APPENDIX A-1

Tablulated List of Current Corrosion Standards, Test Methods, and Recommended Practices Issued by the American Society for Testing and Materials (ASTM) and the National Association of Corrosion Engineers (NACE)

American Society For Testing and Materials, 1916 Race Street, Philadelphia, Pa., 19103.

Designation	Title
A 262–68(3)[1]	Recommended Practices for Detecting Susceptibility to Intergranular Attack in Stainless Steels
A 279–63(3)	Total Immersion Corrosion Test of Stainless Steels
A 296–68(2)	Specification for Corrosion Resistant Iron-Chromium, Iron-Chromium-Nickel, and Nickel Base Alloy Castings for General Application
A 380–57(3)	Recommended Practice for Descaling And Cleaning Stainless Steel Surfaces
A 393–63(3)	Recommended Practice for Conducting Acidified Copper Sulfate Test for Intergranular Attack in Austenitic Stainless Steel
B 117–64(7, 21, 31)	Salt Spray (Fog) Testing
B 287–62(7, 21, 31)	Acetic Acid-Salt Spray (Fog) Testing
B 368–68(7, 21)	Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)
B 380–65(7)	Corrosion Testing of Decorative Chromium Plating by the Corrodkote Procedure
B 537–70(7)	Recommended Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure
B 538–70(7)	Method of FACT (Ford Anodized Aluminum Corrosion Test) Testing
C 464–64(14)	Test for Corrosion Effect of Thermal Insulating Cements on Base Metal
C 621–68(13)	Test for Static Corrosion of Refractories by Molten Glass
C 622–68(13)	Simulated Service Test for Corrosion Resistance of Refractories to Molten Glass
D 69–67(28)	Specification for Friction Tape for General Use for Electrical Purposes
D 130–68(17)	Test for Detection of Copper from Petroleum Products, by the Copper Strip Tarnish Test
D 484–71(17)	Specification for Hydrocarbon Drycleaning Solvents
D 665–60(17)	Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water
D 801–57(20)	Dipentene, Sampling and Testing
D 807–52(23)	Corrosivity Test of Industrial Water (United States Bureau of Mines Embrittlement Detector Method)

[1] Numbers in parentheses indicate the part number of the Book of Standards in which the standard appears, as of 15 July 1971. Standards are also available separately. The number after the dash is the year of adoption or of latest revision.

Designation	Title
D 849–47(20)	Test for Copper Corrosion of Industrial Aromatic Hydrocarbons
D 930–67(22)	Total Immersion Corrosion Test of Water-Soluble Aluminum Cleaners
D 1141–52(23)	Specification for Substitute Ocean Water
D 1261–55(17)	Test for Effect of Grease on Copper
D 1275–67(18, 29)	Test for Corrosive Sulfur in Electrical Insulating Oils
D 1280–67(22)	Total Immersion Corrosion Test for Soak Tank Metal Cleaners
D 1374–57(22)	Aerated Total Immersion Corrosion Test for Metal Cleaners
D 1384–70(22)	Corrosion Test for Engine Antifreezes in Glassware
D 1567–62(22)	Testing Detergent Cleaners for Evaluation of Corrosive Effects on Certain Porcelain Enamels
D 1611–60(15)	Test for Corrosion Produced by Leather in Contact with Metal
D 1616–60(20)	Test for Copper Corrosion by Mineral Spirits (Copper Strip Test)
D 1654–61(21)	Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments
D 1735–62(21)	Water Fog Testing of Organic Coatings
D 1743–64(17)	Test for Rust Preventive Properties of Lubricating Greases
D 1838–64(18, 19)	Test for Copper Strip Corrosion by Liquified Petroleum (LP) Gases
D 2043–69(15)	Test for Silver Tarnishing by Paper
D 2059–63(25)	Test for Resistance of Zippers to Salt Fog
D 2251–67(22)	Test for Metal Corrosion by Halogenated Organic Solvents and Their Admixtures
D 2570–70(22)	Simulated Service Corrosion Testing of Engine Antifreezes
D 2649–70(18)	Determining Corrosion Characteristics of Dry Solid Film Lubricants
D 2688–70(23)	Test for Corrosivity of Water in the Absence of Heat Transfer (Weight Loss Methods)
D 2776–69T(23)	Tests for Corrosivity of Water in the Absence of Heat Transfer (Electrical Methods), Tentative
D 2803–70(21)	Test for Filiform Corrosion Resistance of Organic Coatings on Metal
D 2809–69T(22)	Test for Cavitation-Erosion Corrosion Characteristics of Aluminum Automotive Water Pumps with Coolants, Tentative
G 1–72(31)	Recommended Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens
G 2–67(7, 31)	Recommended Practice for Aqueous Corrosion Testing of Samples of Zirconium and Zirconium Alloys
G 3–68(31)	Recommended Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
G 4–68(3, 31)	Recommended Practice for Conducting Plant Corrosion Tests
G 5–72(31)	Recommended Practice for a Standard Reference Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements
G 7–69T(30)	Recommended Practice for Atmospheric Exposure Testing of Nonmetallic Materials, Tentative

Designation	Title
G 9–69T(21, 30)	Test for Water Penetration into Pipeline Coatings, Tentative
G 11–69T(21, 30)	Test for Effects of Outdoor Weathering on Pipeline Coatings, Tentative
G 15–71(31)	Definitions of Terms Relating to Corrosion and Corrosion Testing
G 16–71(31)	Recommended Practice for Applying Statistics to Analysis of Corrosion Data
G 28–72	Method of Detecting Susceptibility to Intergranular Attack in Wrought Nickel-Rich Chromium Bearing Alloys
G 30–72	Recommended Practice for Making and Using U-Bend Stress Corrosion Test Specimens
G 31–72	Recommended Practice for Laboratory Immersion Corrosion Testing of Metals
G 33–72	Recommended for Recording Data from Atmospheric Corrosion Tests of Metallic Coated Steel Specimens
G 34–72	Standard Method of Test for Exfoliation Corrosion Susceptibility in 7XXX series Copper-Containing Aluminum Alloys (Exco Test)
G 35–73	Recommended Practice for Determining the Susceptibility of Stainless Steel and Related Ni-Cr-Fe Alloys to Stress Corrosion Cracking in Polythionic Acids
G 36–73	Recommended Practice for Performing Stress Corrosion Cracking Tests in a Boiling Magnesium Chloride Solution
G 37–73	Recommended Practice for the Use of Mattsson's Solution of pH 7.2 to Evaluate the Stress Corrosion Susceptibility of Cu-Zn Alloys

National Association of Corrosion Engineers, 2400 West Loop South, Houston, Texas, 77027.

TM–01–69[2]	Laboratory Corrosion Testing of Metals for the Process Industries
RP–01–69	Control of External Corrosion on Underground or Submerged Piping Systems
TM–01–70	Visual Standard for Surfaces of New Steel Airblast Cleaned with Sand Abrasive
RP–01–70	Protection of Austenitic Stainless Steel in Refineries Against Stress Corrosion Cracking by the Use of Neutralizing Solutions During Shut Down
TM–02–70	Method of Conducting Controlled Velocity Laboratory Corrosion Tests
TM–01–71	Autoclave Corrosion Testing of Metals in High-Temperature Water
RP–01–71	Method for Lining of Lease Production Tanks with Coal Tar Epoxy
TM–01–72	Antirust Properties of Petroleum Products Pipeline Cargoes
RP–01–72	Surface Preparation of Steel and Other Hard Materials by Water Blasting Prior to Coating or Recoating
RP–02–72	Direct Calculation of Economic Appraisals of Economic Control Measures

[2] The last two digits indicate the year of adoption. TM denotes a test method and RP a recommended practice.

Designation	Title
RP–03–72	Methods for Lining Lease Production Tanks with Coal Tar Epoxy
RP–04–72	Methods and Controls to Prevent In-Service Cracking of Carbon Steel (P-1) Welds in Corrosive Petroleum Refining Environments
RP–05–72	Design, Installation, Operation, and Maintenance of Impressed Current Deep Ground Beds
TM–01–73	Methods for Determining Water Quality for Subsurface Injection Using Membrane Fitters
RP–01–73	Collection and Identification of Corrosion Products
RP–02–73	Handling and Proper Usage of Inhibited Oil Field Acids

APPENDIX A-2

Selected Tabulation of British, French, and German Standards Concerned with Corrosion Testing Methods and the Evaluation of the Corrosion Resistance of Materials and Products

---

British Standards: Issuing Agency—British Standards Institution

Designation	Title and Description

B.S. 135   Specifications for Benzines and Benzoles
The corrosive sulfur content is specified in terms of the discoloration of a freshly prepared copper strip exposed in a reflux condenser.

B.S. 245   Specifications for White Spirit
Similar to B.S. 135

B.S. 441   Rosin-Cored Solder Wire "Activated" and "Non-Activated" (Non-corrosive)
The corrosive action of flux residue is assessed in terms of the discoloration and possible pitting of a copper sheet exposed to the flux at 35 C for 48 h.

B.S. 489   Specification for Steam Turbine Oils
Specifies corrosivity, rust preventing characteristics, and oxidation behavior in terms of ASTM Standard Methods D 130, D 665, D 943, and D 974.

B.S. 1133   British Standard Packaging Code. Section 6. Temporary Prevention of Corrosion
Salt, humidity, and hydrogen bromide exposure tests are used to evaluate the effectiveness of corrosion inhibiting coatings and solutions.

B.S. 1224   Specification for Electroplated Coatings of Nickel and Chromium
CASS, Corrodkote, and acetic acid salt spray tests (similar to ASTM B 368, B 380, and B 287) are used to evaluate corrosion resistance.

B.S. 1263   Hypodermic Syringes for Use in Medical and Surgical Practice
Autoclaving in steam, boiling in distilled water, and boiling in 0.9 percent sodium chloride solution consecutively for 30 min each, are used to evaluate corrosion resistance.

B.S. 1344 Part 2A   Vitreous Enamels-Group A, Kitchen Equipment
Disks of filter paper saturated with 100 g/l of citric acid are placed onto the surface and the deterioration observed after 20 min at 20 C.

B.S. 1391   Performance Tests for Protection of Light-Gauge Steel and Wrought Iron Against Corrosion
Corrosion tests are described involving either daily exposure to a sea water spray or continuous exposure to vapor condensation above a heated solution of sulfur dioxide. These tests are aimed at evaluating both metallic and paint coatings.

B.S. 1615   Anodic Oxidation Coatings for Aluminum
An acetic salt spray test (similar to ASTM B 287) and the sulfur dioxide test of B.S. 1391 are used to evaluate corrosion resistance and effectiveness of scaling.

B.S. 1706   Specification for Electroplated Coatings of Cadmium and Zinc on Iron and Steel

Designation	Title and Description

The effectiveness of passivation is measured by means of a 95-percent relative humidity exposure at 55 C for 16 h, followed by cooling to 30 C and holding for 1 h.

B.S. 1872 — Specification for Electroplated Coatings of Tin

Exposure to a controlled moist sulfur dioxide atmosphere is used to determine coating discontinuities.

B.S. 1916 — Hypodermic Syringes for Insulin Injection

The same procedure as in B.S. 1263 is used to evaluate corrosion resistance.

B.S. 2011 — Basic Climatic and Durability Tests for Components for Radio and Allied Electronic Equipment

A 2-h synthetic sea water spray at 20 C followed by storage at 35 C and 90–95 percent relative humidity is used. Humidity tests at 55 C and 95 percent humidity with 2 deg C temperature fluctuations four times an hour are also included

B.S. 2056 — Rust, Acid, and Heat Resisting Steel Wire for Springs

Susceptibility to intergranular corrosion is evaluated by a sensitizing heat treatment followed by exposure to a solution of copper sulfate and sulfuric acid.

B.S. 2983 — Hypodermic Dental Needles

A 5-h exposure to 10 percent citric acid solution at room temperature followed by boiling in distilled water for 30 min is used.

B.S. 3116 — Specification for Automatic Fire Alarm Systems in Buildings Part I. Heat-Sensitive (Point) Detectors

A 16-day exposure to condensing sulfur dioxide is used, similar to that detailed in B.S. 1391.

B.S. 3597 — Specification for Electroplated Coatings of 65/35 Tin-Nickel Alloy

A 24-h exposure to a controlled sulfur dioxide atmosphere at room temperature is used to evaluate the presence of discontinuities.

B.S. 3745 — Method for the Evaluation of Results of Accelerated Corrosion Tests on Metallic Coatings

A detailed procedure for the counting and evaluation of corrosion sites observed after acetic acid salt spray, Corrodkote, and CASS tests is described.

B.S. 4601 — Specification for Electroplated Coatings of Nickel Plus Chromium on Plastic Materials

CASS and acetic acid salt spray tests are used.

B.S. 4292 — Specification for Electroplated Coatings of Gold and Gold Alloy

Exposure to sulfur dioxide followed by exposure to hydrogen sulfide is used for coatings greater than 5 μm thick while hydrogen sulfide exposure alone is used for thinner coatings.

B.S. 4758 — Specification for Electroplated Coatings of Nickel for Engineering Purposes

Exposure to sodium chloride and gelatine-soaked filter papers for 10 min followed by dipping into a solution of potassium ferricyanide is used to evaluate coating porosity.

French Standards: Issuing Agency—L'Association Francaise De Normalisation (AFNOR)

NF X 41–002 — Essai au brouillard salin

Gives specifications for both 5 and 20 percent salt spray testing at 35 C and 85–90 percent relative humidity.

Designation	Title and Description
NF A 91–020	Revêtements Métalliques Clichés-Étalons Pour Essais de Corrosion

Provides color photographs showing the difference in behavior of an anodic metal plating (zinc) and a cathodic metal plating (nickel) on steel during salt spray testing.

| NF A 05–159 | Détermination de la Résistance a la Corrosion Intergranulaire des Aciers Inoxydables Austénitiques |

Describes the determination of intergranular corrosion susceptibility in austenitic stainless steels using the Monypenny-Strauss Test (immersion in a solution of sulfuric acid and copper sulfate).

| NF A 91–021 | Méthode d'Évaluation des Résultats des Essais de Corrosion, Applicable aux Dépots Électrolytiques Cathodiques |

Provides a detailed rating procedure and classification system for evaluating (in conjunction with NF A 91–020) the performance of cathodic metal electroplates in accelerated corrosion tests.

| NF A 05–160 | Détermination de la Resistance à la Corrosion Intergranulaire des Aciers Inoxydables Austenitiques Essai de Corrosion en Milieu Nitrique |

Describes the determination of intergranular Corrosion susceptibility in austenitic stainless steels by means of the Huey Test (nitric acid exposure).

German Standards: Issuing Agency—Fachnormenausschuss Materialprüfung im Deutschen Normenausschuss

Designation	Title
DIN 1548	Zinküberzüge runder Stahldrähte
DIN 2444	Entwurf, Zinküberzüge auf Stahlrohren; Technische Lieferbedingungen für Feuerverzinkung in handelsüblicher Qualität
DIN 8565	Rostschutz von Stahlbauwerken durch Metallspritzen
DIN 20578	Zinküberzüge für Förderwagen; Feuerverzinkung der Kästen
DIN 50010	Werkstoff-, Bauelemente- und Geräteprüfung; Klimabeanspruchung, Allgemeines, Begriffe
DIN 50016	Werkstoff-, Bauelemente- und Geräteprüfung; Beanspruchung im Feucht-Wechselklima
DIN 50017	Werkstoff-, Bauelemente- und Geräteprüfung; Beanspruchung in Schwitzwasser-Klimaten
DIN 50018	Werkstoff-, Bauelemente- und Geräteprüfung; Beanspruchung im Schwitzwasser-Wechsel-klima mit schwefeldioxydhaltiger Atmosphäre
DIN 50021	Vornorm Korrosionsprüfungen; Sprühnebelprüfungen mit verschiedenen Natriumchloridlösungen
DIN 50900	Korrosion der Metalle; Begriffe
DIN 50901	Korrosionsgroben bei ebenmässigem Angriff; Begriffe, Formelzeichen, Einheiten
DIN 50902	Entwurf Korrosionsschutz; Behandlung von Metalloberflächen, Begriffe
DIN 50903	Metallische Überzüge; Poren, Einschlüsse, Blasen und Risse, Begriffe
DIN 50905	Korrosionsversuche; Richtlinien für die Durchführung und Auswertung

Designation	Title
DIN 50906	Korrosionsprüfung in kochenden Füssigkeiten (Kochversuch)
DIN 50907	auf Meerklima- u. Meerwasserbeständigkeit, für Leichtmetalle
DIN 50908	Prüfung von Leichtmetallen; Spannungskorrosionsversuche
DIN 50910	Einflussgrössen und Messverfahren bei der Korrosion im Erdboden in Gegenwart von elekrischen Erdströmen
DIN 50911	Prüfung von Kupferlegierungen; Quecksilbernitratversuch
DIN 50914	Prüfung nichtrostender Stähle auf Beständigkeit gegen interkristaline Korrosion; Kupfersulfat-Schwefelsäure-Verfahren
DIN 50930	Vornorm Korrosion der Metalle; Beurteilung des korrosionschemischen Verhaltens kalter Wässer gegenüber unverzinkten und verzinkten Eisenwerkstoffen, Richtlinien
DIN 50932	Prüfung metallischer Überzüge; Bestimmung der Dicke von Zinküberzügen auf Stahl durch örtliches anodisches Ablösen
DIN 50933	Entwurf, Prüfung metallischer Überzüge; Messung der Dicke von Überzügen auf Stahl mittels Feinzeigers
DIN 50938	Entwurf, Korrosionsschutz; Brünieren von Eisenwerkstoffen
DIN 50940	Prüfung von chemischen Entrostungsmitteln und Sparbeizzusätzen (Inhibitoren) für Stahl und Eisen: Laboratoriumsversuche
DIN 50941	Korrosionsschutz; Chromatieren von galvanischen Zink- und Cadmiumüberzügen
DIN 50942	Entwurf, Korrosionsschutz; Phosphatieren von Stahlteilen
DIN 50943	Prüfung von anorganischen nichtmetallischen Überzügen auf Aluminum und Aluminiumlegierungen; mikroskopishe Messung der Schichtdicke
DIN 50944	Prüfung von anorganischen nichtmetallischen Überzügen auf Reinaluminum und Aluminumlegierungen; Bestimmung des Flächengewichtes von Aluminiumoxidschienten durch chemisches Ablösen
DIN 50945	—; Zerstörungstreie Messung der Dicke transparenter Oxidschichten nach dem Differenzverfahren mit dem Mikroskop
DIN 50946	—; Prüfung der Güte der Verdichtung anodisch erzeugter Oxidschichten im Anfärbeversuch
DIN 50947	—; Prüfung anodisch erzeugter Oxidschichten im Korrosionsversuch (Dauertauchversuch)
DIN 50948	Prüfung von anorganischen nichtmetallischen Deckschichten auf Reinaluminum und Aluminiumlegierungen; zerstörungsfreie Messung der Schichtdicke von transparenten Oxidschichten nach dem Lichtschnittverfahren
DIN 50949	Prüfung von anorganischen nichtmetallischen Überzügen auf Reinaluminum and Aluminumlegierungen; Zerstörungsfreie Prüfung von anodisch erzeügten Oxidschichten durch Messung des Scheinleitwertes
DIN 50950	Prüfung galvanischer Überzüge; mikroskopische Messung der Schichtdicke
DIN 50951	Entwurf, Prüfung galvanischer Überzüge; Messung der Dicke galvanischer Überzüge nach dem Strahlverfahren

Designation	Title
DIN 50952	Prüfung metallischer Überzüge; Bestimmung des Flächengewichtes von Zinküberzügen auf Stahl durch chemisches-Ablösen des Überzüges, gravimetrisches Verfahren
DIN 50953	Prüfung galvanischer Überzüge; Bestimmung der Dicke von dünnen Chromüberzügen nach dem Tüpfelverfahren
DIN 50954	Prüfung metallischer Überzüge; Bestimmg. des mittleren Flächengewichtes von Zinnüberzügen auf Stahl durch chem. Ablösen des Überzüges
DIN 50955	Entwurf, Prüfung metallischer Überzüge; Messung der Dicke galvanishcer Überzüge, coulometrisches Verfahren
DIN 50957	Prüfung galvanishcer Bäder; Galvanisierungsprüfung mit der Hull-Zelle, allgemeine Grundsätze
DIN 50958	Entwurf, Prüfung galvanischer Überzüge; Korrosionsprüfung von verchromten Gegenständen nach dem modifizierten Corrodkote-Verfahren
DIN 50960	Korrosionsschutz; galvanische Überzüge, Kurzzeichen, Schichtdicken, allgemeine Richtlinien Bbl. Vornorm, Galvanische Überzüge auf Stahl; allgemeine Hinweise zur Anwendung als Schutz gegen atmosphärische Korrosion in Mittel- und Westeuropa
DIN 50961	Korrosionsschutz; galvanische Zinküberzüge auf Stahl
DIN 50962	Korrosionsschutz; galvanische Kadminiumüberzüge auf Stahl
DIN 50963	Korrosionsschutz; galvanische Nickel- und Nickel-Chrom-Überzüge auf Stahl
DIN 50964	Korrosionsschutz; galvanische Kupfer-Nickel-Chrom-Überzüge auf Zink und Zinklegierungen
DIN 50965	Korrosionsschutz; galvanische Zinn- und Kupfer-Zinn-Überzüge auf Stahl, Kupfer und Kupferlegierungen
DIN 50967	Entwurf, Galvanische Überzüge, Nickel-Chrom-Überzüge auf Stahl, Kupfer und Zinkwerkstoffen sowie Kupfer-Nickel-Chrom-Überzüge auf Stahl und Zinkwerkstoffen
DIN 50971	Entwurf, Bl.1, Elektrolytisch erzeugte Überzüge; Chemikalien für cyanidische Bäder, Anforderungen
DIN 50972	Entwurf, Bl.1, Elektrolytisch erzeugte Überzüge; Kupfersulfat für galvanische Bäder, Anforderungen
DIN 50973	Entwurf, Bl.1, Elektrolytisch erzeugte Überzüge; Säuren für galvanische Bäder, Anforderungen
DIN 50975	Korrosionsschutz; Zinküberzüge durch Feuerverzinken, Richtlinien
DIN 50976	Entwurf, Anforderungen an Zinküberzüge auf Gegenstanden aus Eisenwerkstoffen, die als Fertigteile feuerverzinkt werden
DIN 50980	Entwurf, Prüfung metallischer Überzüge; Auswertung von Korrosionsprüfungen
DIN 51213	Vornorm Prüfung metallischer Überzüge auf Drähten

APPENDIX B

Selected ASTM Standards Referred to Frequently in Book

Designation: A 279 – 63

Standard Method of

TOTAL IMMERSION CORROSION TEST OF STAINLESS STEELS[1]

This Standard is issued under the fixed designation A 279; 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.

1. Scope

1.1 This method describes procedures for making total immersion corrosion tests on stainless steels. No one procedure is rigorously described since the most desirable procedure to follow in any specific case will depend on the particular aim desired.

1.2 When the total immersion test is to serve as a control test for determining whether successive lots of the same material differ significantly in some property from each other, the test conditions should be arbitrarily selected and closely controlled so that any variation in results can safely be attributed to variations in the material being tested.

1.3 When the test is used to assist in the choice of material for a specific use, the test conditions should simulate the conditions of service as closely as practical. Where the temperature and composition of the solution, aeration, and similar factors vary widely under service conditions, rigorous control of these factors is not necessary, provided all of the competing materials are subjected to the same exposure conditions.

1.4 In designing any total immersion test, consideration should be given to the various factors discussed in this method since these factors have been found to be of importance in affecting the results obtained.

2. Apparatus

2.1 Any apparatus capable of providing the proper control of the important factors: aeration, temperature, and velocity, may be used to achieve the required degree of reproducibility in a total immersion corrosion test. Methods for control of temperature and aeration will be essentially the same with all types of apparatus. The principal differences will be with respect to the means of providing a control of the velocity.

2.2 *Velocity:*

2.2.1 Ordinarily, velocity will be fixed at some value which shall be held uniform over the whole surface of the specimen, especially when changes in mechanical properties are to be used as a measure of corrosion; however, for particular purposes it may be desired to vary the velocity from point to point on a specimen. Any device for moving a specimen through a solution, or a solution past a specimen, as through a tube, will be satisfactory provided that relative motion can be held constant and when desired, substantially uniform over the whole surface of the specimen (Note 1). It should be recognized that at very high rates of motion the effects of skin friction will reduce the true velocity below the apparent velocity without, however, interfering with the reproducibility of tests made with the same apparatus.

NOTE 1: *Example*—Specimens may be moved in a vertical, circulating path,[2] or specimens may be

---

[1] This method is under the jurisdiction of ASTM Committee A-1 on Steel, Stainless Steel and Related Alloys, and is the direct responsibility of Subcommittee A01.14 on Methods of Corrosion Testing.
Current edition accepted Sept. 30, 1963. Originally issued 1944. Replaces A 279 – 44 T.

[2] For a description of an apparatus to move specimens in a vertical, circular *path see* Fraser, O. B. J., Ackerman, D. D., and Sands, J. W., "Controllable Variables in the Quantitative Study of the Submerged Corrosion of Metals," *Industrial and Engineering Chemistry*, Vol 19, 1927, pp. 332–338; also Searle, H. E., and LaQue, F. L., "Corrosion Testing Methods," *Proceedings*, Am. Soc. Testing Mats., Vol 35, Part II, 1935, p. 249.

A 279

mounted on a carrier attached to a rotating spindle or mounted on the spindle itself in a way that will ensure substantially uniform velocity over the principal surfaces of the specimens when this is desired.[3] It is also possible to mount a specimen on a rotating disk or spindle, so that the surfaces of the specimens move through the solution at velocities that vary with the distance from the center of rotation. Obviously, this procedure will not measure the effect of a single velocity but rather the combined effect of variable velocities. So long as the other test conditions are kept the same, such variable velocity tests may also be expected to give reproducible results.

2.2.2 The test velocity should approximate that expected in the proposed service use of the alloys or metals being tested. It may be impossible to control the motion of boiling liquids, especially when a reflux condenser is used to prevent rapid loss of some constituent of the testing solution. However, the velocity induced by boiling and aeration together may be sufficient to give satisfactory check results, even though it will not suffice to duplicate service conditions involving high velocity as well as high temperature.

2.2.3 Where velocity appears to be the controlling factor, tests should be made at different velocities, keeping the other conditions constant. In some cases it may be proper to omit any kind of mechanical stirring; however, it should be recognized that zero velocity is difficult to maintain and that stagnant tests shall be subjected to exceptionally careful control to achieve a proper degree of reproducibility.

2.3 *Temperature Control*—The temperature of the corroding solution should be controlled within ±2 F (1 C). For control testing at room temperature, it is suggested that the solution be maintained at 95 ± 2 F (35 ± 1 C) which, being slightly above most room temperatures, is easy to maintain by heat input. When a water bath is used to maintain the proper temperature, the level of the water in the bath should be the same or slightly above the level of the solution in the test jars. The water bath should be large enough to permit free circulation of the water around the test jars.

2.4 *Aeration:*

2.4.1 The degree of aeration should be subjected to close control. It is not possible to do this by depending on diffusion from the surface of the solution to maintain uniform conditions even in a well-agitated solution. To achieve air saturation, the solution should be aerated by

blowing air through it using an Alundum thimble[4] or a sintered glass diffusion disk of medium porosity to break the air stream up into small bubbles. Such air bubbles should be introduced at the base of a glass chimney over each aerator so as to prevent the impingement of the stream of air bubbles on the test specimen.

2.4.2 The rate of air flow required to maintain air saturation will depend on the volume of the testing solution, the area of the test specimen, and its rate of corrosion. The volume of air should be measured and controlled as accurately as possible, preferably within ±10%, by the use of a flowmeter such as a calibrated differential manometer, a rotameter, or other suitable device. The volume of air per litre of testing solution should be at least 20 cm^3/min when the recommended solution volume to specimen area (4 litres/dm^2) is maintained. If the indicated rate of corrosion should exceed 300 mg/$dm^2 \cdot$day, it may be necessary to increase the rate of air flow or to employ some extraordinary means of supplying the oxygen required to maintain saturation. The air should be purified by passing it through some porous packing material, such as wool or excelsior, to remove suspended solids, and then through a solution of sodium hydroxide (approximately 3%) to remove carbon dioxide and sulfur compounds, and finally through a water wash bottle which also serves to humidify the air and avoid crystallization of salts in the pores of the aerator.

2.4.3 When it is desired to maintain the dissolved oxygen concentration at a value lower than the point of saturation with air, this should be accomplished by altering the composition of the saturating gas (as by the addition of nitrogen), rather than by altering the rate of flow of the gas. Similarly, where it is desired to have zero aeration, the solution should be kept saturated with, and under, an atmosphere of an inert gas, such as oxygen-free nitrogen. Merely eliminating aeration will not ensure an air-free solution, nor can reproducible results be expected from such attempts to achieve un-

---

[3] For a description of methods for attaching specimens to a rotating spindle, see *Journal*, Am. Soc. Naval Eng., Vol 55, No. 1, February 1943, pp 64-65.

· [4] Thimble RA-98 of the Norton Co., or equivalent, is satisfactory.

A 279

aerated solutions. As a general rule, corrosion of stainless steels is retarded rather than accelerated by aeration. Conditions favoring oxygen exclusion, therefore, favor corrosion and represent just as adverse testing conditions for stainless steels as fully aerated solutions do for non-ferrous metals, and ordinary irons and steels.

2.5 *Specimen Supports*—Supports for the specimens will vary with the apparatus used, but should be designed so as to insulate specimens from each other, and from any metallic container or supporting device used with the apparatus. The supporting device and container should not be affected by the corroding agent to an extent that might cause contamination of the testing solution so as to change its corrosiveness. The shape and form of the specimen support should be such as to avoid, as much as possible, any interference with free contact of the specimen with the corroding solution. Where it is desired to set up conditions favoring contact corrosion, "deposit attack," or other forms of concentration-cell action, the means by which these types of attack are favored should be such as to ensure exact reproducibility from specimen to specimen and test to test.

3. Test Solution

3.1 Test solutions should be made up accurately, using reagents conforming to the specifications of the Committee on Analytical Reagents of the American Chemical Society, dissolved in distilled water except in special cases, such as naturally occurring solutions, or those taken directly from some plant process.

3.2 The composition of any test solution should be controlled to the fullest extent possible and, in reporting results, it should be described as completely and as accurately as possible. Chemical content should be reported either as weight percent of the solution, grams per litre, or in terms of normality.

3.3 The composition of the test solution should be checked by analysis at the end of the test to determine the extent of any changes in composition, such as might result from evaporation favored by aeration. Evaporation losses, if any, should be made up by means of a constant level device, or by frequent additions of distilled water, or other components as may

be required corresponding in composition with the vapors from the solution, so as to maintain the original volume within $\pm 1\%$.

3.4 The volume of the test solution should be large enough to avoid any appreciable changes in its corrosiveness either through the exhaustion of corrosive constituents, or the accumulation of corrosion products or other contaminants that might affect further corrosion.

3.5 A recommended ratio between the volume of the test solution and the area of the specimen is 250 ml/in.^{2} of specimen area (4 litres/dm^2).

3.6 Whatever volume of test solution is used, possible effects of corrosion on the concentration of corrosive constituents should be determined by analysis, and, when required, appropriate action should be taken by replacing the exhausted constituents or providing a fresh solution.

3.7 When the object of the test is to determine the effect of a metal or alloy on the characteristics of the test solution (for example, the effects of metals on dyes), it is desirable to reproduce the ratio of solution volume to exposed metal area that exists in practice. It is also necessary to take into account the actual time of contact of the metal with the solution. If all of these factors cannot be reproduced directly in the laboratory test, then it will be necessary to make proper allowances as by reducing the time of contact to compensate for necessary decreases in the ratio of volume to area. Any necessary distortion of the testing conditions must be taken into account when interpreting the results.

4. Test Specimens and Their Preparation

4.1 The size and shape of specimen will vary with the purpose of the test, the nature of the materials to be tested, and the testing apparatus to be used. The size may also be limited by the necessity of preserving a proper ratio between the area of the specimen and the volume of the testing solution when the latter must be limited. In general, an effort should be made to have the ratio of surface to mass large and that of edge area to total area small.

4.2 When quantitative determinations of changes in tensile properties are to be used as the principal measure of corrosion, then ten-

A 279

sion test specimens or a piece from which such specimens may be cut after exposure shall be used.[5] In such cases, also, a set of similar specimens should be preserved in a noncorrosive environment for comparison with the exposed corrosion test specimens as to tensile properties.

4.3 The shape and dimensions of specimens shall be such as to permit weighing on an accurate balance and to facilitate accurate measurement and calculation of the area of each specimen. Such measurements of dimensions shall be made to the nearest 0.01 in. (0.25 mm), unless for some special purpose greater accuracy is required.

4.4 All sheared edges should be trimmed beyond the shear marks by sawing, machining, or filing or grinding, with the final cut to be as light as possible so as to minimize hardening and distortion of the edges.

4.5 When the test is being made for engineering purposes and a special finish is specified, it may be desirable to make the surface of the test specimen correspond to the surface to be used in service. In general, however, results that are more reproducible may be expected if a standard surface finish for the test specimens is used.

4.6 It has been shown that more uniform results may be expected if a substantial layer of metal is removed from the specimens to eliminate variations in condition of the original metal surface. This may be done either by a preliminary chemical treatment (pickling) or by surfacing with a coarse abrasive paper or cloth, such as No. 50. The thickness of metal so removed should be at least 0.003 mm or 2 to 3 mg/cm^2 in the case of heavy metals.

4.7 The final treatment should include resurfacing with No. 120 abrasive paper or cloth or equivalent. This resurfacing may be expected to cause some surface work-hardening to an extent that will be determined by the vigor of the surfacing operation, but is not ordinarily significant. The resurfaced specimens should then be degreased by scrubbing with clean pumice powder, followed if necessary, by rinsing in water and a suitable solvent, such as acetone or a mixture of 50% alcohol and 50% ether, and drying. The use of towels for drying may introduce an error through contamination of the specimens with grease or

lint. The dried specimens should then be weighed with an accuracy of ±0.0005 g.

4.8 Surface passivation sometimes has an important effect on the resistance of stainless steels in certain types of nonoxidizing solutions. It is, therefore, sometimes desirable to use pre-passivated specimens in corrosion tests. This passivation may be accomplished by exposure of the finally polished specimens for 1 h in nitric acid (30 weight, %) at 60 C. Such passivated specimens represent the most nearly identical starting conditions possible for a series of stainless steel specimens (Note 2). If a passivation treatment is employed, it must be recognized that subsequent disturbance of the surface by scraping or abrasion may greatly affect the results obtained under certain conditions of exposure.

NOTE 2—Instead of the passivation treatment just described, it may be desirable simply to clean the surface of the specimens chemically by treatment in nitric acid (10 weight, %) at 60 C for 30 min.

4.9 When the proposed application will require welded assemblages, welded specimens approximating the thickness to be used should be included in the test. Such specimens should represent the same condition of heat treatment and finish as contemplated for the service unit.

4.10 To facilitate interpretation of test results and their duplication by others, the details of the methods of preparation of the specimens should be described when reporting the results of a test.

4.11 The test report should include a description of the nature and composition of the specimens (see Section 9). The composition preferably should be that actually determined by analysis of the material from which the specimens were cut. If it should not be practical to provide this information, then reference should be made to the approximate or nominal composition of the material or, as a last resort, the trade name or grade of the material may be given. The form and metallurgical condition of the specimen, including the nature and sequence of any hot or cold working, welding, and heat-treatment should also be described as completely as possible.

---

[5] See Figs. 7 and 8 of ASTM Methods E 8, Tension Testing of Metallic Materials, *Annual Book of ASTM Standards*, Part 31.

5. Number of Specimens

5.1 In general, it is recommended that total immersion tests be made in duplicate. For precise work it may be desirable to test a larger number of specimens and for routine tests a single specimen may be considered sufficient. In certain types of nonoxidizing solutions highly variable results may be obtained on stainless alloys with only slight differences in surface or exposure conditions. This should be borne in mind in conducting tests on stainless alloys in, for instance, sulfuric acid solutions.

5.2 Each specimen should preferably be tested in a separate container, since testing several specimens of either the same material or of different materials in a single container may give erratic results. However, under special conditions it may be permissible, or even desirable, to test more than one specimen in a single container provided it is recognized that the corrosion products from a specimen showing a high rate of attack may accelerate corrosion of another specimen, or specimens, in the same container.

6. Methods of Cleaning Specimens After Test

6.1 It is essential that corrosion products be removed from specimens if changes in weight are to be used as a measure of corrosion. There are many satisfactory means of cleaning specimens after exposure, such as the use of bristle brushes with mild abrasives and detergents, treatment with appropriate chemical solutions, especially suitable with certain metals and corrosion products, and electrolytic methods. The use of bristle brushes should ordinarily be limited to heavily corroded specimens. Drastic cleaning methods should not be used when the specimens are small or the amount of weight change expected is slight. For most tests on stainless alloys scrubbing of the specimen with a rubber stopper under running water has been found adequate.

6.2 Whatever the treatment, its effect in removing metal, if any, should be determined for each material and the results of weight loss determinations should be corrected accordingly. The method of cleaning should be reported.

6.3 An electrolytic cleaning method that has been found to be useful with a large number of

metals and alloys is described in Appendix X1. This method can be used for stainless alloys if desired.

6.4 Other chemical cleaning methods that may be used are:

6.4.1 Treatment in nitric acid (10 weight, %) at 60 C provided no chlorides are present since chlorides will promote attack of the base metal.

6.4.2 Treatment in a hot solution of sodium hydroxide (20%) containing 200 g/litre of zinc dust may be effective in loosening deposits which can then be rubbed off.

6.4.3 Immersion of the specimens in a hot solution of ammonium acetate to remove rust.

7. Duration of Test

7.1 The duration of any test will be determined by its nature and purpose. In some cases it will be desirable to expose a number of specimens so that certain of them can be removed after definite time intervals so as to provide a measure of change of corrosion rates with time. Any procedure that requires removal of solid corrosion products between periods of exposure of the same specimens will not measure accurately normal changes of corrosion with time.

7.2 The higher the rate of corrosion, the shorter may be the testing period.

7.3 Where the object of the test is to predict corrosion rates over a long period, it is obviously desirable to run the test for as long as may be practical, provided that the testing conditions and the corrosive characteristics of the solution can be maintained constant over a long test period.

8. Interpretation of Results

8.1 After the corroded specimens have been cleaned, they should be reweighed with the same accuracy as the original weighing (±0.0005 g). It will then be possible to calculate the loss in weight per unit of area during the test period. This may be used as the principal measure of corrosion.

8.2 Corrosion rates calculated from the loss in weight data should be reported in milligrams per square decimetre per day (24 h), abbreviated mdd. Factors for converting these units to other common corrosion-rate units are given in

A 279

Appendix X2. The expression of corrosion loss as a percentage of original weight is usually valueless.

8.3 Corrosion rates in milligrams per square decimetre per day may be expressed in terms of inches penetration per year (abbreviated ipy) by the following equation:

$$ipy = mdd \times (0.001437/d)$$

where:

d = density of the metal, g/cm^3.

8.4 It should be remembered always that any calculations of corrosion rates, such as "mdd" or "ipy," will be subject to error on account of nonuniform distribution of corrosion and changes of corrosion rates with time. In connection with the latter, it is often desirable to carry out the testing program so as to provide data from which curves can be plotted to illustrate changes in corrosion rates with time.

8.5 After reweighing, the specimens should be examined carefully and the average and maximum depths of pits, if any are present, determined by means of a calibrated microscope, or by direct measurement with a depth gage or sharp pointed micrometers. If the number of pits is very large, it should suffice to report the average depth of the ten deepest pits.

8.6 The depths of pits should be reported in thousandths of an inch for the test period. The size, shape, and distribution of pits should be noted. A distinction should be made between local attack or pitting that occurred underneath supporting devices and those pits that developed at the surfaces that had been exposed freely to the testing solution.

8.7 For special purposes it may be desirable to subject the specimen to simple bending tests and microscopical examination to determine whether any embrittlement or intergranular attack has occurred. Electrical resistance measurements of specimens of special type may be employed for studying these effects by comparing with specimens not subjected to test.

8.8 With suitable specimens, it may be possible to make quantitative mechanical tests comparing the exposed specimens with uncorroded specimens reserved for the purpose. By such means the effects of corrosion may be observed by measuring changes in mechanical properties.

8.9 Under certain conditions the stainless steels are susceptible to stress-corrosion cracking. This effect may be studied by the exposure to the solution of specimens previously stressed to a known degree by some suitable method.

9. Report

9.1 To the fullest extent that may be possible, the investigator should follow the recommendations embodied in the ASTM Manual on Quality Control of Materials.[6] In any event the report should include the following information:

9.1.1 The chemical compositions of the metals and alloys tested (see 4.11),

9.1.2 The exact size, shape, and area of the specimen,

9.1.3 The forms and metallurgical conditions of the specimens,

9.1.4 The treatment used to prepare specimens for test,

9.1.5 The number of specimens of each material tested, whether each specimen was tested in a separate container or which specimens were tested in the same container,

9.1.6 The chemical composition of the testing solution and information as to how and to what extent the composition was held constant or how frequently the solution was replaced,

9.1.7 The temperature of the testing solution and the maximum variation in temperature during the test,

9.1.8 The degree of aeration of the solution in terms of cubic centimetres of air per litre of solution per minute and the maximum variation in this flow, or similar information for any gas or mixture of gases other than air. The type of aerator should also be described,

9.1.9 The velocity of relative movement between the test specimens and the solution and a description of how this movement was effected and controlled,

9.1.10 The volume of the testing solution,

9.1.11 The nature of the apparatus used for the test,

9.1.12 The duration of the test or of each part of it if made in more than one stage,

9.1.13 The method used to clean specimens after exposure and the extent of any error

---

[6] Issued as *Special Technical Publication 15-C*, December 1957.

A 279

introduced by this treatment,

9.1.14 The actual weight losses of the several specimens, depths of pits (plus notes on their size, shape and distribution, as by sketch), data on mechanical properties before and after exposure if determined, results of microscopical examination or qualitative bend tests, and

9.1.15 Corrosion rates for individual specimens calculated in milligrams per square decimetre per day.

APPENDIXES

XI. METHOD FOR ELECTROLYTIC CLEANING OF CORROSION TEST SPECIMENS AFTER EXPOSURE

X1.1 After scrubbing to remove loosely attached corrosion products, treat the specimens as a cathode in hot, diluted sulfuric acid under the following conditions:

Test solution	sulfuric acid (5 weight, %)
Inhibitor	2 ml organic inhibitor/litre of solution
Anode	carbon
Cathode	test specimen
Cathode current density	20 A/dm²
Temperature	165 F (74 C)
Exposure period	3 min

X1.2 After the electrolytic treatment, the specimen should be scrubbed. The weight losses of specimens 0.5 dm² in area treated by the method described have been found to be less than 0.0002 mg.

X1.3 Instead of using 2 ml of any proprietary inhibitor, about 0.5 g/litre of such inhibitors as diorthotolyl thiourea, quinoline ethiodide, beta-naphthol quinoline may be used.

X1.4 It should be noted that this electrolytic treatment may result in the redeposition of adherent metal from reducible corrosion products and thus lower the apparent weight loss. However, general experience has indicated that in most cases of corrosion in liquids the possible errors from this source are not likely to be serious.

X2. CONVERSION FACTORS

Multiply	By	To Obtain
Grams per square inch per hour	372 000	milligrams per square decimetre per day (mdd)
Grams per square metre per year	0.0274	milligrams per square decimetre per day (mdd)
Milligrams per square decimetre	0.0003277	ounces per square foot
Milligrams per square decimetre per day (mdd)	0.00000269	grams per square inch per hour
Milligrams per square decimetre per day (mdd)[1]	0.001437/density of metal in g/cm³	penetration inches per year
Milligrams per square decimetre per day (mdd)	0.0001198/density of metal in g/cm³	penetration inches per month
Milligrams per square decimetre per day (mdd)	36.5	grams per square metre per year
Milligrams per square decimetre per day (mdd)	0.00365/density of metal in g/cm³	penetration centimetres per year
Milligrams per square decimetre per day (mdd)	0.00748	pounds per square foot per year
Ounces per square foot	3052	milligrams per square decimetre
Pounds per square foot per year	133.8	milligrams per square decimetre per day (mdd)

[1] Factors for converting milligrams per square decimetre per day to inches penetration per year, for different AISI types of stainless steels are given in Table X1.

A 279

TABLE X1   Factors for Converting Milligrams per Square Decimetre per Day to Inches Penetration per Year for Different Types of Stainless Steel

AISI Type No.	Multiply Corrosion Rate in mg/dm^2 day by indicated factor to get in. penetration per year
410	0.000186
430	0.000186
446	0.000189
302	0.000182
304	0.000182
308	0.000182
309	0.000182
310	0.000182
316	0.000180
317	0.000180
321	0.000182
347	0.000180

Designation: B 117 – 73

Standard Method of
SALT SPRAY (FOG) TESTING[1]

This Standard is issued under the fixed designation B 117; 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.

This method has been approved by the Department of Defense to replace method 811.1 of Federal Test Method Standard No. 151b and for listing in DoD Index of Specifications and Standards. Future proposed revisions should be coordinated with the Federal Government through the Army Materials and Mechanics Research Center, Watertown, Mass. 02172.

1. Scope

1.1 This method sets forth the conditions required in salt spray (fog) testing for specification purposes. Suitable apparatus which may be used to obtain these conditions is described in Appendix A1. The method does *not* prescribe the type of test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results. Comments on the use of the test in research will be found in Appendix A2.

NOTE 1—This method is applicable to salt spray (fog) testing of ferrous and non-ferrous metals, and is also used to test inorganic and organic coatings, etc., especially where such tests are the basis for material or product specifications.

2. Apparatus

2.1 The apparatus required for salt spray (fog) testing consists of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control. The size and detailed construction of the apparatus are optional, provided the conditions obtained meet the requirements of this method.

2.2 Drops of solution which accumulate on the ceiling or cover of the chamber shall not be permitted to fall on the specimens being tested.

2.3 Drops of solution which fall from the specimens shall not be returned to the solution reservoir for respraying.

2.4 Material of construction shall be such that it will not affect the corrosiveness of the fog.

3. Test Specimens

3.1 The type and number of test specimens to be used, as well as the criteria for the evaluation of the test results, shall be defined in the specifications covering the material or product being tested or shall be mutually agreed upon by the purchaser and the seller.

4. Preparation of Test Specimens

4.1 Metallic and metallic-coated specimens shall be suitably cleaned. The cleaning method shall be optional depending on the nature of the surface and the contaminants, except that it shall not include the use of abrasives other than a paste of pure magnesium oxide nor of solvents which are corrosive or will deposit either corrosive or protective films. The use of a nitric acid solution for the chemical cleaning, or passivation, of stainless steel specimens is permissible when agreed upon by the purchaser and the seller. Care shall be taken that specimens are not recontaminated after cleaning by excessive or careless handling.

4.2 Specimens for evaluation of paints and other organic coatings shall be prepared in accordance with applicable specification(s) for the material(s) being tested, or as agreed upon by the purchaser and supplier. Otherwise, the test specimens shall consist of steel meeting the requirements of ASTM Methods D 609 for Preparation of Steel Panels for Testing

---

[1] This method is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals, and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved March 29, 1973. Published June 1973. Originally published as B 117 – 39 T. Last previous edition B 117 – 64.

Paint, Varnish, Lacquer, and Related Products,[2] and shall be cleaned and prepared for coating in accordance with applicable procedure of Method D 609.

4.3 Specimens coated with paints or nonmetallic coatings shall not be cleaned or handled excessively prior to test.

4.4 Whenever it is desired to determine the development of corrosion from an abraded area in the paint or organic coating, a scratch or scribed line shall be made through the coating with a sharp instrument so as to expose the underlying metal before testing. The conditions of making the scratch shall be as defined in ASTM Method D 1654, Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments,[2] unless otherwise agreed upon between the purchaser and seller.

4.5 Unless otherwise specified, the cut edges of plated, coated, or duplex materials and areas containing identification marks or in contact with the racks or supports shall be protected with a suitable coating stable under the conditions of the test, such as ceresin wax.

NOTE 2—Should it be desirable to cut test specimens from parts or from preplated, painted, or otherwise coated steel sheet, the cut edges shall be protected by coating them with paint, wax, tape, or other effective media so that the development of a galvanic effect between such edges and the adjacent plated or otherwise coated metal surfaces, is prevented.

5. Position of Specimens During Test

5.1 The position of the specimens in the salt spray chamber during the test shall be such that the following conditions are met:

5.1.1 Unless otherwise specified, the specimens shall be supported or suspended between 15 and 30 deg from the vertical and preferably parallel to the principal direction of horizontal flow of fog through the chamber, based upon the dominant surface being tested.

5.1.2 The specimens shall not contact each other or any metallic material or any material capable of acting as a wick.

5.1.3 Each specimen shall be so placed as to permit free settling of fog on all specimens.

5.1.4 Salt solution from one specimen shall not drip on any other specimen.

NOTE 3—Suitable materials for the construction or coating of racks and supports are glass, rubber, plastic, or suitably coated wood. Bare metal shall not be used. Specimens shall preferably be supported from the bottom or the side. Slotted wooden strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained, if necessary by means of secondary support at the bottom of the specimens.

6. Salt Solution

6.1 The salt solution shall be prepared by dissolving 5 ± 1 parts by weight of sodium chloride in 95 parts of distilled water or water containing not more than 200 ppm of total solids. The salt used shall be sodium chloride substantially free of nickel and copper and containing on the dry basis not more than 0.1 percent of sodium iodide and not more than 0.3 percent of total impurities. Some salts contain additives that may act as corrosion inhibitors; careful attention should be given to the chemical content of the salt. By agreement between purchaser and seller, analysis may be required and limits established for elements or compounds not specified in the chemical composition given above.

6.2 The pH of the salt solution shall be such that when atomized at 35 C (95 F) the collected solution will be in the pH range of 6.5 to 7.2 (Note 4). Before the solution is atomized it shall be free of suspended solids (Note 5). The pH measurement shall be made electrometrically at 25 C (77 F) using a glass electrode with a saturated potassium chloride bridge in accordance with Method E 70, Test for pH of Aqueous Solutions with the Glass Electrode[3]; or colorimetrically using bromothymol blue as indicator, or short range pH paper which reads in 0.2 or 0.3 of a pH unit (Note 6).

NOTE 4—Temperature affects the pH of a salt solution prepared from water saturated with carbon dioxide at room temperature and pH adjustment may be made by the following three methods:
(1) When the pH of a salt solution is adjusted at room temperature, and atomized at 35 C (95 F), the pH of the collected solution will be higher than the original solution due to the loss of carbon dioxide at the higher temperature. When the pH of the salt solution is adjusted at room temperature, it is therefore necessary to adjust it below 6.5 so the collected solution after atomizing at 35 C (95 F) will meet the pH limits of 6.5 to 7.2. Take about a 50-ml sample of the salt solution as prepared at room temperature, boil gently for 30 s, cool, and determine the pH. When the pH of the salt solution is adjusted to 6.5 to 7.2 by this procedure, the pH of the atomized and collected solution at 35 C (95 F) will come within this range.

[2] Annual Book of ASTM Standards, Part 21.
[3] Annual Book of ASTM Standards, Parts 16, 22, 30.

B 117

(2) Heating the salt solution to boiling and cooling to 95 F or maintaining it at 95 F for approximately 48 h before adjusting the pH produces a solution the pH of which does not materially change when atomized at 35 C (95 F).

(3) Heating the water from which the salt solution is prepared to 35 C (95 F) or above, to expel carbon dioxide, and adjusting the pH of the salt solution within the limits of 6.5 to 7.2 produces a solution the pH of which does not materially change when atomized at 35 C (95 F).

NOTE 5—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheesecloth to prevent plugging of the nozzle.

NOTE 6—The pH can be adjusted by additions of dilute cp hydrochloric acid or cp sodium hydroxide solutions.

7. Air Supply

7.1 The compressed air supply to the nozzle or nozzles for atomizing the salt solution shall be free of oil and dirt (Note 7) and maintained between 69 and 172 kN/m^2 (10 and 25 psi) (Note 8).

NOTE 7—The air supply may be freed from oil and dirt by passing it through a water scrubber or at least 610 mm (2 ft) of suitable cleaning material such as asbestos, sheep's wool, excelsior, slag wool, or activated[4] alumina.

NOTE 8—Atomizing nozzles may have a "critical pressure" at which an abnormal increase in the corrosiveness of the salt fog occurs. If the "critical pressure" of a nozzle has not been established with certainty, control of fluctuation in the air pressure within plus or minus 0.7 kN/m^2 (0.1 psi), by installation of a suitable pressure regulator valve[5] minimizes the possibility that the nozzle will be operated at its "critical pressure."[6]

8. Conditions in the Salt Spray Chamber

8.1 *Temperature*—The exposure zone of the salt spray chamber shall be maintained at 35 + 1.1 − 1.7 C (95 + 2 − 3 F). The temperature within the exposure zone of the closed cabinet shall be recorded at least twice a day at least 7 h apart (except on Saturdays, Sundays, and holidays when the salt spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

NOTE 9—A suitable method to record the temperature is by a continuous recording device or by a thermometer which can be read from outside the closed cabinet. The recorded temperature must be obtained with the salt spray chamber closed to avoid a false low reading because of wet-bulb effect when the chamber is open.

8.2 *Atomization and Quantity of Fog*—At least two clean fog collectors shall be so

placed within the exposure zone that no drops of solution from the test specimens or any other source shall be collected. The collectors shall be placed in the proximity of the test specimens, one nearest to any nozzle and the other farthest from all nozzles. The fog shall be such that for each 80 cm^2 of horizontal collecting area there will be collected in each collector from 1.0 to 2.0 ml of solution per hour based on an average run of at least 16 h (Note 10). The sodium chloride concentration of the collected solution shall be 5 ± 1 weight percent (Note 11). The pH of the collected solution shall be 6.5 to 7.2. The pH measurement shall be made electrometrically or colorimetrically using bromothymol blue as the indicator.

NOTE 10—Suitable collecting devices are glass funnels with the stems inserted through stoppers into graduated cylinders, or crystallizing dishes. Funnels and dishes with a diameter of 10 cm have an area of about 80 cm^2.

NOTE 11—A solution having a specific gravity of 1.0255 to 1.0400 at 25 C (77 F) will meet the concentration requirement. The concentration may also be determined as follows: Dilute 5 ml of the collected solution to 100 ml with distilled water and mix thoroughly; pipet a 10-ml aliquot into an evaporating dish or casserole; add 40 ml of distilled water and 1 ml of 1 percent potassium chromate solution (chloride-free) and titrate with 0.1 N silver nitrate solution to the first appearance of a permanent red coloration. A solution that requires between 3.4 and 5.1 ml of 0.1 N silver nitrate solution will meet the concentration requirements.

8.3 The nozzle or nozzles shall be so directed or baffled that none of the spray can impinge directly on the test specimens.

9. Continuity of Test

9.1 Unless otherwise specified in the specifications covering the material or product being tested, the test shall be continuous for the duration of the entire test period. Continuous operation implies that the chamber be

---

[4] Registered U. S. Patent Office.
[5] The Nullmatic pressure regulator (or equivalent) manufactured by Moore Products Co., H and Lycoming Sts., Philadelphia, Pa. 19124, is suitable for this purpose.
[6] It has been observed that periodic fluctuations in air pressure of ±3.4 kN/m^2 (0.5 psi) resulted in about a twofold increase in the corrosivity of the fog from a nozzle which was being operated at an average pressure of 110 kN/m^2 (16 psi). Controlling the fluctuations within ±0.7 kN/m^2 (0.1 psi), however, avoided any increase in the corrosivity of the salt fog. See Darsey, V. M. and Cavanagh, W. R., "Apparatus and Factors in Salt Fog Testing," *Proceedings*, ASTEA, Am. Soc. Testing Mats., Vol. 48, 1948, p. 153.

B 117

closed and the spray operating continuously except for the short daily interruptions necessary to inspect, rearrange, or remove test specimens; to check and replenish the solution in the reservoir, and to make necessary recordings as described in Section 8. Operations shall be so scheduled that these interruptions are held to a minimum.

10. Period of Test

10.1 The period of test shall be as designated by the specifications covering the material or product being tested or as mutually agreed upon by the purchaser and the seller.

NOTE 12—Recommended exposure periods are to be as agreed upon by the purchaser and seller, but exposure periods of multiples of 24 h are suggested.

11. Cleaning of Tested Specimens

11.1 Unless otherwise specified in the specifications covering the material or product being tested, specimens shall be treated as follows at the end· of the test:

11.1.1 The specimens shall be carefully removed.

11.1.2 Specimens may be gently washed or dipped in clean running water not warmer than 38 C (100 F) to remove salt deposits from their surface, and then immediately dried. Drying shall be accomplished with a stream of clean, compressed air.

12. Evaluation of Results

12.1 A careful and immediate examination shall be made for the extent of corrosion of the dry test specimens or for other failure as required by the specifications covering the

material or product being tested or by agreement between the purchaser and the seller.

13. Records and Reports

13.1 The following information shall be recorded, unless otherwise prescribed in the specifications covering the material or product being tested:

13.1.1 Type of salt and water used in preparing the salt solution,

13.1.2 All readings of temperature within the exposure zone of the chamber,

13.1.3 Daily records of data obtained from each fog-collecting device including the following:

13.1.3.1 Volume of salt solution collected in milliliters per hour per 80 cm^2,

13.1.3.2 Concentration or specific gravity at 35 C (95 F) of solution collected, and

13.1.3.3 pH of collected solution.

13.4 Type of specimen and its dimensions, or number or description of part,

13.5 Method of cleaning specimens before and after testing,

13.6 Method of supporting or suspending article in the salt spray chamber,

13.7 Description of protection used as required in 4.5,

13.8 Exposure period,

13.9 Interruptions in test, cause and length of time, and

13.10 Results of all inspections.

NOTE 13—If any of the atomized salt solution which has not contacted the test specimens is returned to the reservoir, it is advisable to record the concentration or specific gravity of this solution also.

APPENDIXES

A1. CONSTRUCTION OF APPARATUS

A1.1 Cabinets

A1.1.1 Standard salt-spray cabinets are available from several suppliers, but certain pertinent accessories are required before they will function according to this method and provide consistent control for duplication of results.

A1.1.2 The salt spray cabinet consists of the basic chamber, an air-saturator tower, a salt solution reservoir, atomizing nozzles, specimen supports, provisions for heating the chamber, and suitable controls for maintaining the desired tempera-

ture.

A1.1.3 Accessories such as a suitable adjustable baffle or central fog tower, automatic level control for the salt reservoir, and automatic level control for the air-saturator tower are pertinent parts of the apparatus.

A1.1.4 The cabinet should be of sufficient size to test adequately the desired number of parts without overcrowding. Small cabinets have been found difficult to control and those of less than 0.43-m^3 (15-ft^3) capacity should be avoided.

B 117

A1.1.5 The chamber may be made of inert materials such as plastic, glass, or stone, but most preferably is constructed of metal and lined with impervious plastics, rubber, or epoxy-type materials or equivalent.

A1.2 Temperature Control

A1.2.1 The maintenance of temperature within the salt chamber can be accomplished by several methods. It is generally desirable to control the temperature of the surroundings of the salt spray chamber and to maintain it as stable as possible. This may be accomplished by placing the apparatus in a constant-temperature room, but may also be achieved by surrounding the basic chamber of a jacket containing water or air at a controlled temperature.

A1.2.2 The use of immersion heaters in an internal salt-solution reservoir or of heaters within the chamber is detrimental where heat losses are appreciable, because of solution evaporation and radiant heat on the specimens.

A1.2.3 All piping which contacts the salt solution or spray should be of inert materials such as plastic. Vent piping should be of sufficient size so that a minimum of back pressure exists and should be installed so that no solution is trapped. The exposed end of the vent pipe should be shielded from extreme air currents that may cause fluctuation of pressure or vacuum in the cabinet.

A1.3 Spray Nozzles

A1.3.1 Satisfactory nozzles may be made of hard rubber, plastic, or other inert materials. The most commonly used type is made of plastic. Nozzles calibrated for air consumption and solution atomized are available. The operating characteristics of a typical nozzle are given in Table A1.

A1.3.2 It can readily be seen that air consumption is relatively stable at the pressures normally used, but a marked reduction in solution sprayed occurs if the level of the solution is allowed to drop appreciably during the test. Thus, the level of the solution in the salt reservoir must be maintained automatically to ensure uniform fog delivery during the test.[7]

A1.3.3 If the nozzle selected does not atomize the salt solution into uniform droplets, it will be necessary to direct the spray at a baffle or wall to pick up the larger drops and prevent them from impinging on the test specimens. Pending a complete understanding of air-pressure effects, etc., it is important that the nozzle selected shall produce the desired condition when operated at the air pressure

selected. Nozzles are not necessarily located at one end, but may be placed in the center and can also be directed vertically up through a suitable tower.

A1.4 Air for Atomization

A1.4.1 The air used for atomization must be free of grease, oil, and dirt before use by passing through well-maintained filters. Room air may be compressed, heated, humidified, and washed in a water-sealed rotary pump, if the temperature of the water is suitably controlled. Otherwise cleaned air may be introduced into the bottom of a tower filled with water, through a porous stone or multiple nozzles. The level of the water must be maintained automatically to ensure adequate humidification. A chamber operated according to this method and Appendix will have a relative humidity between 95 and 98 percent. Since salt solutions from 2 to 6 percent will give the same results (though for uniformity the limits are set at 4 to 6 percent), it is preferable to saturate the air at temperatures well above the chamber temperature as insurance of a wet fog. Table A2 shows the temperatures, at different pressures, that are required to offset the cooling effect of expansion to atmospheric pressure.

A1.4.2 Experience has shown that most uniform spray chamber atmospheres are obtained by increasing the atomizing air temperature sufficiently to offset heat losses, except those that can be replaced otherwise at very low-temperature gradients.

A1.5 Types of Construction

A1.5.1 A modern laboratory cabinet is shown in Fig. A1. Walk-in chambers are not usually constructed with a sloping ceiling due to their size and location. Suitably located and directed spray nozzles avoid ceiling accumulation and drip. Nozzles may be located at the ceiling, or 0.91 m (3 ft) from the floor directed upward at 30 to 60 deg over a passageway. The number of nozzles depends on type and capacity and is related to the area of the test space. A 11 to 19-dm³ (3 to 5-gal) reservoir is required within the chamber, with the level controlled. The major features of a walk-in type cabinet, which differs significantly from the laboratory type, are illustrated in Fig. A2. Construction of a plastic nozzle, such as is furnished by several suppliers, is shown in Fig. A3.

---

[7] A suitable device for maintaining the level of liquid in, either the saturator tower, or reservoir of test solution may be designed by a local engineering group, or may be purchased from manufacturers of test cabinets as an accessory.

A2. Use of the Salt Spray (Fog) Test in Research

A2.1 The detailed requirements of this method are primarily for quality acceptance and should not be construed as the optimum conditions for research studies. The test has been used to a considerable extent for the purpose of comparing different materials or finishes with an acceptable standard. The recent elimination of many cabinet variables and the improvement in controls have made the three

ASTM Salt Spray Tests: Method B 117, Method B 287, Acetic Acid-Salt Spray (Fog) Testing,[8] and Method B 368, for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS),[8] into useful tools for many industrial and military production

---

[8] *Annual Book of ASTM Standards*, Part 7.

B 117

and qualification programs.

A2.2 The test has been used to a considerable extent for the purpose of comparing different materials or finishes. It should be noted that there is seldom a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Informed personnel are aware of the erratic composition of basic alloys, the possibility of wide variations in quality and thickness of plated items produced on the same racks at the same time, and the consequent need for a mathematical determination of the number of specimens required to constitute an adequate sample for test purposes. In this connection it is well to point out that Method B 117 is not applicable to the study or testing of decorative chromium plate (nickel-chromium or copper-nickel-chromium) on steel or on zinc-base die castings or of cadmium plate on steel. For this purpose Methods B 287 and B 368 are available, which are also considered by some to be superior for comparison of chemically-treated aluminum

(chromated, phosphated, or anodized), although final conclusions regarding the validity of test results related to service experience have not been reached. Method B 117 is considered to be most useful in estimating the relative behavior of closely related materials in marine atmospheres, since it simulates the basic conditions with some acceleration due to either wetness or temperature or both.

A2.3 When a test is used for research, it may prove advantageous to operate with a different solution composition or concentration or at a different temperature. In all cases, however, it is desirable to control the temperature and humidity in the manner specified, and to make certain that the composition of the settled fog and that of the solution in the reservoir are substantially the same. Where differences develop, it is necessary to control conditions so that the characteristics of the settled fog meet the specified requirements for the atmosphere.

A2.4 Material specifications should always be written in terms of the standard requirements of the appropriate salt-spray method, thereby making it possible to test a variety of materials from different sources in the same equipment.

TABLE A1   Operating Characteristics of Typical Spray Nozzle

Siphon Height, in.	Air Flow, liters/min				Solution Consumption, ml/h			
	Air Pressure, psi				Air Pressure, psi			
	5	10	15	20	5	10	15	20
4	19	26.5	31.5	36	2100	3840	4584	5256
8	19	26.5	31.5	36	636	2760	3720	4320
12	19	26.5	31.5	36	0	1380	3000	3710
16	19	26.6	31.5	36	0	780	2124	2904

Siphon Height cm	Air Flow, dm³/min				Solution Consumption, cm³/h			
	Air Pressure, kN/m²				Air Pressure, kN/m²			
	34	69	103	138	34	69	103	138
10	19	26.5	31.5	36	2100	3840	4584	5256
20	19	26.5	31.5	36	636	2760	3720	4320
30	19	26.5	31.5	36	0	1380	3000	3710
40	19	26.6	31.5	36	0	780	2124	2904

TABLE A2   Temperature and Pressure Requirements for Operation of Test at 95 F

	Air Pressure, psi			
	12	14	16	18
Temperature, deg F	114	117	119	121
	Air Pressure, kN/m²			
	83	96	110	124
Temperature, deg C	46	47	48	49

B 117
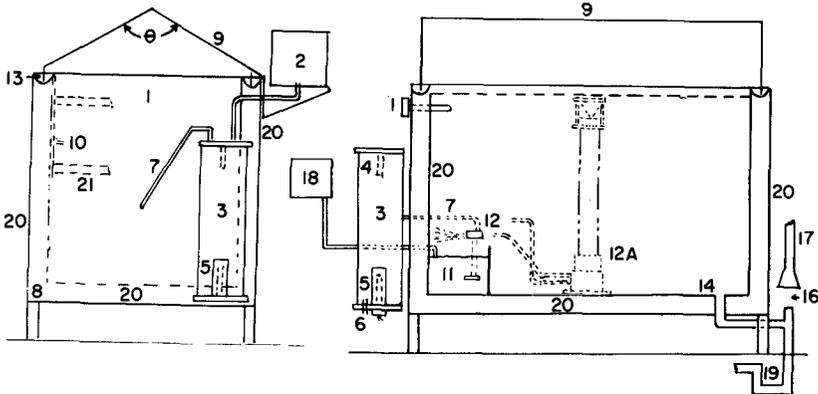
θ — Angle of lid, 90 to 125 deg
1 — Thermometer and thermostat for controlling heater (Item No. 8) in base
2 — Automatic water levelling device
3 — Humidifying tower
4 — Automatic temperature regulator for controlling heater (Item No. 5)
5 — Immersion heater, non-rusting
6 — Air inlet, multiple openings
7 — Air tube to spray nozzle
8 — Strip heater in base
9 — Hinged top, hydraulically operated, or counterbalanced
10 — Brackets for rods supporting specimens, or test table
11 — Internal reservoir
12 — Spray nozzle above reservoir, suitably designed, located, and baffled
12A — Spray nozzle housed in dispersion tower located preferably in center of cabinet
13 — Water Seal
14 — Combination drain and exhaust. Exhaust at opposite side of test space from spray nozzle (Item 12), but preferably in combination with drain, waste trap, and forced draft waste pipe (Items 16, 17, and 19).
16 — Complete separation between forced draft waste pipe (Item 17) and combination drain and exhuast (Items 14 and 19) to avoid undesirable suction or back pressure.
17 — Forced draft waste pipe.
18 — Automatic levelling device for reservoir
19 — Waste trap
20 — Air space or water jacket
21 — Test table or rack, well below roof area

FIG. A1   Typical Salt Spray Cabinet.
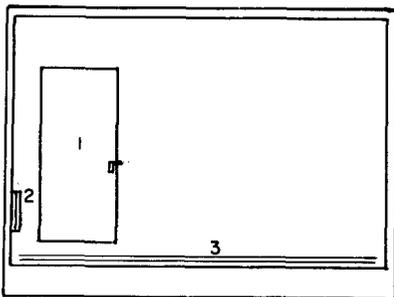
NOTE—The controls are the same, in general as for the laboratory cabinet (Fig. A1), but are sized to care for the larger cube. The chamber has the following features:
(1) Heavy insulation,
(2) Refrigeration door with drip rail, or pressure door with drip rail, inward-sloping sill,
(3) Low-temperature auxiliary heater, and
(4) Duck boards on floor, with floor sloped to combination drain and air exhaust.

FIG. A2   Walk-in Chamber, 1.5 by 2.4 mm (5 by 8 ft) and Upward in Over-all Size.
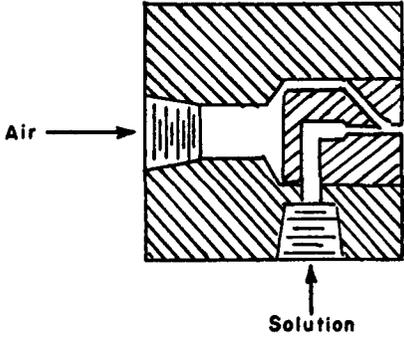
B 117

Air →

Solution

FIG. A3 Typical Spray Nozzle.

By publication of this standard no position is taken with respect to the validity of any patent rights in connection therewith, and the American Society for Testing and Materials does not undertake to insure anyone utilizing the standard against liability for infringement of any Letters Patent nor assume any such liability.

ASTM Designation: G 1 – 72

Standard Recommended Practice for

PREPARING, CLEANING, AND EVALUATING CORROSION TEST SPECIMENS[1]

This Standard is issued under the fixed designation G 1; 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.

1. Scope

1.1 This recommended practice gives suggested procedures for preparing bare, solid metal specimens for laboratory corrosion tests, for removing corrosion products after the test has been completed, and for evaluating the corrosion damage that has occurred. Emphasis is placed on procedures related to the evaluation of corrosion by mass-loss and pitting measurements.

2. Applicable Documents

2.1 *ASTM Standards:*

A 262, Recommended Practice for Detecting Susceptibility to Intergranular Attack in Stainless Steels.[2]

A 279, Total Immersion Corrosion Test of Stainless Steels.[2]

D 1384, Corrosion Test for Engine Antifreezes in Glassware.[3]

3. Methods for Preparing Specimens for Test

3.1 *Surface Condition:*

3.1.1 For laboratory corrosion tests that simulate exposure to service environments, a commercial surface, closely resembling the one that would be used in service, will yield the most significant results.

3.1.2 For more searching tests of either the metal or the environment, standard surface finishes may be preferred. A suitable procedure might be:

3.1.2.1 Degrease in an organic solvent or hot alkaline cleaner.

NOTE 1—Hot alkalies and chlorinated solvents may attack some metals (for example, aluminum).

NOTE 2—Ultrasonic cleaning may be beneficial in both pre-test and post-test cleaning procedures.

3.1.2.2 Pickle in an appropriate solution (in some cases the chemical cleaners described in Section 5 will suffice) if oxides or tarnish are present.

3.1.2.3 Abrade with a slurry of an appropriate abrasive or with an abrasive paper (see Methods A 262 and A 279 and Recommended Practice D 1384). The edges as well as the faces of the specimens should be abraded to remove burrs.

3.1.2.4 Rinse thoroughly and dry.

3.2 *Metallurgical Condition*—When specimen preparation changes the metallurgical condition of the metal, other methods should be chosen or the metallurgical condition must be corrected by subsequent treatment. For example, shearing a specimen to size will cold work and may possibly fracture the edges. Edges should be machined or the specimen annealed.

3.3 The clean, dry specimens should be measured and weighed. Dimensions determined to the third significant figure and mass determined to the fifth significant figure are suggested.

4. Method for Electrolytic Cleaning After Testing

4.1 Electrolytic cleaning is a satisfactory method for many common metals.

4.1.1 The following method is typical; after

---

[1] This recommended practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved May 30, 1972. Published July 1972. Originally published as G 1 – 67. Last previous edition G 1 – 67.

[2] *Annual Book of ASTM Standards*, Part 3.

[3] *Annual Book of ASTM Standards*, Part 22.

scrubbing to remove loosely attached corrosion products, electrolyze the specimen as follows:

Sulfuric acid (H$_2$SO$_4$, sp gr 1.84)	28 ml
Organic inhibitor	2 ml (see Note 3)
Water to make	1 liter
Temperature	75 C (167 F)
Time	3 min
Anode	carbon or lead (see Note 4)
Cathode	test specimen
Current density	20 A/dm^2

NOTE 3—Instead of using 0.2 volume percent of any proprietary inhibitor, about 0.5 g/liter of such inhibitors as diorthotolyl thiourea, quinoline ethiodide, or betanaphthol quinoline may be used.

NOTE 4—If lead anodes are used, lead may deposit on the specimen and cause an error in the mass loss. If the specimen is resistant to nitric acid, the lead may be removed by a flash dip in 1+1 nitric acid. Except for this possible source of error, lead is preferred as an anode as it gives more efficient corrosion-product removal.

4.2 It should be noted that this electrolytic treatment may result in the redeposition of metal, such as copper, from reducible corrosion products and, thus, lower the apparent mass loss.

5. Methods for Chemical Cleaning After Testing

NOTE 5: **Caution**—These methods may be hazardous to personnel. They should not be carried out by the uninitiated or without professional supervision.

5.1 *Copper and Nickel Alloys*—Dip in:

Hydrochloric acid (HCl, sp gr 1.19) or	500 ml
Sulfuric acid (H$_2$SO$_4$, sp gr 1.84)	100 ml
Water to make	1 liter
Temperature	room
Time	1 to 3 min

5.2 *Aluminum Alloys*—Dip in:

Chromic acid (CrO$_3$)	20 g
Phosphoric acid (H$_3$PO$_4$, sp gr 1.69)	50 ml
Water to make	1 liter
Temperature	80 C (176 F)
Time	5 to 10 min

5.2.1 If a film remains, dip in:

Nitric acid (HNO$_3$, sp gr 1.42)	. . .
Time	1 min

5.2.2 Repeat CrO$_3$ dip.
5.2.2.1 Nitric acid alone may be used if there are no deposits.

5.3 *Tin Alloys*—Dip in:

Trisodium phosphate (Na$_3$PO$_4$)	150 g
Water to make	1 liter
Temperature	boiling
Time	10 min

5.4 *Lead Alloys*—Suitable methods include:

5.4.1 Preferably, use the electrolytic cleaning procedure of Section 4.

5.4.2 Dip in:

Acetic acid (99.5 percent)	10 ml
Water to make	1 liter
Temperature	boiling
Time	5 min

5.4.3 Alternatively dip in:

Ammonium acetate	50 g
Water to make	1 liter
Temperature	hot
Time	5 min

5.4.3.1 This removes lead oxide (PbO) and lead sulfate (PbSO$_4$).

5.5 *Zinc*—The following methods are suitable:

5.5.1 Dip in:

Ammonium hydroxide (NH$_4$OH, sp gr 0.90)	150 ml
Water to make	1 liter
Temperature	room
Time	several minutes

5.5.2 Then dip in:

Chromic acid (CrO$_3$)	50 g
Silver nitrate (AgNO$_3$)	10 g
Water to make	1 liter
Temperature	boiling
Time	15 to 20 s

NOTE 6—In making up the chromic acid solution, it is advisable to dissolve the silver nitrate separately and add it to the boiling chromic acid to prevent excessive crystallization of the silver chromate. The chromic acid must be free from sulfate to avoid attack on the zinc.

5.5.2 Dip in:

Hydriodic acid (HI, sp gr 1.5)	85 ml
Water to make	1 liter
Temperature	room
Time	15 s

5.5.2.1 This procedure dissolves a little zinc and corrections must be made as noted in 6.1.

5.6 *Magnesium Alloys*—Dip in:

Chromic acid (CrO$_3$)	150 g
Silver chromate (Ag$_2$CrO$_4$)	10 g
Water to make	1 liter
Temperature	boiling
Time	1 min

5.7 *Iron and Steel*—Suitable methods are:

5.7.1 The hot sodium hydride method is excellent for cleaning iron and steel both from the point of view of ease of removal of corrosion products and minimum attack on the

metal.[4] Because of the hazard involved and the somewhat more sophisticated equipment required, other methods may be preferred. An alternative choice is electrolytic cleaning (see Section 4).

5.7.2 Dip in Clarke's solution:

Hydrochloric acid (HCl, sp gr 1.19)	1 liter
Antimony trioxide (Sb_2O_3)	20 g
Stannous chloride ($SnCl_2$)	50 g
Temperature	room
Time	up to 25 min

5.7.2.1 Solution should be vigorously stirred or the specimen should be rubbed with a nonabrasive implement of wood or rubber.

5.7.3 Dip in:

Sulfuric acid (H_2SO_4, sp gr 1.84)	100 ml
Organic inhibitor	1.5 ml
Water to make	1 liter
Temperature	50 C (120 F)

5.8 *Stainless Steels:*

5.8.1 Methods in 5.7.1 are also applicable

5.8.2 Dip in:

Nitric acid (HNO_3, sp gr 1.42)	100 ml
Water to make	1 liter
Temperature	60 C (140 F)
Time	20 min

5.8.3 Alternatively dip in:

Ammonium citrate	150 g
Water to make	1 liter
Temperature	70 C (158 F)
Time	10 to 60 min

5.9 In place of chemical cleaning, use a brass scraper or brass bristle brush, or both, followed by scrubbing with a wet bristle brush and fine scouring powder. However, this method may not remove all the products from pits.

NOTE 7—Such vigorous mechanical cleaning is applicable where mass losses are large and hence errors in mass loss will produce only small errors in corrosion rates. Blank corrections will be difficult to apply.

5.10 In all the foregoing methods, specimens should be rinsed following cleaning and scrubbed lightly with a bristle brush under running water. The cleaning dip may be repeated as necessary. After the final rinse, specimens should be dried and weighed.

6. Calculation of Corrosion Rate

6.1 Whatever cleaning method is used, the possibility of removal of solid metal is present; this results in error in the determina-

tion of the corrosion rate. To check this, one or more cleaned and weighed specimens may be recleaned by the same method and reweighed. Loss due to this second weighing may be used as an approximate correction to the first one (see Appendix A2 for a more exact method).

6.2 The initial total surface area of the specimen (making allowances for the change in area due to mounting holes) and the mass lost during the test are determined. The average corrosion rate may then be obtained as follows.

$$\text{Corrosion rate} = (K \times W)/(A \times T \times D)$$

where:

K = a constant (see 6.2.1),

T = time of exposure in hours to the nearest 0.01 h,

A = area in cm^2 to the nearest 0.01 cm^2,

W = mass loss in g, to nearest 1 mg, and

D = density in g/cm^3 (see Appendix A1).

6.2.1 Many different units are used to express corrosion rates. Using the above units for T, A, W, and D the corrosion rate can be calculated in a variety of units with the following appropriate value of K:

Corrosion Rate Units Desired	Constant (K) in Corrosion Rate Equation
mils per year (mpy)	3.45×10^6
inches per year (ipy)	3.45×10^3
inches per month (ipm)	2.87×10^2
millimeters per year (mm/y)	8.76×10^4
micrometers per year ($\mu m/y$)	8.76×10^7
picometers per second (pm/s)	2.78×10^6
grams per square meter per hour ($g/m^2 \cdot h$)	$1.00 \times 10^4 \times D^a$
milligrams per square decimeter per day (mdd)	$2.40 \times 10^6 \times D^a$
micrograms per square meter per second ($\mu g/m^2 \cdot s$)	$2.78 \times 10^6 \times D^a$

[a] Density is not needed to calculate the corrosion rate in these units; the density in the constant K cancels out the density in the corrosion rate equation.

NOTE 8—If desired, these constants may also be used to convert corrosion rates from one set of units to another. To convert a corrosion rate in units X to a rate in units Y, multiply by K_Y/K_X for example:

$$15 \text{ mpy} = 15 \times (2.78 \times 10^6)/(3.45 \times 10^6) \text{ pm/s}$$

6.3 Corrosion rates calculated from mass

---

[4] *Technical Information Bulletin SP29-370* "DuPont Sodium Hydride Descaling Process Operating Instructions," available from E. I. duPont de Nemours & Co. (Inc.), Electrochemicals Dept., Wilmington, Del. 19898.

G 1

losses can be misleading when deterioration is highly localized, as in pitting or crevice corrosion. If corrosion is in the form of pitting, it may be measured with a depth gage or micrometer calipers with pointed anvils. Microscopical methods will determine pit depth by focusing from top to bottom of the pit, when it is viewed from above (using a calibrated focusing knob) or by examining a section that has been mounted and metallographically polished. The pitting factor is the ratio of the deepest metal penetration to the average metal penetration (as measured by mass loss).

6.4 Other methods of assessing corrosion damage are:

6.4.1 *Appearance*—The degradation of appearance by rusting, tarnishing, or oxidation.

6.4.2 *Mechanical Properties*—An apparent loss in tensile strength will result if the cross-sectional area of the specimen (measured before exposure to the corrosive environment) is reduced by corrosion. Loss in tensile strength will result if a metasomatic change, such as parting has taken place. Loss in tensile strength and elongation will result from localized attack, such as cracking.

6.4.3 *Electrical Properties*—Loss in apparent conductivity will result from cracking or pitting.

6.4.4 *Microscopical Examination*—Parting, exfoliation, cracking, or intergranular attack may be measured by metallographic examination of suitably prepared sections.

7. Report

7.1 The report should include the compositions and sizes of specimens, their metallurgical conditions, surface preparations, and post-corrosion cleaning methods, as well as measures of corrosion damage such as corrosion rates (calculated from mass losses), maximum depths of pitting, or losses in mechanical properties.

APPENDIXES

A1. DENSITIES FOR A VARIETY OF METALS AND ALLOYS

Aluminum Alloys	Density g/cm³
1100, 3004	2.72
1199, 5005, 5357, 6061, 6062, 6070, 6101	2.70
2024	2.77
2219, 7178	2.81
3003, 7079	2.74
5050	2.69
5052, 5454	2.68
5083, 5086, 5154, 5456	2.66
7075	2.80
Copper Alloys	
Copper	8.94
Brasses:	
Commercial bronze 220	8.80
Red brass 230	8.75
Cartridge brass 260	8.52
Muntz metal 280	8.39
Admiralty 442, 443, 444, 445	8.52
Aluminum brass 687	8.33
Bronzes:	
Aluminum bronze, 5 percent 608	8.16
Aluminum bronze, 8 percent 612	7.78
Composition M	8.45
Composition G	8.77
Phosphor bronze, 5 percent 510	8.86
Phosphor bronze, 10 percent 524	8.77
85-5-5-5	8.80
Silicon bronze 655	8.52
Copper nickels 706, 710, 715	8.94
Nickel silver 752	8.75

Ferrous Metals	Density g/cm³
Gray cast iron	7.20
Carbon steel	7.86
Silicon iron	7.00
Low alloy steels	7.85
Stainless steels:	
Types 201, 202, 302, 304, 304L, 321	7.94
Types 309, 310, 311, 316, 316L, 317, 329, 330	7.98
Type 347	8.03
Type 410	7.70
Type 430	7.72
Type 446	7.65
Type 502	7.82
Durimet 20	8.02
Carpenter Stainless No. 20 Cb-3	8.05
Lead	
Antimonial	10.80
Chemical	11.33
Nickel Alloys	
Nickel 200	8.89
Monel Alloy 400	8.84
Inconel Alloy 600	8.51
Incoloy Alloy 825	8.14
Illium G	8.31
Hastelloy B	9.24
Hastelloy C	8.93
Hastelloy G	8.27

4️⃣5️⃣1️⃣b **G 1**

Other Metals	Density g/cm³	Other Metals	Density g/cm³
Magnesium	1.74	Tin	7.30
Molybdenum	10.22	Titanium	4.54
Platinum	21.45	Zinc	7.13
Silver	10.49	Zirconium	6.53
Tantalum	16.60		

A2. METHOD FOR DETERMINING MASS LOSS WHERE CLEANING MAY ATTACK THE BASE METAL OF THE SPECIMEN

A2.1 Repeat the cleaning procedure a number of times. Weigh after each cleaning and plot the mass loss against the total time of cleaning or the number of cleanings, see Fig. A1. The ordinate at the intersection of the two lines is the mass loss caused by removal of corrosion products alone. The method is particularly applicable to electrolytic cleaning, see Section 4.
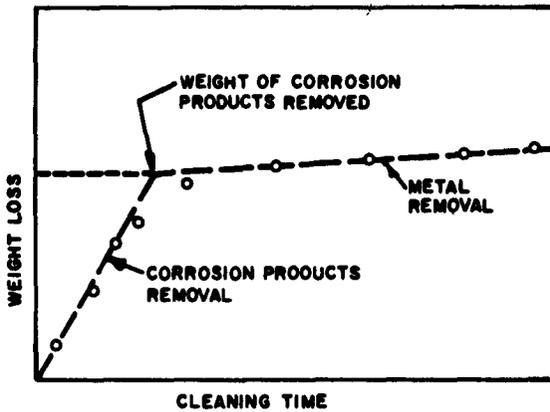

FIG. A1   Mass Loss Versus Exposure Time for Specimens During Cleaning.

ASTM Designation: G 4 – 68

Standard Recommended Practice for
CONDUCTING PLANT CORROSION TESTS[1]

This Standard is issued under the fixed designation G 4; 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.

1. Scope

1.1 This recommended practice outlines procedures for conducting corrosion tests in plant equipment under operating conditions. It is not intended for atmospheric or underground corrosion tests but may possibly apply to other tests under natural conditions where the procedure has proven satisfactory. Corrosion testing by its very nature precludes complete standardization. This recommended practice, rather than a standardized procedure, is presented as a guide so that some of the pitfalls of such testing may be avoided.

NOTE—The values stated in U.S. customary units are to be regarded as the standard.

2. Interferences

2.1 Tests described herein are probably the best means available for approximating the behavior of metals in service, short of actually constructing and operating a piece of equipment. For best results, certain variables must be considered. These include:

2.1.1 Metal specimens immersed in a specific hot liquid may not corrode at the same rate or in the same manner as in equipment where the metal acts as a heat transfer medium in heating or cooling the liquid. In certain services, the corrosion of heat-exchanger tubes may be quite different than that of the shell or heads. This restriction also applies to specimens exposed in gas streams from which water or other corrodents condense on cool surfaces. Such factors must be considered in both design and interpretation of plant tests.

2.1.2 Effects caused by high velocity, abrasive ingredients, etc. (which may be emphasized in pipe elbows, pumps, etc.), may not be easily reproduced in coupon tests.

2.1.3 The behavior of certain metals and alloys may be profoundly influenced by the presence of dissolved oxygen. It is essential that the test coupons be placed in locations representative of the degree of aeration normally encountered in process.

2.1.4 Corrosion products may have undesirable effects on the product. This possibility is frequently recognized in advance. The extent of possible contamination can be estimated from the loss in weight of the specimen, with proper application of the expected relationships among (1) the area of corroding surface, (2) the mass of the product handled, and (3) the duration of contact of a unit of mass of the product with the corroding surface.

2.1.5 Corrosion products from the plant equipment used in the test may influence the corrosion of one or more of the test metals. For example, when aluminum specimens are exposed in copper equipment, corroding copper will exert an adverse effect on the corrosion of the aluminum. Contrariwise, stainless steel specimens can have their corrosion resistance enhanced by the presence of the oxidizing cupric ions.

2.1.6 The accumulation of corrosion products sometimes can have harmful effects. For example, copper, corroding in intermediate

---

[1] This recommended practice is under the jurisdiction of the ASTM Committee G-1 on Corrosion of Metals. This standard is the direct responsibility of Subcommittee G01.12 on In-plant Corrosion Tests.
Current edition effective Sept. 13, 1968. Originally issued as A 224 in 1939. Replaces A 224 – 46.
Revised with the aid of Unit Committee T-5A. Corrosion in Chemical Processes, National Association of Corrosion Engineers and ASTM Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys which formerly held jurisdiction over ASTM Recommended Practice A 224.

G 4

strengths of sulfuric acid will have its corrosion rate increased as the cupric ion accumulates. Phenomena such as this will not be recognized from coupon tests in the plant, and must be anticipated from general knowledge and experience, or studied under controlled (laboratory) conditions.

2.1.7 Coupon corrosion testing is predominantly designed to investigate general corrosion. There are a number of other special types of corrosion phenomena of which one must be aware in the design and interpretation of coupon tests.

2.1.7.1 Galvanic corrosion may be investigated by special devices that couple one coupon to another in electrical contact, as for example, by substituting a spacer made from the more noble metal of the couple in place of an insulating spacer. The behavior of the specimens in this galvanic couple are compared with that of insulated specimens exposed on the same holder and the galvanic effects noted. It should be observed, however, that galvanic corrosion can be greatly affected by the area ratios of the respective metals. The coupling of corrosion coupons then yields only qualitative results, as a particular coupon reflects only the relationship between those two metals at the particular area ratio involved.

2.1.7.2 Crevice or concentration cell corrosion may occur where the metal surface is partially blocked from the corroding liquid, as under a spacer. At times it is desirable to know whether a given metal is subject to crevice corrosion in a given environment, whereas in other cases the spacers can be designed to minimize this effect (see below). An accumulation of debris or corrosion products between the coupons can produce misleading results in either accelerating corrosion or protecting the coupons from corrosion.

2.1.7.3 Selective corrosion at the grain boundaries (for example, intergranular corrosion of sensitized austenitic stainless steels) will not be readily observable in weight loss measurements and often requires microscopical examination of the coupons after exposure.

2.1.7.4 Metasomatic corrosion is a condition in which one constituent is selectively removed from an alloy, as in the dezincification of brass or the graphitization of cast iron. Close attention and a more sophisticated eval-

uation than a simple weight loss measurement is required to detect this phenomenon.

2.1.7.5 Certain metals and alloys are subject to a highly localized type of attack called pitting corrosion. This cannot be evaluated by weight loss. The reporting of nonuniform corrosion is discussed below. It should be appreciated that pitting is a statistical phenomenon and the incidence of pitting can be directly related to the area of metal exposed. For example, a small coupon is not as prone to exhibit pitting as a large one, and it is possible to miss the phenomenon altogether in the corrosion testing of certain alloys, such as the AISI Type 300 series stainless steels in chloride-contaminated environments.

2.1.7.6 All metals and alloys are subject to stress-corrosion cracking under some circumstances. This cracking attack occurs under conditions of tensile stress and it may or may not be visible to the naked eye or upon casual inspection. A metallographic examination will confirm this mechanism of attack. It is imperative to note that this usually occurs with no significant loss in weight of the test coupon, although certain refractory metals are an exception to these observations.

3. Apparatus for Mounting Specimens

3.1 Although it is possible to expose specimens to corrosive environments in operating equipment by attaching them to pieces of string, wire, glass, etc., this is usually inadequate. In general, the method of support should be such as to satisfy the following requirements:

3.1.1 Prevent loss of specimens from causes other than corrosion,

3.1.2 Eliminate the possibility of galvanic effects resulting from metal-to-metal contact between specimens or between the vessel and the specimen exposed therein,

3.1.3 Hold specimens firmly in place, and

3.1.4 Provide for protection of specimens against mechanical damage.

3.2 While it is possible to clamp a specimen near its edge, it is not easy to design a clamping arrangement that will provide the necessary electrical insulation without either completely or partially shielding a fairly large area of the specimen from free contact with the corroding solution. A better arrangement is to drill a hole in the specimen and allow the

G 4

supporting device to pass through the hole. A satisfactory location for the supporting hole is at the center of the specimen so that it cannot be lost from the holder unless either the specimen or the holder is destroyed by corrosion.

3.3 To ensure effective electrical insulation, it is necessary to provide some insulating material between the specimen and the metallic support. This can be accomplished most conveniently by using a tube of some insulating material that will fit over the metal rod. Polyethylene, poly(vinyl chloride), saran, bakelite, ceramics, or fluorinated plastics are satisfactory. The hole in the specimen should be made large enough so that the specimen will slide over the insulating tube. More than one specimen may be exposed on the same holder by insulating or separating the specimens from each other by means of short tubes of insulating material that can be slipped over the insulating tube on the supporting rod. The short tube spacers should be large enough in outside diameter to provide firm support for the specimens without covering more than a small percentage of the total surface. The spacing of the specimens in this arrangement is determined by the length of the insulating spacer. Any insulating material that will withstand the action of the corroding solution may be used. A preferable means of support is the use of individual insulating spacers machined to the desired shape. Figure 1 delineates the dimensions of two types of insulating spacers designed specifically for field corrosion testing. The first type of specimen is designed to minimize concentration cell effects on the face of the specimen. The second type has a sharp shoulder which will tend to lead to crevice corrosion adjacent to the hole in the coupon. The choice between these two types of spacers will lie with the corrosion engineer, based on the type of information he requires.

3.4 Although there are many ways of supporting corrosion coupons in plant apparatus, the following basic types of field corrosion racks are described in some detail as a guide:

3.4.1 A spool rack may be assembled by threading the ends of a supporting rod, and providing end disks or bearing plates against which nuts on the ends of the rod may be turned so as to press the specimens and spacers close together. Two nuts should be used at each end of the rod, the second nut acting as a lock nut (Fig. 2). The end plates used as bearing plates should be made larger than the specimens so that they will act as bumpers to keep the specimens from touching any flat surfaces with which the holder may come in contact. The end plates need not necessarily be made of insulating material. Additional metal rods may be used to connect the end plates at points where they will clear the specimens and can be tightened so as to provide additional reinforcement and rigidity to the entire assembly. The rods also protect the specimens from mechanical injury. Support and bracing rods from $\frac{1}{4}$ in. (6.35 mm) to $\frac{3}{8}$ in. (9.53 mm) have been found suitable. All metal rods used in the assembly should be made of material which is sufficiently corrosion resistant to ensure the assembly remaining intact for the duration of the test. Stainless steel, Monel Alloy 400, or other suitable nonferrous metals are commonly employed. Individual spacers (Fig. 1) may be used, or a separate insulating tube with insulating washers may be employed. All three modes of mounting coupons are illustrated in Fig. 2. Because these spool type racks have the disadvantage of requiring that the equipment not only be out of service but also be gas-free or otherwise made suitable for entry, other types of field corrosion racks (which are preferably in petrochemical process equipment) have been devised.

3.4.2 The insert rack is fabricated by welding suitable rod or strip to a welding disk that can be held within the bolt circle and flange face of a flange in an unused nozzle. A 1.5 to 2-in. (38.1 to 50.8-mm) nozzle is usually convenient for this installation (Fig. 3). Such racks employ a stout member immediately adjacent to the welding disk, for example, 0.375-in. (9.53-mm) Type 316 stainless steel rod with an 0.25-in. (6.35-mm) rod extension that carries the specimens and spacers. Such racks should also be assembled with a lock nut arrangement. Occasionally, racks of this type may be required for nozzles which are not "blind" but are employed for the fastening of piping to the equipment in question. In such cases the disk can be perforated, and the specimens mounted sidewise on the rack, if required, as indicated in Fig. 3.

3.4.3 For larger diameter pipes or nozzles, a "dutchman" type rack may be employed.

Such a rack will consist of a suitable spool piece with the specimens mounted crosswise on a bar as shown in Fig. 4. Both insert and dutchman corrosion racks require that the equipment be out of service, but they may be installed and removed without extraordinary precautions in gas freeing the equipment.

3.4.4 The "slip-in" corrosion rack is ideally suited for effecting the entry and removal of corrosion coupons from operating equipment that is in active service. The slip-in rack requires an unused nozzle of suitable size (usually nominal 1 $^{1}/_{2}$-in. pipe size or greater) and a gate valve. The corrosion rack is then assembled from a short length of pipe or tubing with a suitable flange and a packing-gland arrangement made from the bonnet of a $^{1}/_{2}$-in. (12.7 mm) stainless steel or suitable alloy valve. An alloy rod of appropriate length is used as the specimen mount as shown in Fig. 5. In the "Out" position the specimens are mounted on the rod and drawn into the recessed area provided by the pipe or tube section. When this is bolted to the gate valve and the valve is opened, the assembly of corrosion coupons may be slid into the operating equipment for exposure. When it is desired to remove the specimens, they are withdrawn into the recessed area, the gate valve is closed, and the entire assembly is then physically removed from the operating equipment.

3.4.5 The design of corrosion racks for plant tests is limited only by the imagination and ingenuity of the corrosion engineer. In specific circumstances, for example, it is possible to convert thermowells into corrosion racks by welding a short extension rod on them. Similarly, racks may be designed to clamp onto agitators, thermowells, or other parts of operating equipment.

3.4.6 When the choice of materials of construction has been narrowed to one or two, it may be desirable to investigate heat-transfer effects with a simple bayonet heater of the design shown in Fig. 6. Either a heating or cooling medium is circulated through the tube side of this testing apparatus, and the effect of the hot or cold wall upon corrosion is observed by visual observation, pit depth measurements, micrometer measurements, etc.

4. Test Specimens

4.1 The size and shape of test specimens is influenced by several factors and cannot be rigidly defined. In general, the ratio of surface area to mass should be large so as to favor maximum amount of corrosion loss. This can be accomplished by the use of thin sections. Sufficient thickness, however, should be employed to minimize the possibility of perforation of the specimen during the test exposure. The size of the specimen should be as large as can be conveniently handled, the limitation being imposed primarily by the maximum weight (200 g) that can be handled by an analytical balance and, secondarily, by the problem of effecting entry into operating equipment.

4.2 A convenient size for standard corrosion coupons is 1.5 in. (38.1 mm) in diameter and 0.125 in. (3.18 mm) in thickness with a 0.438-in. (11.1-mm) hole in the center of the round coupon. This size was arrived at as being the maximum size that could easily effect entry through a nominal 1 $^{1}/_{2}$-in. nozzle. However, it is also convenient for larger size nozzle entries as well as for laboratory corrosion testing. A convenient standard coupon for spool-type racks is the 2 by 2 by 0.125 in. (50.8 by 50.8 by 3.18 mm) square, if only a few coupons need be made. A round coupon of 2.11 in. (53.5 mm) by 0.125 in. (3.18 mm), or 2.18 in. (55.5 mm) by 0.062 in. (1.59 mm), is sometimes employed. These last measure 0.500 dm^2 in area.

4.3 Other sizes, shapes, and thicknesses of specimens can be used for special purposes or to comply with the design of a special type of corrosion rack. When the choice of material has been reduced to a few in number in preliminary tests, special coupons should be employed to consider the effect of such factors of equipment construction and assembly as heat treatment, welding, soldering, and cold working or other mechanical stressing.

5. Preparation of Test Specimens

5.1 The edges of test specimens should be so prepared as to eliminate all cold-worked metal except that introduced by stamping for identification. Shearing will, in some cases, cause considerable attack; and, therefore, specimens having sheared edges should not be used. The edges should be finished by machining or polishing. The slight amount of cold working resulting from machining will not

G 4

introduce any serious error.

5.2 Usually no specific finish is of interest except in the sense that uniformity is desirable when comparing data from different tests. Furthermore, it may be necessary to remove dirt or heat-treating scale from the metal surface. It has been found convenient to standardize on a 120-grit surface in most cases. A surface roughness greater than 120 grit should not be used.

5.3 After the test specimens are cut to size, they should be freed from water breaks by suitable cleaning. In isolated cases, it is desirable to expose specimens with a special surface treatment. For example, in some applications, stainless steels may be prepassivated by a 30-min immersion in 10 to 20 percent nitric acid at 60 C. In most cases, however, special pretreatments are unnecessary and undesirable.

5.4 The weight of each specimen should be determined to the nearest 0.1 mg on an analytical balance.

6. Number of Test Specimens

6.1 For statistical validity, it is desirable to expose replicate specimens. When corrosion tests are performed in the laboratory under standard conditions, duplicate specimens will suffice for an accuracy of ± 10 percent. However, this reflects the reproducibility of certain standardized tests, and does not necessarily hold true for plant corrosion testing. It is possible, although not probable, to have rather widely different results on replicate specimens exposed on the same rack in a given test.

6.2 In multiple exposures, it is *probable* that there will be considerable variation in the results from one exposure to the other because of changes in operating conditions. Under such circumstances an evaluation should be based on the statistics of a limited number of observations.[2]

6.3 For a limited number of observations (for example, ten or less) the range w between maximum and minimum values provides more definitive values than does the standard deviation. In practice, it is usually desired to establish a "confidence interval", that is, the distance on either side of the average in which one would expect to find the true value 95 percent of the time. This is established by multiplying the range w by a factor t. The 95

percent confidence interval factors t are as follows:

n	t
2	6.4
3	1.3
4	0.72
5	0.51

Confidence interval $= \bar{x} \pm tw$

where:

x = average of n observations,
w = range, and
t = factor.

For example, if four successive tests give corrosion rates of 15, 20, 25, and 20 mils/year, the average $(\bar{x})$ is 20, and the range is $25 - 15 = 10$. Then the 95 percent confidence interval is 20 ± 10 (0.72) or 20 ± 7.2 mils/year (1 mil/year $= 0.0254$ mm/year). A special consideration can also be applied to evaluate whether a doubtful observation should be disregarded. This is discussed in detail in the referenced article.

7. Identification of Test Specimens

7.1 For purposes of identification, a record should always be made of the relative positions of the test specimens on the holder. If identification marks are obliterated by corrosion, careful handling of the specimens is required to maintain identity.

7.2 Although it may be necessary in special instances to notch the edge of the specimens for identification, it is preferable that they be stamped with a code number. The stamped number has an additional advantage in that, should a specimen show a preferential attack at the stamped area, a warning is given that the material is susceptible to corrosion when cold worked. It is also possible in some instances to detect stress-corrosion cracking emanating from the stamped areas. With such indications, the investigator is forewarned and can reject the material from further consideration or may study further the effects of cold working or stress upon the corrosion behavior. Note, however, that although the presence of such localized attack is a *positive* indication, absence of attack is not a guarantee of immunity from attack in operating equipment.

---

[2] Dean, R. B., and Dixon, W. J., "Simplified Statistics for Small Numbers of Observations," *Analytical Chemistry*, ANCHA, Vol 23, No. 4, April, 1951, pp. 636–638.

8. Installation of Specimen Holder

8.1 The location of the test specimens in the operating equipment will be governed by the information that is desired. This may require tests at more than one location in the same piece of equipment, such as below the level of the test liquid, at the level of the liquid, or in the vapor phase.

8.2 It is desirable to have the specimen holder securely fixed in place. The preferred position of the holder is with the long axis horizontal so as to prevent drippage of corrosion products from one specimen to another. Preferably, the holder should be so placed that any flow of liquid will be against the edges of the specimens. The same condition of agitation of the liquid should then be encountered by all specimens.

9. Duration of Exposure

9.1 The duration of exposure may be based on known rates of deterioration of the materials in use. More often, it is governed by the convenience with which plant operations may be interrupted to introduce and remove test specimens. In many tests, some materials may show little or no attack while other materials may be completely destroyed. In general, the duration of the test should be as long as possible commensurate with the resistance of the materials under test. In special cases, the duration may be established in regard to some specific phase of the operation, as for example to study corrosion in one step of a batch process. Possible changes in the rate of corrosion may be studied either by successive exposures or by the installation of several sets of specimens at the same time, which can be removed one set at a time at different intervals. The minimum duration is commonly defined by the equation:

Minimum hours of test = 2000/mils/year.

10. Removal of Specimens from Test

10.1 The condition and appearance of the holder and specimens after removal from equipment should be noted and recorded. Specimens should then be carefully washed, either in water or in a suitable solvent, to remove all soluble materials from the surface of the specimens. In removing the specimens from the holder, care should be taken to keep

them in proper sequence relative to each other so that any specimen may be identified from the original record of its position on the holder. This is important if corrosion has been so severe that identification marks have been removed.

10.2 A record should be made of the appearance and adhesion of any coatings or films on the surface of the specimens after washing. It may be desirable to photograph the specimens. Color photographs may be of value. Samples of any products or films resulting from corrosion may be preserved for future study.

11. Cleaning[3] and Weighing Test Specimens

1.1 The surfaces of the test specimens should be thoroughly cleaned of all corrosion products. Removal of corrosion products from the specimens may not be a simple procedure. No hard and fast rules can be laid down since the cleaning procedure adopted will depend on the base material as well as the nature of the corrosion products. It will be necessary for the investigator to study the problem and decide upon the most suitable procedure.

11.2 It is essential that the base metal be unattacked either by the cleaning reagent or by compounds formed by reaction between the cleaning reagent and corrosion products or other deposits on the specimen. A preliminary solvent cleaning may be necessary to remove organic deposits. The simplest cleaning procedure is to scrub the specimens with a flat fiber brush using a mild abrasive soap. Care must be taken that no base metal is removed by abrasion. Acid or alkaline solutions of suitable nature and strength may be employed, contingent upon their being noncorrosive to the base metal. For example, a copper flash on stainless steel or Hastelloy Alloy C can be safely removed in concentrated nitric acid. On the other hand, a copper flash on Monel Alloy 400 or Hastelloy Alloy B should be removed with a mixture of peroxide and ammonium hydroxide which will not significantly attack the base metal. A solution of 5 percent stannous chloride and 2 percent antimonious chloride in concentrated hydrochloric acid may be used to remove rust deposits from steel. This

---

[3] ASTM Recommended Practice G 1, for Preparing, Cleaning, and Evaluating Corrosion Test Specimens, which appears in *Annual Book of ASTM Standards*, Part 31.

G 4

solution has an added advantage in that the final disappearance of the last vestige of ferric oxide is readily apparent to the naked eye. For iron, steel, and alloy steels, a hot caustic solution (20 percent) with 200 g of zinc dust added per liter is effective for loosening deposits which can then be scrubbed off.

11.3 A cleaning method used by many investigators for a variety of materials consists in making the specimen the cathode in a hot dilute sulfuric acid solution under the following conditions:

11.3.1 *Solution*—5 weight percent of sulfuric acid plus 2 ml of an appropriate commercial inhibitor per liter of solution.

11.3.2 *Anode Carbon*—cathode-test specimen.

11.3.3 *Cathode Current Density*—20 A/dm^2.

11.3.4 *Temperature*—165 F (73.9 C).

11.3.5 *Exposure Period*—3 min.

11.4 Another method which is sometimes effective for removal of iron oxides is immersion of the specimen in a hot solution of ammonium acetate.

11.5 After cleaning, the weight of each specimen should be determined to the nearest 0.1 mg on an analytical balance and the loss in weight calculated. The corrosion rate in mpy (mils per year) can be calculated using the following equation:

$$\text{mpy} = \frac{\text{weight loss, g} \times 534{,}000}{\text{metal density, g/cm}^3 \times \text{metal area, in.}^2 \times \text{hours exposure}}$$

The corrosion rate may be translated into other terms as discussed below.

12. Examination of Specimen Surface

12.1 The specimen should be carefully examined for type and uniformity of surface attack such as etching, pitting, metasomatic attack, tarnish, film, scale, etc. If pitting is observed, the number, size and distribution, as well as the general shape and uniformity of the pits should be noted. The maximum and minimum depth of the pits can be measured with a calibrated microscope or by the use of a depth gage. Photographs of the cleaned specimens will serve as an excellent record of the surface appearance.

12.2 A distinction should be made between pits occurring under the insulating spacers and those occurring on the boldly exposed surface. As previously noted, pitting at or under the insulating spacers is an indication of the susceptibility of the material to "concentration cell" effects, whereas pitting on the surface is indicative of the intrinsic pitting tendency of the environment.

12.3 In the case of severe pitting of the specimen, the weight loss is of little value and the study of the number, size, and distribution of the pits will be of much more importance. Sometimes a pit-type of corrosion is initiated but is self-healing and stops. A more detailed study of pitting is necessary before a definite conclusion can be reached as to the desirability of rejecting a material because it has a tendency to pit.

12.4 If an alloy is known to be susceptible to localized corrosion on a microscale, such as the phenomenon of intergranular corrosion in stainless steel, dezincification in brass, or stress corrosion cracking of any kind, the specimen should be bent after the previously outlined examination is completed, and any cracks which develop on the surface noted. The results should be compared with those obtained on similar bend tests on unexposed specimens from the same lot of material.

12.5 Microscopical examination of the surface and interior of the specimens may be made if deemed necessary. A low power shop-type binocular microscope is ideal for many of these examinations, although a metallographic examination may be needed.

12.6 The behavior of the metals in galvanic couples can be compared with that of insulated specimens exposed at the same time, and any galvanic effects, including cathodic protection, can be observed. As mentioned earlier, such tests are only qualitative as the magnitude of the galvanic effect will be influenced by the relative areas of the two metals comprising the couple. The results will apply directly only to assemblies where the ratio of areas used in making the tests is similar to the ratio of areas anticipated in the fabricated assembly.

13. Report

13.1 In reporting results of corrosion tests, the conditions of the test should be described in complete detail with special attention being given to the following:

◀⁄᠑⁄ᵖ **G 4**

13.1.1 Corrosive media and concentration,

13.1.2 Type of equipment in which the test was made,

13.1.3 Process carried out in the operating equipment,

13.1.4 Location of specimens in the operating equipment,

13.1.5 Temperature of corrosive media (maximum, minimum, and average),

13.1.6 Oxidizing or reducing nature of corrosive media,

13.1.7 Amount and nature of aeration and agitation of corrosive media,

13.1.8 Duration and type of test (if equipment was operated intermittently during the tests, the actual hours of operation should be stated as well as the total time of the test),

13.1.9 Surface condition of specimen (polished, machined, pickled, 120 grit, etc.), and

13.1.10 Units for expressing corrosion loss. The unit for expressing corrosion rate should be mils penetration per year in cases where the corrosion has been substantially uniform in distribution over the surface of the specimen. If this figure is representative, it may be correlated with the thickness of the equipment in the evaluation of the probable life. It is possible to convert this penetration unit into other terms such as millimeters per year or milligrams per square decimeter per day for comparison with other data.[4] Any such expression will be subject to error to the extent to which nonuniform distribution of corrosion

and changes of corrosion rates with time occur. In connection with the latter, it is often desirable to carry out the testing procedures so as to provide data from which curves can be plotted to illustrate changes in corrosion rates with time.

13.2 The depth of pits should be reported in 0.001 in. (0.02 mm) for the test period and not interpolated or extrapolated to thousandths of an inch per year or any other arbitrary period. The size, shape and distribution of the pits should be noted.

14. Supplementary Tests

14.1 Supplementary laboratory tests should always be made when it is desired to study the effect of one or more of the variables encountered in plant tests. They are particularly desirable if there is any reason to believe that the products of corrosion, or the metal used for the equipment in which the test was conducted, might have had a controlling influence on the behavior of any metal in which there is further interest.

14.2 Special supplementary field tests should be made if there is any reason to believe that stress corrosion cracking, intergranular corrosion, or any other special metallurgical phenomena may be anticipated.

---

[4] See Appendix A2 of ASTM Method A 279, Total Immersion Corrosion Test of Stainless Steels, which appears in the *Annual Book of ASTM Standards*, Part 3.

FIG. 1 Tubular Plastic Spacers.

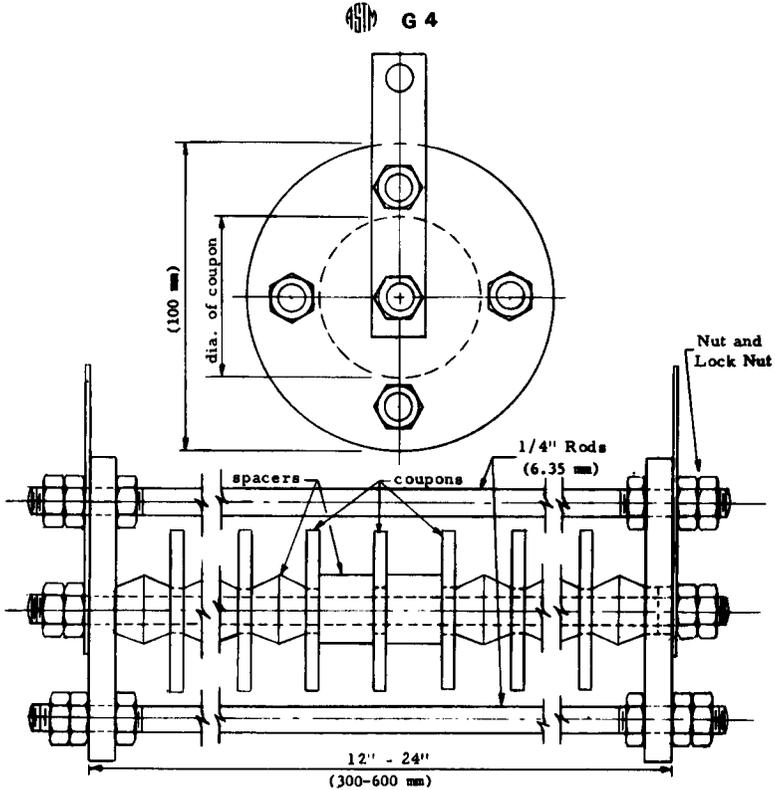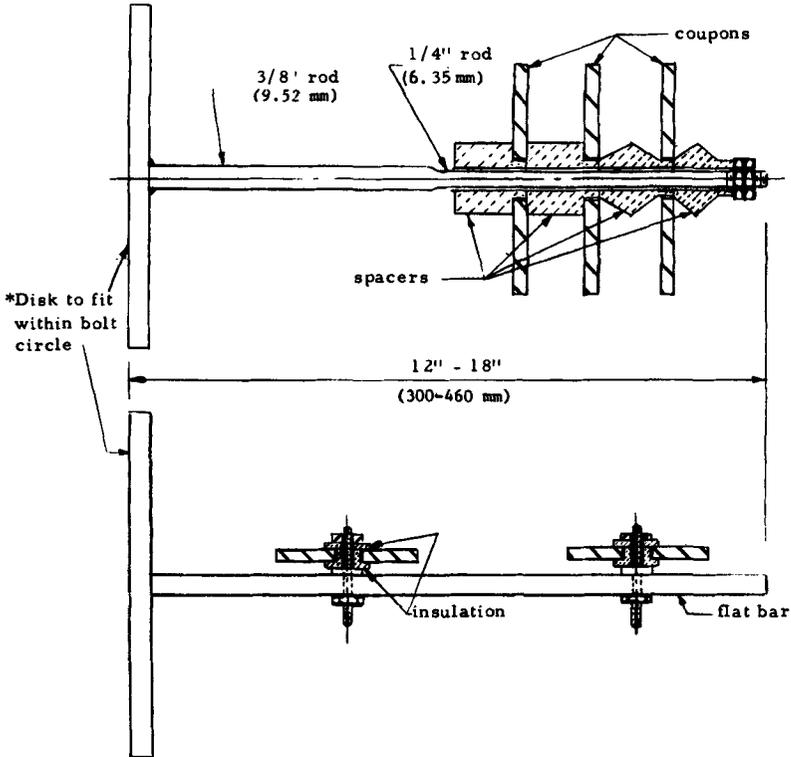**FIG. 2 Spool Rack.**
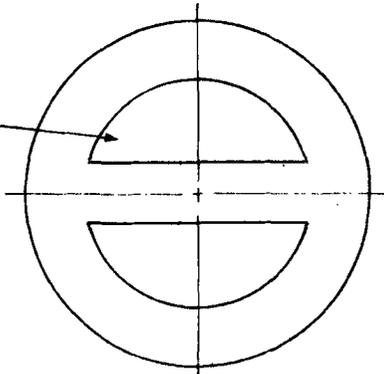
FIG. 3   Insert Racks.

FIG. 4   Dutchman Racks.

FIG. 5   Slip-In Corrosion Test Rack.

ASTM   G 4

3/8" instrument tubing
(9.52 mm)

steam inlet

slip on flange

gasket

1" condenser tubing
(25 mm)

FIG. 6   Hot-Wall Tester.

ASTM Designation: G 15 – 71

Standard Definitions of Terms Relating to
CORROSION AND CORROSION TESTING[1]

This Standard is issued under the fixed designation G 15; 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.

anode—the electrode of an electrolytic cell at which oxidation is the principal reaction. (Electrons flow away from the anode in the external circuit. It is usually the electrode where corrosion occurs and metal ions enter solution.)

anion—a negatively charged ion.

cathode—the electrode of an electrolytic cell at which reduction is the principal reaction. (Electrons flow toward the cathode in the external circuit.)

cathodic corrosion—corrosion of a metal when it is a cathode. It usually happens to amphoteric metals as a result of a rise in pH at the cathode or as a result of the formation of hydrides.

cation—a positively charged ion.

concentration cell—an electrolytic cell, the emf of which is caused by a difference in concentration of some component in the electrolyte. This difference leads to the formation of discrete cathode and anode regions.

corrosion potential—the potential of a corroding surface in an electrolyte relative to a reference electrode measured under open-circuit conditions.

electrolytic cleaning—a process of removing soil, scale, or corrosion products from a metal surface by subjecting it as an electrode to an electric current in an electrolytic bath.

equilibrium (reversible) potential—the potential of an electrode in an electrolytic solution when the forward rate of a given reaction is exactly equal to the reverse rate. The equilibrium potential can only be defined with respect to a specific electrochemical reaction.

galvanostatic—pertaining to an experimental technique whereby an electrode is maintained at constant current in an electrolyte.

inhibitor—a chemical substance or combination of substances, which when present in the proper concentration and forms in the environment, prevents or reduces corrosion.

long-line current—current which flows through the earth from an anodic to a cathodic area of a continuous metallic structure. Usually used only where the areas are separated by considerable distance and where the current results from concentration-cell action.

metallizing—See **thermal spraying.**

open-circuit potential—the potential of an electrode measured with respect to a reference electrode or another electrode when no current flows to or from it.

overvoltage—the change in potential of an electrode from its equilibrium or steady state value when current is applied.

passivator—a type of inhibitor which appreciably changes the potential of a metal to a more noble (positive) value.

potentiostat—an instrument for automatically maintaining an electrode at a constant potential or controlled potentials with respect to a suitable reference electrode.

redox potential—the potential of a reversible oxidation-reduction electrode measured with respect to a reference electorde, corrected to the hydrogen electrode, in a given electrolyte.

stress-corrosion cracking—a cracking process

279

G 15

requiring the simultaneous action of a corrodent and sustained tensile stress. This excludes corrosion-reduced sections which fail by fast fracture. It also excludes intercrystalline or transcrystalline corrosion which can disintegrate an alloy without either applied or residual stress.

thermal spraying—a group of processes wherein finely divided metallic or nonmetallic materials are deposited in a molten or semimolten condition to form a coating. The coating material may be in the form of powder, ceramic rod, wire, or molten materials.

ASTM **Designation: G 16 – 71**

Standard Recommended Practice for

APPLYING STATISTICS TO ANALYSIS OF CORROSION DATA[1]

This Standard is issued under the fixed designation G 16; 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.

1. Scope

1.1 Corrosion scientists and engineers are making increased use of statistical methods, not only in laboratory programs, but often in field failure analysis. Application of statistical methods and interpretation of the results obtained is rendered difficult by the large number of complex techniques that are available and a lack of standardization between analytical methods employed by various groups. Statistics as a discipline applies to nearly all physical, biological, and economic sciences; this has led to development of a large number of methods that are generally applicable and complex. In contrast, the specific application of statistics to corrosion problems often involves simplification and use of a limited number of methods.

1.2 The purpose of this practice is to provide a set of sample procedures that are in current usage in statistical analysis of corrosion experiments. It is recognized that the procedures selected are but a fraction of methods available and that complete agreement on specific methods selected may not be possible. The examples included are intended to provide a method for planning corrosion experiments, analyzing data obtained, and establishing the degree of confidence that can be placed in the results of specific experimental or field applications data. Alternative methods or improved approaches are constantly being developed that may provide more complete analysis and understanding of specific experiments. Accordingly, Subcommittee G01.03 would welcome the comments and criticisms of readers so that future revisions of the procedure may be updated to re-

flect statistical methods that are most relevant to analysis of corrosion experiments.

1.3 The recommended practice includes the following sections:

2. Errors, Their Recognition, and Treatment (1)[2]

2.1 Engineers are frequently faced with the problem of making measurements in the laboratory or in the field that are not completely accurate. It is common practice to repeat a set of measurements; this repetition allows application of statistics to determine the degree of precision obtained. The basis for this approach is that random errors tend to cancel out when a large number of measurements are averaged. For example, suppose that a technician has been asked to prepare a large number of nominally $\frac{1}{8}$-in. thick corrosion samples by cutting them from a large diameter bar of a relatively soft alloy. If the cutting is judged by

---

[1] This recommended practice is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.03 on Statistical Analysis and Planning of Corrosion.

Effective Jan. 8, 1971.

[2] The boldface numbers in parentheses refer to the list of references at the end of this recommended practice.

G 16

eye, it is obvious that not all of the samples will be the same thickness. However, when all the samples are measured and an average thickness is calculated, it will be found that most values lie close to the numerical average or mean.

2.2 Statistical methods cannot eliminate experimental error, but statistics can provide an indication of the magnitude of the possible errors. Statistical methods are particularly useful in establishing the degree of confidence that can be placed in a given measurement or in a value calculated from a measurement. Statistical analysis is based on the premise that errors follow a normal distribution pattern or some special case. The types of errors that occur in experiments arise in measurements or in handling of data. Proper care during the experiment and in subsequent calculations is essential to minimize unnecessary errors and to ensure that all sources of error can be properly identified and calculated.

2.2.1 *Normal Distribution*—In the example above if the number of samples of each measured thickness is plotted against the thickness, a curve called a histogram will be obtained. Frequently this curve will approximate the shape shown in Fig. 1. This is the so-called normal distribution curve. It has certain characteristics. It can be divided into equal-size segments on either side of the mid point which will include a certain fixed percentage of all measurements. The first two are located equal distances from the mid point on either side to include 68.27 percent of the measurements. These represent one standard deviation ($\pm\sigma$); the standard deviation is discussed in the next section. Two standard deviations ($\pm2\sigma$) on the abcissa will then encompass 95.45 percent of the measurements and three standard deviations ($\pm3\sigma$) will encompass 99.73 percent of the measurements. It should be noted that not all experimental error is normally distributed, and it is a good idea to plot histograms to determine if the data fit a normal distribution. This is only possible when a large number of points are available, for example, 20 or more.

2.2.2 *Systematic Error*—In the example above suppose 10 people measured the thickness of each of the corrosion coupons. The plots of the resulting data for each person would generally also produce a normal distri-

bution curve. However, the results for those who pressed down hard with their micrometers on the soft metal would produce a low average and a curve whose maximum was located to the left of that shown in Fig. 1. Those who did not press down firmly would obtain a high average and a curve to the right of that shown. This type of error is a function of the experimental technique and is not a random error. It is called bias and is a systematic error which cannot be handled by statistical analysis alone, although statistical methods can sometimes be used to detect and identify bias.

2.2.3 *Mistakes*—Mistakes either in carrying out an experiment or in calculations are not a characteristic of the population and can preclude statistical treatment of data, or lead to erroneous conclusions if included in the analysis. Sometimes mistakes can be identified by statistical methods by recognizing that the probability of obtaining a particular result is very low.

2.2.4 *Significant Figures:*

2.2.4.1 Care should be exercised in reporting results to show the proper number of significant figures. The location of the decimal point can be used for this purpose. For example the number 2700 can be written as 2.7×10^2 to show two significant digits, that is, an indicated accuracy of ±100. On the other hand 2700. indicates an accuracy of ±1.

2.2.4.2 In carrying out calculations it is good practice to retain one insignificant digit through the calculation to minimize rounding off errors. This insignificant digit should be rounded off in the result. For example the sum of 2700. + 7.07 should be reported as 2707. not 2707.07.

2.2.5 *Propagation of Error in Calculation:*

2.2.5.1 Mathematical operations with experimental data will cause errors in the data to change in predictable ways. Two types of errors are frequently discussed: maximum error and probable error. Estimates of maximum error can usually be found in descriptions of instruments, etc., and generally include systematic as well as random error. Probable error refers to the standard deviation due to random error in systems where it is known or assumed that bias is negligible.

2.2.5.2 Maximum error calculations can be

handled by equation:

$$\Delta Q = \Sigma_{i\,=\,1}{}^n \mid \partial Q / \partial X_i \mid \Delta X_i \qquad (1)$$

where:

Q = calculated quantity of interest, a function of $_n$ measured variables denoted X_i,

ΔQ = maximum error in Q, and

ΔX_i = maximum error in the independent variables.

This expression assumes that all of the X's are independent variables. If this is not true then all partial deviations with dependent variables must be grouped by independent variables inside the absolute value bracket and the sign of the partial deviations considered in each group.

2.2.5.3 If standard deviation information is available then a different equation should be used, namely:

$$\sigma(Q) = [\Sigma_{i\,=\,1}{}^n (\partial Q / \partial X_i)^2 \sigma^2(X_i)]^{1/2} \qquad (2)$$

where $\sigma(X)$ represents the standard deviation of X and all the other terms are defined above.

2.2.5.4 Again we assume that all X's are independent of one another. Both Eqs 1 and 2 can be simplified in specific cases. For example if

$$Q(X_1 X_2 X_3 \cdots X_n) = A(X_1)^a (X_2)^b \cdots (X_n)^j \quad (3)$$

then Eq 1 can be simplified to:

$$\Delta Q / Q = a(\Delta X_1 / X_1) + b(\Delta X_2 / X_2)$$
$$+ \cdots + j(\Delta X_n / X_n) \quad (4)$$

and with simple product functions the percent errors are additive. Another simplification occurs in the case when:

$$Q = aX_1 + bX_2 + \cdots jX_n$$

Then Eq 1 becomes:

$$\Delta Q = a\Delta X_1 + b\Delta X_2 + \ldots + j\Delta X_n \qquad (5)$$

2.2.5.5 For an application of these calculations, consider the errors introduced into the results from limited precision of equipment such as a balance. Weighing is a common measurement in conducting corrosion tests. The difference between initial and final weights is often used to calculate corrosion rates. These weighings are usually conducted on a conventional laboratory analytical balance that is accurate to ±0.2 mg. In weight loss, L equals the initial weight minus the final

weight. From Eq 5 the maximum error between the initial and final weights of a corroded specimen is 0.4 mg. If the observed difference in weight is only 1 mg, the maximum possible error is $(0.4/1) \times 100 = 40$ percent. If the difference is 10 mg, the error is only 4 percent.

2.2.5.6 For another example, the velocity of flowing water is to be measured in a corrosion test. The water will be bypassed into a container for a given period of time and the amount collected will be weighed. Knowing the pipe diameter, the average velocity can then be calculated from the following equation:

$$V_{\text{avg}} = W / tA\rho = 4W / \pi D^2 t\rho \qquad (6)$$

where:

W = weight of water,

A = cross-sectional area,

D = diameter,

t = time, and

ρ = density.

ΔW = ±5 lb—An old scale will be used which is accurate to ±5 lb. About 100 lb will be collected.

Δt = ±1 s—Accuracy of watch and observer is estimated to be ±1 s. Total time will be about 70 s.

ΔD = ±0.03 in.—Out-of-roundness and caliper errors are expected to be ±0.03 in. for 1-in. diameter pipe (inside diameter).

$\Delta \rho$ = ±0.02 lb/ft³—Temperature measurement is expected to be 60 ± 3 F, which corresponds to densities limits of 62.38 and 62.34 lb/ft³. Thus, the error is of the order of 0.1 percent. This is an order of magnitude less than the other errors and thus the error in this term can be neglected.

The maximum error then can be calculated by Eq 1.

$$\Delta V = \left| \frac{4}{t\pi D^2 \rho} \Delta W \right| + \left| \frac{4W}{\pi D^2 \rho t^2} \Delta t \right| + \left| \frac{8W}{t\pi\rho D^3} \Delta D \right|$$

$$= \frac{4 \times 144}{70 \times 3.14 \times 1 \times 62.3} \Delta W$$

$$+ \frac{4 \times 100 \times 144}{3.14 \times 1 \times 62.3 \times (70)^2} \Delta t$$

$$+ \frac{8 \times 100 \times 1728}{70 \times 3.14 \times 62.3 \times 1} \Delta D$$

G 16

$$= 0.042 \, \Delta W + 0.060 \, \Delta t + 100 \, \Delta D$$
$$= 0.52 \, \text{ft/s}$$
$$V_{\text{avg}} = 4W/\pi D^2 t \rho$$

$$= \frac{4 \times 100 \times 144}{3.14 \times 1 \times 70 \times 62.3}$$

$$= 4.2 \, \text{ft/s}$$

Percent error $= (0.522/4.21) \times 100 = 12$ percent

2.2.5.7 A simplified form of Eq 1 can be used in this case because Eq 6 is of the form shown in Eq 3. Rewriting Eq 4 for this case we have:

$$\Delta V/V = | \, \Delta W/W \, | + | \, 2\Delta D/D \, | + | \, \Delta T/T \, |$$

Then by inspection:

$$\Delta V/V = (5/100) + (2 \, (0.03)/1) + (1/70)$$
$$= 0.124 = 12.4 \, \text{percent}$$

and

$$\Delta V = 0.124 \times 4.21 = 0.52 \, \text{ft/s}$$

2.2.5.8 Another advantage to this simplification is that it is dimensionless and so eliminates the need for converting units. Note in the example given D is measured in inches but must be converted to feet to be used in the equation given. Also note that the greatest reduction in error can be made by increasing the accuracy of weighing and of measuring the pipe diameter.

2.2.5.9 Simplification of Eq 2 along the lines shown above are also possible. For example in the above case if the error figures were standard deviation rather than maximum errors the result would become:

$$\sigma(V)/V = [(0.05)^2 + (2)^2 \cdot (0.03)^2 + (1/70)^2]^{1/2}$$
$$= [0.0063]^{1/2} = 0.0795 \, \text{or} \, 8.0 \, \text{percent}$$

Then the standard deviation of V would be 0.34 ft/s.

3. Standard Deviation (2)

3.1 The 24 values listed under x in Table 1 are weight loss data in mg/dm²-day for a particular alloy exposed several months to sea water. An over-all description of data may be expressed as (1) the mean, $\bar{x}$, the sum of all values divided by the total number of values, n; (2) the median, the mid value (the average of the 12th and 13th values since n is even) in ascending order; often more meaningful than the mean when there are one or two values vastly different from the rest, and (3) the standard deviation, σ.

3.2 The standard deviation of a large group of numbers is defined below

$$\sigma = \sqrt{\Sigma d^2/n} \qquad (7)$$

where:
σ = standard deviation,
$d = x - \bar{x}$ (where x = value and $\bar{x}$ = the mean), and
n = total number of observations.

The definition also holds for a small group of numbers if $\bar{x}$ is known independently; however, if $\bar{x}$ is not independently known, then with a limited number of observations, only an estimate of the standard deviation can be made which is:

$$s = \sqrt{\Sigma d^2/(n - 1)} \qquad (8)$$

or

$$s = \sqrt{n\Sigma x^2 - (\Sigma x)^2/n(n - 1)}. \qquad (9)$$

Equation 9 is convenient if a desk calculator is used. The square of the standard deviation, σ^2, is called the variance of the data. The computations have been carried out in Table 1, and the mean and the estimate of the standard deviation are found to be 177.17 and ±10.71 mg/dm²-day, respectively.

If data from the 24 samples follow normal distribution (well-known bell-shaped curve), then
$\bar{x} \pm s$ will include 68.27 percent of the results, on the average
$\bar{x} \pm 2s$ will include 95.45 percent of the results, on the average
$\bar{x} \pm 3s$ will include 99.73 percent of the results, on the average.

4. Probability Curves (2, 3, 4)

4.1 Arithmetic probability paper is so constructed that data from a normal distribution, when plotted on the paper will be randomly distributed about a straight line. To plot the curve, the data must be arranged in ascending order of value and the cumulative percent of tests must be determined for each observation from the following equation:

$$P(\%) = 100 \, [(i - 0.375)/(n + 0.25)] \qquad (10)$$

where:
i = position of data point in total ranking, and
n = total number of data points.

The data from Table 1 are used to calculate $P(\%)$'s which are shown in Table 2.

G 16

4.2 The results are plotted on arithmetic probability paper[3] in Fig. 2. It is not necessary to fit a straight line to these data since the line can be plotted accurately from the mean and the standard deviation. The mean ($\bar{x}$), 177.17, is plotted at 50 percent on the abscissa and the mean plus the standard deviation ($\bar{x} + s$) (177.17 + 10.71) is plotted at 84.13 percent on the abscissa. A straight line through these points establishes the slope of the curve. (The mean minus the standard deviation ($\bar{x} - s$) could also have been used as one of the points.)

4.3 Some data exhibit log-normal distribution. For example, the time to fracture of aluminum alloys in stress-corrosion cracking tests in salt solution follows such a distribution. A plot of these data on log-normal probability paper produces a straight line. Endurance times for the stress-corrosion cracking of an aluminum alloy and the log-normal probability plot of these data are presented in Table 3 and Fig. 3, respectively (3). The probability curves are plotted from the log mean (at 50 percent) and the log mean minus or plus the standard deviation of the logs (at 16 percent, 84 percent) given in Table 3.

4.4 A special form of distribution is extreme value analysis. This type of distribution has been used to analyze maximum pit depths. The pit depth distribution on a given number of corrosion coupons may follow a normal distribution on each coupon, but the maximum pit depths on each of the coupons follow a special distribution of extreme values. The mathematics of extreme values are complex, but practical use of the technique has been simplified by the use of extreme probability paper.

4.5 Aziz (4) has used extreme probability paper in the study of the pitting of aluminum alloys. As an example, consider the maximum pit depths observed for sets of 9 or 10 samples exposed to tap water for exposure periods ranging from 2 weeks to 1 year presented in Table 4. The data are ranked in order of increasing pit depth. The plotting position for each ranking is determined by $R/(n + 1)$ where R = rank and n = total number of specimens. Thus, where 9 specimens were examined, the plotting position for the first specimen is $1/(9 + 1) = 0.100$, for the second is $2/(9 + 1) = 0.200$ etc. For 10 specimens

the plotting position for the first specimen is $1/(10 + 1) = 0.0909$ etc.

4.6 The above data when plotted on extreme probability paper produce the straight lines shown in Fig. 4, thus indicating an extreme value distribution. By extrapolating the plotted lines, one can make certain predictions. For example, with the 2-week data it can be seen that the probability of obtaining a pit 760 μm in depth or less is 0.999 and that the probability of obtaining a pit greater than 760 μm is only one in 1000; whereas the observed deepest pit depth was 580 μm.

5.1 Curve Fitting—Method of Least Squares (5)

5.1 To fit data to a linear plot of the form $y = mx + b$, it is necessary to solve two equations:

$$m\Sigma x^2 + b\Sigma x = \Sigma xy$$
$$m\Sigma x + bn = \Sigma y$$

or

$$m = (n\Sigma xy - \Sigma x\Sigma y)/[n\Sigma x^2 - (\Sigma x)^2]$$
$$b = (1/n)(\Sigma y - m\Sigma x)$$

where:

Σx = sum of all x points,

Σy = sum of all y points,

Σx^2 = sum of squares of x points,

Σxy = sum of x points multiplied by y points, and

n = number of points.

For a parabola of the form $y = ax^2 + bx + c$ three equations must be solved:

$$a\Sigma x^4 + b\Sigma x^3 + c\Sigma x^2 = \Sigma x^2 y$$
$$a\Sigma x^3 + b\Sigma x^2 + c\Sigma x = \Sigma xy$$
$$a\Sigma x^2 + b\Sigma x + cn = \Sigma y$$

5.2 Data for exposure of five replicate specimens of Zircaloy-2 to 750 F-1500 psi steam are presented in Table 5. It is known that the corrosion kinetics of Zircaloy-2 obey two rate laws, an initial cubic-to-parabolic rate followed by a linear rate. In 750 F steam the rate becomes linear after about 42 days. Thus the data in Table 5 comprise the *initial* reaction kinetics which follows a power formula of the general type:

$$W = kt^a$$

where:

W = weight gain (the oxide is extremely adherent),

---

[3] Keuffel and Esser No. 359-24 has been found satisfactory.

k = a rate constant,
t = time, and
a = dimensionless.

The above equation can be expressed in the logarithmic form:

$$\log W = a \log t + \log k$$

A plot of the above logarithms produces a straight line whose slope is a. Thus, the logarithms of the data can be used to fit a curve by the least squares method for a straight line by setting:

$$y = \log W$$
$$x = \log t$$
$$b = \log k$$
$$m = a$$

5.3 The logarithmic forms of the data and the required summations are shown in Table 6 (although only one value is given for x there is one for each value of y, that is five for each exposure time). The calculations of m and b are shown below:

$$m = \frac{m\Sigma xy - \Sigma x\Sigma y}{n\Sigma x^2 - (\Sigma x)^2}$$

$$= \frac{30 \times 41.303 - 27.75 \times 39.92}{30 \times 35.001 - (27.75)^2} = 0.469$$

$$b = \frac{1}{n}(\Sigma y - m\Sigma x)$$

$$\approx \frac{39.92 - (0.469 \times 27.75)}{30} = 0.897$$

$\log k = b = 0.897$
$k = 7.89$
$a = m = 0.469$

Thus, the equation of the curve best fitting the data is

$$W = kt^a$$
$$W = 7.89t^{0.469}$$

5.4 The above curve is plotted on log-log paper in Fig. 5. The curve also shows a plot of the 95 percent ($2s$) limits of data. These were obtained from the estimate of standard deviation of residuals about the curve, calculated from the equation:

$$s = \sqrt{\Sigma d^2/(n - 2)}$$

where $d = y - \hat{y}$ and $\hat{y}$ is the weight gain predicted from the above equation at a given level of exposure time t. The logarithms of data presented in Table 6 were used on the assumption that variance of logarithms of data is in-

dependent of means at each exposure period. (Variance should be independent of means of function of the data (arithmetic, logarithms, exponential, etc.)) The difference, d, from the predicted value, $y - \hat{y}$, was determined for each exposure period. All the d's were squared and summed and s was determined for the entire sample.

$$s = \sqrt{\Sigma d^2/(n-2)} = \sqrt{0.0644/(30-2)} = 0.048$$

At the 95 percent confidence interval $2s = (2)$ (± 0.048) = ± 0.096. Then the calculations of the lines for 95 percent confidence are as follows: (For 1- and 42-day exposures).

$$\log w = \log k + a \log t \pm 2s$$
At 1 day, $\log w = 0.897 + (0.469 \times 0) \pm 0.096$
$$= 0.897 + 0.096 = 0.991$$
$$= 0.897 - 0.096 = 0.803$$
At 42 days, $\log w = 0.897 + (0.469 \times 1.62)$
$$\pm 0.096$$
$$= 0.897 + 0.760 + 0.096$$
$$= 1.753$$
$$= 0.897 + 0.760 - 0.096$$
$$= 1.561$$

The antilogs are:

At 1 day, 0.991 = 9.79 mg/dm²
0.803 = 6.36 mg/dm².
At 42 days, 1.753 = 56.6 mg/dm²
1.561 = 36.4 mg/dm²

5.5 These deviations do not include the effect of variable slope which becomes increasingly important in regions away from the mean. The variable slope could be plotted in Fig. 5 as two lines passing through the data mean with slopes of $m \pm 2s$ (m).

6.1 Estimate of Limits that Include True Value of Mean (Confidence Limits) (6)

6.1 When dealing with a small number of observations, the estimate of the limits that include the true value of the mean can be obtained from:

$$\Delta = \pm t(s/\sqrt{n})$$

where:
s = estimate of standard deviation,
n = number of observations, and
t = student's t from published tables.
For example, the 14-day-75 F steam data for Zircaloy-2 are calculated below:

Five observations: 25.6, 25.5, 24.3, 26.9, and 27.1 mg/dm²
Mean $(\bar{x})$ = 25.9
Estimate of standard deviation $s = \sqrt{\Sigma d^2/(n-1)}$
$= \sqrt{5.29/(5-1)} = 1.15$

G 16

The values of t are obtained from Table 7 at the appropriate degrees of freedom $(n - 1)$. For 4 degrees of freedom the value of $t = 0.741$ at 0.50 probability, 2.776 at 0.95 probability, and 4.604 at 0.99 probability. Then calculating the limits that would include the true mean at particular levels of confidence:

$$\Delta = \pm t (s/\sqrt{n})$$

Confidence Limits, percent	Deviation
50	$\Delta = \pm 0.741 \times (1.15/\sqrt{5}) = \pm 0.38$
95	$\Delta = \pm 2.776 \times (1.15/\sqrt{5}) = \pm 1.42$
99	$\Delta = \pm 4.604 \times (1.15/\sqrt{5}) = \pm 2.36$

	Limits of Mean	
	Lower	Upper
50	25.5	26.3
95	24.5	27.3
90	23.5	28.3

Thus based on the above samples, we could be 99 percent confident that the true mean is contained between 23.5 and 28.3.

7. Comparing Means (5,6,7)

7.1 The means of two sets of replicate observations can be compared by determining the estimate of the limits of the difference between the two means. If the limits include zero, the means are statistically alike; if they do not include zero, the means are different.

7.2 As an example, determine whether two heat treatments of Zircaloy-2 produce a significant difference in corrosion behavior. The heat treatments, corrosion data for 14 days in 750 F steam, and calculations of an estimate of the standard deviation, s, are presented in Table 8. An s for both measurements is calculated as follows:

$$s = \sqrt{(\Sigma d_1^2 + \Sigma d_2^2)/[(n_1 - 1) + (n_2 - 1)]}$$
$$= \sqrt{(5.29 + 24.35)/[(5 - 1) + (5 - 1)]} = 1.92$$

The s for both measurements is now multiplied by $\sqrt{2}$.

Corrected $s = s (\sqrt{2}) = (1.92)(1.41) = 2.70$

(s is multiplied by $\sqrt{2}$ because differences increase s by $\sqrt{2}$.) The limits of the difference between the means is calculated from

$$\Delta = t (s/\sqrt{n})$$
Difference between means $= 27.3 - 25.9 = 1.4$
$$s = 2.70$$
$$n = 5$$
Degrees of freedom $= (5 - 1) + (5 - 1) = 8$

From Table 7,

	50%	95%	99%
$t =$	0.706	2.306	3.355
$\Delta = \pm t (s/\sqrt{n}) =$	± 0.85	± 2.79	± 4.05
Upper limit	2.2	4.2	5.4
		-1.4	-2.6

The above limits of the differences between the two means do not include zero in the first column so that the two means are statistically different at the 50 percent confidence level. The limits of the differences between the two means include zero in the last two columns and the two means are not significantly different at the 95 percent and 99 percent confidence levels.

7.3 An alternative method of comparing means has been described by Freeman (7) who uses the equation:

$$t = \frac{\bar{d}}{\sqrt{(n_x s_x^2 + n_y s_y^2/n_x + n_y - 2)[(1/n_x) + (1/n_y)]}}$$

where:
$\bar{d}$ = difference between the means of x and y,
n_x = number of variates of the x's,
s_x^2 = variance of the x's,
n_y = number of variates of the y's, and
s_y^2 = variance of the y's

The degrees of freedom are $n_x + n_y - 2$.

The calculated value of t is compared with the tabulated values of t (see Table 7) for the appropriate degrees of freedom. If the calculated value is larger than the value from tables, the difference is significant at that confidence level. If the calculated value is smaller, the difference is not significant at that confidence level. For example, assume that the calculated t for two means obtained from five samples in each is 2.604. Then in Table 7 examine the values of t at 8 degrees of freedom $(5 + 5 - 2)$. The calculated value 2.604 is greater than the tabulated value of 0.706 at the 50 percent confidence level and greater than 2.306 at the 95 percent confidence level, so that the means are significantly different at these levels. However, at the 99 percent confidence level the means are not significantly different because the calculated value of 2.604 is smaller than the tabular value of 3.355.

G 16

8. Comparison of Data on Probability Curves (8)

8.1 Data can be compared by plotting confidence limits for each curve on probability paper and determining whether they overlap. As an example, the log-probability distribution of stress-corrosion cracking endurance of aluminum alloys with and without silver additions are plotted in Fig. 6. Not all specimens failed. The estimate of standard deviation, s, is obtained from the plot as the difference between the log endurances at 16.2 and 50 percent probability. At the median (50 percent) the 95 percent confidence limits are calculated to be:

$$\pm\, t\,(s/\sqrt{n})$$

where:

t = student's t (see Table 7), and
n = number of failed specimens,

At one standard deviation ($\pm s$) the limits must be expanded by adding $\pm t(s/\sqrt{2n})$ to $\pm t(s/\sqrt{n}.)$ Lines through these points to the limits at the median establish approximate confidence limits.

8.2 Since not all specimens have failed, $\pm t(s/\sqrt{n})$ is plotted one standard deviation from the median of specimens that have failed. This results in limits being rather broad at high and low probabilities, a consequence of lack of data in this region.

8.3 Although confidence limits overlap in Fig. 6, the lines from either sample are not included in confidence limits of the other; therefore, within the approximate range of probability of 2 to 50 percent, silver addition has a significant effect on stress-corrosion cracking.

9. Sample Size (9)

9.1 One of the most frequently asked questions in corrosion work is "How many samples should I test for each condition?" The statisticians usual answer is "What are the limits you wish to put on the results?"

9.2 Assume that it is desired to determine the corrosion behavior of a new alloy in a chemical environment and that prior tests with similar alloys have produced an estimate of the standard deviation (s) of 10 mg/dm^2. Furthermore, it is desired, at the 95 percent confidence level, that the limits that include the true value of the mean do not exceed 5 mg/

dm^2. From Section 6 the desired equation is

$$\Delta = \pm\, t(s/\sqrt{n})$$

where:

Δ = limits that include the true value of the mean (at a particular level of confidence),
t = student's t,
s = estimate of the standard deviation, and
n = sample size.

Thus,

$$\sqrt{n} = ts/\Delta \quad \text{or} \quad n = t^2 s^2/\Delta^2$$

In the above equation, t is a function of n. For a first approximation assume that $n = 16$; then $t = 2.131$ (from Table 7).

$$n = (2.131)^2 (10)^2/(5)^2 = 18.2$$

Substituting the value of t corresponding to a sample size of 18 and recalculating for n would give a more accurate value for n. Actually, the value of t for 18 samples at the 95 percent confidence level is not greatly different from that for 16 samples. Thus, under the above conditions it is estimated that a sample size of 18 would be required to assure that, at the 95 percent confidence level, the limits of the true value of the mean would not exceed ± 5 mg/dm^2 with an assumed estimate of the standard deviation of 10 mg/dm^2.

9.3 A reasonably small number of specimens can be used if a corrosion experiment is so designed that replicate specimens are exposed to an environment and specimens are removed periodically for evaluation (such as descaling). If there are, say, six to eight exposure periods, the removal of triplicate specimens at each period can furnish statistically significant results, by calculating a standard deviation based on all the data rather than for a single exposure period. That is, determine the mean for each exposure period; determine the difference, d, from the mean for the specimens at that exposure period; square the d's and determine the estimate of the standard deviation by the usual equation:

$$s = \sqrt{\Sigma d^2/(n - 1)}$$

This approach assumes that the variances at the several exposure times are the same. The s determined by this method provides a better estimate of the standard deviation for a given exposure period than that obtained from the three replicate samples examined at each period.

ASTM **G 16**

9.4 On the other hand, extremely large sample sizes are required to obtain significant results if the evaluation is a go-no-go type, such as pitting versus no pitting or cracking versus no cracking. Snedecor has assembled probabilities for observation of these types. They are listed in Table 9. As an illustration assume that ten tubes were selected randomly from a heat exchanger and were examined thoroughly for stress-corrosion cracking. If cracks were found in only one of the ten tubes, it would be predicted at the 95 percent confidence level that between 0 and 45 percent of the remainder of the tubes would contain a stress-corrosion crack. On the other hand, if none of the ten tubes contained a crack that it would still be predicted at 95 percent confidence that between 0 and 31 percent of the remaining tubes would contain a crack. It can be seen from Table 9 that no cracks in 100 tubes would reduce the predicted percentage to 0 to 4 for the remainder of the tubes.

10. Comparison of Effects—Analysis of Variance (10)

10.1 The data presented in Table 10 are the results of laboratory impingement tests in 3 percent NaCl solution. Copper alloy specimens 1 by 4 by 0.05 in. were bolted radially to the periphery of nonmetallic disks. Each disk carried four specimens of each of four alloys. The maximum peripheral velocities of the outer edge of the specimens were 20, 25, and 40 ft/s. The test was run for 10 weeks and the maximum pit depth was obtained for each specimen. The whole test was then repeated. There were the following sources of variation: 4 alloys, 3 velocities, 2 tests, and 4 replicate specimens. There were the following main effects: "among alloys," "among velocities," "between tests"; the following two-way interactions: alloys-velocities, alloys-tests, velocities tests; one three-way interaction: alloys-velocities-tests; and an error term (derived from the variation among replicate specimens). The equations are:

For main effects,

$$SS = (1/n) \, \Sigma t^2 - (T^2/N)$$

For two-way interactions,

$$SS = (1/n) \, [(\Sigma t_1)^2 + (\Sigma t_2)^2 + \cdots + (\Sigma t_z)^2] - [(T^2/N) + SS \text{ for each of the main effects}]$$

For three-way interactions,

$$SS = (1/n) \, [(\Sigma t_1)^2 + (\Sigma t_2)^2 + \cdots + (\Sigma t_z)^2] - [(T^2/N) + SS \text{ for all main effects and interactions}]$$

For error term,

$$\Sigma t^2 - [(T^2/N) + SS \text{ for all main effect and interactions}]$$

where:

SS = sum of squares,

n = number of data within each level being compared,

t = sum of data common to a given level of the main effect,

$t_1, t_2 \cdots t_z$ = test results common to a given combination of the levels of the two main effects (two-way interaction) or three main effects (three-way interaction). For example, the alloy and test interaction Σt's is the sum of 12 data points for a given alloy and test and $n = 12$,

T = sum of all the data, and

N = total number of observations.

10.2 The sum of squares and mean square are determined for each main effect, two-way interaction, three-way interaction, and error term. The mean square, MS = SS/DF, where DF = degrees of freedom. The degrees of freedom are:

Tests: $(2 - 1) = 1$
Alloys: $(4 - 1) = 3$
Velocities: $(3 - 1) = 2$
Tests-alloys: $(2 - 1) \times (4 - 1) = 3$
Tests-velocities: $(2 - 1) \times (3 - 1) = 2$
Alloys-velocities: $(4 - 1) \times (3 - 1) = 6$
Tests-alloys-velocities: $(4 - 1) \times (3 - 1) \times (2 - 1) = 6$
Error term: $(96 - 1) - $ (the sum of the DF's of all main effects and interactions) $= 72$

10.3 The mean square for each effect is divided by the mean square of the most significant interaction containing that effect or the error if none of the interactions are significant. The result is compared with values from F tables which may be found in most text books on statistics (see Table 11). The F value is found by locating the degrees of freedom in the error term down in the table. If the calculated value is greater than the F value, the effect is significant. If it is less than the F value, the effect is not significant.

10.4 Calculation of the sum of squares and mean squares is shown in Table 12. Analysis

of variance is shown in Table 13.

11. Two-Level Factorial Design (8)

11.1 The two-level factorial design experiment is an excellent method for determining which variables have an effect on the outcome. The significance of each effect can be determined by analysis of variance.

11.2 As many variables as possible that may be expected to have an effect on the outcome should be included in the original experiment. In order to simplify the following example, only three variables will be used.

11.3 Assume that the stress-corrosion cracking endurance of aluminum alloys is being evaluated on alternate immersion tests in 3 percent NaCl. Suppose that one alloy contains silver and another does not, and in addition, that the effects of cold working and overaging are to be studied. The following nomenclature is then assigned:

A+ Alloy with silver   A− Alloy without silver
B+ With cold work   B− Without cold work
C+ With overage   C− Without overage

	A+		A−	
	B+	B−	B+	B−
C+				
C−				

11.4 This experiment requires eight entirely different sets of conditions. In order to determine the within-sample error more accurately, it is wise to replicate each condition. It is thus necessary to perform a minimum of 16 separate tests. In this particular example, the outcome is the log of the endurance of each stress-corrosion specimen.

	A+		A−	
	B+	B−	B+	B−
C+	1.86	2.54	2.01	3.02
	1.95	2.43	2.32	2.89
C−	1.65	2.32	1.98	2.56
	1.73	2.25	1.87	2.60

Each response can be identified by its location. For example, y_{A+B+C+} has two responses, which are 1.86 and 1.95. They can

be further subscripted as y_{A+B+C+_1} and y_{A+B+C+_2}. The error sum of squares for the experiment is:

$$\sum_{ijk_l}^{A\pm B\pm C\pm} \left(\sum_{l=1}^{2} y(ijk)_l^2 - \left[\sum_{l=1}^{2} y(ijk) \right]^2 \right)$$

Each pair of responses must be squared, then added, and also added then squared. For example, the responses for A+B+C+ would be treated in the following manner:

$$(1.86)^2 + (1.95)^2 - \frac{1}{2} [1.86 + 1.95]^2 = 0.004$$

Each of these figures is then summed, to give the error sum of squares, which in this example is 0.0789. The error degree of freedom is $(2 - 1) (8) = 8$. The 2 is the number of times the response is replicated, and the 8 is the number of pairs.

11.5 In studying the effects of variables it is mathematically easier to work with differences between levels rather than with means at each level. The difference is referred to as a contrast:

$$\hat{A} = (1/N) [\Sigma y_+ - \Sigma y_-]$$

where:
$\hat{A}$ = contrast or effect of silver,
N = number of tests, which is 16,
y_+ = any response in the A+ columns, and
y_- = any response in the A− columns.

$\hat{B}$ and $\hat{C}$ are calculated in a similar manner. The interactions $\hat{AB}$, $\hat{AC}$, $\hat{BC}$, and $\hat{ABC}$ use the same procedure, except the signs for the responses are determined by products of the signs for the variables. For example, (A+) (B+) is $\hat{AB}(+)$ and (A+) (B+) (C−) is $\hat{ABC}$−. For $\hat{AB}$, A+B+ is (+), A+B− is (−), A−B+ is (−), and A−B− is (+). The absolute value of each response remains the same. Each effect or contrast has $(2 - 1)$ degrees of freedom. The 2 is for the levels at each condition.

11.6 Each contrast is squared and multiplied by the number of tests (16) to obtain the sum of squares. Table 14 shows the values as they are used in analysis of variance. F is the ratio of the sum of squares of the effect to the error sum of squares. An F distribution table shows that for 1 degree of freedom for the greater sum of squares (numerator) and 8 degrees of freedom for the lesser sum of squares (denominator), the 5 percent and 1 percent levels of F are 5.32 and 11.25, respectively.

G 16

Thus, in this example, there is less than 1 percent probability that the B effect is caused by random error. On the other hand, the remainder of the effects are not significant.

11.7 If this were a true problem, it would show that materials without cold work were not as susceptible to stress-corrosion cracking as materials with cold work. The addition of silver and overaging had no significant effect. Note that this is a hypothetical example.

11.8 Each time an additional variable is to be studied, twice as many experiments must be performed to complete the two-level factorial design. When many variables are involved, the number of experiments becomes prohibitive.

11.9 Fractional replication can be used to reduce the amount of testing. When this is done, the amount of information that can be obtained from the experiment is also reduced.

11.10 The example of the factorial design with three variables will be used. However, the negative side of the $\widehat{ABC}$ contrast will not be included.

		A+		A−	
		B+	B−	B+	B−
C+		1.86			3.02
		1.95			2.89
C−			2.32	1.98	
			2.25	1.87	

11.11 With the previous method for analysis of variance it is found that ABC cannot be obtained, because the negative values are missing, and that contrasts $\hat{A} = \widehat{BC}$, $\hat{B} = \widehat{AC}$, and $\hat{C} = \widehat{AB}$. In this particular example, an assumption that all the interaction effects are unimportant is correct and it is possible to arrive at the same conclusions that were obtained from the full factorial design experiment. In some cases, it may be that the interaction effects are much greater than the effects of the main variables, in which case an assumption would lead to drastically wrong conclusions. It is wise to have some idea about the effect of interactions before fractional replication is used.

REFERENCES

(1) Mickley, H. S., Sherwood, T. K., and Reed, C. E., Editor, *Applied Mathematics in Chemical Engineering Second Edition*, McGraw-Hill Book Co., New York, N.Y., 1957, pp. 46–99.

(2) Douglas, D. M., ARMCO Steel Corp. "Are You Using or Abusing Data?," paper presented at the 1965 joint Northeast-North Central Regional Meeting National Association of Corrosion Engineers, Pittsburgh, Pa., Oct. 4–6, 1965.

(3) Booth, F. F., and Tucker, G. E. G., "Statistical Distribution of Endurance in Electrochemical Stress-Corrosion Tests," *Corrosion*, CORRA, Vol 21, 1965, pp. 173–177.

(4) Aziz, P. M., "Application of Statistical Theory of Extreme Values to the Analysis of Maximum Pit Depth Data for Aluminum," *Corrosion*, CORRA, Vol 12, 1956, pp. 495–506t.

(5) Hanes, H. D., "The Effect of Gas-Pressure Bonding on the Corrosion Resistance of Zir-

caloy-2," a thesis presented in partial fulfillment of the requirements for the degree of Master of Science, The Ohio State University, 1961.

(6) Youden, W. J., *Experimentation and Measurement*, National Science Teachers Assn., Washington, D. C., 1962.

(7) Freeman, H. A., *Industrial Statistics*, John Wiley & Sons, Inc., New York, N.Y., 1942.

(8) Haynie, F. H., Vaughan, D. A., Phalen, D. I., Boyd, W. K., and Frost, P. D., "A Fundamental Investigation of the Nature of Stress-Corrosion Cracking in Aluminum Alloys," AFML-TR-66-267, June 1966.

(9) Snedecor, G. W., "Statistical Methods Applied to Experiments in Agriculture and Biology," 4th Ed., Iowa State College Press, Ames, Iowa, 1946.

(10) Contribution by D. H. Thompson, Anaconda American Brass Co., Waterbury, Conn.

ASTM G 16

TABLE 1 Computing Standard Deviation

x	d	d^2	x	d	d^2	x	d	d^2
190	13	169	178	1	1	178	1	1
195	18	324	162	15	225	164	13	169
169	8	64	162	15	225	189	12	144
185	8	64	171	6	36	178	1	1
180	3	9	192	15	225	171	6	36
178	1	1	172	5	25	172	5	25
170	7	49	195	18	324	156	21	441
179	2	4	181	4	16	185	8	64

$\bar{x} = 177.17$

$d = |x - \bar{x}| \quad s = \sqrt{\Sigma d^2/(n-1)} = \sqrt{2642/23} = 10.71$

TABLE 2 $P(\%) = 100[(i - 0.375)/(n + 0.25)] =$ **Cumulative Probability (see Fig. 2)**

i	$P(\%)$	Data	i	$P(\%)$	Data	i	$P(\%)$	Data
1	2.6	156 MDD	9	35.5	172 MDD	17	68.5	181 MDD
2	6.7	162	10	40	172	18	72.5	185
3	10.8	162	11	44	178	19	77	185
4	15	164	12	48	178	20	81	189
5	19	169	13	52	178	21	85	190
6	23	170	14	56	178	22	89.2	192
7	27	171	15	60	179	23	93.3	195
8	31.5	171	16	64.5	180	$n = 24$	97.4	195

TABLE 3 Endurances of Aluminum-5 percent Magnesium Stress-Corrosion Specimens Exposed Anodically in 3 percent NaCl Solution (see Fig. 3)

Intensiostatic 40 mA/in.²	66, 70, 72, 73, 75, 75, 76, 77, 80, 80, 82, 82, 82, 88, 89, 90, 91, 91, 92, 92, 93, 93, 94, 94, 94, 95, 96, 96, 96, 97, 97, 97, 97, 99, 99, 100, 100, 100, 101, 106, 106, 106, 107, 107, 107, 108, 108, 110, 111, 115, 116, 116, 116, 116, 117, 117, 118, 119, 120, 122, 122, 122, 123, 126, 127, 128, 130, 130, 132, 133, 135, 135, 136, 140, 147, 150, 152.
	Geometric mean = 103.2 Mean of $\log_{10}$ endurance = 2.014 Standard deviation of $\log_{10}$ endurance = 0.0844
Potentiostatic −0.34 V (S.C.E.)[a]	50, 52, 57, 60, 60, 60, 62, 63, 63, 64, 66, 66, 67, 67, 67, 67, 67, 68, 68, 69, 69, 70, 70, 70, 70, 70, 71, 71, 71, 71, 72, 72, 72, 72, 72, 72, 72, 73, 74, 74, 74, 74, 75, 75, 75, 76, 76, 76, 76, 76, 76, 77, 77, 77, 78, 78, 78, 78, 78, 78, 80, 80, 80, 80, 80, 81, 81, 81, 82, 82, 82, 83, 83, 83, 83, 84, 84, 85, 85, 85, 85, 85, 86, 86, 86, 86, 86, 87, 88, 88, 89, 90, 90, 92, 92, 92, 92, 92, 93, 93, 94, 94, 95, 95, 97, 97, 97, 98, 98, 99, 99, 99, 99, 99, 100, 100, 100, 102, 105, 105, 108, 112, 112, 115.
	Geometric mean = 80.15 Mean of $\log_{10}$ endurance = 1.90387 Standard deviation of $\log_{10}$ endurance = 0.0697

[a] Saturated Calomel Electrode.

⚛Ⱳ **G 16**

TABLE 4 Ordered Maximum Pit Depths Developed on Alcan 3S-O Coupons Immersed in Kingston Tap Water for the Time Periods Shown Together with Their Ranks and Plotting Positions (see Fig. 4)

Rank	2 Weeks	Plotting Position	1 Month	Plotting Position	2 Months	Plotting Position	4 Months	Plotting Position	6 Months	Plotting Position	1 Year	Plotting Position
1	330	0.1000	570	0.0909	600	0.1000	620	0.0909	640	0.0909	700	0.0909
2	460	0.2000	620	0.1818	670	0.2000	620	0.1818	650	0.1818	70C	0.1818
3	500	0.3000	640	0.2727	770	0.3000	670	0.2727	670	0.2727	750	0.2727
4	500	0.4000	640	0.3636	790	0.4000	680	0.3636	700	0.3636	770	·.3636
5	530	0.5000	700	0.4545	790	0.5000	720	0.4545	720	0.4545	700	0.4545
6	540	0.6000	740	0.5454	830	0.6000	780	0.5454	730	0.5454	810	0.5454
7	560	0.7000	780	0.6363	860	0.7000	780	0.6363	750	0.6363	820	0.6363
8	560	0.8000	810	0.7272	930	0.8000	800	0.7272	770	0.7272	830	0.7272
9	580	0.9000	840	0.8181	1030	0.9000	830	0.8181	780	0.8181	830	0.8181
10	...	...	910	0.9090	...	...	920	0.9090	850	0.9090	930	0.9090

TABLE 5 Weight Gain of Zircaloy-2 in 750 F Steam at Time Indicated, mg/dm²

		Days			
1	3	7	14	28	42
9.8	11.8	20.3	25.6	34.8	47.2
7.2	11.8	19.7	25.5	36.0	49.2
6.6	10.5	19.0⁻	24.3	34.1	47.3
8.5	13.8	22.3	26.9	34.8	48.6
9.9	13.9	22.4	27.1	41.7	52.2

TABLE 6 Least Squares Calculation Zircaloy-2 in 750 F Steam

Log of time (x):	0	0.48	0.85	1.15	1.45	1.62
Log of weight gain (y):	0.99	1.07	1.31	1.41	1.54	1.67
	0.86	1.07	1.29	1.41	1.56	1.69
	0.82	1.02	1.28	1.39	1.53	1.67
	0.93	1.14	1.35	1.43	1.54	1.69
	1.00	1.14	1.35	1.43	1.62	1.72

$\Sigma x = 27.75$ $\Sigma y = 39.92$ $\Sigma xy = 41.303$ $\Sigma x^2 = 35.0015$

TABLE 7 Distribution of t

Degrees of Freedom	Probability		
	0.50	0.95	0.99
1	1.000	12.706	63.657
2	0.816	4.303	9.925
3	0.765	3.182	5.841
4	0.741	2.776	4.604
5	0.727	2.571	4.032
6	0.718	2.447	3.707
8	0.706	2.306	3.355
15	0.691	2.131	2.947
30	0.683	2.042	2.750
99	0.676	1.984	2.626
∞	0.674	1.960	2.576

TABLE 8 Comparing Means—Zircaloy-2 for 14 Days in 750 F Steam

1450 F WQ		1650 F WQ	
25.6	Mean = 25.9	25.5	Mean = 27.3
25.5	Σd^2 = 5.29	26.8	Σd^2 = 24.35
24.3	s = 1.15	26.8	s = 2.46
26.9		27.2	
27.1		30.5	

$s_{1.2}$ for both measurements

$= \sqrt{(\Sigma d_1^2 + \Sigma d_2^2)/[(n_1 - 1) + (n_2 - 1)]}$

$= \sqrt{(5.29 + 24.35)/[(5 - 1) + (5 - 1)]} = 1.92$

Correct $s = s_{1.2}(\sqrt{2}) = 1.92(\sqrt{2}) = 2.70$

⚗ **G 16**

TABLE 9   95 percent Confidence Interval (Percent) for Binomial Distribution

Number Observed, X	Size of Sample 10		15		20		30		50		100		Fraction Observed, X/n	Size of Sample 250		1000	
0	0	31	0	22	0	17	0	12	0	07	0	4	0.00	0	1	0	0
1	0	45	0	32	0	25	0	17	0	11	0	5	0.01	0	4	0	2
2	3	56	2	40	1	31	1	22	0	14	0	7	0.02	1	5	1	3
3	7	65	4	48	3	38	2	27	1	17	1	8	0.03	1	6	2	4
4	12	74	8	55	6	44	4	31	2	19	1	10	0.04	2	7	3	5
5	19	81	12	62	9	49	6	35	3	22	2	11	0.05	3	9	4	7
6	26	88	16	68	12	54	8	39	5	24	2	12	0.06	3	10	5	8
7	35	93	21	73	15	59	10	43	6	27	3	14	0.07	4	11	6	9
8	44	97	27	79	19	64	12	46	7	29	4	15	0.08	5	12	6	10
9	55	100	32	84	23	68	15	50	9	31	4	16	0.09	6	13	7	11
10	69	100	38	88	27	73	17	53	10	34	5	18	0.10	7	14	8	12
11			45	92	32	77	20	56	12	36	5	19	0.11	7	16	9	13
12			52	96	36	81	23	60	13	38	6	20	0.12	8	17	10	14
13			60	98	41	85	25	63	15	41	7	21	0.13	9	18	11	15
14			68	100	46	88	28	66	16	43	8	22	0.14	10	19	12	16
15			78	100	51	91	31	69	18	44	9	24	0.15	10	20	13	17
16					56	94	34	72	20	46	9	25	0.16	11	21	14	18
17					62	97	37	75	21	48	10	26	0.17	12	22	15	19
18					69	99	40	77	23	50	11	27	0.18	13	23	16	21
19					75	100	44	80	25	53	12	28	0.19	14	24	17	22
20					83	100	47	83	27	55	13	29	0.20	15	26	18	23
21							50	85	28	57	14	30	0.21	16	27	19	24
22							54	88	30	59	14	31	0.22	17	28	19	25
23							57	90	32	61	15	32	0.23	18	29	20	26
24							61	92	34	63	16	33	0.24	19	30	21	27
25							65	94	36	64	17	35	0.25	20	31	22	28
26							69	96	37	66	18	36	0.26	20	32	23	29
27							73	98	39	68	19	37	0.27	21	33	24	30
28							78	99	41	70	19	38	0.28	22	34	25	31
29							83	100	43	72	20	39	0.29	23	35	26	32
30							88	100	45	73	21	40	0.30	24	36	27	33
31									47	75	22	41	0.31	25	37	28	34
32									50	77	23	42	0.32	26	38	29	35
33									52	79	24	43	0.33	27	39	30	36
34									54	80	25	44	0.34	28	40	31	37
35									56	82	26	45	0.35	29	41	32	38
36									57	84	27	46	0.36	30	42	33	39
37									59	85	28	47	0.37	31	43	34	40
38									62	87	28	48	0.38	32	44	35	41
39									64	88	29	49	0.39	33	45	36	42
40									66	90	30	50	0.40	34	46	37	43
41									69	91	31	51	0.41	35	47	38	44
42									71	93	32	52	0.42	36	48	39	45
43									73	94	33	53	0.43	37	49	40	46
44									76	95	34	54	0.44	38	50	41	47
45									78	97	35	55	0.45	39	51	42	48
46									81	98	36	56	0.46	40	52	43	49
47									83	99	37	57	0.47	41	53	44	50
48									86	100	38	58	0.48	42	54	45	51
49									89	100	39	59	0.49	43	55	46	52
50									93	100	40	60	0.50	44	56	47	53

[a] If X exceeds 50, read 100 − X = number observed and subtract each confidence limit from 100.
[b] If X/n exceeds 0.50, read 1.00 − X/n = fraction observed and subtract each confidence limit from 100.

ⴺ⅏ⵙ **G 16**

TABLE 10   Maximum Depth of Pitting, Mils

Velo- city, ft/s	Test 1			Test 2		
	20	25	40	20	25	40
Alloy A	24	19	33	32	21	37
	24	18	35	27	20	44
	22	20	31	34	21	42
	23	21	34	26	19	43
Alloy B	23	21	31	29	18	40
	22	19	36	32	21	37
	22	19	30	31	20	36
	20	19	33	27	20	38
Alloy C	5	28	21	2	11	6
	4	30	19	3	9	8
	5	20	24	4	18	7
	5	23	18	3	13	6
Alloy D	10	3	7	6	7	11
	6	13	14	14	4	12
	10	4	8	11	6	12
	7	3	9	11	7	10

TABLE 11   Partial Table of the Distribution of F (5 percent)-Top (1 percent)-Bottom

DF of Error MS	DF of Effect MS									
	1	2	3	4	5	6	7	8	9	10
1	161	200	216	225	230	234	237	239	241	242
	4052	4999	5403	5625	5764	5859	5928	5981	6022	6056
2	18.51	19.00	19.16	19.25	19.30	19.33	19.36	19.37	19.38	19.39
	98.49	99.00	99.17	99.25	99.30	99.33	99.34	99.36	99.38	99.40
3	10.13	9.55	9.28	9.12	9.01	8.94	8.88	8.84	8.81	8.78
	34.12	30.82	29.46	28.71	28.24	27.91	27.67	27.49	27.34	27.23
4	7.71	6.94	6.59	6.39	6.26	6.16	6.09	6.04	6.00	5.96
	21.20	18.00	16.69	15.98	15.52	15.21	14.98	14.80	14.66	14.54
5	6.61	5.79	5.41	5.19	5.05	4.95	4.88	4.82	4.78	4.74
	16.26	13.27	12.06	11.39	10.97	10.67	10.45	10.27	10.15	10.05
10	4.96	4.10	3.71	3.48	3.33	3.22	3.14	3.07	3.02	2.97
	10.04	7.56	6.55	5.99	5.64	5.39	5.21	5.06	4.95	4.85
25	4.24	3.38	2.99	2.76	2.60	2.49	2.41	2.34	2.28	2.24
	7.77	5.57	4.68	4.18	3.86	3.63	3.46	3.32	3.21	3.13
50	4.03	3.18	2.79	2.56	2.40	2.29	2.20	2:13	2.07	2.02
	7.17	5.06	4.20	3.72	3.41	3.18	3.02	2.88	2.78	2.70
70	3.98	3.13	2.74	2.50	2.35	2.23	2.14	2.07	2.01	1.97
	7.01	4.92	4.08	3.60	3.29	3.07	2.91	2.77	2:67	2.59
80	3.96	3.11	2.72	2.48	2.33	2.21	2.12	2.05	1.99	1.95
	6.96	4.88	4.04	3.56	3.25	3.04	2.87	2.74	2.64	2.55
100	3.94	3.09	2.70	2.46	2.30	2.19	2.10	2.03	1.97	1.92
	6.90	4.82	3.98	3.51	3.20	2.99	2.82	2.69	2.59	2.51
∞	3.84	2.99	2.60	2.37	2.21	2.09	2.01	1.94	1.88	1.83
	6.64	4.60	3.78	3.32	3.02	2.80	2.64	2.51	2.41	2.32

⚙ASTM G 16

TABLE 12   Calculations Based on Section 10 and Examples of Table 10

T^2/N = $(1811)^2/96$ = 34163.76041

SS for tests = $\frac{1}{48}[(895)^2 + (916)^2] - T^2/n$
= 4.59375   DF = 1
                                        MS = 4.59375

SS for alloys = $\frac{1}{24}[(670)^2 + (644)^2 + (292)^2 + (205)^2] - T^2/N$
= 7124.78125   DF = 3
                                        MS = 2374.92708

SS for velocities = $\frac{1}{32}[(524)^2 + (515)^2 + (772)^2] - T^2/N$
= 1329.52084   DF = 2
                                        MS = 664.76042

SS for tests-alloys = $\frac{1}{12}[(304)^2 + (366)^2 + \ldots + (111)^2] - [T^2/N + 4.59375 + 7124.78125]$
= 811.78125   DF = 3
                                        MS = 270.59375

SS for tests-velocities $\frac{1}{16}[(232)^2 + (280)^2 + \ldots + (389)^2] - [T^2/N + 4.59375 + 1329.52084]$
= 172.31250   DF = 2
                                        MS = 86.15625

SS for alloys-velocities = $\frac{1}{8}[(212)^2 + (159)^2 + (299)^2 + (206)^2 + (157)^2 + (281)^2 + (31)^2 + (152)^2 + (109)^2 + (75)^2 + (47)^2 + (83)^2] - [T^2/N + 7124.78125 + 1329.52084]$
= 1924.56250   DF = 6
                                        MS = 320.76041

SS for tests-alloys-velocities = $\frac{1}{4}[(93)^2 + (78)^2 + \ldots + (45)^2] - [T^2/N + 4.59375 + 7124.78125 + 1329.52084 + 811.78125 + 172.31250 + 1924.56250]$
= 129.43750   DF = 6
                                        MS = 21.57291

SS for error = $[(24)^2 + (24)^2 + (22)^2 + \ldots + (10)^2]$ $[T^2/N + $ SS for all main effects and interactions]
= 438.2500   DF = 72
                                        MS = 6.08680

〔ASTM〕 **G 16**

TABLE 13 Analysis of Variance

	Degrees of Freedom	Calculated MS Ratio	95 percent Level		99 percent Level	
			F Value	Signi- ficant?	F Value	Signi- ficant?
Tests	1,3	$\dfrac{4.59375}{270} = <1$	10.13	no	34.12	no
Tests, Alloys						
Alloys	3,6	$\dfrac{2374.92708}{320} = 7.45$	4.76	yes	9.78	no
Alloys, Velocities						
Velocities	2,6	$\dfrac{664.76042}{320} = 2.07$	5.14	no	10.92	no
Alloys, Velocities						
Tests, Alloys	3,6	$\dfrac{270.59375}{21.57} = 12.5$	4.76	yes	9.78	yes
Tests, Alloys, Velocities						
Tests, Velocities	2,6	$\dfrac{86.15625}{21.57} = 4.0$	5.14	no	10.92	no
Tests, Alloys, Velocities						
Alloys, Velocities	6,6	$\dfrac{320.76041}{21.57} = 14.5$	4.28	yes	8.47	yes
Tests, Alloys, Velocities						
Tests, Alloys, Velocities	6,72	$\dfrac{21.57291}{6.08680} = 3.54421$	2.23	yes	3.07	yes
Error						

TABLE 14 Analysis of Variance

Effect	Contrast	Sum of Squares	Degrees of Freedom	F
$\hat{A}$	−0.1575	0.397	1	5.03
$\hat{B}$	−0.3275	1.716	1	21.75
$\hat{C}$	+0.1287	0.265	1	3.36
$\widehat{AB}$	+0.0337	0.018	1	0.23
$\widehat{AC}$	−0.025	0.010	1	0.13
$\widehat{BC}$	−0.013	0.0028	1	0.04
$\widehat{ABC}$	+0.0188	0.0056	1	0.07
Error		0.0789	8	
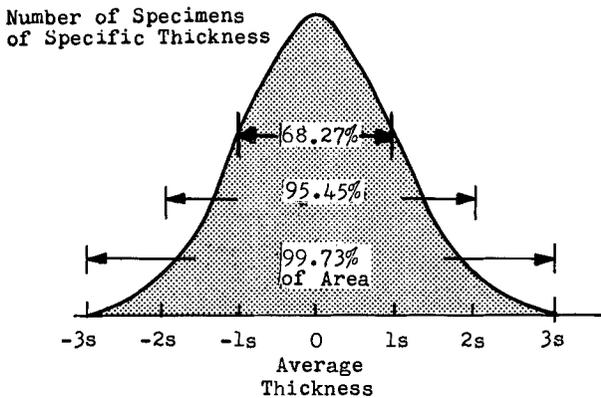
FIG. 1 Normal Distribution Curve.
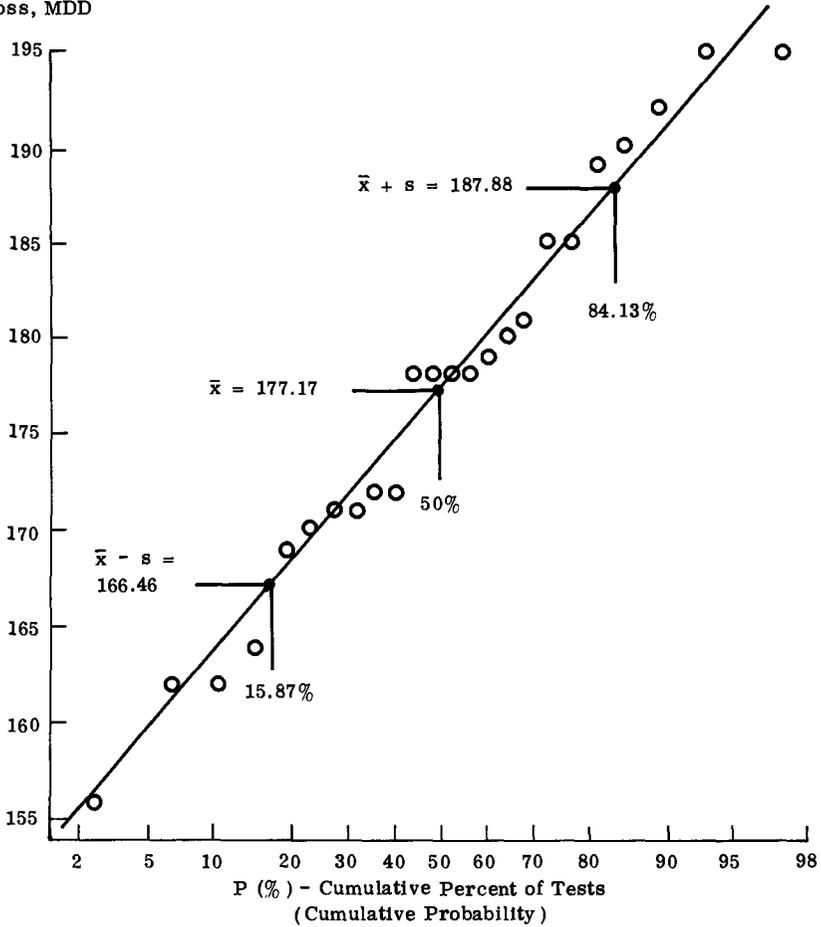
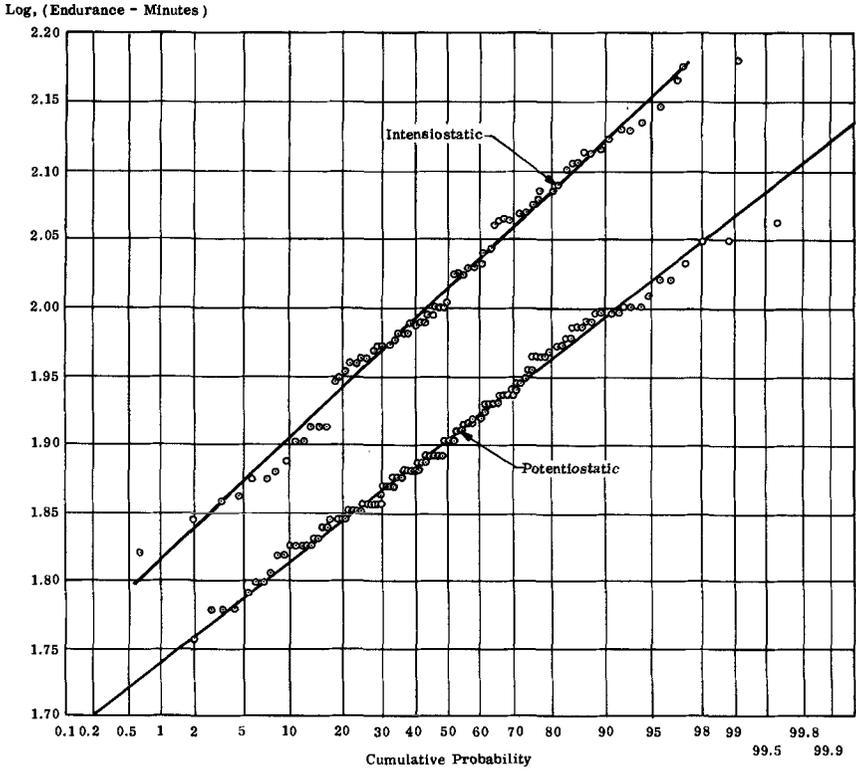FIG. 2 Cumulative Frequency Distribution of 24 Corrosion Tests (see Table 2).

FIG. 3 Endurance of Aluminum—5 Percent Magnesium Alloys Exposed Anodically in 3 Percent NaCl Solution (See Table 3).
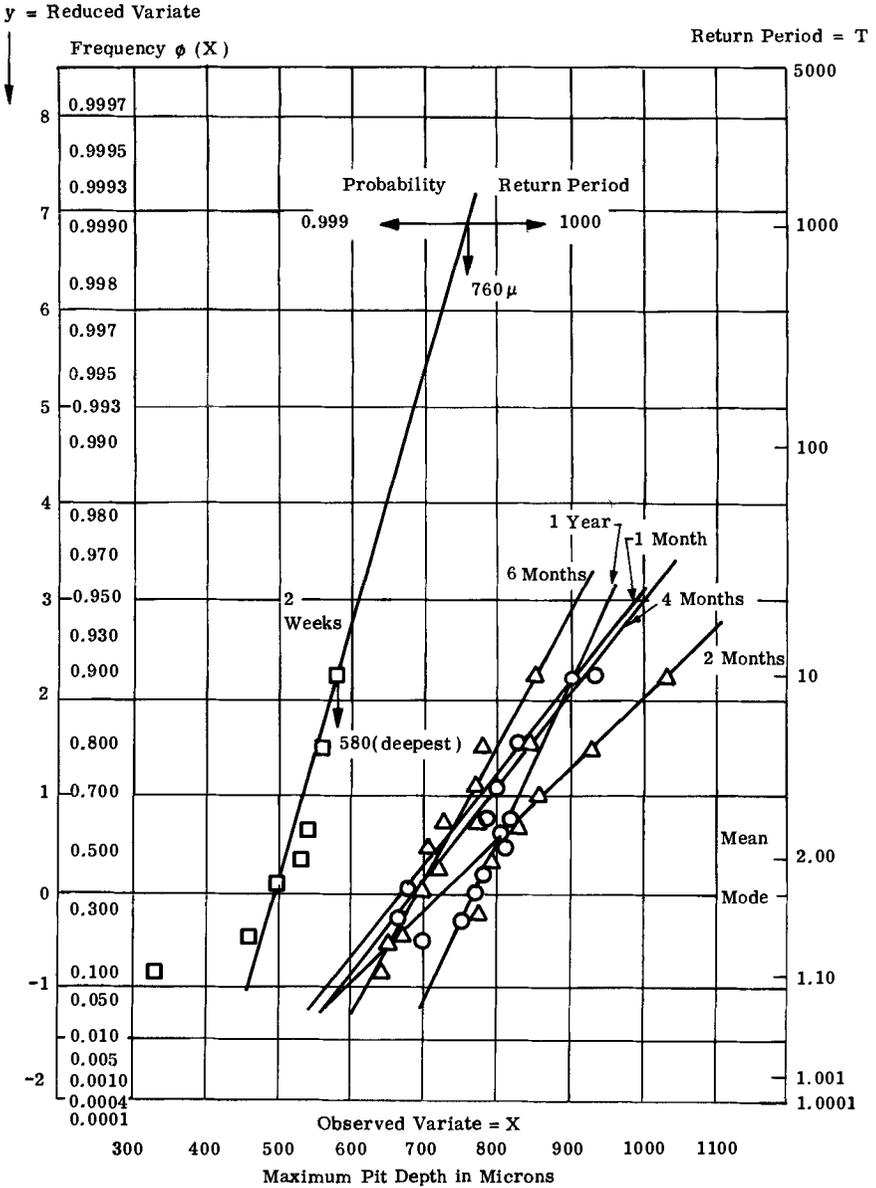
$ASTM$ **G 16**

FIG. 4 Maximum Pit Depth Data for Alcan 3S-O (AA3003-O) Immersed in Kingston Tap Water for the Time Periods Shown Plotted Against Their Cumulative Relative Frequencies (See Table 4).
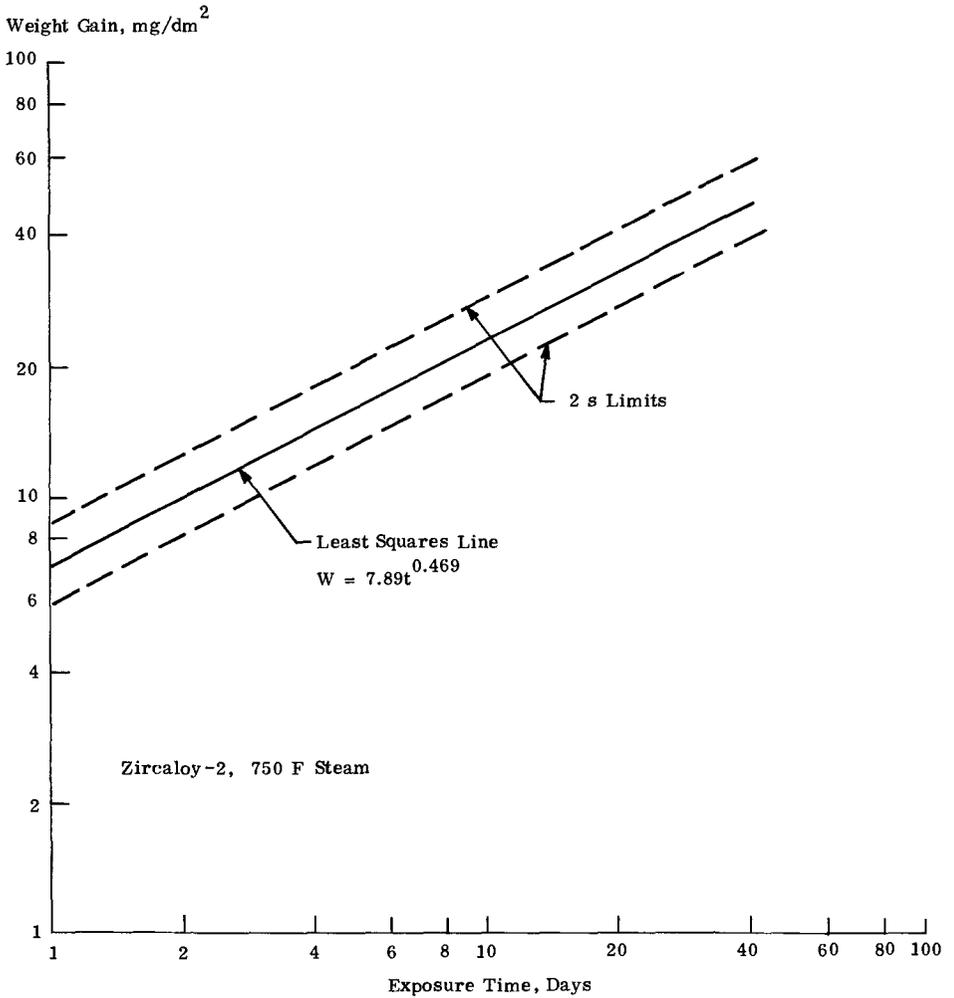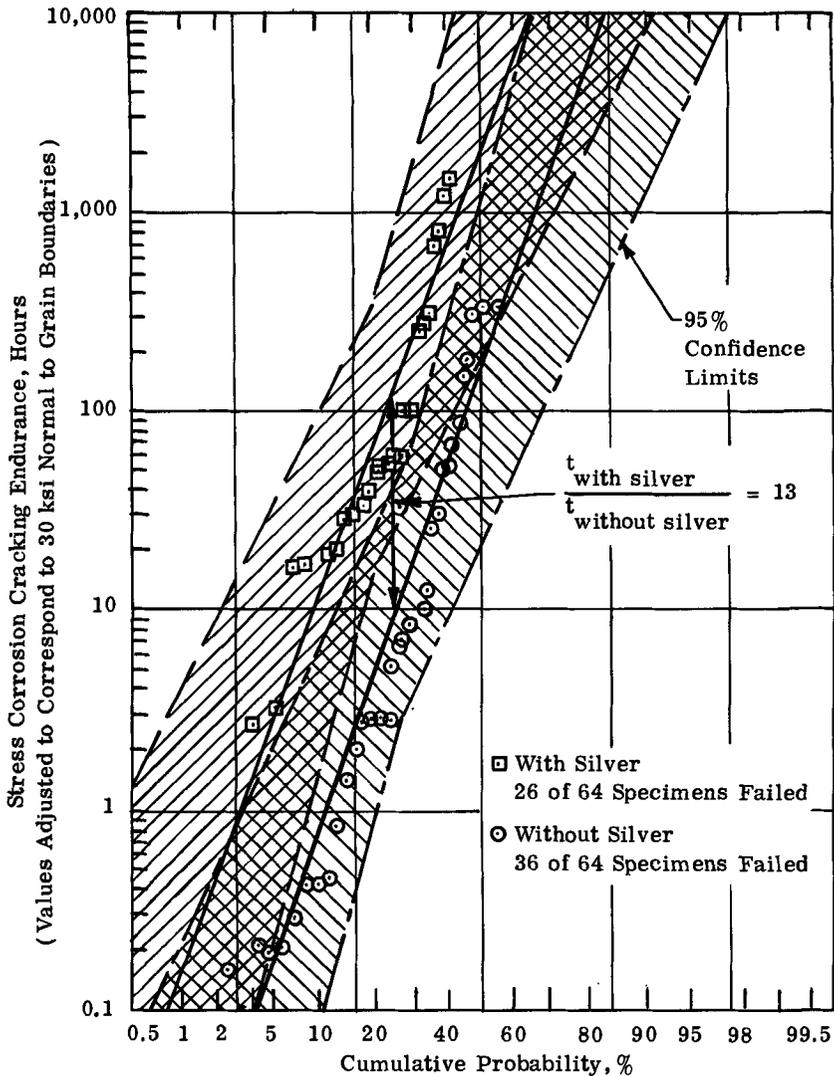
◀§▶   **G 16**

Weight Gain, mg/dm^2

FIG. 5   Zircaloy-2 Exposed to 750 F-1500 psi Steam.

ASTM   G 16

FIG. 6   Effect of the Addition of Silver on Stress-Corrosion-Cracking Behavior of 7079 Type Aluminum Alloys.

Confidence limits code:
    ▨ Region within 95 percent confidence limits on best fit line for endurances of alloys containing silver
    ▨ Region within 95 percent confidence limits on best fit line for endurances of alloys without silver
    ▨ Region common to both