scanner records the real surface points in relation to the sticker target position. The collected data can be reconstructed to form the 3-D rendering of the corrosion pits using compatible laser scan software.

5.5.5.3 White light interferometry uses the light interference produced by the surface roughness of the specimen. The source emits white light that is separated by the beam splitter into measurement and reference beams. The reference beam is reflected from the reference plane using a mirror, and the measurement beam is incident to the specimen surface. The interference pattern in the charged-coupled device (CCD) image sensor is formed by the reflected beam that passed through the reference mirror. The data can be reconstructed to form the 3-D rendering of the corrosion pits using compatible interferometry software (4, 6).

5.5.5.4 A digital 3-D optical microscope can be used to form the profile of the corrosion pit by stacking two-dimensional (2-D) images taken at different vertical heights (along z-axis). The optical microscope captures the data in the 2-D plane (*x*-axis and *y*-axis) with single- image acquisition at each step along the vertical height (*z*-axis). A digital 3-D rendered image is reconstructed using compatible software.

5.5.5.5 Results from 3-D rendering can often be useful in analyzing pit size, pit shape, and pit density.

5.5.6 <u>Caveats</u>—<u>NoneSome</u> of these nondestructive test methods <u>may not</u> provide satisfactory detailed information about pitting. They can be used to locate pits and to provide some information about the size of pits, but <u>they generally are not some may not</u> <u>be</u> able to detect small pits, and confusion may arise in attempting to differentiate between pits and other surface blemishes. Most of these methods were developed to detect cracks or flaws in metals, but with more refined development they may become more applicable to pitting measurements.

6. Extent of Pitting

6.1 *Mass Loss*—Metal mass loss is not ordinarily recommended for use as a measure of the extent of pitting unless general corrosion is slight and pitting is fairly severe. If uniform corrosion is significant, the contribution of pitting to total metal loss is small, and pitting damage cannot be determined accurately from mass loss. In any case, mass loss can only provide information about total metal loss due to pitting but nothing about depth of penetration. However, mass loss should not be neglected in every case because it may be of value; for example, mass loss along with a visual comparison of pitted surfaces may be adequate to evaluate the pitting resistance of alloys in laboratory tests.

6.2 Pit Depth Measurement:

6.2.1 *Metallographic*—Pit depth can be determined by sectioning vertically through a pre-selected pit, mounting the cross-sectioned pit metallographically, and polishing the surface. The depth of the pit is measured on the flat, polished surface by the use of a microscope with a calibrated evepiece. measurement system (for example, evepiece reticle or digital imaging. The method is very accurate, but it requires good judgment in the selection of the pit and good technique in cutting through the pit. Its limitations are that it is time consuming, the deepest pit may not have been selected, and the pit may not have been sectioned at the deepest point of penetration.

6.2.2 Machining (7, 8): (2, 3):

6.2.2.1 This method requires a sample that is fairly regular in shape, and it involves the destruction of the specimen. Measure the thickness of the specimen between two areas that have not been affected by general corrosion. Select a portion of the surface on one side of the specimen that is relatively unaffected; then machine the opposite surface where the pits are located on a precision lathe, grinder, or mill until all signs of corrosion have disappeared. (Some difficulty from galling and smearing may be encountered with soft metals, and pits may be obliterated.) Measure the thickness of the specimen between the unaffected surface and subtract from the original thickness to give the maximum depth of pitting. Repeat this procedure on the unmachined surface unless the thickness has been reduced by 50 % or more during the machining of the first side.

6.2.2.2 This method is equally suitable for determining the number of pits with specific depths. Count the visible pits; then

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machine away the surface of the metal in measured stages and count the number of visible pits remaining at each stage. Subtract the number of pits at each stage from the count at the previous stage to obtain the number of pits at each depth of cut.

6.2.3 Micrometer or Depth Gage:

6.2.3.1 This method is based on the use of a pointed needle attached to a micrometer or calibrated depth gage to penetrate the pit cavity. Zero the instrument on an unaffected area at the lip of the pit. Insert the needle in the pit until it reaches the base where a new measurement is taken. The distance traveled by the needle is the depth of the pit. It is best to use constant-tension instruments to minimize metal penetration at the base of the pit. It can be advantageous to use a stereomicroscope in conjunction with this technique so that the pit can be magnified to ensure that the needle point is at the bottom of the pit. The method is limited to pits that have a sufficiently large opening to accommodate the needle without obstruction; this eliminates those pits where undercutting or directional orientation has occurred.

6.2.3.2 In a variation of this method, attach the probe to a spherometer and connect through a microammeter and battery to the specimen (38, 49). When the probe touches the bottom of the pit, it completes the electrical circuit, and the probe movement is a measurement of pit depth. This method is limited to very regularly shaped pits because contact with the side of the pit would give a false reading.

6.2.4 *Microscopical*—This method is particularly valuable when pits are too narrow or difficult to penetrate with a probe type of instrument. The method is amenable to use as long as light can be focused on the base of the pit, which would not be possible in the case of example (e) in Fig. 1.

6.2.4.2 Locate a single pit on the metal surface and center under the objective lens of the microscope at low magnification (for example, $50\times$). Increase the objective lens magnification until the pit area covers most of the field under view. Focus the specimen surface at the lip of the pit, using first the coarse and then the fine-focusing knobs of the microscope. Record the initial reading from the fine-focusing knob. Refocus on the bottom of the pit with the fine-focusing knob and record the reading. The difference between the initial and the final readings on the fine-focusing knob is the pit depth.

6.2.4.3 Repeat the steps in $\frac{5.2.4.26.2.4.2}{100}$ to obtain additional measurements or until satisfactory duplication has been obtained. The repeatability of pit depth measurements on a single pit at four magnifications is shown in Annex A1.

6.2.4.4 A variation of the microscopical technique <u>employsinvolves</u> the use of an interference microscope. A beam of light is split, and one portion is projected on the specimen and the other on a reference mirror surface. The reflected light from these two surfaces is recombined, and interference fringes are formed that provide a topographical map of the specimen surface. These fringes can be used to measure vertical deviations on the metal surface. However, the method is limited to the shallower pits, that is, less than 25 μm, because the number of fringes increases to the point where they are difficult to count.

6.2.5 3-D Optical Microscopy Method—This method is distinguishable from 6.2.4.2 in that analysis does not require manual use of the fine focus of a microscope. Rather, microscopes with computer-controlled capabilities are commercially available with corrosion pit measurement as an intended application. There are numerous advantages to this type of analysis, including the reduction in time of analysis and the ability to scan larger surfaces. Such digital equipment has the additional advantage that it allows quantification of pitted surface areas and pit diameters as well as provides depths and pit shapes. Asymmetric pits and tunneling processes that distort the pit shape will be as difficult to detect using this method as the manual method above. As with standard optical microscopy, one needs to: (1) use sufficient magnification to observe pit features, and (2) check the validity of the equipment using pits with depths validated by independent means. Care should be taken to ensure the system is properly calibrated and attention should be paid to the influence of sample type and surface condition on how accurately and reproducibly the system detects the correct number of pits, pit depths, areas, volumes, and so forth.

6.2.6 *Laser Scanning Methods* (see ISO 25178-604:2013(E))—This profilometry method uses lasers to scan the metal surface and measure pit depth relative to the metal surface. The use of lasers allows the user to scan large areas for inspection. However, the resolution required for pit depth measurement may require parameter optimization (for example, scan speed/scan interval). In addition to ensuring proper calibration of the system, attention should be paid to the reproducibility of measurements within a given sample, and the validity of the equipment should be confirmed using pits with depths evaluated using independent means. While this technology has been used successfully to evaluate surface roughness/topography (10), real-world users have communicated