AUTOCLAVE CORROSION INHIBITOR EVALUATION

Cormetrics Job# 23-XXX

Activity	Date	Testing	Reviewed by
Report	November 5, 2022	M. Koldijk	F. Hornsby

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TABLE OF CONTENTS

1 INTRODUCTION

Cormetrics was retained to perform sweet autoclave testing on three batch and two continuous corrosion inhibitors. The goal of the testing was to evaluate the relative performance of the inhibitors against a blank.

2 BRINE

Synthetic brine was prepared for use in the testing with the following brine chemistry.

Cations				Anions		
Sodium (mg/L)	Potassium (mg/L)	Calcium (mg/L)	Magnesium (mg/L)	Sulphate (mg/L)	Bicarbonate (mg/L)	Chloride (mg/L)
13,273	121	45	122	12	500	21,700

Table 1: Synthetic Brine Composition

The original brine composition was altered by lowering the reported bicarbonate content from 2216 mg/L to 500 mg/L to reduce the calcite scaling tendency and increase the corrosivity. The prepared brine is a low TDS brine with a moderate bicarbonate content resulting in an average buffering capability. The synthetic brine was prepared fresh prior to use in the test and was pre-purged with pure CO_2 for a minimum of 2 hours. The pH of the pre- and post-test fluids was measured and recorded.

3 CHEMICAL SAMPLES

The following table lists the inhibitors received and the product application guideline.

Inhibitor Name	Application	Treatment Rate
Continuous A	Continuous	20, 100 & 300 ppm
Continuous B	Continuous	100 ppm
Batch A	Batch	1:1 w/ Diesel
Batch B	Batch	1:1 w/ Diesel
Batch C	Batch	1:1 w/ Diesel

Table 2: Inhibitors Received

4 AUTOCLAVE TESTING

The inhibitors were evaluated under sweet conditions in the autoclave apparatus. A blank cell was included to provide a baseline corrosion rate and evaluate relative inhibitor performance.

4.1 Gas Composition

System gas analysis indicates a 6 mol% CO_2 content with a total pressure of 1000 kPa resulting in a partial pressure of 60 kPa CO_2. There is no H_2S with the produced gas. Autoclave charge pressures used in the evaluation are listed in the following table:

Total Pressure (kPa)	CO_2 (kPa)	N_2 (kPa)
1000	60	Balance

Table 3: Autoclave Gas Pressures

4.2 Test Temperature

Temperature specified in protocol was 30°C. Sealed autoclaves are placed inside individual mantles and heated to the test temperature. Proportional temperature controllers accurate to +/- 2°C are used to maintain set temperature. The test duration was increased from a typical 4-day exposure to 7 days with the intention of better differentiation for pitting attack inhibition.

4.3 Inhibitor Application

The continuous inhibitors were dosed directly into the brine at the specified treatment rates.

The batch corrosion inhibitors were diluted at a 1:1 ratio with diesel for electrode filming. The electrodes were immersed in the batch inhibitor solution for 10 seconds followed by a 1-minute drip and two consecutive one-minute rinses in purged synthetic brine.

The autoclaves were then sealed and purged in cell for 15 minutes with pure CO_2 to sweep the head space.

4.4 Autoclave Test Apparatus

The autoclaves used by Cormetrics are constructed of UNS N10276 and have a capacity of approximately 300 mL. Tests were conducted with 250 mL of synthetic brine in each cell, approximately 80% full. A three-electrode assembly is suspended from the lid of the autoclave, keeping the bottom clear for a Teflon-coated magnetic stir-bar. Configuration of the electrodes is a closely spaced equilateral triangle, with each cylindrical electrode having a 6.3 ø x 38 mm geometry. Reference electrodes are made from UNS N10276, while working and counter electrodes are UNS G10180 carbon steel. Electrodes are solvent rinsed and weighed prior to the

commencement of the test period. A surface area of 7.92 cm^2 is used in corrosion rate calculations. Weight loss corrosion rates are reported as an average of the working and counter electrodes.

The temperature of the fluid in the autoclave is sensed by a thermistor probe, held at the center of the cell by a UNS N10276 sleeve. Charging of the autoclave is by means of an offset UNS N10276 tube, fitted with a pressure gauge and sour-service valve.

LPR measurements were obtained at 30-minute intervals by connecting each cell to a Gamry Interface 1000 potentiostat, via a Gamry ECM8 multiplexer.

Assembled autoclave test cells with heater and stirrer, test cell base and lid with LPR poles

5 DISCUSSION

Appendix I contains graphs of the Linear Polarization Resistance (LPR) corrosion rate measurements. Weight loss corrosion rates and electrode photographs are presented in Appendix II.

5.1 Autoclave Discussion

The following tables summarize the data from the autoclave corrosion test:

Inhibitor/Dose	Initial pH	Final pH	Post-Test Fluid Observations
Blank	5.49	6.06	Clear
Continuous Inhibitor A (20 ppm)		5.47	Clear
Continuous Inhibitor A (100 ppm)		5.42	Clear
Continuous Inhibitor A (300 ppm)		5.49	Slightly Hazy
Continuous Inhibitor B (100 ppm)		5.50	Clear
Batch Inhibitor A (1:1 w/ Diesel)		5.48	Clear
Batch Inhibitor B (1:1 w/ Diesel)		5.61	Clear
Batch Inhibitor C (1:1 w/ Diesel)		5.52	Clear

Table 4: AC Testing pH Data Summary

The initial pH was mildly acidic which decreased the potential for scaling in the test conditions and increased general corrosivity. There was minor change in pH in the inhibited cells due to the low general corrosion rates.

Inhibitor/Dose	First 4 Hours Average LPR (mpy)	Last 4 Hours Average LPR (mpy)	Average LPR (mpy)	Weight Loss (mpy)	Weight Loss Protection (%)	Pitting Rate (mpy)
Blank	22.93	13.92	47.25	22.16	-	N/A
Continuous Inhibitor A (20 ppm)	2.04	1.20	1.29	1.28	94.2	183
Continuous Inhibitor A (100 ppm)	1.84	0.13	0.45	0.81	96.3	156
Continuous Inhibitor A (300 ppm)	1.94	0.06	0.36	0.76	96.6	N/A
Continuous Inhibitor B (100 ppm)	2.31	0.12	0.53	1.04	95.3	156
Batch Inhibitor A (1:1 w/ Diesel)	0.05	0.02	0.02	0.69	96.9	N/A
Batch Inhibitor B (1:1 w/ Diesel)	0.12	0.22	0.23	0.70	96.8	209
Batch Inhibitor C (1:1 w/ Diesel)	0.16	0.03	0.07	0.85	96.1	N/A

Table 5: AC Test Corrosion Data Summary

The blank displayed an initial LPR corrosion rate that trended up to a high of ~90 mpy before trending down and stabilising around 14 mpy. The decreasing LPR trend and stabilisation indicates the formation of a passivating corrosion product scale. The high LPR corrosion rate indicates the potential for aggressive corrosion on unprotected steel in the conditions as evaluated. The post-test electrodes had an overall moderate general corrosion with a weight loss corrosion rate of 22.16 mpy.

The Continuous Inhibitor A at 20 ppm displayed an initial LPR corrosion rate of 2 mpy that trended down slowly and stabilised just above 1 mpy. Typically, an LPR corrosion rate that trends below 1 mpy indicates a good performing inhibitor in the conditions as tested. The post-test electrodes had a light patchy etching attack with edge attack and pitting. The edge attack occurred where a crevice is formed between the electrode and the mounting sleeve which is a more difficult area to mitigate corrosion. The pitting attack indicates an area of weaker or incomplete inhibitor film on the electrode surface. The maximum measured pit depth was 3.5 mils equivalent to a 183 mpy pitting rate. The weight loss corrosion rate was 1.28 mpy equal to a 94.2% protection level compared to the blank, indicating a low general corrosion rate with pitting attack.

Increasing the treatment rate of Corrosion Inhibitor A to 100 ppm reduced the general corrosion rate but was unable to prevent the localised pitting attack noted at the 20-ppm dose. The LPR corrosion rate trended down from an initial LPR corrosion rate of 2.5 mpy and stabilised well below 1 mpy. The weight loss corrosion rate was 0.81 mpy equal to a protection level of 96.3% as compared to the blank. The pitting attack of 156 mpy indicates this inhibitor is not able to eliminate localised corrosion activity at this treatment level under the test conditions.

Continuous Inhibitor A at 300 ppm treatment rate had the lowest average LPR and weight loss corrosion rate of the continuous products evaluated. The LPR corrosion rate trended below 1 mpy after 13 hours exposure and stabilised below 0.1 mpy at the end of the 7-day exposure. This indicates the presence of good protective inhibitor film. The post-test electrodes displayed light patchy corrosion and mild edge attack. There was no pitting visible on the electrodes. The weight loss corrosion rate was 0.76 mpy equal to a 96.6% protection level.

Continuous Inhibitor B at 100 ppm had an initial LPR corrosion rate of 3.84 mpy that trended down and stabilised around 0.1 mpy. The LPR trend indicates it was slower to film out than the Corrosion Inhibitor A product at the same treatment rate. The post-test electrodes had a light patchy etching with edge attack and pitting. The maximum measured pit depth was 3 mils equivalent to a 156 mpy pitting rate. The weight loss corrosion rate was 1.04 mpy equal to a 96.9 protection level.

Batch inhibitor A had a low stable LPR corrosion rate that trended below 0.1 mpy for the duration of the test. This indicates the inhibitor provided a good protective film. Inhibitor A had the lowest average LPR, and weight loss corrosion rate of the batch products evaluated. The post-test electrodes displayed an overall dull electrode surface with mild general corrosion and no localised attack. The weight loss corrosion rate was 0.69 mpy equal to a 96.9% protection level.

Batch inhibitor B had an LPR corrosion rate that trended up initially prior to stabilising near 0.2 mpy. The initial increase in corrosion rate may indicate a weakening or incomplete batch film as there was localised pitting attack present on the post-test electrodes. The maximum measured pit depth was 4 mils equivalent to a 209 mpy pitting rate. The general corrosion rate was low at 0.70 mpy as the remainder of the electrode had a light patchy etching attack.

Batch inhibitor C displayed a low LPR corrosion rate that trended down from an initial high of 0.22 mpy and stabilised slightly above that of batch inhibitor A. The low stable LPR corrosion rate indicates a good protective batch film. The post-test electrodes displayed an overall dull general corrosion with no localised pitting attack. The weight loss corrosion rate was 0.85 mpy equal to a 96.1% protection rate.

6 CONCLUSION

The blank indicated moderately corrosive conditions as tested with a weight loss corrosion rate of 22.2 mpy. The test conditions with 60 kPa CO_2 and 30°C test temperature provided a strong baseline corrosion rate for inhibitor evaluation.

Both continuous inhibitors provided good general corrosion protection at the evaluated treatment rates with weight loss protection levels all above 94%. Increasing the treatment rate of Continuous Inhibitor, A improved its performance where the 300-ppm treatment level was able to prevent pitting attack. The presence of pitting downgrades the performance ranking for an inhibitor at the tested treatment level. Continuous Inhibitor B was slightly less effective when comparing the 100-ppm treatment level although the difference in performance was mild.

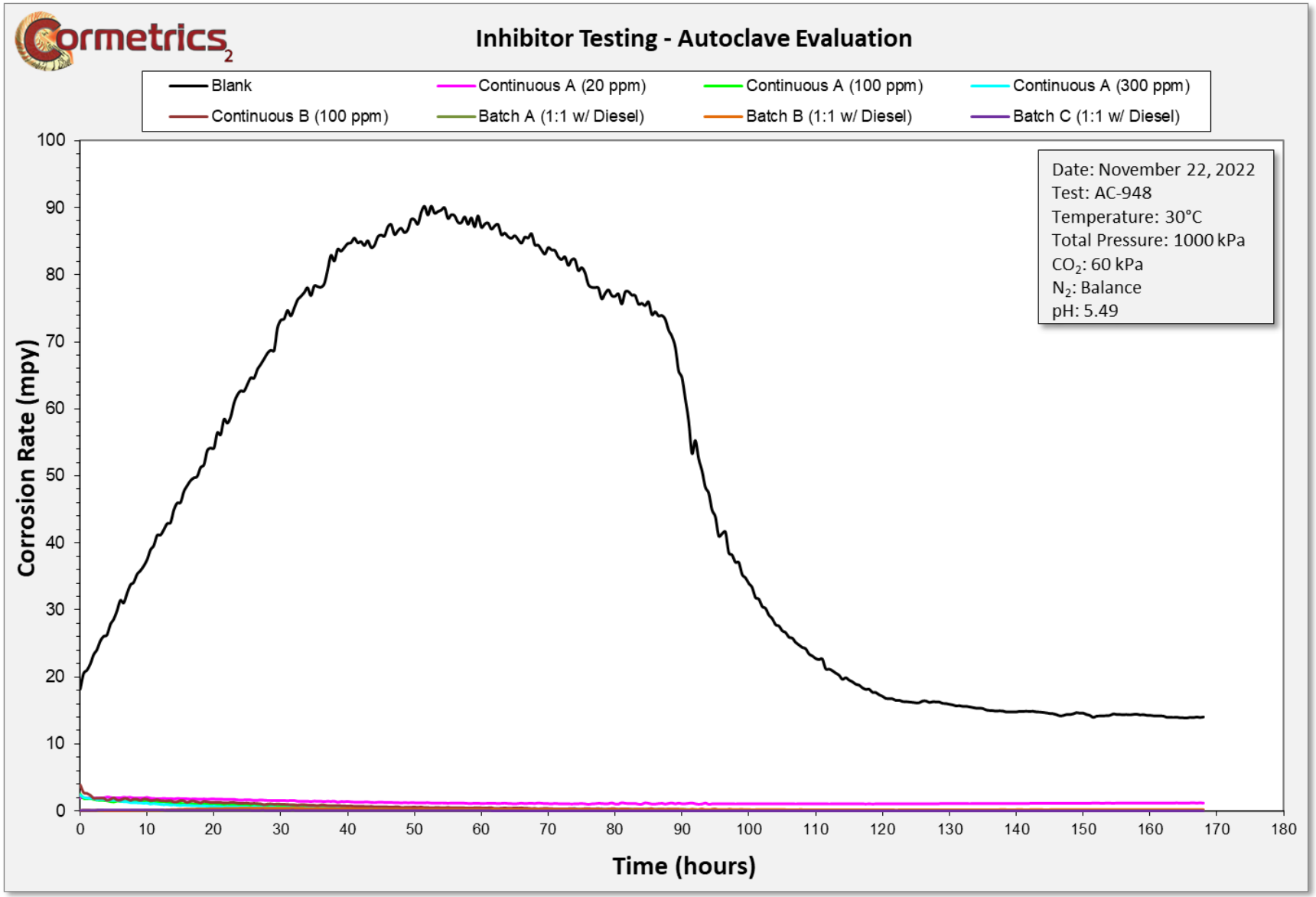
Batch inhibitors A and C provided good inhibition in the conditions as evaluated with low weight loss corrosion rates and no pitting attack. Batch Inhibitor A is a slightly better performer as it had the lowest LPR, and weight loss corrosion rates of the batch products evaluated. Batch Inhibitor B displayed pitting attack and its performance ranking is significantly reduced.

Respectfully,

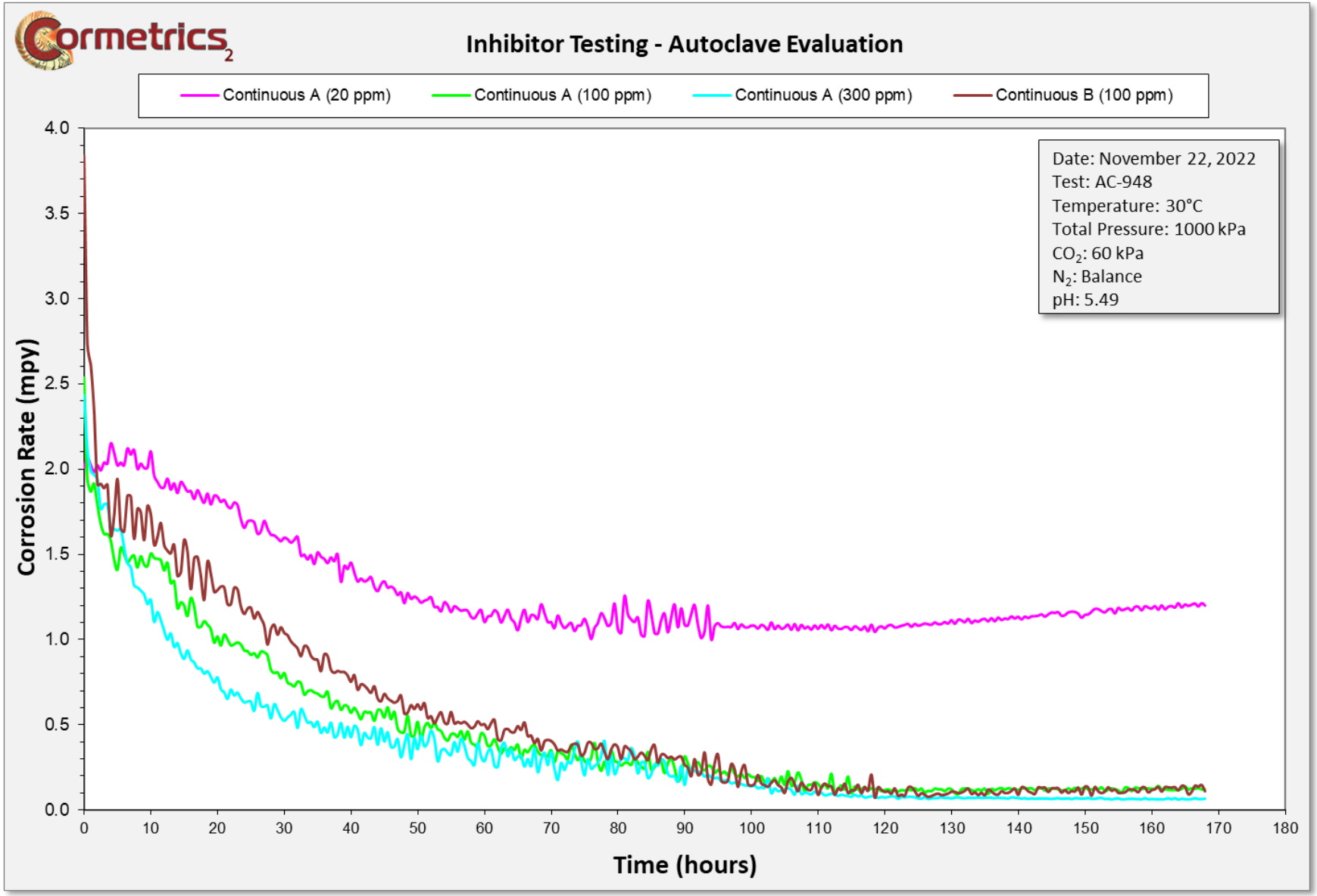
Frank Hornsby
President – **Cormetrics 2 Ltd**

Please note, all inhibitor samples and electrodes are stored for 6 months prior to disposal.

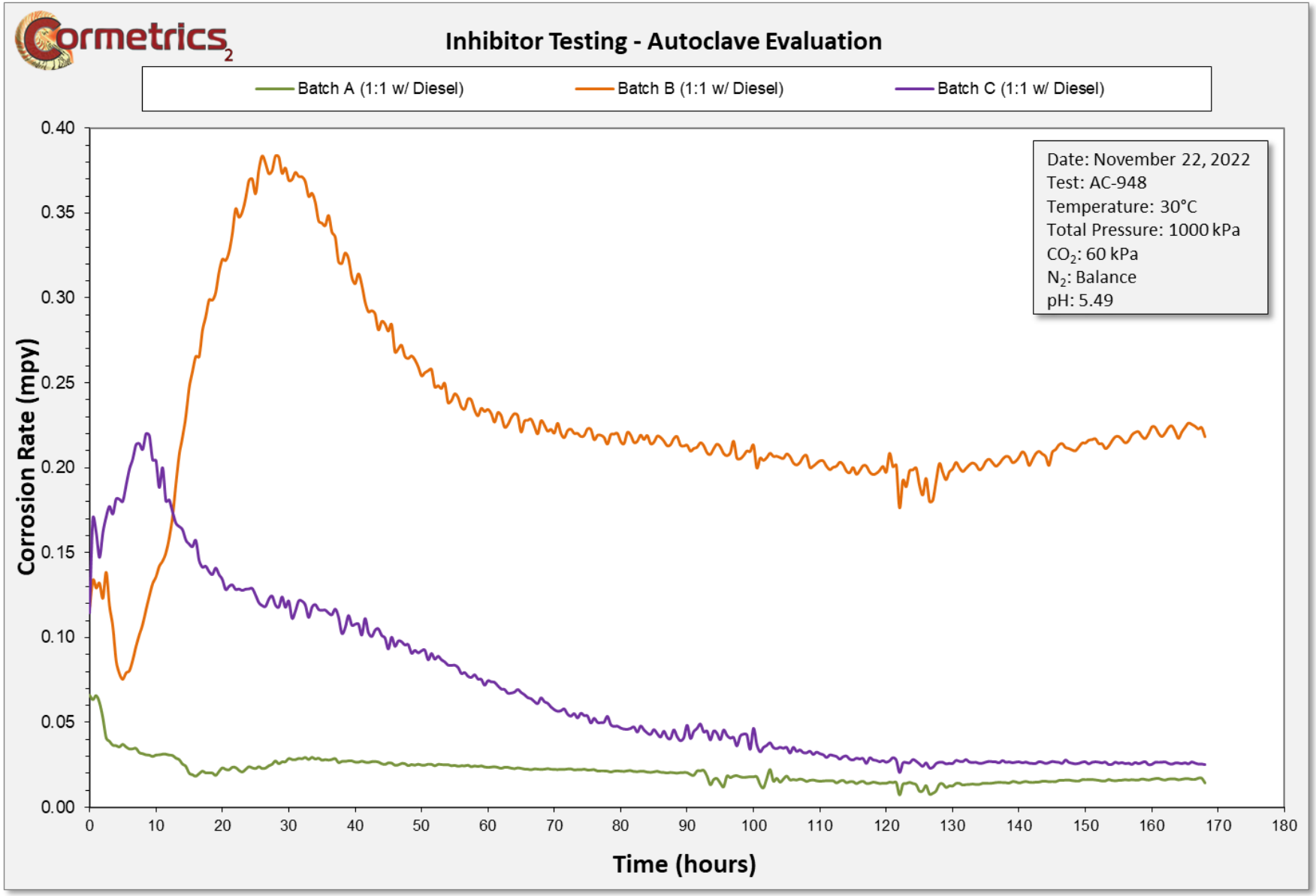
APPENDIX I – LPR DATA



Graph 1: AC Test LPR Data



Graph 2: AC Test LPR Data (Continuous Inhibitors Only)



Graph 3: AC Test LPR Data (Batch Inhibitors Only)

APPENDIX II – WEIGHT LOSS & VISUAL DATA

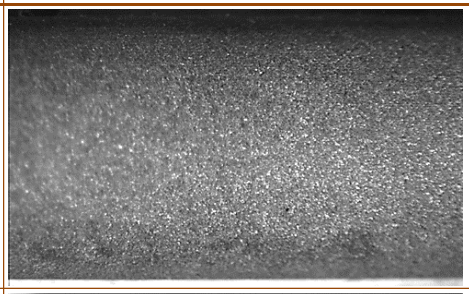
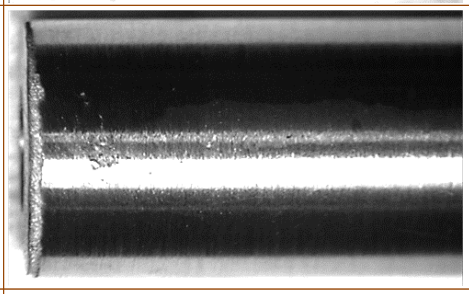
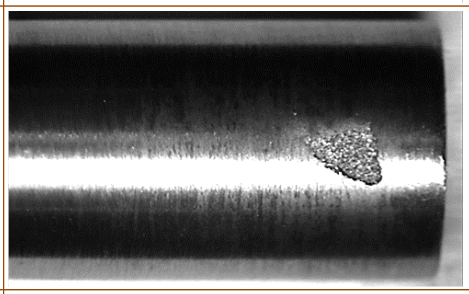
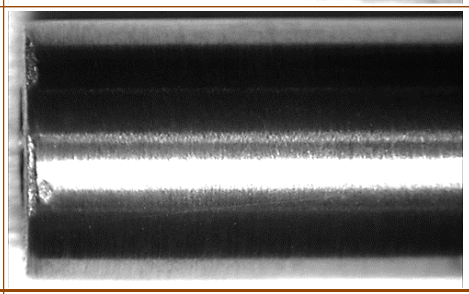
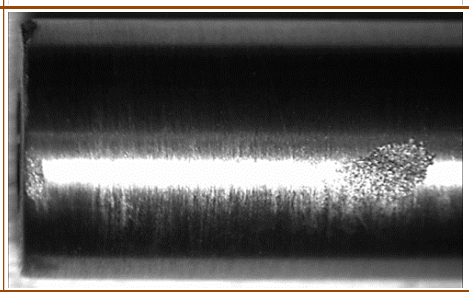
Inhibitor	Weight Loss		Visual Description	Pit Depth		Electrode Photo	
	(mg)	(mpy)		(mils)	(mpy)	(Full)	(Close-Up)
Blank	67.13	22.16	Overall moderate general corrosion	N/A	N/A		
Continuous A (20 ppm)	3.87	1.28	Light patchy etching w/ edge attack & pitting	3.5	183		
Continuous A (100 ppm)	2.47	0.81	Light patchy etching w/ pitting	3.0	156		
Continuous A (300 ppm)	2.30	0.76	Light patchy etching w/ edge attack	N/A	N/A		
Continuous B (100 ppm)	3.16	1.04	Light patchy etching w/ edge attack & pitting	3.0	156		

Table 6: AC Weight Loss Data & Electrode Photographs (Continuous Inhibitors)

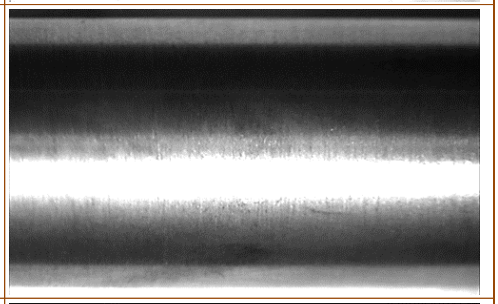
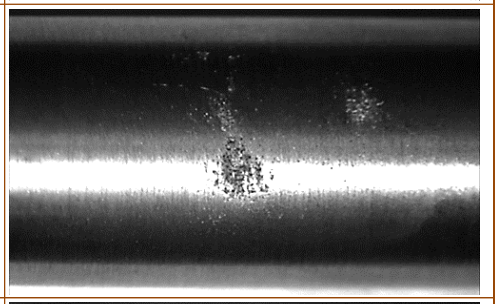
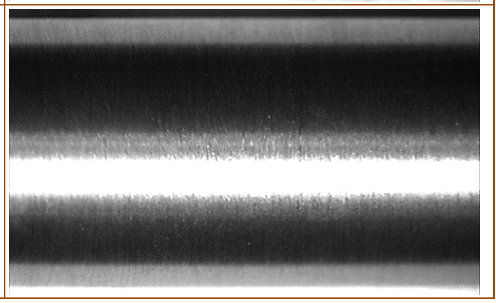
Inhibitor	Weight Loss		Visual Description	Pit Depth		Electrode Photo	
	(mg)	(mpy)		(mils)	(mpy)	(Full)	(Close-Up)
Blank	67.13	22.16	Overall moderate general corrosion	N/A	N/A		
Batch A (1:1 w/ Diesel)	2.10	0.69	Overall dull general corrosion	N/A	N/A		
Batch B (1:1 w/ Diesel)	2.13	0.70	Light patchy etching w/ pitting	4.0	209		
Batch C (1:1 w/ Diesel)	2.58	0.85	Overall dull general corrosion	N/A	N/A		

Table 7: AC Weight Loss Data & Electrode Photographs (Batch Inhibitors)