5 Corrosion Evaluation

CHEMICAL CLEANING PROCEDURES FOR REMOVAL OF CORROSION PRODUCTS

Material	Solution	Time	Temperature	Remarks
Aluminum and Aluminum Alloys	50 mL phosphoric acid (H_3PO_4, sp gr 1.69) 20 g chromium trioxide (CrO_3) Reagent water to make 1,000 mL	5 to 10 min	90 °C to Boiling	If corrosion product films remain, rinse, then follow with nitric acid procedure (C.1.2).
	Nitric acid (HNO_3, sp gr 1.42)	1 to 5 min	20 to 25 °C	Remove extraneous deposits and bulky corrosion products to avoid reactions that may result in excessive removal of base metal.
Copper and Copper Alloys	500 mL hydrochloric acid (HCl, sp gr 1.19) Reagent water to make 1,000 mL	1 to 3 min	20 to 25 °C	Deaeration of solution with purified nitrogen will minimize base metal removal.
	4.9 g sodium cyanide (NaCN) Reagent water to make 1,000 mL	1 to 3 min	20 to 25 °C	Removes copper sulfide corrosion products that may not be removed by hydrochloric acid treatment.

Material	Solution	Time	Temperature	Remarks
	100 mL sulfuric acid (H_2SO_4, sp gr 1.84) Reagent water to make 1,000 mL	1 to 3 min	20 to 25 °C	Remove bulky corrosion products before treatment to minimize copper redeposition on specimen surface.
	120 mL sulfuric acid (H_2SO_4, sp gr 1.84) 30 g sodium dichromate ($Na_2Cr_2O_7 \cdot 2H_2O$) Reagent water to make 1,000 mL	5 to 10 s	20 to 25 °C	Removes redeposited copper resulting from sulfuric acid treatment.
	54 mL sulfuric acid (H_2SO_4, sp gr 1.84) Reagent water to make 1,000 mL	30 to 60 min	40 to 50 °C	Deaerate solution with nitrogen. Brushing of test specimens to remove corrosion products followed by re-immersion for 3 to 4 s is recommended.
Iron and Steel	1,000 mL hydrochloric acid (HCl, sp gr 1.19) 20 g antimony trioxide (Sb_2O_3) 50 g stannous chloride ($SnCl_2$)	1 to 25 min	20 to 25 °C	Solution should be vigorously stirred or specimen should be brushed. Longer times may be required in certain instances.
	50 g sodium hydroxide (NaOH) 200 g granulated zinc or zinc chips Reagent water to make 1,000 mL	30 to 40 min	80 to 90 °C	Caution should be exercised in the use of any zinc dust since spontaneous ignition upon exposure to air can occur.
	200 g sodium hydroxide (NaOH) 20 g granulated zinc or zinc chips Reagent water to make 1,000 mL	30 to 40 min	80 to 90 °C	Caution should be exercised in the use of any zinc dust since spontaneous ignition upon exposure to air can occur.
	200 g diammonium citrate ($(NH_4)_2HC_6H_5O_7$) Reagent water to make 1,000 mL	20 min	75 to 90 °C	Depending upon the composition of the corrosion product, attack of base metal may occur.
	500 mL hydrochloric acid (HCl, sp gr 1.19) 3.5 g hexamethylene tetramine Reagent water to make 1,000 mL	10 min	20 to 25 °C	Longer times may be required in certain instances.

Material	Solution	Time	Temperature	Remarks
	Molten caustic soda (NaOH) with 1.5–2.0% sodium hydride (NaH)	1 to 20 min	370 °C	For details refer to Technical Information Bulletin SP29-370, "DuPont Sodium Hydride Descaling Process Operating Instructions."
Lead and Lead Alloys	10 mL acetic acid (CH_3COOH) Reagent water to make 1,000 mL	5 min	Boiling	...
	50 g ammonium acetate (CH_2COONH_4) Reagent water to make 1,000 mL	10 min	60 to 70 °C	...
	250 g ammonium acetate (CH_2COONH_4) Reagent water to make 1,000 mL	5 min	60 to 70 °C	...
Magnesium and Magnesium Alloys	150 g chromium trioxide (CrO_3) 10 g silver chromate (Ag_2CrO_4) Reagent water to make 1,000 mL	1 min	Boiling	The silver salt is present to precipitate chloride.
	200 g chromium trioxide (CrO_3) 10 g silver nitrate ($AgNO_3$) 20 g barium nitrate ($Ba(NO_3)_2$) Reagent water to make 1,000 mL	1 min	20 to 25 °C	The barium salt is present to precipitate sulfate.
Nickel and Nickel Alloys	150 mL hydrochloric acid (HCl, sp gr 1.19) Reagent water to make 1,000 mL	1 to 3 min	20 to 25 °C	...
	100 mL sulfuric acid (H_2SO_4, sp gr 1.84) Reagent water to make 1,000 mL	1 to 3 min	20 to 25 °C	...
Stainless Steels	100 mL nitric acid (HNO_3, sp gr 1.42) Reagent water to make 1,000 mL	20 min	60 °C	...
	150 g diammonium citrate ($(NH_4)_2HC_6H_5O_7$) Reagent water to make 1,000 mL	10 to 60 min	70 °C	...

Material	Solution	Time	Temperature	Remarks
	100 g citric acid ($C_6H_8O_7$) 50 mL sulfuric acid (H_2SO_4, sp gr 1.84) 2 g inhibitor (diorthotolyl thiourea or quinoline ethyliodide or betanaphthol quinoline) Reagent water to make 1,000 mL	5 min	60 °C	...
	200 g sodium hydroxide (NaOH) 30 g potassium permanganate ($KMnO_4$) Reagent water to make 1,000 mL *followed by* 100 g diammonium citrate ($(NH_4)_2HC_6H_5O_7$) Reagent water to make 1,000 mL	5 min	Boiling	...
	100 mL nitric acid (HNO_3, sp gr 1.42) 20 mL hydrofluoric acid (HF, sp gr 1.198–48%) Reagent water to make 1,000 mL	5 to 20 min	20 to 25 °C	...
	200 g sodium hydroxide (NaOH) 50 g zinc powder Reagent water to make 1,000 mL	20 min	Boiling	Caution should be exercised in the use of any zinc dust since spontaneous ignition upon exposure to air can occur.
Tin and Tin Alloys	150 g trisodium phosphate ($Na_3PO_4 \cdot 12H_2O$) Reagent water to make 1,000 mL	10 min	Boiling	...
	50 mL hydrochloric acid (HCl, sp gr 1.19) Reagent water to make 1,000 mL	10 min	20 °C	...
Zinc and Zinc Alloys	150 mL ammonium hydroxide (NH_4OH, sp gr 0.90) Reagent water to make 1,000 mL	5 min	20 to 25 °C	...

Material	Solution	Time	Temperature	Remarks
	followed by 50 g chromium trioxide (CrO_3) 10 g silver nitrate ($AgNO_3$) Reagent water to make 1,000 mL	15 to 20 s	Boiling	The silver nitrate should be dissolved in water and added to the boiling chromic acid to prevent excessive crystallization of silver chromate. The chromic acid must be sulfate free to avoid attack of the zinc base metal.
	100 g ammonium chloride (NH_4Cl) Reagent water to make 1,000 mL	2 to 5 min	70 °C	…
	200 g chromium trioxide (CrO_3) Reagent water to make 1,000 mL	1 min	80 °C	Chloride contamination of the chromic acid from corrosion products formed in salt environments should be avoided to prevent attack of the zinc base metal.
	85 mL hydriodic acid (HI, sp gr 1.5) Reagent water to make 1,000 mL	15 s	20 to 25 °C	Some zinc base metal may be removed. A control specimen (3.1.1) should be employed.
	100 g ammonium persulfate ($(NH_4)_2S_2O_8$) Reagent water to make 1,000 mL	5 min	20 to 25 °C	Particularly recommended for galvanized steel.
	100 g ammonium acetate (CH_3COONH_4) Reagent water to make 1,000 mL	20 to 5 min	70 °C	…

Source: ASTM G1, "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens" (West Conshohocken, PA, USA: ASTM International, 2000). Reprinted with permission, copyright ASTM.

ELECTROLYTIC CLEANING PROCEDURES FOR REMOVAL OF CORROSION PRODUCTS

Material	Solution	Time	Temperature	Remarks
Iron, Cast Iron, Steel	75 g sodium hydroxide (NaOH) 25 g sodium sulfate (Na_2SO_4) 75 g sodium carbonate (Na_2CO_3) Reagent water to make 1,000 mL	20 to 40 min	20 to 25 °C	Cathodic treatment with 100 to 200 A/m^2 current density. Use carbon, platinum or stainless steel anode.
	28 mL sulfuric acid (H_2SO_4, sp gr 1.84) 0.5 g inhibitor (diortho-tolyl thiourea or quinoline ethyliodide or betanaphthol quinoline) Reagent water to make 1,000 mL	3 min	75 °C	Cathodic treatment with 2,000 A/m^2 current density. Use carbon, or platinum or lead anode.
	100 g diammonium citrate ($(NH_4)_2HC_6H_5O_7$) Reagent water to make 1,000 mL	5 min	20 to 25 °C	Cathodic treatment with 100 A/m^2 current density. Use carbon or platinum anode.
Lead and Lead Alloys	28 mL sulfuric acid (H_2SO_4, sp gr 1.84) 0.5 g inhibitor (diortho-tolyl thiourea or quinoline ethyliodide or betanaphthol quinoline) Reagent water to make 1,000 mL	3 min	75 °C	Cathodic treatment with 2,000 A/m^2 current density. Use carbon, platinum or lead anode.
Copper and Copper Alloys	7.5 g potassium chloride (KCl) Reagent water to make 1,000 mL	1 to 3	20 to 25 °C	Cathodic treatment with 100 A/m^2 current density. Use carbon or platinum anode.

Material	Solution	Time	Temperature	Remarks
Zinc and Cadmium	50 g dibasic sodium phosphate (Na_2HPO_4) Reagent water to make 1,000 mL	5 min	70 ˚C	Cathodic treatment with 110 A/m^2 current density. Specimen must be energized prior to immersion. Use carbon, platinum or stainless steel anode.
	100 g sodium hydroxide (NaOH) Reagent water to make 1,000 mL	1 to 2 min	20 to 25 ˚C	Cathodic treatment with 100 A/m^2 current density. Specimen must be energized prior to immersion. Use carbon, platinum or stainless steel anode.
General (excluding Aluminum, Magnesium and Tin Alloys)	20 g sodium hydroxide (NaOH) Reagent water to make 1,000 mL	5 to 10 min	20 to 25 ˚C	Cathodic treatment with 300 A/m^2 current density. A S31600 stainless steel anode may be used.

Source: ASTM G1, "Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens" (West Conshohocken, PA, USA: ASTM International, 2000). Reprinted with permission, copyright ASTM.

ETCHANTS FOR REVEALING MICROSTRUCTURES IN SELECTED ALLOYS

Alloy	Etchant	Uses
Aluminum and Al Alloys	0.5–25 g NaOH 1 g zinc chloride 100 mL water	General purpose etch. Grain boundary delineation. Immerse up to 2 min.
	1 mL HF (48%) 200 mL water	Outlines microconstituents. Immerse for 30–40 s.
	12.5 mL HNO_3 (conc.) 2.5 mL HF (48%) 85 mL water	General purpose etch. Grain boundary delineation. Immerse up to 1 min.
	2 mL HF (48%) 3 mL HCl (conc.) 20 mL HNO_3 (conc.) 175 mL water	Modified Keller's Rgnt. General purpose etch for Al & Al alloys. Immerse 10–60 s. Wash in warm water, blow dry.
Copper and Copper Alloys	10 mL NH_4OH 10 mL H_2O_2 (3%) Can dilute up to 20 mL water	General purpose etch. Grain boundary delineation. Use fresh, swab, or immerse up to 1 min.
	10 g $(NH_4)_2S_2O_8$ 90 mL water	General purpose etch. Grain boundary delineation. Immerse up to 1 min.
	10 g Cr_2O_3 4 drops HCl 75–100 mL water	Swab or immerse up to 30 s.
Nickel and Nickel Alloys	20 mL HNO_3 60 mL HCl	AquaRegia. Grain boundary, carbide, and σ contrast. Use fresh and under hood. Discard after use. Swab or immerse up to 1 min.
	3 parts glycerol 2–3 parts HCl 1 part HNO_3	Glyceregia. Popular etch. Use fresh and under hood. Discard after use. Swab or immerse up to 1 min.
	10 g $CuSO_4$ 50 mL HCl 50 mL water	Marble's Reagent. Grain boundary delineation. Swab or immerse up to 1 min. A few drops of H_2SO_4 increase etch activity.

Alloy	Etchant	Uses
Iron and Iron Alloys	2 mL HNO_3 98 mL Ethanol	Nital. Gives good pearlite-ferrite-grain boundary contrast in carbon and low alloy steels. Swab or immerse up to 1 min.
	4 g picric acid 100 mL Ethanol 4–5 drops of zephiran chloride (wetting agent)	Picral. Promotes good resolution of pearlite, bainite. martensite, and carbides. Swab or immerse up to 1 min.
	100 mL Picric acid (sat.) 1 g tridecylbenzene	Reveals prior austenitic grain boundaries in martensitic steels.
Stainless Steel	1 part HNO_3 1 part HCl 1 part water	General purpose etch for stainless steels. Promotes grain boundary contrast. Immerse in a gently stirred solution.
	1 g picric acid 5 mL HCl 100 mL Ethanol	Vilella's Reagent. Outlines carbides, σ and δ. Immerse up to 1 min.
	1 part glycerol 3 parts HCl 1 part HNO_3	Glyceregia for SS's. Reveals grain structure. Outlines σ and carbides. Use fresh and under hood. Discard after use. Swab or Immerse up to 1 min.
	10 g oxalic acid 100 mL water	Electrolytic etch (sample is anode). Use at 1–6 V@ 0.1–1.0 A/cm^2. Resolves σ in 5–10 s. Resolves carbides in 15–30 s. Resolves grain boundaries in 45–60 s.

Source: Manual 20, *Corrosion Tests and Standards: Application and Interpretation* (West Conshohocken, PA, USA: ASTM International, 1995). Reprinted with permission, copyright ASTM.

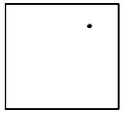
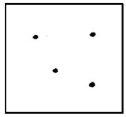
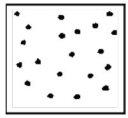
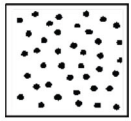
COMPARISON OF SURFACE ANALYSIS TECHNIQUES

Category Technique	Incident Particle	Analyzed Particle	Measured Quantity	Analysis Depth in Monolayers	Sample Damage	Sensitivity (Atomic Fraction)	Standard-less Quan-tification
Auger	Electron	Auger Electron	Energy	2–10	No-Mod	10−3	fair
XPS-ESCA	X-Ray	Photo-Electron	Energy	2–10	No-Min	10−3	fair
Dynamic Sims	Ions	Sec Ions	Mass	10–20	Mod-Ext	10−7	poor
Static Sims	Ions	Sec Ions	Mass	1–2	Mid-Mod	10−6	poor
SNMS	Ions	Neutrals	Mass	5–10	Min-Ext	10−7	fair
SALI	Ions	Neutrals	Mass	1–2	Min-Ext	10−7	fair
RBS	Ions	Input Ions	Energy	many	Min-Mod	10−3	good
ISS	Ions	Input Ions	Energy	1	No	10−3	good

Notes—No: None; Mod: Moderate; Med: Medium; Ext: Extensive

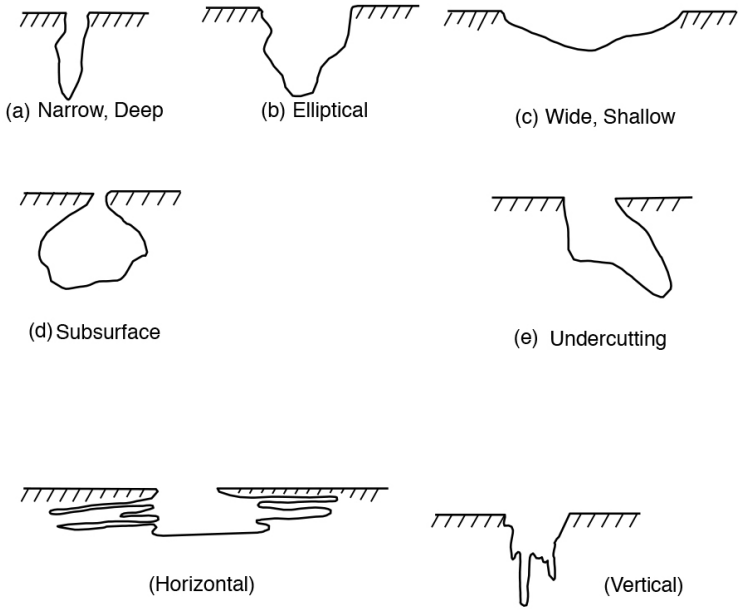
Source: Manual 20, *Corrosion Tests and Standards: Application and Interpretation* (West Conshohocken, PA, USA: ASTM International, 1995). Reprinted with permission, copyright ASTM.

STANDARD RATING CHART FOR PITS

	A	B	C
	Density	Size	Depth
1			
	$2.5 \times 10^3/m^2$	$0.5\ mm^2$	0.4 mm
2			
	$1 \times 10^4/m^2$	$20\ mm^2$	0.8 mm
3			
	$5 \times 10^4/m^2$	$80\ mm^2$	1.6 mm
4			
	$1 \times 10^5/m^2$	$12.5\ mm^2$	3.2 mm
5			
	$5 \times 10^5/m^2$	$24.5\ mm^2$	6.4 mm

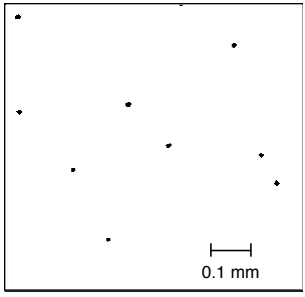
Source: ASTM G46, "Standard Guide for Examination and Evaluation of Pitting Corrosion" (West Conshohocken, PA, USA: ASTM International, 2000). Reprinted with permission, copyright ASTM.

VARIATIONS IN THE CROSS-SECTIONAL SHAPE OF PITS

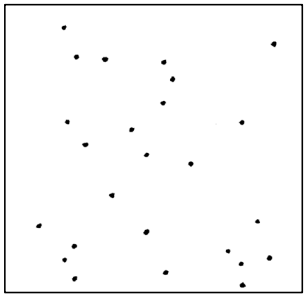


Source: ASTM G46, "Standard Guide for Examination and Evaluation of Pitting Corrosion" (West Conshohocken, PA, USA: ASTM International, 2000). Reprinted with permission, copyright ASTM.

STANDARD DOT PATTERNS FOR THE NUMBER OF CORROSION PITS (cm^2) OBSERVED AT 100X

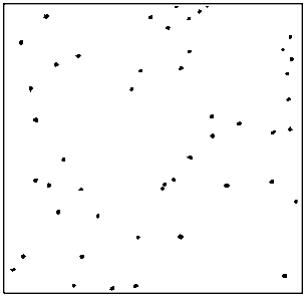


1,000 pits/square cm at 100 ×

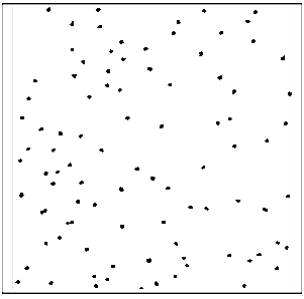


2,500 pits/square cm at 100 ×

RANDOM DOT PATTERNS



5,000 pits/square cm at 100 ×



10,000 pits/square cm at 100 ×

Source: ASTM B627 (withdrawn). "Test Method for Electrolytic Corrosion Testing." (West Conshohocken, PA, USA: ASTM International, 2000). Reprinted with permission, copyright ASTM.

STANDARD COATING RATINGS SYSTEM

Standard	Scale	Description
ASTM D714-56, Evaluating	Size of Blister	
the Degree of Blistering	10	No blister
Paints	8	Pinpoint
	6	Pinpoint to $^1/_{16}$ in.
	4	$^1/_8$ in.
	2	$^3/_8$ in. or larger
	Frequency of Blisters	
	10	None
	8	Few
	6	Medium
	4	Medium-Dense
	2	Dense
ASTM D659-44, Evaluating	10	No chalk or discolor
Degree of Resistance to		on cloth
Chalking of Exterior	8	Slight discoloration
Paints (wool cloth pressed on	6	Light discoloration
surface and turned 180 degrees)	2	Completely opaque chalk
ASTM D660, Evaluating	10	No checking
Degree of Resistance	9	Very minor checking
to Checking (checking	8	Few checks
is a break in the surface not	6	Moderate
penetrating to the substrate)	4	Almost continuously checked
	2	Completely checked
ASTM D661, Evaluating the	10	No cracking
Degree of Resistance to	9	Very minor cracking
Cracking of Exterior	8	Few cracks
Paints (cracking extends	6	Moderate
through coating to substrate)	4	Almost continuously cracked
	2	Completely cracked
ASTM D772-47, Evaluating	10	No flaking
the Degree of Flaking	8	Few flakes
(scaling) of Exterior	6	Moderate flaking
Paint	4	20 to 25% of surface flaked
	2	40 to 50% of surface flaked

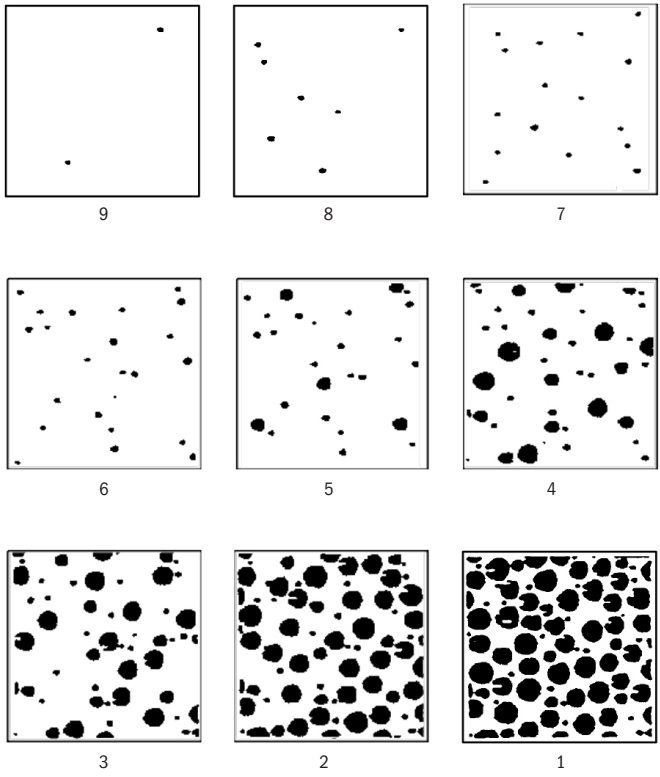
Source: C.G. Munger, *Repairing Protective Coatings: Effect of Coating Types, Plant Engineering* (Houston, TX, USA: NACE International, 1977).

RATING OF PAINTED SURFACE ASTM-D610/SSPC-Vis 2

Scale and Description of Rust Grades

Rust Grades*	Description	ASTM-SSPC Photographic Standard
10	no rusting or less than 0.01 percent of surface rusted	unnecessary
9	minute rusting less than 0.03 percent of surface rusted	No. 9
8*	few isolated rust spots. less than 0.1 percent of surface rusted	No. 8
7	less than 0.3 percent of surface rusted	none
6*	extensive rust spots but less than 1 percent of surface rusted	No. 6
5	rusting to the extent of 3 percent of surface rusted	none
4*	rusting to the extent of 10 percent of surface rusted	No. 4
3*	approximately one sixth of the surface rusted	none
2	approximately one third of the surface rusted	none
1	approximately one half of the surface rusted	none
0*	approximately 100 percent of the surface rusted	unnecessary

See following page for ASTM-SSPC Photographic Standards.



Source: A. Marshall, *NACE Coating Inspector's Logbook*, 3rd ed. (Houston, TX, USA: NACE International, 1996).

ABBREVIATIONS DESCRIBING DEFECTS

Types of Failure

R = corrosion (rusting) of the basis metal. (Permanent or massive type of basis metal corrosion such as that in pinholes, bare, or flaked areas, or in craters of broken blisters.)

Rs = stain due to basis metal corrosion products, such as rust stain, which can be removed readily with a damp cloth or chamois and mild abrasive revealing a sound bright surface.

S = stains or spots other than that of *obvious* basis metal corrosion products.

Sp = surface pits. Corrosion pits probably not extending through to the basis metal—that is absence of *obvious* basis metal corrosion products bleeding therefrom.

F = flaking or peeling of deposit. B = blistering.
C = cracking. Z = crazing.
W = crow's feet.

Degree or Extent of Pinhole Rusting, Staining, Surface Pitting, Flaking, Etc.

vs = very slight amount.
s = slight amount.
i = intermediate or moderate amount.
x = excessive amount.

Description of Blisters

s = less than about 0.5 mm in diameter.
i = about 0.5 to 2.0 mm in diameter.
x = greater than about 2.0 mm in diameter.
vf = 5 or fewer.
f = 5+ to 10.
i = 10+ to 25.
m = 25+ to 50.
ym = over 50.

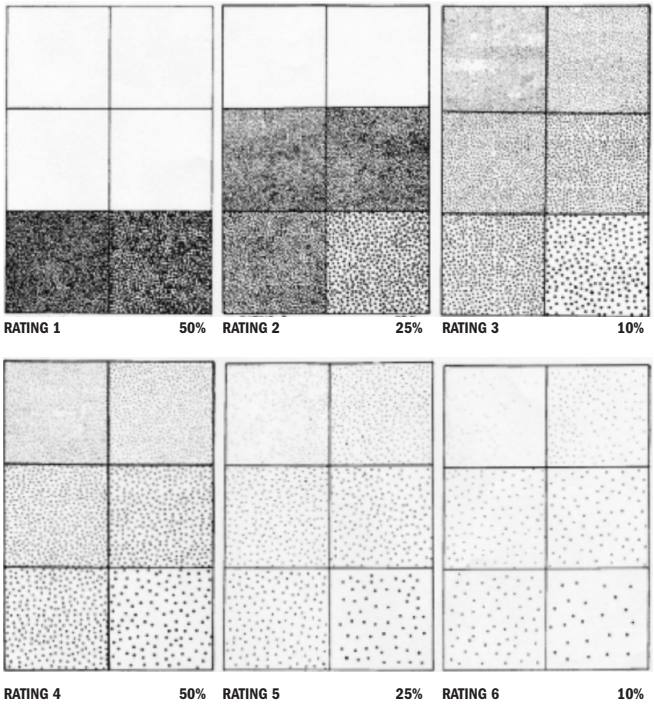
Description of Location of Defects

e = edge.
g = general.

Source: ASTM B537, "Standard Practice for Rating of Electroplated Panels Subjected to Atmospheric Exposure" (West Conshohocken, PA, USA: ASTM International, 2000). Reprinted with permission, copyright ASTM.

RATING OF ELECTROPLATED PANELS SUBJECTED TO ATMOSPHERIC EXPOSURE

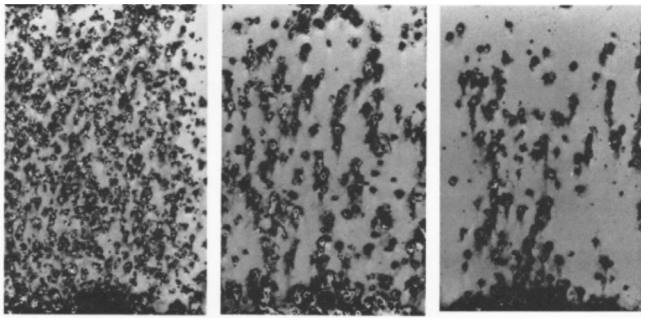
B 537-70 (2007)



RATING OF ELECTROPLATED PANELS SUBJECTED TO ATMOSPHERIC EXPOSURE

B 537-70 (2007)

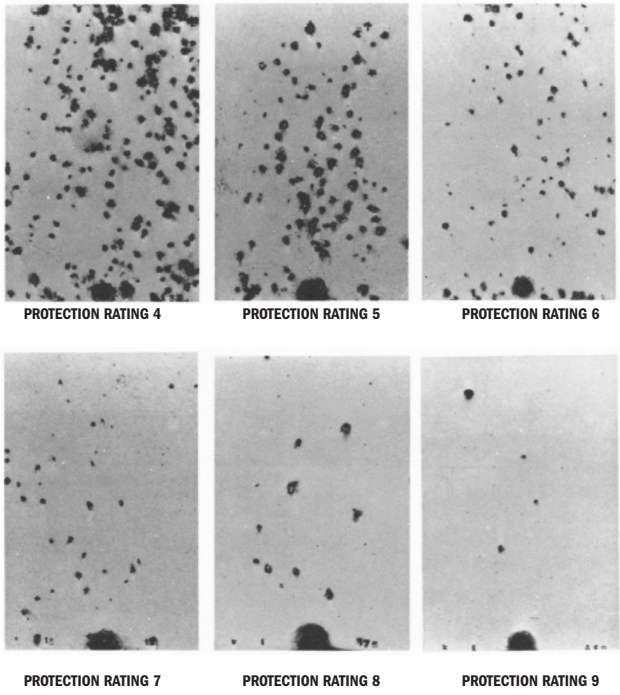
RATING 7 0.5% RATING 8 0.25% RATING 9 0.1%



PROTECTION RATING 1 PROTECTION RATING 2 PROTECTION RATING 3

RATING OF ELECTROPLATED PANELS SUBJECTED TO ATMOSPHERIC EXPOSURE

B 537-70 (2007)



PROTECTION RATING 4 | PROTECTION RATING 5 | PROTECTION RATING 6

PROTECTION RATING 7 | PROTECTION RATING 8 | PROTECTION RATING 9

Source: ASTM B 539-70, "Standard Test Methods for Measuring Resistance of Electrical Connections" ((West Conshohocken, PA, USA: ASTM International, 2007).