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Evaluating Hydrostatic Test Corrosion Inhibitors and Hydrostatic Test Conditions

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ABSTRACT

Systems such as pipes, heat exchangers, and storage tanks are subject to hydrostatic testing after fabrication. Corrosion inhibitors may be added to the hydrostatic test solution to provide corrosion protection prior to the installation and use of these systems. Effectiveness of these corrosion inhibitors has been well documented and proven, however the parameters of the hydrostatic test and the effect of those parameters on the performance of the corrosion inhibitor performance are not well studied. This paper will assess a unique test method to simulate hydrostatic testing and the corrosion protection offered by a corrosion inhibitor. The effects of various hydrostatic test parameters on the performance of the corrosion inhibitor will also be studied.

Key words: Corrosion Inhibitor, Volatile Corrosion Inhibitor, VCI, Hydrostatic Test, Hydrotest,

INTRODUCTION

Hydrostatic testing is a universally accepted process in which systems such as pipes, heat exchanges, storage tanks, and other vessels are filled with water and pressurized to test the system for strength and leaks. This process is vital for quality control during product and use of these different systems.¹

The use of water in hydrostatic testing is economical and efficient, however because most of these systems are manufacturing using carbon steel the introduction of water into these systems can cause corrosion on the metals' surface. The electrochemical corrosion reaction occurs in three stages at the

metal surface, forming iron (II) hydroxide (*Reaction 1*), iron (III) hydroxide (*Reaction 2*), and eventually various iron oxides, or rust (*Reaction 3*).²



It is known that temperature, pH, oxygen concentration, dissolved salts, and acids can affect the rate, and possibly the mechanism, of this reaction.^{2,3} Corrosion inhibitors have been used for decades to prevent corrosion in these systems, especially in the oil and gas industry.⁴ These corrosion inhibitors may be classified as inorganic or organic corrosion inhibitors, and more specifically as inorganic – anodic, inorganic – cathodic, inorganic – mixed, and organic – adsorption. These organic corrosion inhibitors which protect against corrosion by adsorbing on to the metal surface, forming a hydrophobic layer that effectively inhibits or stops *Reaction 1*. The efficacy of these inhibitors depends on factors like the chemical structure, concentration in the surrounding media, affinity for the metal surface, and so on. The concentration of corrosion inhibitor in the media is critical to its ability to protect a metal surface.⁵

As organic corrosion inhibitors, specifically volatile organic corrosion inhibitors (VCIs) have shown to be useful in hydrostatic testing applications,⁶ work has not been done to study the effects of different hydrostatic testing parameters on corrosion inhibitor function. Parameters like conditioning time (length of time of the hydrostatic test), temperature of the test fluid/water, test pressure, and water quality are considered important variables to control in a hydrostatic test, but the effect of these parameters on corrosion inhibitor efficiency has not been studied. While corrosion inhibitors or VCIs will provide varying levels of protection based on concentration and formulation, this study will focus specifically on the effect of water quality and corrosion inhibitor concentration on a specific VCI hydrostatic test additive (henceforth referred to as “VCI” or “VCI Product”).

Chlorides are a common corrosive contaminate in hydrostatic test water, as brackish or sea water is often used for hydrostatic testing in areas where fresh water in sufficient quantities is unavailable or too expensive. It has been shown that a likely mechanism for chloride catalysis of corrosion reactions is through adsorption onto the metals surface.³ Because organic VCIs, and the VCI Product used in this experiment, operate by adsorption, it could be expected that these mechanisms for catalysis and corrosion protection compete in solution. A better understanding of this reaction and relationship between the competing adsorption of chlorides and the corrosion protection offered by VCIs can provide valuable insight into the optimization of VCI dosage and better understanding of corrosion inhibitor needs with different levels of water quality. The best corrosion protection should be visualized at lowest chloride and higher VCI concentrations and the worst corrosion protection should be visualized at the highest chloride and lowest VCI concentrations.

This paper will use standard test procedures, including immersion testing based on ASTM⁽¹⁾ G31⁷ and electrochemical Tafel polarization testing, as well as a novel test procedure which is used to better simulate the conditions after a hydrostatic test in order to better evaluate the corrosion protection provided by the VCI Product. The testing conducted will assess the corrosion protection provided by VCI used at a variety of concentrations and at a variety of chloride levels.

EXPERIMENTAL PROCEDURE

To evaluate the relationship between chloride concentration and corrosion inhibitor concentration, several solutions of varying chloride and corrosion inhibitor concentration are required. Solutions were made using deionized water with varying levels of chloride content and corrosion inhibitor content.

⁽¹⁾ ASTM International, 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

Sodium chloride was selected as the chloride source as it is the most common source of chlorides in natural water systems.

Table 1
Sodium Chloride Used for Solution Preparation

Chloride Concentration [ppm]	Sodium Chloride Used per 1000 grams of solution [grams]
0	0
100	0.165
300	0.495
500	0.824
1000	1.65
5000	8.24

Table 2
VCI Product Used for Solution Preparation

VCI Product Concentration	VCI Product Used per 1000 grams of solution [grams]
0.0%	0
0.3%	3.0
0.5%	5.0
0.75%	7.5
1.0%	10.0

The following solutions were made for evaluation in Immersion Testing, Electrochemical Corrosion Testing, and Simulation of Hydrostatic Testing. The following solutions were prepared and are referred to in the form of ([Salt Concentration], [VCI Concentration]). The following solutions were created:

Table 3
Salt/Corrosion Inhibitor Solutions Used

- (0, 0.3)
- (100, 0.3)
- (300, 0.3)
- (500, 0.3)
- (1000, 0.3)
- (5000, 0.3)
- (0, 0.5)
- (100, 0.5)
- (300, 0.5)
- (500, 0.5)
- (1000, 0.5)
- (5000, 0.5)
- (0, 0.75)
- (100, 0.75)
- (300, 0.75)
- (500, 0.75)
- (1000, 0.75)
- (5000, 0.75)
- (0, 1.0)
- (100, 1.0)
- (300, 1.0)
- (500, 1.0)
- (1000, 1.0)
- (5000, 1.0)

Standard steel panels were used for the simulation of hydrostatic testing and immersion corrosion testing. Test panels (1.59 cm x 2.54 cm x 7.62 cm [0.63" x 1" x 3"] SAE⁽²⁾ 1008/1010 cold rolled steel) were prepared by hand polishing both faces to a consistent finish with 240 grit aluminum oxide abrasive paper. Panels were cleaned with methanol prior to use and are hereafter referred to as "test panels".

Simulation of Hydrostatic Testing

A novel method of testing was used to evaluate the effectiveness of the solutions as an additive to hydrostatic testing. Test panels were placed into each test solution in *Table 3* and allowed to condition. Three panels were tested in each test solution. After the conditioning time, panels were removed from the solution and immediately placed into a cylindrical glass cell, with a 12.7 cm [5"] height and 5.08 cm [2"] diameter, while still wet. At this point, 2.0 g $\pm$ 0.2 g of the test solution were placed into the test cell to maintain wet and humid conditions and simulate residual hydrostatic test water left in a system after the hydrostatic test. Glass test cells were sealed with a lid and thread sealant tape, then placed in an oven to accelerate corrosion.

The following parameters were kept constant throughout the testing, in order to isolate the interaction between product concentration and chloride concentration:

- Conditioning Time: 1 hour
- Solution Temperature: 21 °C $\pm$ 2 °C
- Pressure: Ambient
- Oven Temperature: 40 °C $\pm$ 2 °C

⁽²⁾ SAE International (SAE), 400 Commonwealth Dr., Warrendale, PA 15096

Test cells were inspected daily for signs of corrosion appearing on panels. After the first 24 hours panels were inspected and corrosion was graded according to ASTM D610⁸. The time to the appearance of visual corrosion was noted. After six weeks of time in the 40 °C oven panels were removed, and a final inspection and grading was performed according to ASTM D610.

Immersion Corrosion Testing

Test was performed with combination of chloride concentration and inhibitor concentrations in *Table 3*, including a control solution with no inhibitor and no chlorides. Each solution used in immersion test was used after the Hydrostatic Testing, detailed above. As such, some solutions used for immersion testing (particularly at chloride concentrations of 1000 ppm and 5000 ppm) contained some unmeasured level of iron oxide.

The solutions were held in a cylindrical glass cell with a 12.7 cm [5"] height and 5.08 cm [2"] diameter. Approximately 200 g of each solution (enough to fully immerse the test panel) was used in each test cell to ensure full immersion of the test panels. Panels were cleaned with methanol, weighed to the nearest 0.1 mg, and placed into the respective solutions. Test cells were placed into a 40 ± 2 °C oven for approximately one week. At this time, panels were removed, rinsed with methanol, and oxides were removed from the surface with a cleaning solution detailed in ASTM G1⁹, Section A1 C.3.5. Panels were immersed for 5 minutes to remove oxides then neutralized using an alkaline cleaner and cleaned with methanol. The panels were examined for mass loss and the rate of corrosion was calculated using the following formula from ASTM G31 (equation 2, Section 12.4):

$$\text{Corrosion Rate (mpy)} = \frac{3.45 \times 10^6 \times W}{A \times T \times D} \quad (1)$$

'W' represents the mass loss in grams, 'A' is the panel surface area in cm², 'T' is the duration of the test in hours, and 'D' is the metal density in grams per cubic centimeter (7.87 g/cm³ for SAE 1008/1010 steel). The corrosion rate reduction was also calculated by comparing the control sample to the test samples with the following formula:

$$\text{Corrosion Rate Reduction} = \left(1 - \frac{R}{C}\right) \times 100\% \quad (2)$$

Where 'C' represents the corrosion rate of the control sample and 'R' is the corrosion rate of the test sample.

A single panel was prepared for each tested solution, along with a single panel to be tested in deionized water as a control. A single prepared, but untested, steel panel was prepared and subjected to the cleaning procedure as above. The mass loss of the cleaning procedure was determined, and this mass loss was subtracted from the mass loss measured in each tested panel, in order to determine the true mass loss from the immersion corrosion testing.

Because testing was performed in "rounds", a different control panel (with no chlorides or corrosion inhibitor) was tested with each set of testing. The average corrosion rate for each control was used for calculations of the corrosion rate reduction.

Electrochemical Corrosion Testing

Testing was performed on samples with a combination of chloride concentrations and inhibitor concentrations as defined in *Table 1* and *Table 2*, including control solutions with no inhibitor. One liter of these solutions was placed in a glass electrochemical cell as described in ASTM G5¹⁰ and stirred at 300 rpm with a triangular stir bar throughout the experiment. SAE 1018 carbon steel plugs were prepared

by ultrasonic cleaning in hexanes for three minutes prior to being wiped dry with lint-free laboratory tissues and placed in the electrochemical cell with the test solution.

Tafel polarization curves were taken utilizing a freshly prepared steel plug for each measurement. The plugs were polarized with a potentiostat across a potential range of ± 250 mV from the Open Circuit Potential (OCP). Polarization began after a three-minute equilibration time once the variation in OCP settled to ± 0.5 mV over 5-10 seconds. Tafel polarization proceeded at a rate of 1 mV/s with one measurement every two seconds. At each measured voltage, the corresponding current was recorded and plotted. Once the polarization measurement was complete, curve fitting was used to determine the anodic and cathodic Tafel slopes as well as the corrosion current density and corresponding corrosion rate.

RESULTS

Simulation of Hydrostatic Testing

For the hydrostatic testing simulation, panels were assessed by their time to failure (i.e. first visible signs of corrosion in the test cell), their corrosion rating (according to ASTM D610) after 24 hours, and a final evaluation of their corrosion rating (according to ASTM D610) after six weeks. The scale according to ASTM D610 starts at “10” indicating no corrosion going down to “0”, showing the most severe corrosion. Additional indications of “P”, “G”, and “S” indicate the type of corrosion as “pinpoint”, “general”, and “spot” corrosion, respectively. *Table 4* below indicates the definition of corrosion grades according to ASTM D610. *Table 5*, summarizes data collected from this testing, providing the average time to corrosion and average corrosion rating after 24 hours and after six weeks; complete data can be found in *Appendix, Tables A1-A4*. Panels were inspected visually with the help of visual aids in the ASTM D610 standard to match the appearance of the corrosion.

Table 4
ASTM D610 Corrosion Grades

Rust Grade	Percent of Surface Rusted
10	<0.01%
9	0.01% - 0.03%
8	0.03% - 0.1%
7	0.1% - 0.3%
6	0.3% - 1.0%
5	1.0% - 3.0%
4	3.0% - 10.0%
3	10.0% - 16.0%
2	16.0% - 33.0%
1	33.0% - 50.0%
0	>50.0%

The first inspection after testing was made at varying times after the test procedure was carried out, typically within 24 hours. At the time of the first inspection (as shown below in *Tables A1-A4*), several different test cells showed corrosion. For this reason, if a test cell failed between the time of the test procedure and first inspection, the failure was listed as “less than 24 hours”. For testing carried out at 0.3% VCI concentration, the first inspection was not performed until 66 hours later (the test was set up at the end of day prior to a weekend); based on the trends found at other VCI concentrations, the time to failure was still listed as less than 24 hours for these tests.

Table 5
Hydrostatic Test Data Summary

Cl ⁻ Concentration [ppm]	VCI Concentration	Average Time to Corrosion [hours]	Average Corrosion Rating [24 hour]	Average Corrosion Rating [6 weeks]
0	0.3%	573.7	10.0	9.0
100		46**	9.7	9.0
300		<24	7.0	6.3
500		<24	7.0	6.3
1000		0	4.3	3.7
5000		0	3.0	2.0
0	0.5%	>1000*	10.0	9.7
100		544	10.0	7.3
300		<24	6.7	7.0
500		<24	6.3	5.7
1000		<24	5.0	4.3
5000		0	2.3	1.0
0	0.75%	>1000*	10.0	9.3
100		262	10.0	8.7
300		38.7**	9.3	7.7
500		<24	7.3	6.0
1000		<24	6.0	5.0
5000		0	2.0	2.0
0	1.0%	>1000*	10.0	10.0
100		894.7	10.0	9.0
300		293.3	9.0	8.0
500		<24	8.7	6.0
1000		<24	6.7	5.7
5000		0	3.0	2.3

*Some or all of the tested panels did not show failure over the 6-week test period.

**Two of three panels showed failure in less than 24 hours. The average time was calculated based on a "Time to Corrosion" of 24 hours.