Standard
Test Method

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 are widely used to provide temporary corrosion protection for the surfaces of ferrous and nonferrous metal parts. "Temporary" refers to conditioning the environment enclosing the metal parts for a period, usually months to years, before the parts are put to their ultimate use, or before a "permanent" coating such as paint is applied. VCI materials compete with alternative temporary corrosion protection methods that include oils, greases, and waxes sometimes called rust preventives or corrosion preventive compounds; various aqueous solutions and coatings; and combinations of these coatings with desiccants and "barrier" packaging. Some of these alternatives may incorporate a VCI function, and others claim or imply VCI function in a name, but may function primarily by contact-inhibiting properties. A test of VCI efficacy must reasonably separate VCI effects (the combination of vapor transport and corrosion protection) from other mechanisms.

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 subsequently herein called the "VIA-Ferrous" test. It can be performed reproducibly with relatively simple and low-cost apparatus. The VIA-Ferrous test provides for standard conditions in a test jar of water-saturated, warm air without the presence of accelerating contaminants. This test method evaluates the combination of (1) vapor transport across a gap containing air, water vapor, and VCI, and (2) corrosion protection. Two options are included to discern the possible desiccating effects of some types of VCI materials, such as VCI paper, as a refinement of this test method. This test method uses two standard low-carbon steels as representative of the broad class of ferrous metals. An optional compatibility check that can be performed to determine whether a VCI-treated barrier material (e.g., film, paper) that is intended for temporary protection of ferrous metals causes corrosion of copper is included. The test method is based largely on U.S. MIL-STD-3010A, Test Method 4031,[1] with modifications and options from related standards[2–5] and practices of members of Technology Exchange Group (TEG) 093X. These practices included details of preparation of VCI material samples and steel specimens and interpretation of results. Similar VIA test methods are currently in wide use for basic qualification of VCI materials.[6–8]

This standard test method is intended for use by VCI material manufacturers and users. It can be used for basic pass/fail qualification tests by production, quality assurance, user, or corrosion specialist laboratories to determine the VIA of VCI materials to protect ferrous metal surfaces from corrosion (rusting), including the component of ferrous metal protection afforded by multimetal VCI materials.

This VIA-Ferrous test is the first of a planned sequence of three standard test methods to evaluate the VIA of VCI materials. Completion and approval of this standard is a prerequisite for developing a standard test method to evaluate the VIA of VCI materials to protect representative nonferrous metal surfaces from corrosion (VIA-Nonferrous test), and another test method to include the presence of atmospheric contaminants that can accelerate corrosion of the metals.

This standard was prepared by Task Group (TG) 215—Volatile Corrosion Inhibitors (VCIs), which is administered by Specific Technology Group (STG) 61—Inhibition: Corrosion and Scaling and sponsored by STG 46—Building Systems. It is published by NACE International under the auspices of STG 61.

In NACE standards, the terms *shall, must*, *should,* and *may* are used in accordance with the definitions of these terms in the *NACE Publications Style Manual.* The terms *shall* and *must* are used to state a requirement, and are considered mandatory. The term *should* is used to state something good and is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

NACE International
Standard Test Method

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Contents

Section 1: General

1.1 This standard describes a test method for evaluating the VIA of various forms of VCI materials to reduce corrosion on the surface of ferrous metals. This standard is applicable to VCI materials in the form of thin flexible, rigid, or laminate materials. Examples of thin flexible VCI materials are VCI-treated paper or VCI-impregnated plastic films, sheets, and bags. Examples of more rigid materials include thicker plastic sheeting of 1,000 µm (40 mil) or more in thickness as well as profile board and molded trays, boxes, and other rigid forms. This standard is also applicable to other forms of VCI material such as powders, tablets, plastic pellets, vapor capsules, diffusers, and emitters, as well as liquids that have vapor-inhibiting properties, perhaps in addition to contact inhibitor effects.

1.2 The essence of this standard is a relatively quick, inexpensive, and basic laboratory test in a reusable sealed jar to determine the VIA of various VCI materials to reduce corrosion on the surface of ferrous metals (VIA-Ferrous test). A competent laboratory should be able to achieve reasonable and reproducible results in distinguishing between VCI materials that have VIA-Ferrous properties and those that do not, including the VIA-Ferrous performance component of multimetal VCI materials. Materials that perform well in this test exhibit a combined effect of (1) vapor transport (without contact between the VCI material sample and the test surface of the steel specimen) and (2) corrosion inhibition on the surface of the steel specimen exposed to conditions of water-saturated, warm air followed by condensing water.

1.3 This VIA-Ferrous test method provides a basic qualitative test with limited differentiation among VCI materials. Finer differentiation and comparisons of relative performance among VCI materials is beyond the scope of this test method.

1.4 A numerical rating system has been established for describing and reporting the VIA efficacy of the VCI material sample tested. The user of this VIA-Ferrous test method may specify a pass/fail criterion associated with this numerical rating system. The user must select the pass/fail criterion, as described in Paragraph 6.6, to be used for the VIA-Ferrous test.

1.5 An optional compatibility check may be performed to determine whether a VCI-treated barrier material (e.g., film, paper) that is intended for temporary protection of ferrous metals causes corrosion of copper (see Section 7). The copper compatibility check is not required unless specified by manufacturer or user of the VCI-treated barrier material. Further tests related to nonferrous metals are beyond the scope of this VCI-Ferrous test standard.

Section 2: Definitions

Corrosion: The deterioration of a material, usually a metal, that results from a reaction with its environment.

Corrosion inhibitor: A chemical substance or combination of substances that, when present in the environment, prevents or reduces corrosion.

Rust: Corrosion product consisting of various iron oxides and hydrated iron oxides. (This term properly applies only to iron and ferrous alloys.)

Sample: A volatile corrosion inhibitor (VCI) material prepared for evaluation in this vapor-inhibiting ability (VIA) test.

Specimen: A metal part of the VIA-Ferrous test apparatus whose test surface is prepared and evaluated to indicate VIA performance of a VCI material 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.

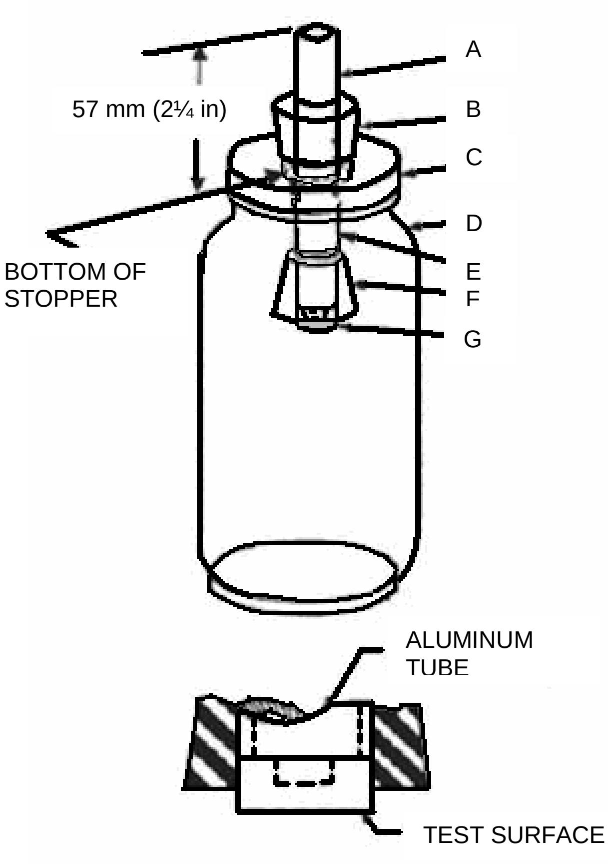
Volatile corrosion inhibitor (VCI): A chemical substance that acts to reduce corrosion by a combination of volatilization from a VCI material, vapor transport in the atmosphere of an enclosed environment, and condensation onto surfaces in the space, including absorption, dissolution, and hydrophobic effects on metal surfaces, where the rate of corrosion of metal surfaces is thereby inhibited; also called vapor-phase inhibitor, vapor-phase corrosion inhibitor, and vapor-transported corrosion inhibitor.

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 jar lids are shown and described in Figures 2 and 3.

3.1.2 Any changes to the materials used in the VIA-Ferrous test apparatus, or the other equipment and materials used to handle the specimens (see Paragraph 3.2), must be tested to verify that the new materials do not have adverse effects on test results, and that VIA tests of VCI material samples with new materials in the test apparatus provide comparable corrosion results to the specified materials.



A. Aluminum tube of length 114 mm (4½ in), OD 16 mm (⅝ in), and ID 13 mm (½ in). The tube shall have a capacity of 16 mL of water at 24 ±2°C (75 ±3°F). Reusable.

B. Rubber stopper, size #6½ with 13-mm (½-in) diameter hole drilled through the center. Reusable.

C. Jar lid. See Figures 2 and 3 for details. Reusable.

D. Jar, clear glass, liter (quart) size with approximate sizes: mouth diameter 60 mm (2⅜ in), height 178 mm (7 in), ID 83 mm (3¼ in). Reusable.

E. Insulating sleeve, high-purity silicone rubber tubing, ID 13 mm (½ in), length 38 mm (1½ in). Reusable.

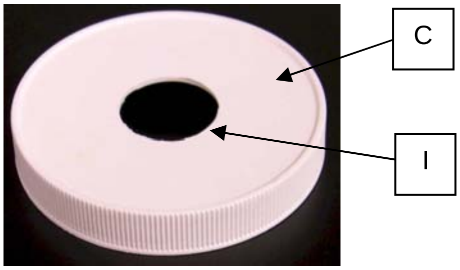
F. Rubber stopper, size #6½ with 13-mm (½-in) diameter hole drilled through the center. Reusable.

G. Steel specimen, OD 16 mm (⅝ in), length 13 mm (½ in), with flat bottom hole drilled in center to depth 10 mm (⅜ in). Reusable.

Lower drawing: Enlarged view of bottom of specimen holder assembly.

FIGURE 1: Jar-test apparatus with enlarged view of bottom of specimen holder assembly. All parts of the specimen holder assembly shall be in contact with the adjacent parts.[(1)]

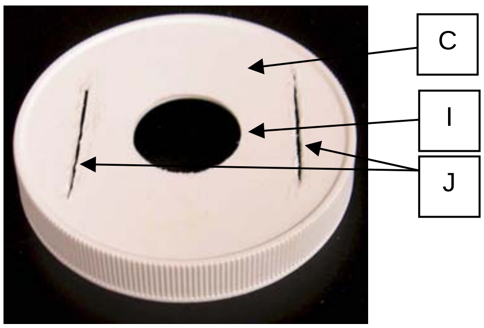
[(1)] Reprinted with permission from U.S. Department of Defense Standard MIL-STD-3010.



C. Lid, plastic, with screw threads to fit the standard liter (quart) size jar (Figure 1, Item D), with drilled center hole

I. Center hole, diameter 30 mm (1 3/16 in)

FIGURE 2: Jar lid used when VCI films and papers are taped to the inside of lid.(2)



C. Lid, plastic, with screw threads to fit the standard liter (quart) size jar (Figure 1, Item D), with drilled center hole

I. Center hole, diameter 30 mm (1 3/16 in)

J. Slots cut through lid, typical length 38 mm (1½ in) and maximum width 6 mm (¼ in)

FIGURE 3: Jar lid used when VCI films and papers are taped to the outside of lid.(3)

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 mineral spirits and methanol to clean steel specimens
- Drill or lathe for optional specimen preparation (see Paragraph 4.2.2)
- Plastic spindle, made from round rod and tapered to a tight fit to the opening in the back of the steel specimen, to hold the specimen in the chuck of a lathe or drill press (see Paragraph 4.2.2)
- Tubing, high-purity silicone rubber, 13 mm (½ in) inside diameter (ID)—insulating sleeve (see Figure 1, Item E); also may be substituted for rubber stopper (see Figure 1, Item F).
- 240 grit and 400 grit abrasive paper, silicon carbide or aluminum oxide for either grit
- Hot plate
- Forceps
- 150-mL or 250-mL beakers
- Watch glass covers to fit beakers
- Absorbent lint-free laboratory wipes
- Heat gun or hair dryer (optional)
- Graduated cylinders: 10 mL, 50 mL, 100 mL
- Low-acid transparent adhesive tape
- Small weighing boats
- Plastic sample holders (see Figure 4)
- Desiccator with drying agent
- Dataloggers (see Figure 4). Specifications: Temperature Measurements—Accuracy: ±1°C (±2°F), Resolution: ±0.1°C (±0.2°F); RH Measurements—Accuracy: ± 5%, Resolution:

(2) Figures 2-11 furnished by Northern Technologies International Corporation, 4201 Woodland Rd., PO Box 69, Circle Pines, MN 55014.
(3) See Footnote (2) for source information.

±0.4%; Setting: Capture pairs of temperature—% relative humidity (RH) at no more than one such pair of readings per minute. Must dissipate no more than 2 mW of power for more than 20 ms when it captures individual pairs of temperature and %RH data.

- Water bath
- Glass stirring rods
- Latex, vinyl, or rubber gloves
- Safety glasses
- Reagents: mineral spirits, methanol, glycerin, distilled water
- Alternate reagents: ethanol, isopropanol, acetone (may be used as alternates to methanol for rinsing specimens in Paragraph 4.3). Use of one of these alternate reagents constitutes a change in the materials used in the VIA-Ferrous test. Specimens rinsed with an alternate reagent must be tested for detectable VIA-Ferrous effects as described in Paragraph 3.1.2.

3.3 Number of Jars Required

3.3.1 For Option 1 (see Paragraph 4.2.1) that uses hand grinding and polishing of steel specimens. 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 material sample to be tested (triplicates) plus two jars for controls (duplicates) for each set of one or more different VCI material samples, or a total of 3n + 2 jars, where n is the number of different VCI material samples of the same type to be tested at the same time with the identical test setup.

3.3.2 For Option 2 (see Paragraph 4.2.2) that uses machine-aided grinding and polishing of steel specimens. For each different type of VCI material to be tested at the same time with the identical test setup, prepare at least two, preferably three, jars for each VCI material sample to be tested (duplicates or triplicates) plus two jars for controls (duplicates) for each set of one or more different VCI material samples, or a total of 2n + 2 (preferably 3n + 2) jars, where n is the number of different VCI material samples of the same type to be tested at the same time with the identical test setup. For example, prepare a minimum of (2 × 2) + 2 = 6 jars, but preferably (3 × 2) + 2 = 8 jars, for a test of two different VCI material samples of the same type and the controls.

Enlarged view of plastic sample holder

FIGURE 4: Datalogger inside the jar-test apparatus, held in place with low-acid transparent adhesive tape.[4]

3.4 Cleaning of Jar-Test Apparatus

3.4.1 Wash each jar-test apparatus item, except the steel specimen, using laboratory grade detergent prior to initial use and each reuse. Note: Steel specimen preparation and cleaning procedures are in Section 4.

[4] See Footnote (2) for source information.

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 prior to use.

Section 4: Specimens, Preparation, and Cleaning

4.1 Specimens

4.1.1 VIA-Ferrous test specimens shall be made from one of the following two types of low-carbon, cold-finished steel round bar stock

- Specimen Type 1010: meeting requirements of Federal Specification QQ-S-698,[9] Condition 5
- Specimen Type 1018: meeting requirements of AISI[(5)] Type 1018 or C1018

NOTE: Carbon content range for Type 1010 and Type 1018 steel is 0.08 to 0.20%.

4.1.2 The selected round bar stock shall be machined to fit into the rubber stopper or silicone tubing of the specimen holder assembly (see Figure 1, Items B and F).

4.2 Specimen Preparation

4.2.1 Option 1. Hand Grinding and Polishing of Steel Specimens

The following specimen preparation procedure shall be used when Grade 4 (see Paragraphs 6.6.1 and 6.6.2) is the criterion specified by the user to judge whether a VCI material sample passes the VIA-Ferrous test. This specimen preparation procedure produces polished and shiny test surfaces with high reproducibility of finish.

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 (similar to 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 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 a single upward or downward grinding stroke along one side of the abrasive paper. Make subsequent grinding strokes parallel to and in the same direction as the previous stroke, with the steel specimen held in the same position each time. Continue until the test surface of the steel specimen is considered to be 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 that is about 90 degrees to the position in which it was held in Paragraph 4.2.1.4. Repeat as needed.

4.2.1.6 Remove the steel specimen from the rubber stopper, wipe the steel specimen clean with surgical gauze, and examine the polished test surface under 10X to 20X magnification. Usable steel specimens shall have a test surface with no abrasion marks or corrosion spots present, no traces of scratches from the 240 grit abrasive, and a surface roughness less than approximately 250 nm (10 µin) root mean square (rms).

4.2.1.7 Clean and protect each usable steel specimen immediately as described in Paragraph 4.3.

4.2.2 Option 2. Machine-Aided Grinding and Polishing of Steel Specimens

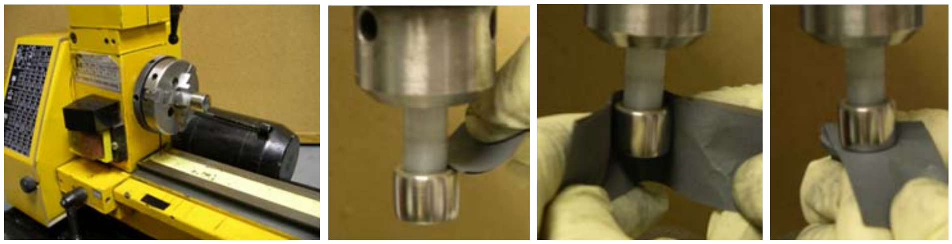
The following specimen preparation procedure shall be used when either Grade 3 or Grade 2 (see Paragraphs 6.6.1 and 6.6.2) is the criterion specified by the user to judge whether a VCI material sample passes the VIA-Ferrous test. This specimen preparation procedure produces adequately polished and shiny surfaces for repeatable quality inspections of VCI material production and development samples.

[(5)] American Iron and Steel Institute (AISI), 1140 Connecticut Ave. NW, Suite 705, Washington, DC 20036.

4.2.2.1 Hand clean inside each steel specimen with any abrasive paper to remove residual rust.

4.2.2.2 Check the setting on the drill press or lathe to ensure rotation at the highest speed. Place a plastic spindle in the chuck with the steel specimen on the spindle (see Figure 5).

4.2.2.3 Turn on the drill press or lathe. Clean the steel specimen outside surfaces (undrilled end test surface and sides) using the 240 grit abrasive paper for rough grinding (see Figure 5).



(a) (b) (c) (d)

FIGURE 5: Steel specimen on spindle in chuck of a horizontal lathe (a) and in chuck of a vertical drill press being polished on the top (b), side (c), and bottom (d) with abrasive paper.[6]

4.2.2.4 Change to 400 grit abrasive paper for final polishing (see Figure 5). Turn off the drill press or lathe when polishing is complete.

4.2.2.5 Remove the steel specimen from the plastic spindle, wipe the steel specimen clean with surgical gauze, and examine the polished test surface visually. Usable steel specimens shall have a fully polished test surface with a very shiny mirror-like finish when examined by unaided normal vision.

4.2.2.6 Clean and protect each usable steel specimen immediately as described in Paragraph 4.3.

4.3 Specimen Cleaning

The following specimen cleaning procedure uses solvents and methods that reflect historical baselines of test data in individual laboratories that have evolved from successful use of U.S. MIL-STD-3010A, Test Method 4031.

4.3.1 Use clean forceps to place the prepared steel specimens into a 150-mL or 250-mL beaker (see Figure 6). Place this cleaning beaker under a ventilated hood, and add a sufficient volume of mineral spirits to cover the specimens completely.

[6] See Footnote (2) for source information.

FIGURE 6: Forceps being used to place prepared steel specimens into the cleaning beaker.[7]

4.3.2 Place the cleaning beaker on a hot plate under the ventilated hood, cover with a watch glass, and turn on the hot plate (see Figure 7). Heat until the mineral spirits start to get hot (small bubbles appear), then turn the hot plate to low. Leave the cleaning beaker containing hot mineral spirits and specimens on the hot plate for 30 seconds, and then remove the cleaning beaker from the hot plate.

FIGURE 7: Hot plate with cleaning beaker covered with watch glass to reduce evaporation.[8]

4.3.3 Pour methanol into another beaker under the ventilated hood. Use clean forceps to remove each specimen from the cleaning beaker containing mineral spirits. Gently shake off excess mineral spirits (to minimize contamination of the methanol), and then slowly and carefully place the hot specimen into this rinsing beaker containing methanol. A hot specimen may cause methanol to spray or spatter. Wear appropriate personal protective equipment, especially to protect hands and eyes.

4.3.4 Check that methanol completely covers all specimens after they have been placed in the methanol rinsing beaker. Add more methanol if needed. Place rinsing beaker with methanol on a hot plate under the ventilated hood. Turn hot plate on low and bring methanol to a boil. Methanol boils at very low temperature and its vapors are flammable.

[7] See Footnote (2) for source information.
[8] See Footnote (2) for source information.

4.3.5 Boil specimens in methanol for 60 seconds, and then turn off the hot plate.

4.3.6 Use clean forceps to remove a single specimen. Air dry the specimen for at least 30 seconds using either a heat gun or hair dryer (see Figure 8).

FIGURE 8: Specimen, held with clean forceps in a gloved hand, being dried by a heat gun. Several dried specimens are temporarily held on an absorbent lint-free laboratory wipe.[(9)]

4.3.6.1 Use of a heat gun or hair dryer 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. Contamination samples should be compared with uncontaminated controls.

4.3.7 Handle specimens only with clean forceps or a gloved hand with an absorbent lint-free laboratory wipe between the specimen and the glove. Place the dried specimens on an absorbent lint-free laboratory wipe after cleaning.

4.3.7.1 Use of 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. Contamination samples should be compared with uncontaminated controls.

4.3.8 Repeat the steps in Paragraphs 4.3.6 and 4.3.7 for the remaining specimens in the methanol rinsing beaker.

4.3.9 Store cleaned specimens in a desiccator. If cleaned specimens are stored for a period greater than 24 hours, 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).

4.3.10 Changes in Specimen Cleaning Procedure. Any change in the above 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 material samples with revised specimen cleaning procedures provide comparable corrosion results as VIA tests with the above specified specimen cleaning procedures. Any change in specimen cleaning procedure must be noted in the test report (see Paragraph 8.1.2).

4.3.10.1 Ultrasonic cleaning (cavitation agitation) with mineral spirits should not be used. Hot mineral spirits adequately remove contamination from polished specimens.

4.3.10.2 Methanol is the specimen rinsing solvent preferred by most laboratories. Other laboratories may prefer to replace methanol with ethanol, isopropyl alcohol, or acetone.

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(9) See Footnote (2) for source information.

Section 5: Test Solution

5.1 The test solution shall be a 26 percent 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. 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, although the mixture may be preserved with 0.1% copper sulfate.

5.3 Prepare a batch of glycerin-water mixture as follows:

5.3.1 Measure the required volume of glycerin in one graduated cylinder and pour it into a beaker with sufficient capacity to hold the desired volume of glycerin-water mixture. To ensure that all the glycerin is removed from the graduated cylinder, pour 10 mL of distilled water into the graduated cylinder used for the glycerin; shake carefully and pour the solution into the beaker. Do this several times until the glycerin is fully transferred from the graduated cylinder, keeping track of the amount of distilled water used.

5.3.2 Using another clean graduated cylinder, measure the remaining volume of distilled water required to prepare the desired volume of glycerin-water mixture and pour it into the beaker.

5.3.3 Use a glass rod to stir the viscous glycerin and the distilled water until the solution is homogenously mixed.

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 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 (see Paragraph 6.1.3).

6.1.1.1 When the VIA-Ferrous test is to be performed on flat, solid VCI material samples that are likely to have desiccant action 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 (see Paragraph 6.3.1) is selected, sample strips of each control material sample must also be prepared. The control material sample shall be made from the same base material from which the VCI material sample is made, but without the VCI additive.

6.1.2 Use an absorbent lint-free laboratory wipe and 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 9). Do not add any silicone grease or other lubricant to facilitate this insertion. Repeat for all jars of samples to be tested.

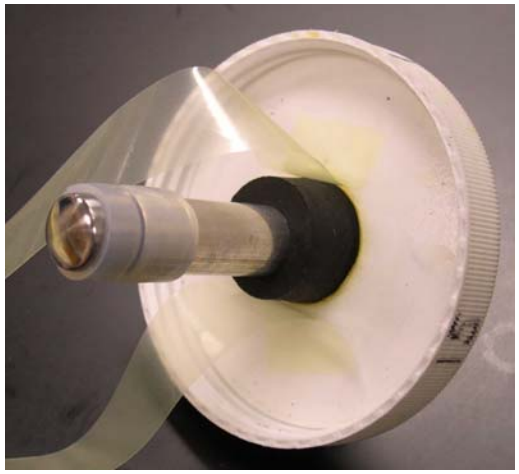


FIGURE 9: VIA-Ferrous test jar lid (internal view) showing steel specimen inserted in the end of the silicone tubing with VCI material sample strips held in place with low-acid transparent adhesive tape.[10]

6.1.3 Secure the pair of VCI material sample strips to the underside of the unslotted jar lid (see Figures 2 and 9) with low-acid transparent adhesive tape. Alternatively, use a jar lid with slots (see Figure 3) and insert the VCI material sample strips through these slots in the lid. Hold these sample strips in position by forming a 6-mm (¼-in) tab at one end of each strip and tape these tabs to the outer surface of the lid (see Figure 10). The low-acid transparent tape used to secure the sample strips in Figure 10 must be placed so that the tape overlaps all edges of the sample strips and also completely seals the slots in the jar lid.

FIGURE 10: VIA-Ferrous test jar lid (external view) showing VCI material sample strips inserted through slots in lid and held in place with low-acid transparent adhesive tape.[11]

[10] See Footnote (2) for source information.
[11] See Footnote (2) for source information.

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 material sample strips. Control jar lids must be unslotted or the slots must be completely sealed with low-acid transparent tape.

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 weigh boat. (If VCI pellets are in a pouch, 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 11). Alternatively, a small beaker, used to hold the sample, may be placed directly into the glycerin-water mixture on 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 weigh boat, onto the sample holder in the test jar (see Figure 11). 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.

(a) (b) (c)

FIGURE 11: Examples of sample holders for powder, pellet, and liquid forms of VCI materials.[12]
- **(a) Jar on left contains VCI pellets in a small pouch that is 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) Typical plastic sample holder with holes next to an example of a weighing boat.**

[12] See Footnote (2) for source information.

6.2.4 Use an absorbent lint-free laboratory wipe and gloved fingers to insert the prepared and cleaned steel specimen into the stopper or optional silicone tubing leaving 6 mm (¼ in) of exposed steel length (see Figure 9). 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. Repeat steps in Paragraphs 6.2.4 and 6.2.5 for all jars of samples to be tested.

6.2.6 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 (see Figure 11).

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. 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 material samples. The control material samples shall be made from the same base material from which the VCI material 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 4). The datalogger must be positioned vertically so that droplets of condensed water from the 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 hours. During this period of time, the %RH should increase and stabilize at the level determined by the composition of the glycerin-water mixture (see 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, one at a time, in the warm water bath for 5 to 20 seconds. 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 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.

Note: The temperature of the glycerin-water rises only a few degrees during this brief warming, but this small change has a significant effect on accelerating 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 hours at 22 ±3°C (72 ±5°F). During this period of time, 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-hour specimen-conditioning period has elapsed, remove the lids from the jars. Condensation of water should be visible on the steel specimens.

6.5.1.1 If there is no condensation of water observed on the steel specimen, the VIA-Ferrous test of the VCI material 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 (see Paragraph 6.4.1); (b) warmer water bath (see Paragraph 6.4.3); (c) longer time in water bath (see Paragraph 6.4.4); or (d) a change in preparation of the steel specimen until corrosion occurs on the control specimens in the jars having no VCI material sample. When modified test conditions for which the control specimens exhibit significant corrosion have been established, repeat the VIA-Ferrous tests of VCI material samples under these modified conditions.

6.5.2 Examine and rate (see Paragraph 6.6) the steel specimens that were exposed in the jars with no VCI material sample to establish that these control specimens 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.

6.5.3 If the control specimens are corroded, examine and rate (see Paragraph 6.6) the steel specimens that were exposed in jars with VCI material samples and those that were exposed in jars that contained the control material samples (additional controls used for test setup Option A—see Paragraph 6.3.1).

6.6 Rating System for Steel Specimens

6.6.1 Assign a numerical rating (grade) to each steel specimen exposed in the test in accordance with the corrosion criteria set forth in Table 2.

6.6.1.1 Grades 0 through 3 shall be assigned based on visual examination by unaided normal vision. Steel specimens evaluated using visual examination may have been prepared by machine-aided grinding and polishing, in accordance with Option 2 (see Paragraph 4.2.2).

6.6.1.2 Grade 4 shall be assigned based on a microscopic examination at a magnification of 10X. NOTE: Steel specimens evaluated using magnification and microscopic examination shall have been prepared by hand grinding and polishing of steel specimens in accordance with Option 1 (see Paragraph 4.2.1).

Table 2: Corrosion Criteria for Rating Steel Specimens

Corrosion Criteria	Grade 4	Grade 3	Grade 2	Grade 1	Grade 0
Number of corrosion spots	≤3 (A)	0	≤3 (B)	>3(C)	>5(D)
Corroded surface area (% of total)	Not Applicable	0	<1	1 to 10	>10

(A) Based on microscopic examination in accordance with Paragraph 6.6.2.1
(B) Based on visual examination by unaided normal vision in accordance with Paragraph 6.6.2.3
(C) Based on visual examination by unaided normal vision in accordance with Paragraph 6.6.2.4
(D) Based on visual examination by unaided normal vision in accordance with Paragraph 6.6.2.5

6.6.1.3 A spot of corrosion is defined as a localized change in the test surface, such as pitting or etching or the formation of loose or granular particles, accompanied by the red-orange appearance associated with corrosion products commonly called rust. A more subtle distinction may be made in judgment of Grades 3 and 4 at the earliest onset of corrosion. The distinction is whether a relatively uniform and slight stain has begun to form over a large portion of the test surface of the specimen, or whether a more localized rust-colored area is accompanied by the slightest detectable change of surface finish. Each laboratory must ultimately determine how it will consistently distinguish between a slight stain and a barely detectable rust spot.

6.6.2 The numerical rating system (Grades 0 through 4) also may be used to summarize the performance of the group of steel specimens used to evaluate each VCI material sample. For this usage, the ratings (grades) are defined below. Figure 12 illustrates typical visual patterns for this assessment.[4]

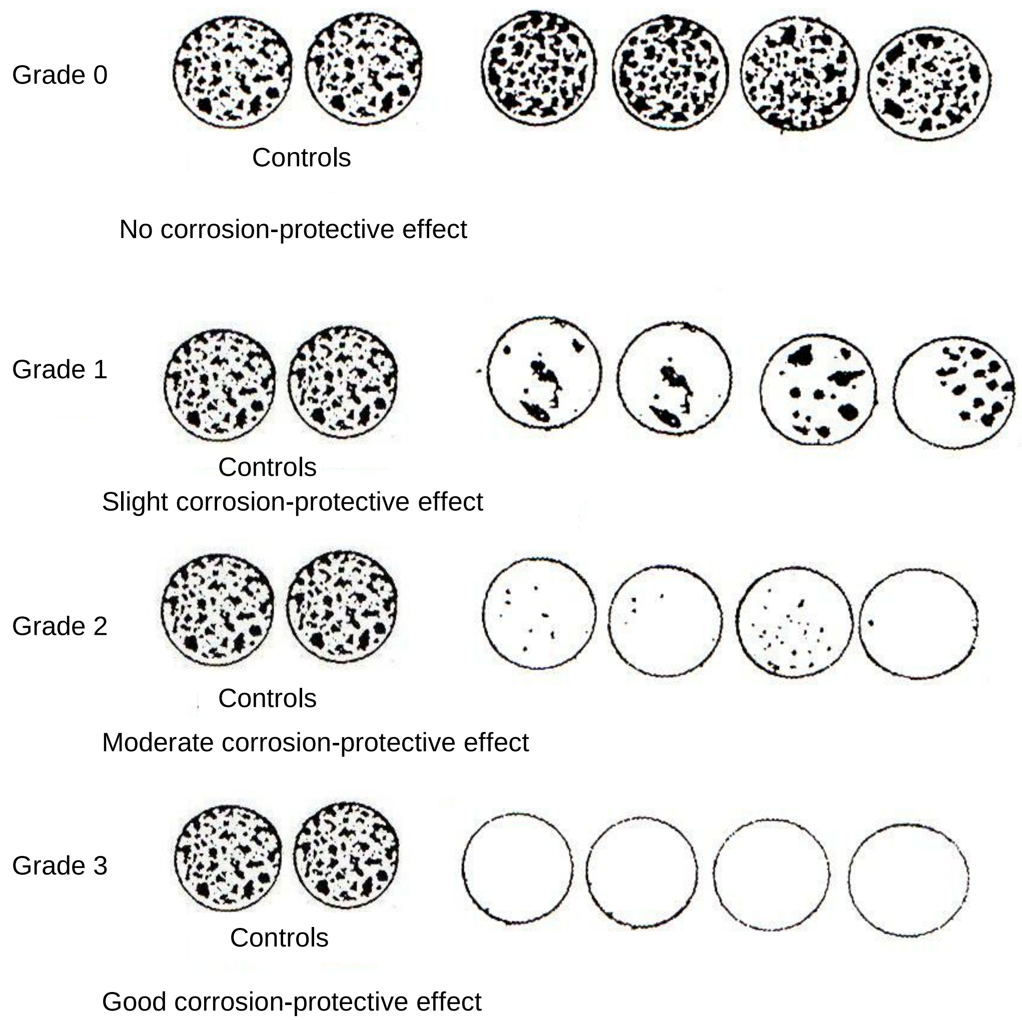
6.6.2.1 Grade 4—Microscopic examination at a magnification of 10X shows no more than three total spots of corrosion, the largest being less than 7.6 µm (300 µin) in diameter, on the best three of four tested steel specimens prepared by hand grinding and polishing in accordance with Option 1 (see Paragraph 4.2.1). Grade 4 indicates an excellent corrosion protective effect.

6.6.2.2 Grade 3—Visual examination by unaided normal vision shows no corrosion on tested steel specimens. Grade 3 indicates a good corrosion-protective effect.

6.6.2.3 Grade 2—Visual examination by unaided normal vision shows no more than three spots of corrosion, the largest being less than 1 mm in diameter, with a clear and significant difference in corrosion between control specimens and all specimens exposed to the VCI material sample within the extremes of normal statistical variability in corrosion effects. Grade 2 indicates a moderate corrosion-protective effect.

6.6.2.4 Grade 1—Visual examination by unaided normal vision shows more than three spots greater than 1 mm in diameter, a total area of corrosion of approximately 1 to 10% of the steel specimen test surface area. There may be relatively small differences in corrosion between control specimens and all specimens exposed to the VCI material sample within the extremes of normal statistical variability in corrosion effects. Grade 1 indicates a slight corrosion-protective effect.

6.6.2.5 Grade 0—Visual examination by unaided normal vision shows no significant difference in corrosion between control specimens and all specimens exposed to the VCI material sample within the extremes of normal statistical variability in corrosion effects. Grade 0 indicates no corrosion-protective effect.



Grade 4 Same as Grade 3 except examined under 10X magnification.

Excellent corrosion-protective effect.

FIGURE 12: Typical visual patterns for rating the group of specimens.[(13)]

6.7 Evaluation of Desiccation Effects

6.7.1 Option A

Option A for evaluating desiccation effects utilizes two sets of controls—the blank control (with no test material sample in the test jar) and the base material control (with the base material 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.

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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, corrosion effects of the VCI test sample specimens should be compared with those of the base material controls. If there are no clear differences in corrosion effects between the VCI test sample specimens 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 utilizes 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% ±2% by the end of the period of moisture saturation (see Paragraph 6.4.1) or a value of greater than or equal to 90% by the end of the specimen conditioning period (see Paragraph 6.4.5), the sample may have some desiccant action, and the VIA-Ferrous test of the associated VCI material 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 which options, alternate test setups, alternate reagents, and 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 material sample.

6.8.3 Attempt to record the visual appearance of each specimen by one or more of the following options:

6.8.3.1 Photographs—It is sometimes difficult to make a photographic record that accurately reveals the corrosion observed on the specimens.

6.8.3.2 Contact transfers—It may be possible to obtain a record by pressing a piece of transparent adhesive tape onto the specimen test surface. This tape is then peeled off with any adhering corrosion products and pressed onto white paper.

6.8.3.3 Retain specimens—Enclose the specimens in well-sealed VCI packaging.

Section 7: Compatibility of VCI-Treated Barrier Material with Copper

7.1 Purpose

The following compatibility check may be used to determine whether chemical components from a VCI-treated barrier material (e.g., film, paper) that is intended for temporary protection of ferrous metals cause corrosion of copper.[7] The copper compatibility check is not required unless it is specified by the manufacturer or user of the VCI-treated barrier material.

7.2 Preparation of Copper Test Panels

7.2.1 Cut three flat panels of cold-rolled, hard-temper copper conforming to ASTM[(14)] B 152[10] and measuring 1.5 mm x 13 mm x 75 mm (in × ½ in × 3 in).

7.2.2 Polish each cut panel with 240 grit aluminum oxide abrasive paper to remove pits and irregularities from all surfaces. The use of “wet or dry” paper is prohibited. Iron oxide abrasives shall not be used. The final abrasion shall be in a direction parallel to the length of the panel.

7.2.3 Bend each polished panel into a “U” shape having a radius of 6 mm (¼ in) and a distance of 13 mm (½ in) between side walls at the ends.

7.2.4 Tightly wrap a sample of VCI-treated barrier material measuring 18 mm x 88 mm (¾ in x 3½ in) around each U-shaped panel with the treated or effective side to match, so that the material is perpendicular to the longitudinal axis and at the base of the open section of the “U.” Secure the VCI-treated barrier material with white nylon thread.

7.3 Test Procedure

7.3.1 Pour 50 mL of a solution of synthetic glycerin and distilled water having a specific gravity of 1.103 at 24 ±3°C (75 ±5°F) into a 500-mL (1-pint) glass jar measuring 68 mm (2½ in) in height. This glycerin-water mixture is intended to produce a vapor with a relative humidity of 85 ±3% above the test solution in the jar during the test.

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(14) ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

7.3.2 Place an inverted glass vessel inside the test jar for use as a stage. Place the three wrapped copper U-shaped panels in the test jar around the perimeter of the stage with both legs of the inverted "U" resting on the stage, avoiding contact with the glycerin solution. Seal the test jar with a screw cap using an aluminum foil gasket.

7.3.3 Place the sealed test jar in a circulating air oven at 66 ±2°C (150 ±4°F) for 7 days.

7.3.4 Remove the test jar from the oven, and allow it to cool to room temperature. Remove the wrapped copper "U"-shaped panels from the test jar and remove the VCI-treated material wrapping from each U-shaped panel.

7.3.5 Examine each U-shaped copper panel on the inside surface of the "U" for evidence of corrosive effects and report results in accordance with ASTM D 130.[11]

Section 8: Test Report

8.1 The test report shall include the following:

8.1.1 Description of VCI material samples.

8.1.2 Description of VIA-Ferrous test procedures including options, alternate test setups, and changes to test apparatus, other equipment, materials, and procedures used.

8.1.3 Specimen type used (see Paragraph 4.1.1).

8.1.4 Rating assigned to each specimen exposed to VCI material samples and controls.

8.1.5 Optional photographic or contact transfer records.

8.1.6 Rating assigned to group of specimens used to assess the effectiveness of each VCI material sample tested.

8.1.7 Results of optional copper compatibility test of VCI-treated barrier material, if performed.

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