

Laboratory Test to Evaluate the Vapor-Inhibiting Ability of Volatile Corrosion Inhibitor Materials for Temporary Protection of Ferrous Metal Surfaces

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Foreword

Volatile corrosion inhibitor (VCI) materials provide temporary corrosion protection for the surfaces of metal parts that are not in contact with the inhibitor. Temporary protection is afforded as long as there is a moderately sealed enclosure containing the metal parts and the source of the VCI, which may be in the enclosing package itself. The duration of protection may be months to years before the parts are removed from the enclosure and put to use, or before a more “permanent” coating such as paint is applied. A referenced NACE Standard Practice SP0487 includes VCI in the context of guidance and best practices for users of interim or temporary corrosion protection methods.

The purpose of this basic test of VCI function is to provide a repeatable one-day assessment of the Vapor Inhibiting Ability (VIA) of VCI materials to protect ferrous metal surfaces from corrosion (rusting), including protection of ferrous metal afforded by components of VCI classified for multi-metal protection. The essence of the test is to: (1) prescribe simple standard conditions of enclosure with vapor transport across a gap containing air, warm water vapor, and the VCI molecules, and (2) provide qualitative visual grading of the efficacy of corrosion protection of standardized low carbon steel specimens, as generally representative of ferrous metals. The method is derived from the military methods in U.S. MIL-STD-3010¹⁻⁴ propagated since the 1970s. It describes practices used for relatively high throughput VIA testing by industrial producers and users of VCIs for quality control and comparisons in product development among competing VCI materials. Provision is also made to test for possible confounding effects of VCI and water absorption (desiccant effects) of VCI paper materials. Several other closely derived test standards were also propagated since the 1970s in other international regions, e.g., German military⁵⁻⁶ and Japanese JIS standards. The variation among these methods can result in different outcomes.⁷⁻⁸ The apparatus and grading scale of the test method are also used in comparisons of VCIs with corrosive contaminants.⁹ Considerations of possibly analogous VIA testing for protection of non-ferrous metals have not proved feasible.¹⁰

Scope

This standard test method evaluates the vapor-inhibiting ability (VIA) of various forms of VCI materials for temporary corrosion protection of ferrous metal surfaces, which is also called the “VIA-Ferrous” test.

Rationale

This VIA test method is for the purpose of qualifying materials that provide VCI functionality using current commercial practices for reproducibility and productivity. This revision no longer includes details that duplicate the 2013 revision of Reference 1 used for military procurement qualification. In particular, this revision no longer includes a Grade 4 level of “no corrosion” by microscopic inspection. It also removes a test in Reference 1 that determines if copper is stained or corroded in contact with VCI materials, since VCI chemistries for which that test was developed are now essentially obsolete.

Referenced Standards and Other Consensus Documents

Unless specifically dated, the latest edition, revision, or amendment of the documents listed in the table below shall apply.

AMPP/NACE/SSPC, www.ampp.org:

NACE SP0487	Considerations in the Selection and Evaluation of Rust Preventives and Vapor Corrosion Inhibitors for Interim (Temporary) Corrosion Protection
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Japanese Standards Association Group (JSA), www.jsa.or.jp/en:

JIS Z 1519	Volatile corrosion inhibitor for iron and steel
JIS Z 1535	Volatile Corrosion Inhibitor for Treated Papers for Iron and Steel
JIS Z 1542	Corrosion inhibitor treated film for iron and steel

In AMPP standards, the terms *shall* and *must* are used to state requirements and are considered mandatory. The term *should* is used to state something that is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

Section 1: General

- 1.1 This standard describes a test method for evaluating the VIA of various forms of materials for providing temporary corrosion inhibition on the surface of ferrous metals by vapor transport of VCI in an enclosed sealed space.
- 1.2 The essence of this standard test method is a relatively quick, inexpensive, and basic laboratory test. The test is labeled as quick, requiring only 24 hours. It is inexpensive, the test apparatus being a reusable sealed jar, compared to testing of parts or subassemblies in long-term controlled atmosphere chamber conditions to simulate conditions in a specific application, or field tests. An experienced laboratory should achieve reasonable and reproducible results in distinguishing between VCI materials that have VIA-Ferrous properties.
- 1.3 This VIA-Ferrous test method is characterized as a basic qualitative test with limited differentiation among VCI materials. Finer differentiations between VCI materials must be made only within a single test. Comparisons between tests run at different times should only use the grading scale described in Figure 9.
- 1.4 A numerical graded rating system is established for describing and reporting the VIA-Ferrous functionality of the VCI sample tested. The user of this VIA-Ferrous test method may specify a pass/fail grade among the three levels described in [Paragraph 6.6](#).

Section 2: Definitions

Desiccant Effects: A VCI material may be packaged with or embedded in paper or corrugated packaging or other substrate that absorbs moisture, acting as a desiccant. This reduction in the humidity level within the sealed test apparatus may confound the results of a VIA test so that a tested VCI may appear to be more effective. Paragraph 6.7 provides for separating VIA and desiccant effects.

Sample: A portion of material taken from a larger quantity as representative of the whole for test purposes. And, for the purposes of this test method, this is a material for evaluation in this vapor-inhibiting ability (VIA) test to determine whether it exhibits VCI properties under these standard test conditions.

Specimen: A prepared portion of a carbon steel cylindrical rod with which a VIA test is to be performed as part of the VIA-Ferrous test apparatus whose test surface is prepared and evaluated to indicate the VIA performance of a VCI sample.

Test Surface: The specifically prepared surface of the steel specimen that is exposed to the enclosed environment in the VIA-Ferrous test apparatus and evaluated for the presence of corrosion (rust) after the specified exposure period.

Vapor-Inhibiting Ability (VIA): The basic and quick (24 hours) pass/fail test for the combination of vapor transport across a gap containing air, distilled water vapor, and VCI molecules, all within an enclosed volume, and corrosion inhibition by the vapor transported component.

Vapor-Inhibiting Ability of Ferrous Metal (VIA-Ferrous): A test of VIA functionality using a standardized steel specimen surface.

Volatile Corrosion Inhibitor (VCI): A chemical substance that acts to reduce corrosion by a combination of (1) volatilization, evaporation, or sublimation from a material, (2) vapor transport of the volatile component in the humid atmosphere of an enclosed environment, and (3) condensation of the volatile component onto surfaces in the space, including adsorption, dissolution and hydrophobic effects on metal surfaces within the space, where the rate of corrosion of metal surfaces is thereby reduced or inhibited. The VCIs are variously called a vapor corrosion inhibitor, a vapor phase inhibitor, a vapor phase corrosion inhibitor, and vapor-transported corrosion inhibitor. VCI materials may be in the form of thin flexible, rigid, or laminate materials, such as VCI-treated paper or VCI-impregnated plastic films, sheets, and bags. Examples of rigid forms of these materials include thicker plastic sheeting of 1,000 µm (40 mils) or more in thickness as well as profile board and molded trays, boxes, or other rigid forms. Other forms of VCI material are powders, tablets, plastic pellets, vapor capsules, diffusers, and emitters, as well as liquids

Water Bath: A pan or tray with tap water at controlled temperature, specified in [Paragraph 6.4](#), in which sealed Test Apparatus jars are placed for stability of their interior ambient temperature.

Section 3: Test Apparatus, Equipment, and Materials

3.1 Test Apparatus

- 3.1.1 The VIA-Ferrous test shall be performed in the “jar-test” apparatus shown and described in Figure 1.¹ Details of the jar lid are shown in [Figure 2](#).
- 3.1.2 Any minor changes to the test apparatus and specimen preparation or the other equipment and materials used to handle the specimens ([Paragraph 3.2](#)) must be tested to verify that the changes do not have adverse effects on test results. A test should be conducted where the “regular” and “alternate” reagents shall be tested in parallel, for verification of suitability of the “alternate” reagent. All changes must be reported as deviations from the standard.

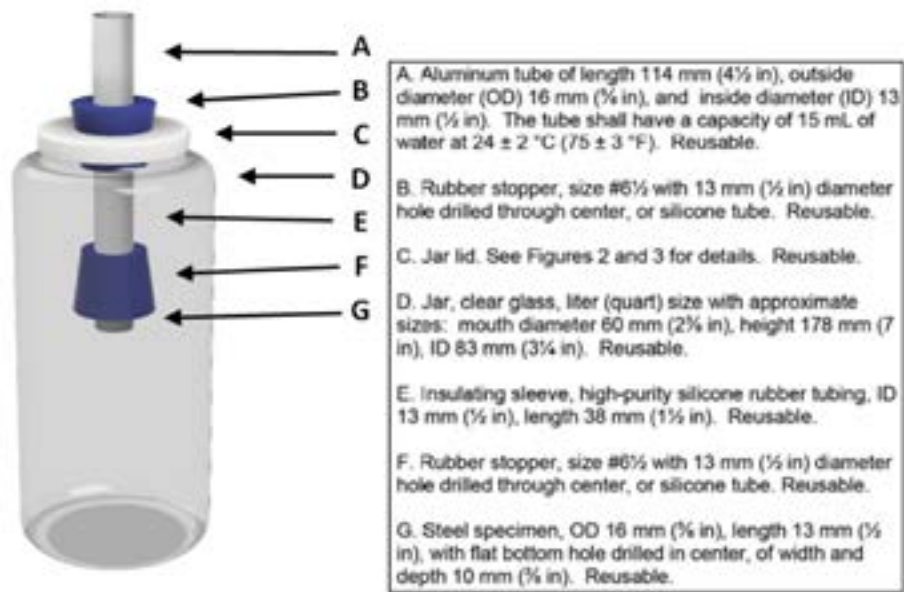


Figure 1: Jar-Test Apparatus Assembly. All Parts of the Specimen Holder Assembly Shall be in Contact with the Adjacent Parts⁽¹⁾

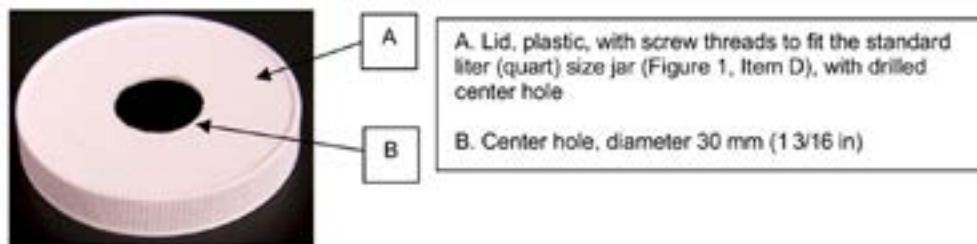


Figure 2: Jar Lid⁽²⁾

3.2 Other Equipment and Materials

Other equipment and materials used to perform this VIA-Ferrous test method include:

- Fume hood, for use when heating methanol to clean steel specimens
- Tubing, high-purity silicone rubber, 13 mm (½ in) ID—insulating sleeve (see Figure 1, Item E)
- 240 grit and 400 grit abrasive paper, silicon carbide, or aluminum oxide for either grit for specimen preparation ([Paragraph 4.2](#))
- Hot plate
- Forceps
- 150 mL or 250 mL beakers
- Watch glass covers to fit beakers
- Absorbent lint-free laboratory wipes
- Graduated cylinders: 10 mL, 50 mL, 100 mL

⁽¹⁾ Reprinted in part from MIL-STD-3010 with permission of U.S. Department of Defense.

⁽²⁾ All figures are used with permission of Northern Technologies International Corporation, 4201 Woodland Rd., PO Box 69, Circle Pines, MN 55014.

- Low-acid transparent adhesive tape
- Small weighing boats
- Plastic sample holders (Figures 3 and 8)
- Desiccator with drying agent
- Water bath
- Latex, vinyl, or rubber gloves
- Safety glasses
- Reagents: methanol (reagent grade $\geq 99.8\%$), glycerin (reagent grade $\geq 99\%$), distilled water, or deionized water.
 - Methanol, while being an ideal solvent for this application, is a hazardous material, highly flammable and toxic through ingestion, skin contact, and inhalation of the fumes. Care should be taken to wear appropriate personal protective equipment and to use a fume hood while methanol is in use.
 - Any alternative reagent used, such as ethanol or isopropyl alcohol, must be clearly reported as a deviation from the standard test method along with the purity used. Acetone has been found to be an unacceptable solvent for this test standard.^{7,8}
- Optional datalogger (Figure 3a) or another internal sensor may be used inside enclosed test jars to monitor temperature and humidity. The datalogger must be designed for use in high humidity. Temperature measurement recommended minimum accuracy: $\pm 1\text{ }^{\circ}\text{C}$ ($\pm 2\text{ }^{\circ}\text{F}$), Resolution: $\pm 0.1\text{ }^{\circ}\text{C}$ ($\pm 0.2\text{ }^{\circ}\text{F}$); Relative Humidity (RH) measurement recommended minimum accuracy: $\pm 5\%$, Resolution: $\pm 0.4\%$; Setting: Capture pairs of temperature and % RH at no more than one such pair of readings per minute with dissipation of no more than 2 mW of power for more than 20 ms when it captures individual pairs of temperature and % RH data.



Figure 3a: Datalogger Inside the Jar-Test Apparatus, Held with Low-Acid Transparent Adhesive Tape



Figure 3b: Example of a Plastic Sample Holder

3.3 Number of Jars Required

- 3.3.1** For each different type of VCI material to be tested at the same time with the identical test setup, prepare three jars for each VCI sample to be tested (triplicates), plus one jar as a control for each set of one or more different VCI samples, or a total of $3n + 1$ jars, where n is the number of different VCI samples of the same type to be tested at the same time with the identical test setup.

3.4 Cleaning of Jar-Test Apparatus

- 3.4.1** Wash each jar-test apparatus item, except the steel specimen, using laboratory-grade detergent before initial use and each reuse. Note: Steel specimen preparation and cleaning procedures are in Section 4.
- 3.4.2** Rinse each item at least three times with tap water, followed by a rinse with distilled water.
- 3.4.3** Dry each item before use.

Section 4: Specimens, Preparation, and Cleaning

4.1 Test Specimens

- 4.1.1** VIA-Ferrous Test Specimens shall be made from one of the following two types of low-carbon, steel round bar stock:
- Unified Numbering System (UNS)⁽³⁾ G10100 (Specimen Type 1010): meeting requirements of Federal Specification QQ-S-698,11 Condition 5
 - UNS G10180 (Specimen Type 1018): meeting requirements of UNS G10180 (AISI⁽⁴⁾) Type 1018 or C1018)
- 4.1.2** For Option 2, specimen preparation by automated machine grinding ([Paragraph 4.2.3](#)), specimens are recommended to be nickel-plated clean steel of a type specified in Paragraph 4.1.1. The automatic polisher does not polish the sides and back of the specimen. High phosphorus electroless nickel plating is recommended but not required to provide corrosion resistance and negligible galvanic effects.

4.2 Test Specimen Preparation

⁽³⁾ Unified Numbering System for Metals and Alloys (UNS). UNS numbers are listed in *Metals & Alloys in the Unified Numbering System*, 14th ed. (Warrendale, PA: SAE International and West Conshohocken, PA: ASTM International, 2023).

⁽⁴⁾ American Iron and Steel Institute (AISI), 1140 Connecticut Ave. NW, Suite 705, Washington, DC 20036, www.steel.org.

4.2.1 Option 1: Hand Grinding and Polishing of Steel Specimens

- 4.2.1.1 Wash each steel specimen using laboratory-grade detergent, rinse with distilled water, rinse with methanol, and allow to air dry.
- 4.2.1.2 Hand clean the sides and inside of each steel specimen with any abrasive paper to remove residual rust.
- 4.2.1.3 Mount the steel specimen in a rubber stopper (like that used in the jar-test apparatus, see [Figure 1](#), Item B or F) such that the flat, undrilled end of the steel specimen (the test surface) and some of the side surface is still visible. The rubber stopper is used as a holder for the specimen during the hand grinding and polishing steps.

NOTE: The abrasive cleaning steps will make the rubber stopper very dirty, so it must not be reused for any subsequent steps without thorough cleaning as described in [Paragraph 3.4](#).

- 4.2.1.4 Firmly hold the rubber stopper with the steel specimen such that the flat, undrilled end of the steel specimen (the test surface) is parallel to the surface of 240-grit abrasive paper. Make even grinding strokes across the abrasive paper with the steel specimen held in the same position. Continue until the test surface of the steel specimen is considered uniformly ground and smooth.
- 4.2.1.5 Change to 400-grit abrasive paper and continue to polish the test surface using firm strokes along separate parallel lines. Hold the steel specimen test surface in a fixed position rotated 90 degrees from where it was held in Paragraph 4.2.1.4. Repeat until uniformly ground and smooth and all traces of 240 grit sanding can no longer be seen.
- 4.2.1.6 Remove the steel specimen from the rubber stopper, wipe the steel specimen clean with surgical gauze.
- 4.2.1.7 Clean and protect each usable steel specimen immediately as described in [Paragraph 4.3](#).

4.2.2 Option 2: Grinding using a rotating table (Figure 4)

- 4.2.2.1 This method allows using the rotating table as a replacement for manual grinding. Opposite to the hand grinding (Option 1, Paragraph 4.2.1), where the metal plug is moved, here, sandpaper is rotating while the specimen is held.

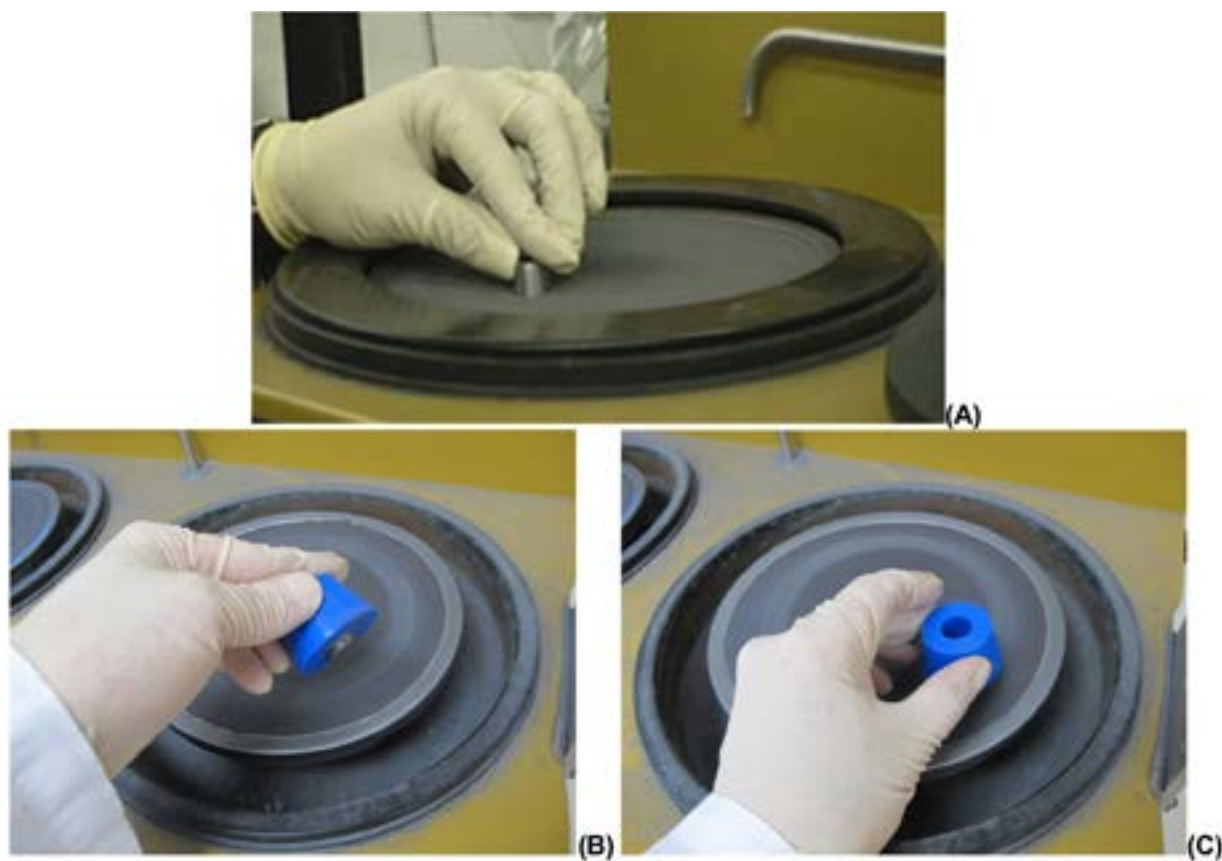


Figure 4: Rotating Table with Handheld Plug

4.2.2.2 Hand clean the sides and inside of each steel specimen with any abrasive paper to remove residual rust.

4.2.2.3.1 (Option A) Mount the steel specimen in a rubber stopper (like that used in the jar-test apparatus, see [Figure 1](#), Item B or F) such that the flat, undrilled end of the steel specimen (the test surface) and some of the side surface is still visible. The rubber stopper is used as a holder for the specimen during the hand grinding and polishing steps (Figure 4A).

NOTE: The abrasive cleaning steps will dirty the rubber stopper, so it must not be reused for any subsequent steps without a thorough cleaning, as described in [Paragraph 3.4](#).

4.2.2.3.2 (Option B) Hold the sample by hand (Figures 4B and 4C).

4.2.2.4 Hold the steel specimen so that the flat, undrilled end of the steel specimen (the test surface) is parallel to the surface of 240 grit abrasive paper. Hold the specimen firmly while the table is on. Do not turn or move. Continue until the test surface of the steel specimen is considered uniformly ground and smooth.

4.2.2.5 Change to 400-grit abrasive paper and continue to polish the test surface using the same procedure as above (Paragraph 4.2.2.4). Repeat until uniformly ground and smooth and all traces of 240-grit sanding can no longer be seen.

4.2.2.6 Remove the steel specimen from the rubber stopper (if used), wipe the steel specimen clean with surgical gauze.

4.2.2.7 Clean and protect each usable steel specimen immediately as described in [Paragraph 4.3](#).

4.2.3 Option 3: Automated Machine Grinding and Polishing of Steel Specimens

This specimen preparation procedure produces reproducibly polished surfaces with high productivity for routine evaluation of production quality and development samples of VCI materials. Figures 5 and 6 show a wheel fixture with metal specimens held firmly in place for an automated grinding and polishing process. The wheel fixture and a platen, to which a sheet of grit sandpaper is adhered by an adhesive backing, move under controlled pressure in contra rotation.

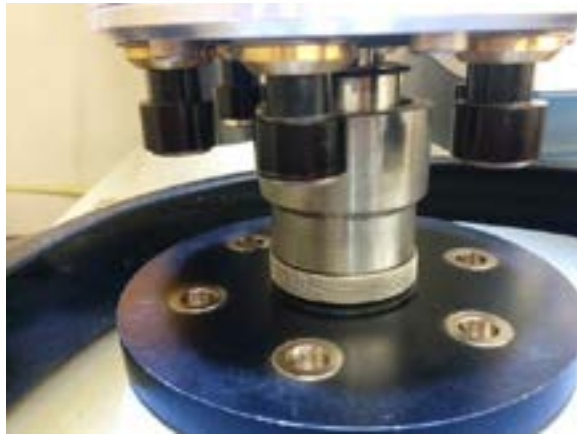


Figure 5: Custom Wheel Fixture of Anodized Aluminum for Firmly Holding Several Metal Specimens

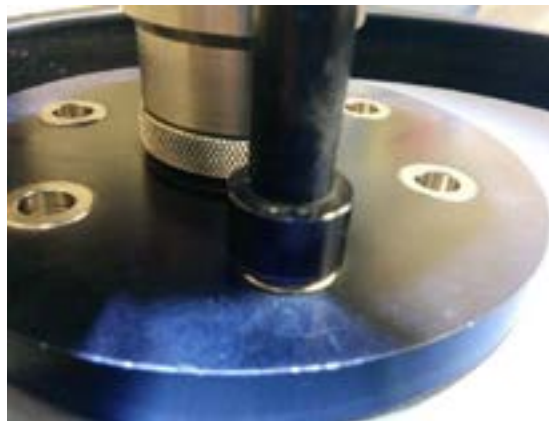


Figure 6: Pistons are Lowered and the Wheel and Sandpaper Platen Turns in Counter Rotation. Generally, the Machine must Be Started before All Pistons are Lowered

4.2.3.1 The following settings of the automated setup are recommended. Alternative settings must be clearly stated in the report: Force 4 lbs, Force type Single, Head speed 60 rpm, Platen speed 150 rpm, and Contra Rotation. Use adhesive-backed 240-grit and 400-grit sandpaper that is appropriate for the automated machine being used. If no automatic holder is available, one may alternatively hold the specimen on the automatic sander. Heavy-duty gloves are recommended to protect hands from injury.

- 4.2.3.2** For the first polishing round, the 240-grit paper must adhere to the aluminum polishing platen. Lower the head and place the test specimens into the slots. Sand these plugs for 1 minute or until uniformly smooth.
- 4.2.3.3** For the second round of polishing, the 400-grit sandpaper must adhere to the aluminum platen. Repeat the polishing steps, increasing the polish time to 2 minutes or until uniformly smooth.
- 4.2.3.4** After the 400 grit polish has finished, the steel specimens are wiped with a methanol-soaked piece of gauze to remove grit debris. As an option, these coupons may then be placed in a beaker filled with methanol.

4.3 Specimen Cleaning

- 4.3.1** Degreasing of newly received specimen: New specimens may be covered in oil and need to be degreased before use. Any degreasing method may be used. Specimens must be free of any oil and should be washed with laboratory-grade detergent and completely dried before the methanol cleaning steps.
- 4.3.2** Cleaning procedure for all steel test specimens
 - 4.3.2.1** Use clean forceps to place the prepared steel specimens into a 150 mL or 250 mL beaker. Place this cleaning beaker under a ventilated hood and add a sufficient volume of methanol to cover the specimens completely.
 - 4.3.2.2** Place the cleaning beaker on a hot plate under the ventilated hood, and cover with a watch glass. Turn the hot plate on low and bring the methanol to a boil. Methanol boils at a very low temperature, 65 °C (150 °F), and its vapors are flammable.
 - 4.3.2.3** Boil specimens in methanol for 60 seconds, and then turn off the hot plate.
 - 4.3.2.4** Pour fresh methanol into another beaker under the ventilated hood. Use clean forceps to remove each steel specimen from the cleaning beaker. Slowly and carefully place the hot specimen into this rinsing beaker containing fresh methanol. A hot specimen may cause methanol to spray or spatter. Wear appropriate personal protective equipment, especially to protect hands and eyes.
 - 4.3.2.5** Check that methanol completely covers all specimens after they have been placed in the rinsing beaker. Add more methanol if needed. Place the rinsing beaker on a hot plate under the ventilated hood. Turn the hot plate on low and bring the methanol to a boil. Methanol boils at a very low temperature, 65 °C (150 °F), and its vapors are flammable.
 - 4.3.2.6** Boil specimens in methanol for 60 seconds, and then turn off the hot plate.
 - 4.3.2.7** Use clean forceps to remove a single specimen and air dry. Several dried specimens can be temporarily held on a clean watch glass or absorbent lint-free laboratory wipe.
 - 4.3.2.8** An absorbent lint-free laboratory wipe, lens paper, or other lint-free laboratory paper is a potential source of contaminants on the specimen test surface that must be tested for detectable VIA-Ferrous effects as described in Paragraph 3.1.2. Contaminated steel specimens should be compared with uncontaminated controls.
 - 4.3.2.9** Repeat the steps in Paragraph 4.3.2.7 for the remaining specimens in the methanol rinsing beaker.

4.3.2.10 If not immediately used, store cleaned specimens in a desiccator. If cleaned specimens are stored for a period longer than 24 h, the specimen preparation and cleaning procedures shall be repeated starting with the final polishing using 400-grit abrasive paper (i.e., [Paragraph 4.2.1.5](#) or [4.2.2.4](#)).

4.3.3 Changes in Specimen Cleaning Procedure. As noted also in [Paragraph 3.1.2](#), any change in the specimen cleaning procedure must be tested to verify that the changes do not have adverse effects on test results and that VIA tests of VCI samples with revised specimen cleaning procedures provide corrosion results comparable to those of VIA tests with the above-specified specimen cleaning procedures. Any change in specimen cleaning procedure must be noted in the test report (Paragraph 8.1.2).

4.3.3.1 Methanol is the preferred specimen rinsing solvent. Should methanol be replaced with ethanol or isopropyl alcohol for this rinse, then any such change must be verified as described in Paragraph 3.1.2 and the change noted in the test report described in Paragraph 8.1.2.

Section 5: Test Solution

5.1 Prepare glycerin solution. The test solution shall be a 26% by volume mixture of reagent-grade glycerin in distilled water, which is intended to produce a vapor with approximately 90% RH in the jar vapor space during the test. This mixture is 30.6 mass-%, its refractive index is about 1.371, and its specific gravity is 1.076, both at about 20 to 25 °C (68 to 77 °F). Table 1 provides examples of the volumes of glycerin and distilled water required to prepare the desired volume of this glycerin-water mixture.

Table 1
Examples of the Volumes of Glycerin and Distilled Water Required to Prepare the Desired Volume of Glycerin-Water Mixture

Approximate Total Volume of Mixture (mL)	Volume of Glycerin (mL)	Volume of Water (mL)		Approximate Total Volume of Mixture (mL)	Volume of Glycerin (mL)	Volume of Water (mL)
50	13	37		300	78	222
100	26	74		350	91	259
150	39	111		400	104	296
200	52	148		450	117	333
250	65	185		500	130	370

5.2 A total of 10 mL of glycerin-water mixture shall be used for each jar. The total volume (mL) of mixture for a test group should be at least $10 \times (n + 1)$, where n is the number of jars in the test group. A fresh batch of glycerin-water mixture should be prepared for each test group and used within 24 hours of production.

Section 6: Test Procedure

6.1 Setup of Jar Test for VCI Film, VCI Paper, or Other Flat, Solid VCI Materials

6.1.1 For each jar, prepare a pair of sample strips from the VCI sample to be tested. Each cut sample strip shall be 25 mm x 150 mm (1.0 in x 6.0 in). Sample strips shall be placed immediately with the remainder of the original material from which they were cut, in their respective protective bags, and closed. Alternatively, each pair of sample strips may be placed into a separate polyethylene bag and closed to prevent possible loss of VCI components. The sample strips shall remain enclosed until just prior to being attached to a jar lid (Paragraph 6.1.3).

6.1.1.1 When the VIA-Ferrous test is to be performed on flat, solid VCI samples that are likely to have desiccant effects by the nature of their porous structure (e.g., thick paper, cardboard, and foam), select one of the test setup options in [Paragraph 6.3](#) to eliminate desiccation effects. When test setup Option A ([Paragraph 6.3.1](#)) is selected, sample strips of each control sample must also be prepared. The control sample shall be made from the same base material from which the VCI sample is made but without the VCI additive.

6.1.2 The following steps should be done quickly and without delay between steps. Use an absorbent lint-free laboratory wipe and clean gloved fingers to insert the prepared and cleaned steel specimen into the rubber stopper or optional silicone tubing with 6 mm (¼ in) of exposed steel length (see Figure 7). Do not add any silicone grease or other lubricant to facilitate this insertion. Repeat for all jars of samples to be tested.



Figure 7: VIA-Ferrous Test Jar Lid (Internal View) Showing Steel Specimen Inserted in the End of the Silicone Tubing with VCI Sample Strips Held in Place with Low-Acid Transparent Adhesive Tape

- 6.1.3** Secure the pair of VCI sample strips to the underside of the jar lid with low-acid transparent adhesive tape.
- 6.1.4** Place the lid on the jar without tightening. Label the lid with an identification number for the sample. Repeat steps in Paragraphs 6.1.3 and 6.1.4 for all jars of samples to be tested.
- 6.1.5** Remove the lid and transfer 10 mL of the glycerin-water mixture into the bottom of each jar, taking care that none of this mixture contacts anything other than the bottom of the jar. Place the lid back onto each jar and tighten securely.
- 6.1.6** For each different type of VCI material to be tested at the same time with the identical test setup, prepare control jars using this same procedure, but without VCI sample strips.
- 6.1.7** Minor alternative procedures that offer incremental productivity improvement may be used. For example, add the glycerin-water mixture to the jar and cover the jar with the inverted lid. Immediately insert the steel specimen and screw the lid tightly onto the jar.

6.2 Setup of Jar Test for VCI Powder, VCI Pellets, VCI Liquid, and VCI Pellets in Pouches

- 6.2.1** Weigh out 50 ± 5 mg of each sample of powder, pellets, liquid, or similar forms of VCI material to be tested into a small weighing boat. (If VCI pellets are in a pouch, the pouch must be a porous material to allow VCI to migrate into the vapor space of the test jar).
- 6.2.1.1** For large pouches of VCI pellets, open the pouch to obtain a sample of the pellets. Small pouches of pellets may be used directly in the test if their mass varies by 10% or less.
- 6.2.1.2** It may be necessary to pulverize and blend large pellets of VCI material to obtain a small representative sample.
- 6.2.2** Transfer 10 mL of the glycerin-water mixture into each jar. Then place an inert sample holder with legs, to support the sample above the glycerin-water mixture level, into the bottom of each jar (see Figure 8). Alternatively, a small beaker, used to hold the sample, may be placed directly into the glycerin-water mixture at the bottom of the jar.
- 6.2.3** If using an inert sample holder, place the weighed sample, either in a small pouch or in a weighing boat, onto the sample holder in the test jar (Figure 8). If using a small beaker, transfer the sample into the beaker. Place the lid on the jar without tightening. Label the lid with an identification number for the sample. Repeat for all jars of samples to be tested.

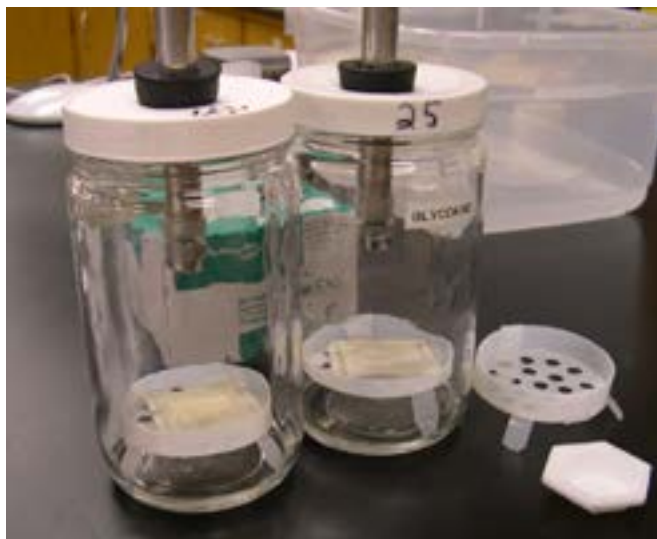


Figure 8: Examples of Sample Holders for Powder, Pellet, and Liquid Forms of VCI Materials.

- (a) Jar on Left Contains VCI Pellets in a Small Pouch Placed on a Sample Holder with Legs to Maintain Position above the Level of the Glycerin-Water Mixture;**
(b) Jar with Control Sample, an Empty Pouch on an Identical Sample Holder; and
(c) Plastic Sample Holder with Holes next to an Example of a Weighing Boat

- 6.2.4** Use an absorbent lint-free laboratory wipe and clean gloved fingers to insert the prepared and cleaned steel specimen into the stopper or optional silicone tubing leaving 6 mm ($\frac{1}{4}$ in) of exposed steel length (Figure 7). Do not add any silicone grease or other lubricant to facilitate this insertion.
- 6.2.5** Place the lid on the jar and tighten securely.

- 6.2.6** Repeat steps in Paragraphs 6.2.4 and 6.2.5 for all jars of samples to be tested.
- 6.2.7** For each different setup used (e.g., with different types of inert sample holders), prepare control jars using this same procedure but without the weighed sample (or small pouch) of VCI material ([Figure 8](#)).

6.3 Test Setup Options for Eliminating Desiccation Effects

There may be special cases when it is necessary to differentiate between VCI and desiccant action for a VCI material that is being tested for its VIA-Ferrous effects.⁷ Examples when this may be needed are for heavy-gauge VCI paper, VCI powder, and some VCI foams that may exhibit temporary desiccating effects in addition to longer-term VCI activity. Two test setup options are available for eliminating the desiccation effects. Option A involves the use of additional control jars to determine the VCI effects by comparing directly between test results with samples of the same base material that are made with and without a given VCI additive. Option B involves the use of a temperature-humidity datalogger, which can provide guidance to ensure that a reasonable degree of saturation of the test sample has been achieved. These two test setup options detailed below serve different purposes to augment the basic test method described in this standard.

- 6.3.1** Option A. Prepare four jars for controls: the normal two jars with no test sample material inside, and two additional jars that contain control samples. The control samples shall be made from the same base material from which the VCI sample is made, but without the VCI additive.
- 6.3.2** Option B. Install a small datalogger (temperature-humidity sensor) on the side of a plastic sample holder and secure it to the inside of the test jar (see [Figure 3a](#)). The datalogger must be positioned vertically so that droplets of condensed water from the test specimen do not fall onto the sensor. The recorded history of temperature and % RH may be used to infer the validity of the test when these data confirm that temperature and % RH conditions described in Paragraph 6.4 are achieved.

6.4 Conditioning of Steel Specimens

- 6.4.1** VCI and moisture saturation period: allow the tightly closed jars to remain undisturbed at 22 ± 3 °C (72 ± 5 °F) for at least 20 h. During this period, the % RH should increase and stabilize at the level determined by the composition of the glycerin-water mixture (Paragraph 5.1).
- 6.4.2** Approximately 30 minutes before the end of the saturation period, prepare at least 16 mL of ice water per jar. Place ice cubes in a container and add water, allowing adequate time for the water to become as cold as possible, typically 0 to 2 °C (32 to 35 °F).
- 6.4.3** Prepare a warm water bath at a temperature of 50 ± 2 °C (122 ± 4 °F). The water level shall be 50 ± 6 mm (2.0 ± 0.2 in). The temperature of the water bath shall be within the stated range limits for all jars.
- 6.4.4** Place each test jar, singular or multiple if able, in the warm water bath for 20 ± 5 s. The length of time in the warm water bath may be varied within this range to compensate for room temperature, a few seconds longer for relatively cooler room temperatures. Remove the jar(s) from the warm water bath and immediately fill its aluminum tube (see [Figure 1](#), Item A) with ice water. Repeat the steps in this paragraph for all jars. The temperature of the bath must be monitored and adjusted as needed to stay within the range specified.

Note: The temperature of the glycerin-water mixture rises only a few degrees during this brief warming, but this small change has a significant effect on accelerating the condensation of water on the steel specimen after the addition of ice water to cool it.
- 6.4.5** Specimen conditioning period: allow the tightly closed jars to remain undisturbed for 3 h at 22 ± 3 °C (72 ± 5 °F). During this period, the RH should increase and stabilize at a level greater than or equal to 90%.

6.5 Visual Observation

- 6.5.1** After the 3 h specimen conditioning period has elapsed, remove the lids from the jars. Condensation of water should be visible on the steel specimens and corrosion should have also occurred on the control specimens, as in Paragraph 6.5.2.
- 6.5.1.1** If there is no visible condensation of water observed on the steel specimen, and no corrosion has occurred on the control specimens, the VIA-Ferrous test of the VCI sample in that jar shall be considered invalid.
- 6.5.1.2** An invalid test must be repeated under other, more severe test conditions. Modifications that may be made to increase the severity of the test conditions include (a) longer saturation time ([Paragraph 6.4.1](#)); (b) warmer water bath ([Paragraph 6.4.3](#)); (c) longer time in the water bath ([Paragraph 6.4.4](#)); or (d) a change in preparation of the steel specimen until condensation and corrosion occur on the control specimens in the jars having no VCI sample. When modified test conditions for which the control specimens exhibit significant corrosion have been established, repeat the VIA-Ferrous tests of VCI samples under these modified conditions. All modifications shall be noted in the report.
- 6.5.2** Examine and rate (Paragraph 6.6) the steel specimens that were exposed in the jars with no VCI sample to establish that these control specimens are corroded.
- 6.5.2.1** The rating for the control specimens must be no better than Grade 0 for the VIA-Ferrous test to be considered valid, i.e., control specimens must be clearly “rusty” for comparison to validate that VCI specimens in other jars have a relative and significant protective effect.
- 6.5.3** If the control specimens are corroded, examine and rate (Paragraph 6.6) the steel specimens that were exposed in jars with VCI samples and those that were exposed in jars that contained the control samples (additional controls used for test setup Option A—see Paragraph 6.3.1).

6.6 Rating System for Steel Specimens

- 6.6.1** The numerical rating system (Grades 0 through 3) shall be used to summarize the performance of the group of steel specimens for evaluation of each VCI sample that is tested. The ratings (grades) are defined below. Figure 9 illustrates typical visual patterns for this assessment.
- 6.6.2** Grade 3 (illustrated in [Figure 9](#)) — Visual examination by unaided normal vision shows no corrosion over essentially all of the surface on tested steel specimens. Grade 3 indicates a good corrosion-protective effect from vapor-transported inhibitor action. Some edge corrosion may be present but must be minimal.
- 6.6.3** Grade 2 (illustrated in Figure 9) — Visual examination by unaided normal vision shows at most a few spots or very small areas of corrosion with a clear difference in corrosion between control specimens and all specimens exposed to the VCI sample. Grade 2 indicates a moderate corrosion-protective effect.
- 6.6.4** Grade 1 (illustrated in Figure 9) — Visual examination by unaided normal vision shows some areas of corrosion distributed across the specimen surface that may include both larger and smaller spots. There may be relatively small differences in corrosion between the control specimens and all of the Test Specimens exposed to the VCI sample. Grade 1 indicates a slight corrosion-protective effect.
- 6.6.5** Grade 0 (illustrated in Figure 9) — Visual examination by unaided normal vision shows all specimens are significantly corroded, with no significant difference in corrosion among control speci-

mens and all Test Specimens that have been exposed to the VCI sample. Grade 0 indicates no corrosion-protective effect. Note Paragraph 6.5.2 requires that all control specimens must be no better than Grade 0 for the VIA-Ferrous test to be considered valid.

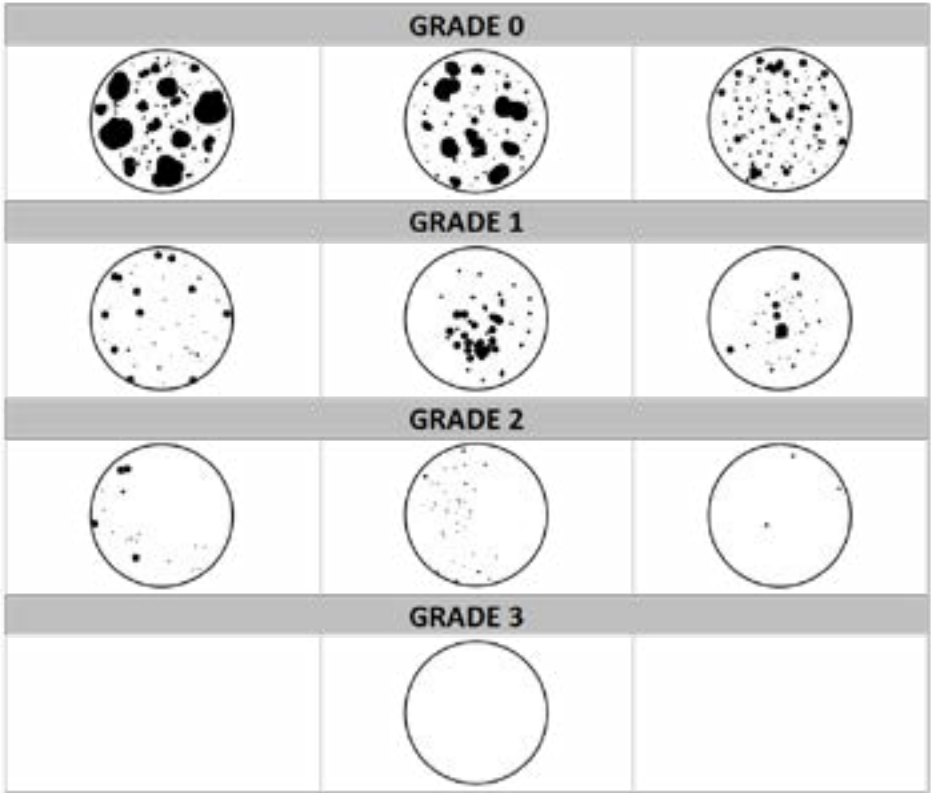


Figure 9: Typical Visual Patterns for Rating the Group of Specimens According to Paragraph 6.6

6.7 Evaluation of Desiccation Effects

6.7.1 Option A

Option A for evaluating desiccation effects uses two sets of controls—the blank control (with no VCI test sample in the test jar) and the base material control (with the base sample in the test jar). Based on the specimen rating results, if the base material control specimens and the blank controls show similar corrosion effects, it may be concluded that desiccation effects are not a factor for the base material used in manufacturing the VCI material. In this case, the corrosion effects of the VCI test sample specimens may be evaluated normally, based on the observed differences between the test and control specimens.

If, however, these two sets of specimens exhibit distinct differences in corrosion effects, with a lower grade rating for the blank control specimens, it may be concluded that desiccation effects exist and that desiccating actions are likely also affecting the corrosion effects observed for the VCI test sample specimens. In this case, the corrosion effects of the VCI test samples should be compared with those of the base material controls. If there are no clear differences in corrosion effects between the VCI test samples and the base material control specimens, the test may be invalid and should be repeated under other more severe test conditions, as described in [Paragraph 6.5.1.2](#).

6.7.2 Option B

Option B for evaluating desiccation effects uses a small datalogger (temperature-humidity sensor) inside the test jar to record temperature and % RH. These data may be used to infer the validity of the test by confirming that temperature and % RH conditions described in [Paragraph 6.4](#) are achieved. If the datalogger shows that the % RH does not reach a value of $90\% \pm 2\%$ by the end of the period of moisture saturation ([Paragraph 6.4.1](#)) or a value greater than or equal to 90% by the end of the specimen conditioning period ([Paragraph 6.4.5](#)), the sample may have some desiccant action, and the VIA-Ferrous test of the associated VCI sample shall be considered invalid.

An invalid test must be repeated under other more severe test conditions, as explained in [Paragraph 6.5.1.2](#).

6.8 Records

- 6.8.1** Record Test Procedures including which options, alternate test setups, alternate reagents, changes to test apparatus, other equipment, materials, and procedures were used.
- 6.8.2** Record the rating assigned to each steel specimen, including those used as normal controls, those additional controls exposed to the base material of the VCI sample without the VCI additive for Option A, and those exposed to the VCI sample.
- 6.8.3** Record the visual appearance of each specimen by photograph.

Section 7: Test Report

7.1 The test report shall include the following:

- 7.1.1** Description of VCI samples.
- 7.1.2** Description of Records (see 6.8.1) including test procedures, preparation options, alternate test setups, and changes to test apparatus, other equipment, materials, and procedures used.
- 7.1.3** Description of specimen type used (see [4.1.1](#)).
- 7.1.4** Description of Rating assigned (see 6.8.2) to each specimen exposed to VCI samples and controls.
- 7.1.5** Photographic records (see 6.8.3).
- 7.1.6** Rating assigned to the group of specimens to assess the effectiveness of each VCI sample tested.

Other Referenced Documents

1. MIL-STD-3010 (latest revision), "Test Procedures for Packaging Materials and Containers," Test Method 4031, "Vapor Inhibiting Ability (VIA) of Volatile Corrosion Inhibitor (VCI) Materials" (Philadelphia, PA: Department of Defense Single Stock Point).
2. MIL-PRF-3420 (latest revision), "Wrapping Materials, Volatile Corrosion Inhibitor Treated, Opaque" (Philadelphia, PA: DODSSP).
3. MIL-PRF-22019 (latest revision), "Barrier Materials, Transparent, Flexible, Sealable, Volatile Corrosion Inhibitor Treated" (Philadelphia, PA: DODSSP).

4. MIL-DTL-22020 (latest revision), "Bags, Transparent, Flexible, Sealable, Volatile Corrosion Inhibitor Treated" (Philadelphia, PA: DODSSP).
5. TL 8135-0002 (latest edition), "Technical Specification for Packaging Material, Corrosion Inhibiting Paper, VCI Paper" (Federal Republic of Germany: Federal Office for Military Technology and Procurement).
6. J. Brekan, N. Smith, and K. Lanzo, "Understanding the Usefulness of German Test Method TL 8135-002 for Various Steel Alloys," CORROSION 2023, paper no. 19169, Houston, TX: AMPP, 2023.
7. J. Brekan, K. Lanzo, and T. Kovacs-Taborda, "Volatile Corrosion Inhibitor (VCI) Testing – Pitfalls and Surprises in Routine Test Methods," CORROSION 2021, paper no. 16323, Houston, TX: AMPP, 2021.
8. M. Fillbach, M. Roe, D. Neuburger, B. Nygaard, and M. Shahlari, "Investigating Specimen Preparation and Cleaning Variables Influencing NACE TM0208-2018 Jar Test Performance," CORROSION 2022, paper no. 17985, Houston, TX: AMPP, 2022.
9. B. Bavarian, A. Avanesian, R. Yakani, L. Reiner, and B. Miksic, "Application of Vapor Phase Corrosion Inhibitors for Contaminated Environments," CORROSION 2019, paper no. 12772, Houston, TX: NACE, 2019,
10. A.J. Henderson and E.Y. Lyublinski, "Evolving Standard Test Methods for Vapor Inhibiting Ability (VIA) for Corrosion Protection," CORROSION 2010, paper no. 10146, Houston, TX: NACE, 2010.
11. Federal Specification QQ-S-698 (latest revision), "Steel, Sheet and Strip, Low Carbon" (Philadelphia, PA: DODSSP).

Bibliography

Commercial Item Description A-A-59441 (latest revision). "Vapor Corrosion Inhibitors." Philadelphia, PA: DODSSP.

Nathan, C.C., ed., *Corrosion Inhibitors*. Houston, TX: NACE, 1973. D.F. Knaack and D. Brooks. "Inhibitors for Temporary Protection, Part 2—Vapor Phase Corrosion Inhibitors," pp. 224–227.

Reve, R.W., ed., *Uhlig's Corrosion Handbook*. 2nd ed. New York, NY: John Wiley & Sons, Inc., 2000. Chapter 59, "Corrosion Inhibitors."

Roberge, P.R., ed., *Handbook of Corrosion Engineering*. New York, NY: The McGraw-Hill Companies, 2000. Chapter 10, "Corrosion Inhibitors."