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Wheel Test Method Used for Evaluation of Film-Persistent Corrosion Inhibitors for Oilfield Applications

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Foreword

NACE technical committee reports are intended to convey technical information or state-of-the-art knowledge regarding corrosion. In many cases, they discuss specific applications of corrosion mitigation technology, whether considered successful or not. Statements used to convey this information are factual and are provided to the reader as input and guidance for consideration when applying this technology in the future. However, these statements are not intended to be recommendations for general application of this technology, and must not be construed as such.

The current technology for assessing the effectiveness of corrosion inhibitors proposed for batch treatment of oil wells/continuous treatment of transportation pipelines is a subject of much discussion and, in many cases, disagreement. The purpose of this report is primarily to explain a test method that has been used to evaluate film-persistent corrosion inhibitors for a laboratory ranking of various inhibitors under specified conditions of oilfield environments and also to verify/assure that the best laboratory candidate and the field production test inhibitor are the same, they shall be both finger printed. An alternate procedure which combines dynamic and static components in a single test is also presented. The test methods described in this report are not uniformly accepted throughout the oil industry, but are outlined here to make information available on at least one commonly used type of wheel test, the variables involved, and some sources of variation and inaccuracy. All data contained herein are presented for informational purposes only.

This report is intended for use by professionals in the oil and gas industry (including producers, service companies, and testing laboratories) for the evaluation of corrosion inhibitors that are designed for batch applications.

This NACE technical committee report was originally prepared in 1982 by Task Group T-1D-8, a component of Unit Committee T-1D on Corrosion Monitoring and Control of Corrosion Environments in Petroleum Production Operations. It was reviewed by T-1D and reaffirmed in 1995 under the auspices of Group Committee T-1 on Corrosion Control in Petroleum Production and in 2005 by Specific Technology Group (STG) 31 on Oil and Gas Production—Corrosion and Scale Inhibition. It was revised in 2017 by TG 528 "Revise NACE Publication 1D182." This report is published under the auspices of STG 31.

Introduction

The test methods presented herein are based on the so-called “wheel test”[1-3] which is versatile in that it has often been adjusted to test a variety of inhibitors, and has been performed on various test specimens. Its versatility has also enabled modifications to specific test variables, such as temperature, film life, surface conditions, concentration of corrosive agents, and oil-to-water ratios. With respect to its versatility, the test is similar to other tests with the same general intent. The low level of reproducibility of test results is considered by some to offset the advantages of its versatility. Reproducibility problems seem to be encountered when the test is performed by more than one technician. A single technician appears to be more likely to reproduce his/her own results on a regular basis.

This report describes the wheel test and discusses the various conditions in which it has been used. The wheel test is a dynamic test performed by placing fluids (oil, water, and inhibitor) in a 200 mL (6.80 oz) beverage bottle with a metal test specimen, purging with a corrosive gas, and capping the bottle. The bottle generally has then been agitated for a period of time by securing it to the circumference of a “wheel” and rotating the wheel. After agitation, the test specimens have been transferred to another bottle containing no inhibitor (only corrosive fluids) and agitated for a longer period of time. At the end of this time, the metal test specimen has been removed and cleaned and the mass loss has been measured.

Techniques used in both test procedures are described.

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Section 1: Fluids and Chemicals

The oil used has generally been crude oil. It has been purged with inert gas or one of the corrosive gases such as carbon dioxide (CO_2) or hydrogen sulfide (H_2S) before it has been measured into the test container. Every possible effort has been made to exclude air. When crude oil has not been available, refined oil has been used. Refined oils contained oxygen and sometimes contained polar additives. Both of these materials have appeared to contribute to poor results and were removed.

Polar material has been removed by filtration through bentonite or fuller's earth. Oxygen has been removed by blanketing the oil with oxygen-free nitrogen (99.99%) and agitating the oil with an aqueous solution of catalyzed sodium sulfite (Na_2SO_3). This has been done with 150 mL (5.10 oz) of 1% catalyzed sodium sulfite per 3 L (3.20 qt) of oil.

Brines have been purged with nitrogen to remove oxygen, and then sparged with CO_2 or H_2S to simulate produced water. The synthetic brine is commonly 9.62% sodium chloride (NaCl), 0.305% calcium chloride ($CaCl_2$), 0.186% magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$), and 89.89% distilled water; however, synthetic seawater in accordance with ASTM[(1)] D11414 has been used for special purposes.

Traces of oxygen have probably been responsible for many of the inconsistent test results; therefore, the following practices have been used to remove oxygen and prepare the brine for testing: the brine has been sparged with nitrogen (2 L/min). A 30 min sparge has usually been used for 4 L (4.20 qt) of brine. Catalyzed sodium sulfite (4 mg/L) has been added to scavenge any residual oxygen.

For sweet tests (nonsulfide), 4 L (4.20 qt) of brine have been purged with CO_2 for about 30 min. The pH of this brine generally has been 4.5 to 4.8.

For sour tests (with H_2S), a reproducible amount (500 mg/L) of H_2S has been generated in situ by adding 1,700 mg/L of acetic acid and 3,530 mg/L of fresh, reagent grade sodium sulfide ($Na_2S \cdot 9H_2O$) to the brine. Higher concentrations of H_2S have been obtained by saturating the brine at a given temperature and pressure, using bottled H_2S.

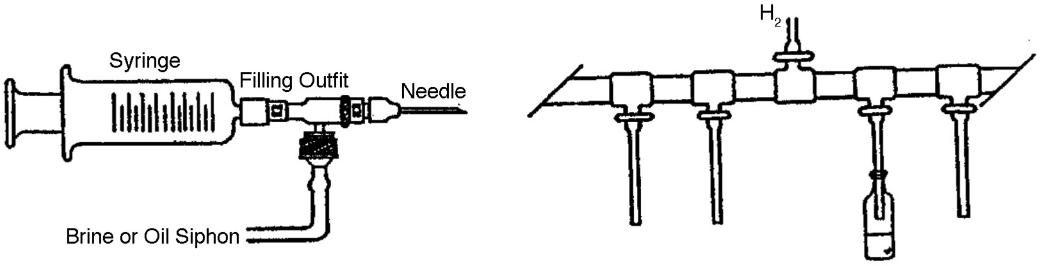
Inhibitors have generally been added neat, because the inhibitor concentrations were 1 to 5% based on oil used in the filming portion of the test. For low concentrations of 50 to 100 ppm, inhibitors have first been diluted to 1% with kerosene. Xylene has been used if the inhibitor was not soluble or was not readily dispersed in kerosene.

Section 2: Test Specimens

There has been a wide variation among various laboratories in the choice of test specimens. Shim stock, 0.13 x 12.7 x 76 mm (0.005 x 0.500 x 3.0 in), has been used for most sour testing, because this material is readily observed for pitting tendency. Drill rod specimens, 1.5 x 150 mm (0.060 x 6.0 in), have often been used after sandblasting for sweet and sour tests when pitting has not been of primary concern. Test specimens have been cleaned with benzene, wiped dry with a clean cloth, and stored in a desiccator until ready for weighing. Fingerprints have been avoided by handling with clean gloves, forceps, or a magnet. Because special surface preparation of the shim stock, other than degreasing, seemed to make the test specimen surface sufficiently nonreproducible, the shim stock has been used as is.

Section 3: Test Equipment

Test containers most often used have been 200 mL (6.80 oz) beverage bottles with coated or plastic-lined caps. The test fluids have been metered into the bottles while the bottles were being purged with nitrogen to prevent oxygen entry. Several methods of avoiding oxygen contamination have been used. One inexpensive method using syringes and a purging manifold is shown in Figure 1.



Typical Bottle-Filling Outfit

Purging Manifold Made from Polyvinyl Chloride (PVC) Tees, Nipples, and Nylon Tubing Connections

Figure 1: One Method of Avoiding Oxygen Contamination

Section 4: Procedure

Generally, the inhibitor to be tested has been added to the bottle first. Then the bottle has been purged of air. First the brine, then the oil, have been metered or measured into the bottle. Then the test specimen has been added, and the bottle has been capped and placed on the wheel. Generally, two oil/water ratios and two inhibitor concentrations have been tested in triplicate in order to obtain adequate information for each inhibitor. The oil/water ratios most often chosen have been 50/50 and 10/90 oil-to-water. The inhibitor concentrations based on oil volume have been 1.0 and 5.0%.

The capped bottles have been placed on holders on the wheel and rotated at the desired temperature for one or two hours to enable the test specimens to be “filmed” by the inhibited fluids. The test specimens then have been carefully removed from the bottles, transferred to another bottle of purged fluids, capped, and put back on the wheel to be rotated another hour to rinse off loose inhibitor clinging to the test specimen. The transfer has been easily made with a magnetized bar on a tube string or any magnetized retriever without disturbing the protective film. The rinsed test specimen has then been transferred to another bottle of purged fluids containing no inhibitor, capped, placed on the wheel, and rotated at temperature for 24 to 72 hours. Some inhibitors have produced sufficient films to enable the filmed test specimen to be transferred into fresh, uninhibited fluids again for a second or third 24 h period. Blanks with no inhibitor film have usually been transferred daily if extended tests were performed. Blanks have not generally been transferred during the film and rinse transfer steps of the inhibited tests.

At the end of the test, the test specimens have been retrieved from the bottles, rinsed with isopropyl alcohol/xylene to remove oil and inhibitor film, and wiped dry. If a sour environment was used, the sulfide film has been removed by a 10 to 15 s dip in inhibited 10% hydrochloric acid (HCl). A steel wool pad has been used to scrub any remaining corrosion product from the test specimen. The test specimen has then been rinsed in water and dried by an acetone or alcohol dip. It has then been desiccated and weighed on an analytical balance to the nearest 0.1 mg.

Section 5: Calculation of Results

The mass losses have been calculated and the results of triplicate specimens have been averaged. In the case of a wild result (one that is more than four times the average deviation), it usually has been rejected and the results of the remaining two specimens have been averaged. The percent protection has then been calculated as shown in Equation (1):

$$\%\,\text{Protection} = \frac{(\text{mg blank mass loss} - \text{mg test specimen mass loss})}{\text{mg blank mass loss}} \times 100 \qquad (1)$$

Section 6: Continuous Treatment

Simulation of a continuous treatment by a constant concentration test has been accomplished by adding 100 ppm of inhibitor based on total fluids to test bottles prepared as blanks. A good inhibitor has given 90% protection.

Section 7: Summary

Many variables, such as the effect of oil/water ratios on corrosion rates, the effect of various contaminants in the water, as well as the effect of these variables on inhibition by one or more inhibitors, have been studied using this test method.