LABORATORY TESTING OF VOLATILE CORROSION INHIBITORS

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ABSTRACT

For control of top of line corrosion (TLC) in multiphase wet gas lines, the best technique currently available on the market is the TLCC-PIG (or spray pig). Its main disadvantages are firstly the necessity of monthly treatment, which may not be compatible with the production requirements, and secondly relatively low efficiency, which is around 70-75 %, if the treatment is monthly. The author's company(1) decided to work with a corrosion inhibitor manufacturer(2) in order to develop volatile corrosion inhibitors, with the development project starting in 2004. After a series of preliminary tests in an independent laboratory, one promising molecule was selected for loop testing, which were finalised in 2007. The volatile product is mixed with a bottom line corrosion inhibitor, tested separately, in order to have a single injection point for bottom line and top of line corrosion inhibitors. The formulated product was field tested in 2008 and early 2009. This paper summarises the preliminary results and corrosion loop test results. The results of field testing will be presented separately.

Key words: top of line corrosion, TLC control, volatile corrosion inhibitor, inhibitor testing.

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INTRODUCTION

TLC is quite a new, since the late 90’s, and significant risk for wet gas pipelines that mainly occurs in South East Asia. Some recent publications also mention TLC cases in the North Sea and the Middle East. In fact, oil and gas producers have developed many fields without taking TLC risk into account. Consequently, many pipelines are currently subjected to TLC and need to be protected using whatever is available on the market for corrosion control and monitoring.

Beginning in the late 90’s and extending to 2004, batch treatments using a double pig were applied with limited success, largely due to the uncertainty of contact between the corrosion inhibitor and the top of the line surface. Preparation for field operations was difficult and it was furthermore necessary to shut down production for batch preparation. The TLCC-PIG or spray pig was ready in 2004 and since then it has been used by all companies for such batch treatments.

Based on the corrosion monitoring by chemical analysis and also recent field evaluations using a cooled probe, it has been established that the efficiency of monthly batch treatments by spray pig, using the appropriate corrosion inhibitor, is about 70 – 75 %, particularly as the corrosion inhibitor film is not maintained (different with continuous injections) until the next treatment. Batch treatment frequency is normally conducted once every month. If it is done once every two months (what is practically done by some operators) the efficiency would be much lower than 50 %.

In order to improve the efficiency of the treatment, simplify it, create safer chemical inhibition and reduce production loss, the author’s Company decided to cooperate with a corrosion inhibitor manufacturer to develop volatile corrosion inhibitors. Due to limited storage and injection capacities/facilities on the offshore platforms, it was decided to mix the volatile corrosion inhibitor, if development of the product was a success, with the bottom line corrosion inhibitor, which was already developed and tested separately.

First, the corrosion inhibitor manufacturer selected some molecules able to remain in the gas phase and thus protect the top of the pipe surface. Then some preliminary corrosion protection tests were carried out in an independent laboratory. One of the promising molecules was used for the formulation of the mixed product. The formulated product, containing both bottom line and volatile corrosion inhibitors was tested in a corrosion loop. The results of these preliminary and loop tests are summarized below. After loop testing, the product was tested in one gas field in Thailand and these results are presented separately.

EXPERIMENTAL SET UP

Two independent laboratories were contracted to perform different series of evaluation tests. The study itself can be divided in three parts:

Part 1: Selection of the inhibitor formulation by electrochemical study in a corrosion cell.

This first part presents results obtained at an independent laboratory in France on a corrosion cell especially designed to reproduce condensing conditions. Several formulations of the inhibitor were tested via pH and electrochemical measurements.

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1. Bongkot field, Gulf of Thailand
2. CORREX Laboratories - France
Part 2: Confirmation of basic inhibitor properties in a glass cell

The formulation selected in Part 1 was used in another series of glass cell tests and was also focused on basic liquid/vapor equilibrium and corrosion study. This second part of the study was performed at a research institute in the US\(^5\).

Part 3: Large scale flow loop experiments

The results obtained in parts 1 & 2 were used to select experimental conditions for a series of large scale flow loop tests. This last step was also performed at a research institute in the US and concluded the laboratory study.

Details of each part are as follows:-

**Part 1: Selection of the Inhibitor Formulation by Electrochemical Study in a Corrosion Cell**

A corrosion cell was designed, allowing the formation of aqueous films at a controlled temperature by condensation of water vapor. The solution used to produce the vapor is characterized by the following parameters: chemical composition of the water phase, saturation gas, and temperature. The device then is constituted to include two main elements:

- The reactor with an aqueous solution producing the vapor
- The condensation cell, in which a specific device has been introduced to perform electrochemical measurements.

A sketch of the experimental set-up is given in Figure 1.

Corrosion measurements were carried out under different conditions, varying either the chemical composition in the reactor (with and without acetic acid) or condensation rate / temperature. The mitigation of corrosion by addition of a non-volatile neutralizing agent in the aqueous phase of the reactor was studied for comparisons with volatile inhibitor performance.

The solution in the reactor was prepared from deionizer water (conductivity ≤ 1.2 \(\mu\)S.cm\(^{-1}\)) and glacial acetic acid (100\% PROLABO quality NORMAPUR, analytical reagent). Water was heated in the reactor up to 90 deg C. and additions of reagents were made after saturation of the solution by CO\(_2\) using a syringe, to avoid stripping of reagent vapors. The concentration of the acetic acid was kept constant during experiments (condensed water is re-circulated). Deaeration was performed by argon bubbling. The solution was then saturated by continuously bubbling CO\(_2\). CO\(_2\) plays the role of a gas carrier for the vapor produced from the solution. The gas flow rate was then controlled and adjusted to obtain the required rate of condensation, allowing maintaining of a water film on the specimens in the corrosion cell. The rate of condensation was regulated at 0.6 ml/m\(^2\).sec.

The specimens (three samples of X65) were embedded in a resin contained in a cylindrical mould made of stainless steel. The mould or one of the specimens may be used as counter-electrode. The reference electrode was connected to the cell by an electrolytic bridge located in the centre of the mould.

\(^5\) Institute for Corrosion and Multiphase Technology - Ohio University - USA
resin surface. Before each experiment, the surface was polished under water with Sic grit paper (grade 1200) and carefully dried to prevent rusting of the specimen before introduction in the cell. The chemical composition of the specimens is given in Table 1.

The pH of the solution in the reactor was continuously monitored as well as the pH of the condensed phase. The rate of formation of the condensed solution was measured using a burette and the free potentials of the specimens were monitored. The polarization resistance was measured through the LPR technique after a continuous condensed water film formed on the electrodes. Coupons were also used to measure the pitting tendency of the volatile products. After obtaining the base line data, the volatile corrosion inhibitor was injected into the reactor and new measurements were taken to evaluate the efficiency of the product.

Several types of molecules have been screened and finally three neutralizing amines were selected. In the following, the results of the best volatile amine, PX 4803 R, are presented (Figure 2).

For the evaluation of the selected volatile product, some tests were carried out with a non-volatile product for comparison, with N-methyl-diethanolamine (MDEA) selected for this purpose.

Addition of MDEA in the reactor increased the pH only in the reactor. The pH of the condensed water did not change (Figure 3) confirming that if no volatile amine was added to the reactor there would be no change in the corrosivity of the condensed water (Figure 4). Even with 1000 ppm of MDEA addition in the reactor, the value of Rp measured into the condensed water remained almost constant, around 500 Ω.cm². In fact, once the protectivity of iron carbonate layer was affected by organic acids, MDEA did not directly provide protection to the top of line surface. It would require some time to remove the traces of acetic acids from the steel surface (especially at low water condensation rates) to reform a protective layer.

Addition of the volatile inhibitor in the reactor increased not only the pH in the reactor but also the pH of the condensed water (from 3.8 to 4.8) as shown on Figure 5. There was also an important increase of the condensed water Rp (Figure 6 and Figure 7). Without addition of acetic acid, the polarization resistance in the condensed water was 1600 Ω.cm². The addition of acetic acid in the reaction contributed to the decrease of Rp value in the condensed water to 500 Ω.cm² and the condensed water became more corrosive. The addition of 400 ppm of PX 4803 R in the reactor made the Rp in the condensed water increase to around 1200 Ω.cm². At 600 ppm of PX 4803 R, the value of Rp in condensed water reached 1400 Ω.cm². The addition of PX 4803 R, in the reactor, can inhibit the corrosivity of condensed water. It is clear that the volatile inhibitor has some neutralising properties and provides some protection through the increase of the pH by helping the formation of an iron carbonate layer (it takes about 20 hours to reach the maximum protection level at 400 ppm). It is not clear if these products have some filming properties.

Based on above results, a corrosion inhibitor containing volatile product (PX 4803R) and bottom line corrosion inhibitor was formulated (PX 4856 R). Figure 8 shows the test results with the new product. At 1000 ppm of acetic acids, the volatile inhibitor had little effect on pH of the condensed water but the polarisation resistance increased significantly with the increase of volatile inhibitor concentration. This is an interesting result showing that even at very high acetic acid concentrations, part of the volatile inhibitor passed into the gas phase and provide protection (mode of protection seems to be more than just neutralising).

A comparison between the performance of volatile inhibitor alone and that of the combined product is shown on Figure 9. Combined product has even better performance than the volatile product alone.
Coupon results with 1000 ppm of acetic acid are shown on Figure 10. Severe corrosion was observed on the coupon without a volatile inhibitor. No localised corrosion was visible on the coupon surface after addition of the volatile inhibitor; only minor uniform corrosion was observed.

In order to check the performance of bottom line corrosion, some polarization resistance experiments were carried out in the reactor. Figure 11 shows the results of the combined product for the bottom line protection in presence of 1000 ppm of acetic acid.

**Part 2: Confirmation of Basic Inhibitor Properties in a Glass Cell**

The commercial form of the inhibitor PX 4856 R selected in the previous part (mixed with a standard bottom of line inhibitor and containing about 90%wt of PX 4856 R) was sent to another independent laboratory for further evaluation.

In this section, the properties of the volatile inhibitor were tested in glass cell for a limited liquid/vapor equilibrium and corrosion study.

The reasoning behind these tests was to perform a few simple tests in a glass cell, thus evaluating the volatility of the inhibitor at different pHs, and then to complete a few short term corrosion tests, still in a glass cell, in order to narrow down the range of inhibitor concentration offering the optimum corrosion protection. Once these conditions are determined, more cumbersome and expensive large scale flow loop tests (Part 3) could be initiated with a better chance of success.

The liquid/equilibrium properties of the inhibitor were evaluated in a glass cell setup equipped with a condenser (Figure 12). The temperature in the main cell where the inhibitor was introduced (tank) was set at 70ºC and the pH was adjusted using hydrochloric or acetic acid. Nitrogen was used a carrier gas in order to flow the vapor phase through the condenser. The condensed liquid was trapped at the end of the condenser and sampled for measurement. The inhibitor concentration was measured in the condensed liquid using an extraction method and the pH in the tank was varied in order to study its influence on the volatility of the inhibitor.

Each test lasted a couple of hours and the results are shown in Figure 13. The volatility of the inhibitor is the ratio of the concentrations of inhibitor measured in the condensed liquid and the concentration of inhibitor measured in the tank. It shows that around pH 5 and at 70 ºC, the concentration of inhibitor in the condensed liquid was about 16% of the one injected in the tank.

Once the liquid/vapour equilibrium properties of the inhibitor were better understood, small scale corrosion tests were carried out in a two-litre glass cell equipped with a reflux condenser (Figure 14). The inhibitor solution, deionizer water and acetic acid were continuously purged with CO₂ gas. The exiting gas/vapor mixture went through a condenser which caught the inhibitor/acid vapors and sent the condensed liquid back to the cell. This way, the volatile species were not be depleted and the composition of the solution in the cell remained constant during the entire test. A heater stirrer was used to control the temperature of the liquid. The pH of the solution in the cell and the gas and liquid temperatures were monitored continuously. The corrosion rate was measured using weight loss probes made of X-65, and Electrical Resistance (ER) probes. A carbon steel coupon was used primarily to investigate the occurrence of localized attack while the ER probe was used to follow the trend of the general corrosion. Both probes were flushed mounted to the bottom face of the lid. Two identical sets of cooling coils, in contact with the top face of the lid, were installed around each probe in order to create artificial cooling conditions. The temperature of the corroding metal could only be measured with the ER probe and enabled the calculation of a local condensation rate. The only way to ensure that the condensation rates were the same as the surface of the ER probe and that of the
surface of the weight loss coupon was to ensure that the cooling conditions (controlled by the cooling liquid flow rate) were identical in each case.

The Figure 15 shows how the condensation process happened on the lid of the glass cell. Large droplets (5 mm diameter) formed on the lid and the corrosion coupons.

Several tests were performed and the conditions are summarized below:

- Pressure: 1 bar
- CO₂ partial pressure: 0.7 bar
- Test duration: two weeks
- Condensation rate: 0.25 ml/(m².s)
- Free acetic acid content: 500 ppm
- Temperature: 70 deg C.
- Corrosion rate measurement: ER probe and X-65 coupons (polished 600 grid sand paper)
- Inhibitor concentration: 0, 500 and 2000 ppm (formulated product containing bottom line and volatile corrosion inhibitors).
- Test duration: 14 days

The ER probe results are presented in Figure 16. The baseline test (0 ppm of inhibitor) presents a general corrosion rate increasing in the first 4 days up to 2.5 mm/year and then stabilizing. The weight loss coupon gave a rate of 1.4 mm/year. The pH in the bulk liquid phase remained around 4.4 and the undissociated acetic acid concentration was calculated at 200 ppm in the bulk liquid phase.

The addition of 500 ppm of inhibitor increased the pH of the solution from 4.5 to 6 but had a relatively small effect on the general corrosion rate which remained above 2 mm/year with the ER probe and around 1.5 mm/year with the weight loss method. The undissociated acetic acid concentration fell to 30 ppm. All together, the presence of 500 ppm of inhibitor in the bulk liquid had little influence on the scale of the corrosion attack (other than increasing the pH and limiting the amount of free acetic acid available for evaporation).

Finally, the addition of 2000 ppm of inhibitor did have a very strong effect on the general corrosion rate which dropped to 0.3 mm/year with both ER and weight loss method. It is important to note that the introduction of such a quantity of inhibitor increased the pH to 6.8 and basically turned all the free acetic acid into acetate ions (nullifying the influence of the acetic acid). However, the remaining corrosivity as a result of the presence of CO₂ seemed to have been effectively stopped (The efficiency was around 90%). It is worth mentioning that in the present case the formulated product was used. The concentration of the volatile part of the product in the bulk liquid phase should be closer to 1700 ppm.

Coupons surfaces placed in the glass cell for different concentrations of inhibitor are presented in Figure 17. In the absence of an inhibitor, the corrosion product layer was expected to be made entirely of FeCO₃. While some parts of the coupon were indeed covered with FeCO₃, most of them showed the presence of a Fe₃C layer. This is usually the case when the saturation conditions in FeCO₃ cannot be met (because of too much condensed liquid) and it is most probably due to the specificity of the experimental setup itself and the combination of stagnant condition and low pressure. Consequently, even though the steel surface appeared corroded, no clear localized corrosion could be identified in this baseline condition.
The addition of 500 ppm of inhibitor did not have any significant effect on the characteristics of the corrosion product layer, which seemed to be exclusively made of Fe₃C. Once again, no localized corrosion was observed.

Finally, in the presence of 2000 ppm of inhibitor, the corrosion product layer did show very different characteristics compared with the two previous tests. At the end of the test, some parts of the weight loss coupons seemed not to have been affected by corrosion and they remained in their original shiny polished state. The layer appeared to be made of a mixture of Fe₃C and another amorphous compound, showing the peaks of Fe, O and C. However, no typical FeCO₃ crystals could be found. No localized corrosion could be found in this glass cell test.

Part 3: Large Scale Flow Loop Experiments

The previous parts of the research work showed that 2000 ppm inhibitor PX 4856 R could effectively control top of the line corrosion when simulated in small scale apparatus under the conditions selected. The next logical step of the study was to perform some experiments in a large scale flow loop where the hydrodynamics of a typical field situation could be more closely matched.

The volatile inhibitor was tested in the 4" (0.102 m) ID, 30-metre long flow loop made of stainless steel (Figure 18). A full description of the experimental setup as well as more details on the experimental procedures can be found elsewhere.

A typical test lasted approximately four weeks and the inhibitor was injected into the tank after a few days of testing. The tests conditions presented in this paper are summarised below.

- Free HAc in the bulk: 500 ppm
- Inhibitor concentration: 2000 ppm in the tank
- Condensation rate: 0.25 ml/m².s
- CO₂ partial pressure: 7 bars
- Temperature: 70 deg C
- Corrosion rate measurement: ER probe and X-65 coupons (polished 600 grid sand paper)
- Test duration: 4 weeks
- Gas flow velocity: 5 m/s

Corrosion rates were measured by ER probes and weight loss coupons made of X65. For comparison, coupons and probes were installed at both bottom line and top of line of the test sections. Weight loss coupons were introduced and replaced at different times during the test, ensuring that a baseline corrosion rate without an inhibitor was always measured before the injection of this inhibitor. It is also worth mentioning that the ER probe was removed from the loop before the injection of the inhibitor. It was cleaned, slightly polished and reinstalled immediately after the chemical injection.

The results obtained with the ER probe located at the top of the line are shown on Figure 19. The ER probe predicted an un-inhibited corrosion rate at 4 mm/year while weight loss coupons gave only 2 mm/year for a similar exposure time (2 days). The fact that the corrosion prediction of the ER probe is not very accurate is in a way common of this kind of method in TLC application. The presence of droplets on the metal surface and the consequent intermittent wetting may lead to conditions difficult to process with regard to ER technology. However, it did react strongly to the injection of inhibitor with the corrosion rate dropping by 50%. The general corrosion rate stabilized after 28 days of testing at
1.2 mm/year for the ER probe. The weight loss corrosion rate at the top of the line was measured around 0.4 mm/year after 28 days of exposure to the inhibited environment. In most of the conditions tested, the average corrosion at the top of the line measured with weight loss decreased by 40 to 60%.

The surface analysis of a few top of the line coupons is shown in Figure 20 which presents pictures of the weight loss coupons immediately after their removal from the loop (with the corrosion product layer intact) and also after removal of the corrosion layer (for the corrosion measurements).

Without an inhibitor, the coupons were covered with a dense grey layer typical of top of the line experiments. Clear evidence of localized attack can be seen on the surface of the coupons which was confirmed after cleaning of the coupons. The corrosion film was made mostly of FeCO₃ as was expected in these conditions (high pH and high Fe²⁺ in the droplets of condensed liquid lead to supersaturation in FeCO₃). The corrosion was more severe around the edges where condensed water accumulated due to gas flow.

The morphology of the corrosion product film was very different when 2000 ppm of inhibitor was present in the bulk liquid phase (tank). No FeCO₃ crystals could be found on the metal surface. Instead, a seemingly amorphous layer covered the entire surface of the coupon. The peak of sulfide was seen on the EDS analysis which was once again unexpected as the element was not supposed to be present in the experimental conditions. The chemical composition of the inhibitor is not known but sulfur compound is typically encountered in this type of chemical. This could be another indication that the inhibitor can indeed travel to the top of the line. The extent of localized corrosion observed on the coupons was also significantly decreased in the presence of inhibitor.

As mentioned above, some coupons were installed at the bottom line although bottom of the line corrosion was not the primary focus of the study. It is important to mention that only the vapor phase containing the volatile inhibitor was circulated in the loop and that no liquid carry-over from the tank was allowed. In this way, a small stream of condensed liquid formed and flowed over the weight loss coupons at the bottom of the line. In many cases, the liquid stream was not large enough to fully cover the coupon surface, leaving some areas more corroded than others. However, when the coupons surface was fully immersed in the condensation water stream, very good protection was achieved as shown in the pictures of the coupons in Figure 21. Theses photos show clearly that the condensed water is properly inhibited as there is no sign of general or localized corrosion.

The potential efficiency of the volatile inhibitor was confirmed by these sets of glass cell and flow loop experiments, freeing the way for the initiation of field testing.

**DISCUSSION**

By simulating TLC in the presence of acetic acid with 0.6 ml/m².s of condensation rate, it was demonstrated that MDEA can only neutralize the acidity of bottom line (in the reactor in the present paper). In the field, the reduction of the bottom line acidity reduces the corrosivity of the gas phase and consequently the corrosivity of the condensed water but MDEA cannot directly protect top of line corrosion.

The selected molecules, PX 4802 R, PX 4803 R and PX 4804 R can neutralize the pH of the reactor (bottom line surface in the line) and the pH of the condensed water (top of line surface in the pipe). The best neutralizing product is PX 4803R. The values of Rp measured on condensed water show
that PX 4803 R can also inhibit the top of line corrosion. This product was selected for further evaluation in the corrosion loop and then in the field.

Corrosion loop tests confirmed the preliminary laboratory tests. The general corrosion rate at the top of the line was clearly reduced by the presence of the volatile inhibitor and more importantly the extent of localized corrosion was lowered as well. In addition, no localized corrosion coupled with very low average corrosion rates was observed on the coupons placed at the bottom of the line. It is worth mentioning that the water at the bottom of the line was the condensed water containing volatile inhibitor.

After loop testing, the combined product was field tested in a gas field in Thailand\(^3\). The results are presented in a separate paper which also will be presented at NACE 2010\(^2\).

**CONCLUSIONS**

A comprehensive research study was successfully implemented in order to select and test the efficiency of a volatile inhibitor. It involved glass cell tests for the inhibitor selection and large scale flow loop experiments for the evaluation of the inhibitor performance. The possibility of TLC control by volatile inhibitors is demonstrated.

An acceptable protection level was achieved in the laboratory. Based on these results a combined product containing a volatile inhibitor and a bottom line inhibitor was formulated and tested. The performance of the combined product was found to be better than the volatile inhibitor alone.

Loop tests confirmed the preliminary laboratory tests. Localized corrosion rate and general corrosion rates were significantly reduced when the volatile inhibitor was injected.

Results of the field testing are presented in a separate paper at NACE 2010\(^2\).

**ACKNOWLEDGEMENTS**

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**REFERENCES**

### TABLE 1: COMPOSITION OF THE TESTED SPECIMENS

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![FIGURE 1: Volatile Inhibitor Testing Equipment for the Preliminary Testing](image-url)
FIGURE 2: pH of Condensed Water Versus Dosage of the 3 Selected Neutralizing Molecules

FIGURE 3: Evolution of the pH in the Reactor and in the Corrosion Cell With MDEA Concentration
FIGURE 4: Evolution of the Rp of the Condensed Water With MDEA Addition in the Reactor

FIGURE 5: Evolution of the pH in the Reactor and in the Corrosion Cell With Volatile Inhibitor Concentration
**FIGURE 6**: Evolution of the $R_p$ of the Condensed Water With Volatile Inhibitor Addition in the Reactor

**FIGURE 7**: Evolution of the $R_p$ of the Condensed Water With Increasing Inhibitor Concentration
FIGURE 8: Condensed Water pH and Polarization Resistance With 1000 ppm of Acetic Acid in the Reactor

FIGURE 9: Comparison of Volatile Product and Combined Product Performances.

FIGURE 10: Coupons Without (Left) and With (right) Volatile Product in Presence of 1000 ppm Acetic Acid.
FIGURE 11: Performance of the Combined Product for Bottom Line Protection.

FIGURE 12: Experimental Set-up of Liquid/vapor Equilibrium Study
**FIGURE 13:** Inhibitor Volatility* Versus pH in the Tank  
* Ratio of the concentrations of inhibitor measured in the condensed liquid and in the tank

**FIGURE 14:** Experimental Set-up of the Small Scale Corrosion Study
FIGURE 15: Experimental Set-up of the Small Scale Corrosion Study
View of the Glass Cell lid (No Inhibitor Present)

FIGURE 16: Glass Cell Experiments. ER Probe Results With Different Inhibitor Concentrations
FIGURE 17: Coupons Surfaces After 2 Weeks of Exposure to 0 ppm (left), 500 ppm (Middle) and 2000 ppm (right) of Corrosion Inhibitor (Same Magnification of X 50)

FIGURE 18: TLC Loop Used for the Testing of the Volatile Corrosion Inhibitor.
FIGURE 19: Flow Loop Test - ER Probe Results

Corrosion rate (mm/year)

Exposure time (days)

Inhibitor injection

Top of the line

FIGURE 20: Coupon Placed in the TLC Loop at Top of the Line
No Inhibitor (left) and With 2000 ppm of Inhibitor in the Bulk Solution (Right)