

Investigating Specimen Preparation and Cleaning Variables Influencing the NACE TM0208-2018 Jar Test Performance

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ABSTRACT

The NACE TM0208-2018 standard jar test is a widely used tool for measuring the efficacy of volatile corrosion inhibitor (VCI) packaging materials to provide off-contact corrosion protection of steel surfaces. However, while ever improving, the standard still lacks sufficient technical specification for some of the materials used during the test procedure, which likely contributes to inconsistent results observed both within and between laboratories. For example, the use of mineral spirits to clean the steel specimen surfaces is unique to the TM0208 standard, but no comment or requirement is made about which of the various types and grades of mineral spirits should be used. In this paper, we investigate how cleaning the steel specimen surfaces with different grades of mineral spirits impacts their corrosion protection by VCI chemistries. In addition, we further explore how changes in the temperature and duration of the mineral spirits bath, together with covering the bath during cleaning, impact the sensitivity of the steel surfaces to corrosion. Ultimately, we correlate corrosion sensitivity with surface energy as measured by the contact angle of water droplets deposited on the surface of the steel specimens after completion of the TM0208 test.

Key words: NACE standard TM0208, VIA test, volatile corrosion inhibitors, VCI, surface preparation, mineral spirits, contact angle, jar test.

INTRODUCTION

The NACE TM0208-2018 Jar Test remains an industry-wide recognized standard for evaluating the vapor-inhibiting ability (VIA) of raw materials and finished products to provide off-contact corrosion protection of steel surfaces¹. It is particularly useful for comparing the efficacy of different VCI chemistries, as well as for monitoring performance consistency between productions of VCI functionalized materials. However, ambiguity regarding technical specifications for select materials used in the test, together with the sensitivity of the test to relatively slight and often unintended deviations from specific steps, has led to inconsistent results not only between laboratories, but also between individuals within the same laboratory, thus reducing the utility of the NACE TM0208 standard¹.

The extent to which select ambiguities within the TM0208 standard may contribute to disparate levels of corrosion inhibition on steel test specimens was recently investigated by Brekan and colleagues². In addition to the quality of sandpaper (3M: 314D vs 211K) used to abrade the steel specimen surface, and the temperature at which the jars were maintained during the 20 hour conditioning period (70°F vs. 80°F), the authors found that the choice of allowable solvents used to clean the post-abraded steel specimen surface can also impact the extent of corrosion inhibition observed. Specifically, while steel specimens boiled in methanol, ethanol and isopropanol were equally protected from corrosion by the three different VCI paper substrates tested, those boiled in acetone were protected in a substrate-dependent manner.

In addition to the use of polar organic solvents, the NACE TM0208 standard also calls for cleaning the abraded steel specimen surfaces with hot mineral spirits (MS). However, the lack of any technical specification for the type or grade of MS to be used allows for sizeable variance in chemical composition. For example, while catalytic hydrodesulfurization of the initial petroleum distillate greatly reduces the sulfur content of type I MS, or Stoddard Solvent, the aromatic concentrations of these solvents are unaffected and remain high³. Conversely, solvent extraction of the distillate greatly reduces aromaticity and is used to generate type II MS solvents³. Still a third type (type III) is produced by catalytic hydrogenation of the distillate fraction such that the resulting MS contains only fully saturated hydrocarbons, as well as having reduced aromatic content³. Each of these types is further categorized into three grades according to the flash point of the distillate: low flash, regular flash, and high flash³. The increasing flashpoints correlate with a shift in composition towards higher molecular weight hydrocarbons and increased cycloaliphatic content. To the best of our knowledge the impact that these variances in MS may have on the TM0208 jar test results has yet to be investigated.

In this paper, we begin to evaluate how cleaning steel specimen surfaces with MS of differing chemical composition effects corrosion in the NACE TM0208-2018 Jar test. Specifically, we compare the corrosivity of steel specimens cleaned with one-of-five different MS that represent unique combinations of types and grades from separate suppliers (Table 1). Notably, type II was not included as it is no longer commonly produced. In addition, to address further variables in the cleaning phase we perceive to be particularly vulnerable to variance, we also explore how increasing the duration and temperature of the MS bath impacts the extent and consistency of corrosion inhibition. Similarly, we investigate the effect that covering the heated MS bath during cleaning has on the corrosivity of the steel specimen surfaces.

Table 1
Examples of available MS with their CAS, Type and Flash point listed.

Name	Type	Flash Point	Aromatics Contents
Sigma-Aldrich⁽¹⁾ White Spirit	I	Regular (> 30°C)	< 17%
Stoddard Solvent	Stoddard (I)	Regular (> 30°C)	< 25%
Citgo⁽¹⁾ Mineral Spirits 66/3	III	Regular (> 30°C)	< 1%
Calumet⁽¹⁾ 142 Flash	III	High (> 55°C)	< 1%
Sigma-Aldrich Mineral Spirits	III	High (> 55°C)	< 2%

EXPERIMENTAL PROCEDURE

While most of the testing was conducted according to the NACE TM0208-2018, some significant changes were made to test specific variables that are explained here. In addition, note that all the glassware and specimens were brand new and set aside specifically for the purposes of this paper.

Equipment used for these experiments includes: Hot plate, assorted beakers, metal forceps or tongs for specimen handling, counter rotation automated polisher, 1 liter Jars (24), 24 lids (constructed according to NACE TM0208), hot water bath heated to 50°C, microscope, flashlight tester, Nikon⁽¹⁾ 5300D DSLR camera, Tamron⁽¹⁾ SP AF 90mm f/2.8 Di Macro Lens for camera, photography light box, 10 mL capacity pipet.

Materials used for these experiments include: Honeywell⁽¹⁾ Laboratory Plus 99.8% methanol, Calumet⁽¹⁾ 142 Flash Mineral Spirits (142 Flash MS), Citgo Mineral Spirits 66/3 (MS 66/3), Sigma Aldrich 86460-2.5L White Spirits (White Spirits), Sigma-Aldrich⁽¹⁾ 262560-4L Mineral Spirits (Sigma MS), Supelco⁽¹⁾ SX0995-1 Stoddard Solvent (Stoddard Solvent), gauze, laboratory lint free tissue, ice.

The specimens were prepared using a counter rotational, automatic polishing system as described in TM0208, Section 4.2.3, Option 3. The specimens were 1018 steel, electroless nickel plated to prevent corrosion in the recessed section of the Specimens. The specimens were polished for a 5-minute session initially, to remove all traces of nickel from the test surface of the Specimen. For each subsequent test, the specimens were polished wet, using a 1-minute polish session on 240-grit silicon carbide sandpaper, followed by a 2-minute session on 400-grit silicon carbide.

The polishing system allows 6 specimens to be polished at a time. At the end of each polish of 6, the specimens were wiped with methanol-soaked gauze to remove polishing residue and residual water. The specimens were then placed into a beaker containing the same methanol used to soak the gauze. This polishing method allows for consistency of the specimens.

Most experimental variation happened in the steps involved with solvent cleaning the specimens. To quickly review the method, specimens were submerged in methanol and were placed onto a hot plate along with a beaker containing MS, and a second beaker containing methanol. When the methanol beakers were boiling and the MS fuming, but not boiling, the specimens were transferred to the beaker containing MS. Fuming here refers to the point at which vapors were visible above the surface of the liquid MS. One minute after the last specimen was added to the MS, the specimens, starting with the first added, were removed, and placed into the second beaker containing methanol. After 60 seconds, starting with the first added, the specimens were removed, and placed onto a clean dry watch glass.

⁽¹⁾ Tradename

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Variation 1: Testing High Flash, Regular Flash, and Methanol Only. Cleaning solvent systems were investigated. Initial testing was a simple test comparing 142 flash MS, MS 66/3, and a triple methanol cleaning system based on MIL-STD-3010C Test 4031⁴. All steps were the same, however when transferring specimens to the MS bath specimens were broken into 3 groups. Group 1 was added to a beaker containing 142 Flash MS, group 2 was added to the beaker containing MS 66/3, and group 3 of the specimens was added to a second and third methanol bath. It should be noted here that the two MS have different boiling points and thus different temperatures at which the MS cleaning bath was conducted. Specifically, the MS 66/3 has an initial boiling point (IBP) of 318.9°F (159.4°C), the high flash MS has an IBP of 375°F (190.6°C) and the methanol has an IBP of 148.5°F (64.7°C).

Variation 2: Effect of Different MS Types, Flash points, and Sources. Four MS were tested, White Spirits, Sigma MS, Stoddard Solvent, and the MS 66/3. Twenty-Four specimens were broken into 4 groups and each group was cleaned in an identical way except for the MS cleaning step, for which a fresh beaker of each MS was used. The rest of the test was run in accordance with NACE TM0208-2018.

Variation 3: Time and Temperature of Cleaning. Preparation was conducted by altering the time that specimens were exposed to the cleaning bath. This testing was conducted using only the MS 66/3. All steps were the same to the point at which the specimens were added to the beaker containing MS. The specimens were divided into 3 groups. Group 1 of the specimens were added to fuming MS for 1 minute. Group 2 was added also at the fume point, but the MS were then allowed to come to a boil, the boil was maintained for 1 minute before specimens were removed. Group 3 was treated similarly to the second, but the boil was maintained for 5 minutes.

Variation 4: Time and Temperature of Different MS Types. The White Spirits were selected for a boiling time test and were tested against the MS 66/3. The samples were divided into 4 groups. group 1 was exposed to fuming White spirits for 1 minute, group 2 was exposed to boiling white spirits for 5 minutes, group 3 was exposed to fuming MS 66/3 for 1 minute, and group 4 was exposed to boiling MS 66/3 for 5 minutes. The rest of the test was run in accordance with NACE TM0208-2018.

Variation 5: Effect of Solvent Reflux by Covering the Cleaning Bath. Specimens were prepared according to NACE TM0208 until the MS cleaning step. The specimens were divided here into four groups. Group 1 was prepared by submersion in a fuming MS 66/3 bath for 1 minute without covering the beaker, group 2 was similarly prepared, but the beaker was covered. Group 3 was placed into the MS 66/3 at fuming, then brought to a boil for 3 minutes, the beaker was uncovered. Group 4 was treated identically to group 3, however the beaker was covered.

Variation 6: Time and Temperature with Refluxing Solvent Cleaning Bath. Specimens were once again broken into 4 groups and treated identically up until the MS step. In this variation all beakers were covered for the duration of the specimens' time in the MS bath. Group 1 was placed into fuming MS 66/3 for 1 minute while covered. Group 2 was placed into fuming MS 66/3, allowed to come to a boil and then boiled for 1 minute. Group 3 and group 4 were identical to group 2, but were boiled for 2 and 3 minutes, respectively. All Baths were covered.

Following the preparation steps, TM0208-2018 procedures, sections 4.3 through section 6.8, were followed. The specimens were placed into the stopper holders suspended over a water glycerin mixture meant to create a 90% RH environment. Samples which were designated positive controls (samples positive for VCI) had an aluminum weigh boat containing 0.05g of ground sodium nitrite placed in the bottom of the jar. The Positive Controls were added to bracket the performance of specimens exposed to an environment negative for VCI and performance in the presence of a known VCI at an effective dose. The jars were allowed to condition for 20 hours. After the conditioning period the jars were placed, 8 at a