

Autoclave Evaluation of Three Continuous Corrosion Inhibitors

Prepared for:

ABC Company

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Introduction

ABC Company submitted three chemicals for autoclave testing according to the protocol developed for the XYZ facility evaluation. The products were labeled 'A', 'B', and 'C' and were tested at a concentration of 1000ppm, based on the volume of water in the cell (200ml).

The running order of the test is shown below in Table 1.

Table 1. Experimental design for autoclave testing.

	Test
Cell 1	Blank
Cell 2	Product 'A'
Cell 3	Product 'B'
Cell 4	Product 'C'

Test Protocol

Brine

Produced water from the XYZ system was used as the test fluid. The water chemistry is shown below in Table 2.

Table 2. XYZ Facility Produced Water Chemistry

Cations	Concentration (mg/l)	Anions	Concentration (mg/l)
Sodium	20,000	Chloride	31,500
Potassium	500	Bicarbonate	800
Calcium	1600	Sulfate	5500
Magnesium	750		
Barium	0.5		
Strontium	50		

Two litres of produced brine were charcoal filtered and purged with CO₂ for two hours. The brine was then purged with the test gas mixture (28% CO₂, balance H₂S) for 30 minutes. Any precipitates and charcoal fines were removed from the water by paper filtration and the colorless, solids-free brine was then re-purged for 30 minutes with the acid gas mixture.

Autoclave Cell

The autoclaves are constructed of Hastelloy 276-C and have a capacity of approximately 300ml. The tests were carried out with 200ml of brine in each cell, i.e. the autoclaves were approximately two-thirds full.

A three-electrode assembly is suspended from the lid of the autoclave, keeping the bottom clear for a Teflon-coated magnetic stir-bar. The configuration of the electrodes is a closely spaced equilateral triangle, with each cylindrical electrode having 1.5 " x 0.25" geometry. The reference electrodes are made from Hastelloy 276-C, while the working and counter electrodes are 1018 carbon steel. The electrodes are purchased from Stellar Manufacturing and are solvent rinsed and weighed prior to the commencement of the test. The electrodes have a surface area of 7.92cm² and this value is used throughout this report for corrosion rate calculations.

The temperature of the fluid in the autoclave is sensed by a thermistor probe, held at the center of the cell by a Hastelloy sleeve. Purging and charging of the test liquid is accomplished by means of an offset Hastelloy tube, fitted with a pressure gauge and sour-service needle valve. Each of the four cells is also equipped with a pressure relief valve in order to purge the test liquids directly in the cells once they have been filled and sealed.

LPR measurements were obtained at 30-minute intervals by connecting each cell to a Gamry PC4-300 potentiostat and controller, via a Gamry ECM8 multiplexer. Data acquisition was by means of Gamry's DC105 software package.

Autoclave filling and pressuring procedure

200ml of purged brine are placed in a separating funnel, followed by 20ml of XYZ condensate. After 1000ppm of the appropriate inhibitor product has been injected into the aqueous phase, the funnel is sealed and vigorously shaken. The fluids are then allowed to settle until a clear interface has developed (typically 3-5 minutes). The aqueous phase (200ml) is drawn off into an autoclave cell.

When all four cells have been filled and sealed the test liquid is purged with the same acid gas mixture used to prepare the brine (28% CO₂, balance H₂S). The autoclave is charged to 240 psi and allowed equilibrate for 30-40 minutes. Any reduction in pressure is then restored, by recharging the autoclave from the mixed gas cylinder. At this point, the total pressure is brought up to 850psi (at room temperature) with high-pressure methane.

When all four cells have been prepared, they are placed inside individual heating mantles and brought to temperature (65°C) via proportional temperature controllers. This takes 5-10 minutes. Gentle stirring of the autoclave fluids is achieved by placing the heating mantles on stirrer plates. Finally the Gamry instrument is programmed to start collection of corrosion potential and LPR data.

Results and Discussion

Figure 1 below, illustrates the corrosion potential of the working electrode in each cell. In two of the three cells (the ones inhibited with products A and C), the corrosion potential becomes more noble with time, eventually providing a 'steady-state' potential (vs. Hastelloy C) of -8 to -15 mV. While the corrosion potential of the cell inhibited with product B also becomes more noble initially, it starts to fluctuate about 20 hours into the test, before becoming more anodic at -100 mV or so (vs. Hastelloy C).

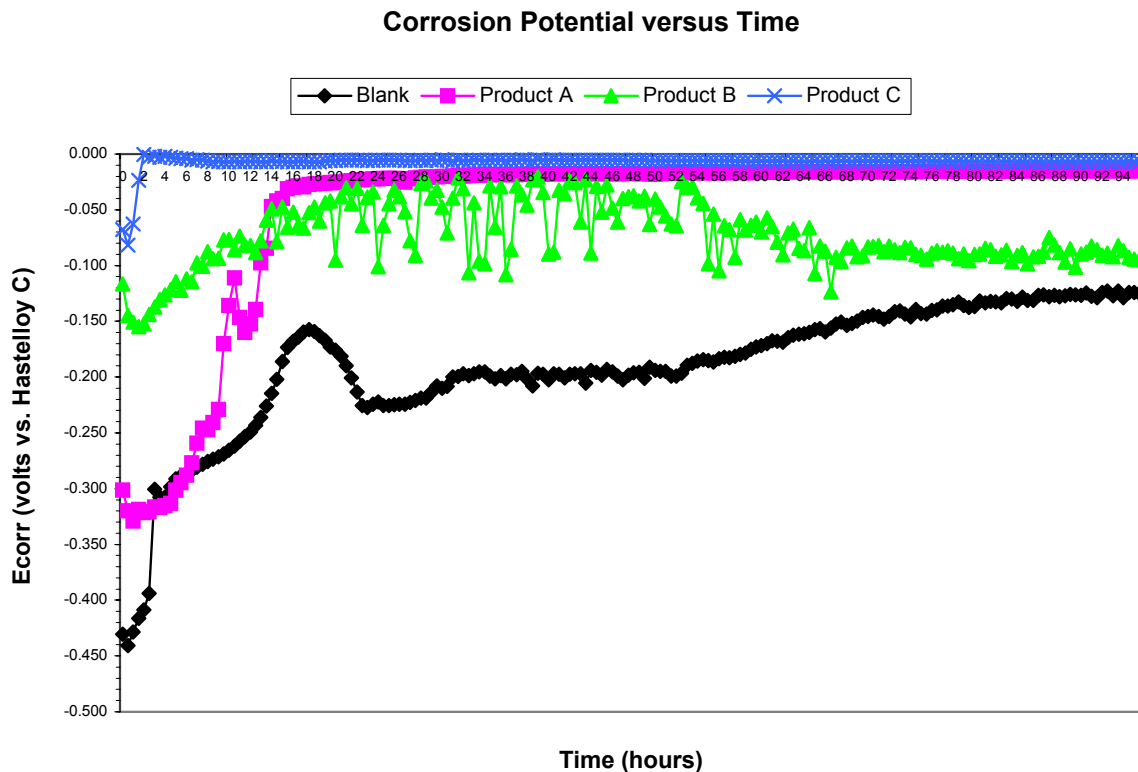


Figure 1. Corrosion potential *versus* time.

The rate at which the steady state corrosion potential is reached is important. Here there are clear differences between effectiveness of the three products. Product 'B' was the weakest, with E_{corr} in cell B fluctuating and never reaching values as cathodic as those in cells A and C. Product 'C' was the strongest product (as judged by the trend in E_{corr} with time), with cell 'C' reaching an E_{corr} value of -10 mV or so (vs. Hastelloy C) within a couple of hours of the start of the test.

The LPR data collected from all 4 cells is shown below in Figures 2 and 3. Figure 2 shows clearly that the corrosion rate in the 'blank' cell is significantly greater than in the inhibited cells. The same data are provided on an expanded scale in Figure 3 to highlight the differences between the three products.

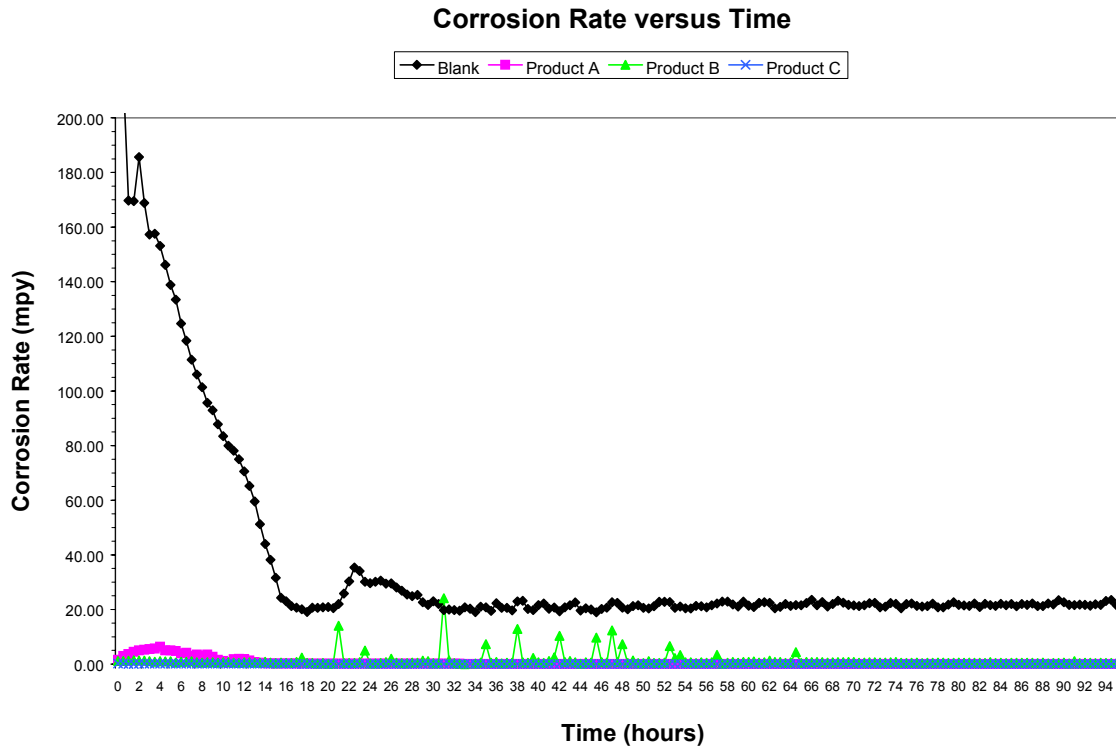


Figure 2. Corrosion rate vs. time

Figure 3 demonstrates clearly the differences in corrosion rate seen in the three inhibited cells. Product C has the greatest effectiveness, with the corrosion rate recorded by LPR staying very low and steady throughout the test. Product A achieves a very low steady-state corrosion rate which takes approximately 14 hours for such to occur. The fluctuations in the corrosion potential in the cell inhibited with product B are also seen in the polarization resistance, resulting in the extremely 'noisy' trace seen in Figure 3.

Both the E_{corr} and LPR data suggest the following rankings for the three inhibitor products: $C > A > B$.

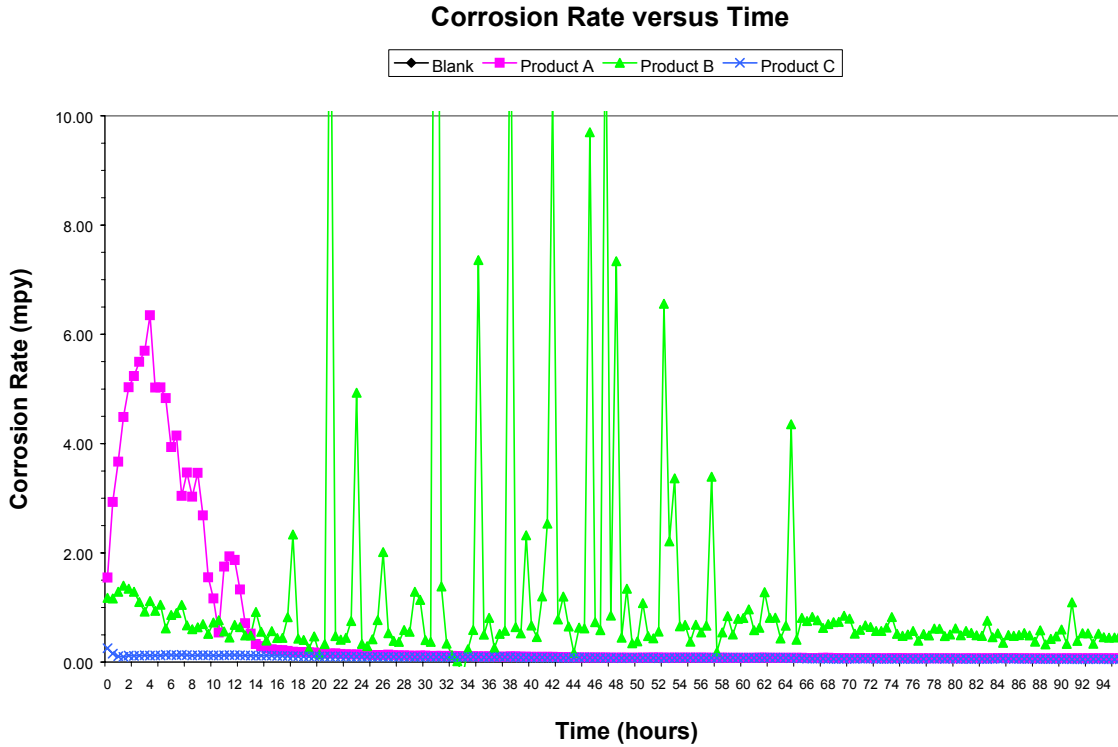


Figure 3. Corrosion rate vs. time (expanded scale).

Gravimetric and visual inspection results from the inhibited cells are found below in Table 3.

Table 3. Gravimetric and visual examination results for the blank and three continuous inhibitors.

Electrode	Wt. Loss (mg)	C.R. (mpy)	Visual
Blank	22.0	22.1	Extensive general corrosion. Broad, 'saucer-like' H ₂ S pits developing.
Product A	1.0	1.0	One area of etching with pit initiation sites; one smaller area of pit development.
Product B	1.4	1.4	One broad area of etching, with pit initiation sites on one side.
Product C	0.7	0.7	Scattered, small areas of light etching.

The damage on each of the working electrodes is provided in more detail in Figures 4 through 7 below.

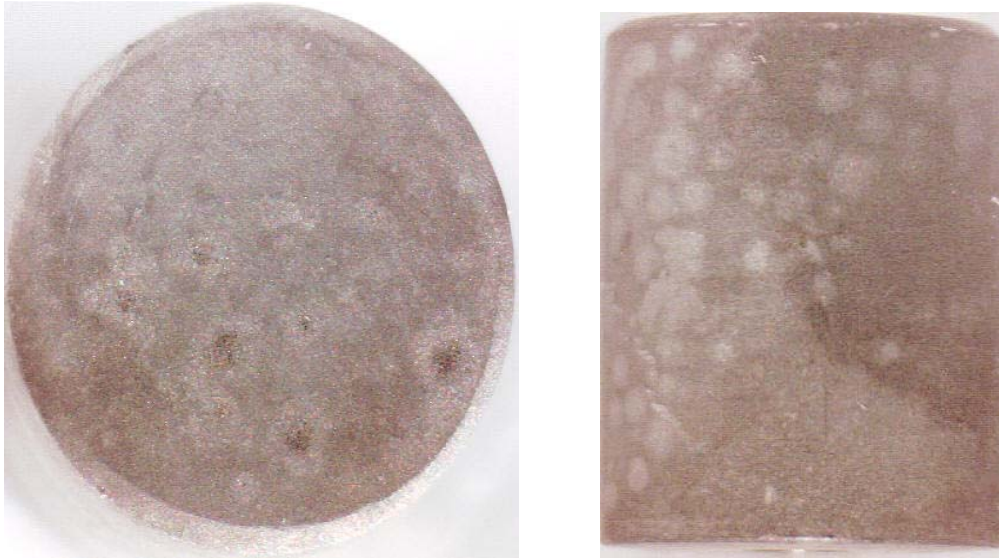


Figure 4. Blank

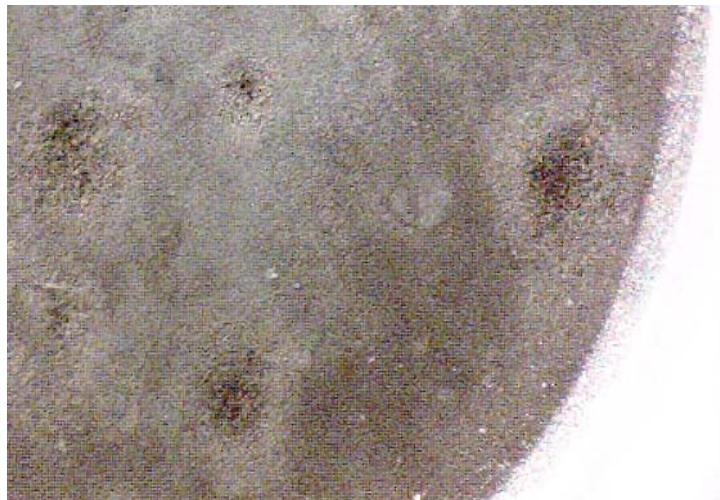


Figure 4A. Blank. Damaged area at x20 magnification.



Figure 5. Product A.



Figure 6. Product B.

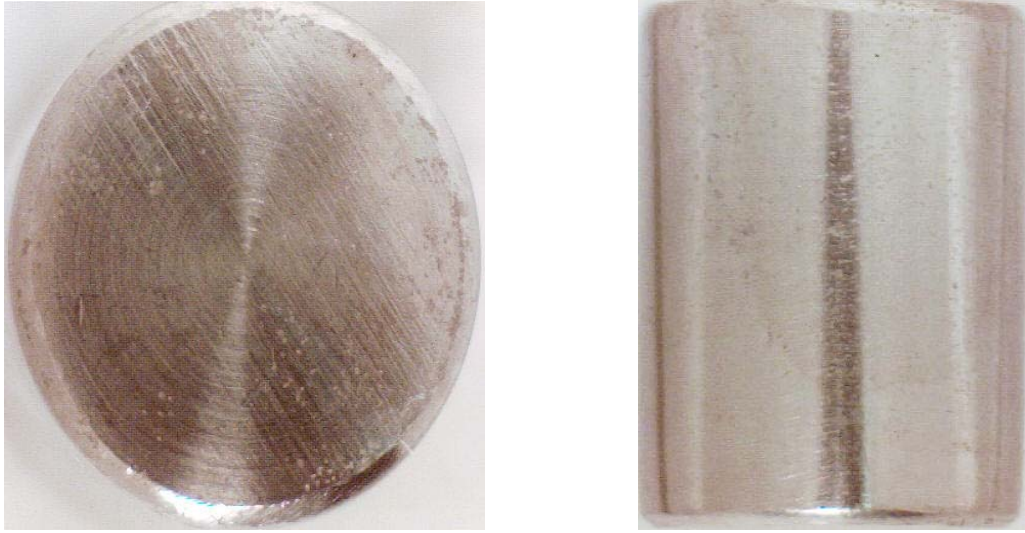


Figure 7. Product C

Summary & Conclusions

1. The blank corrosion rate was 22.1 mpy (by gravimetric), 20-22 mpy (by LPR).
2. The blank electrode had extensive general corrosion damage and 'saucer-like' H₂S pits developing.
3. The corrosion potential data showed several features. Two of the three inhibited cells (A and C) achieved 'steady-state' E_{corr} values of -8 to -15mV (vs. Hastelloy C), suggesting low, stable corrosion rates. Cell C achieved this 'steady state' value of E_{corr} very quickly. The ranking of the inhibitors according to this criterion is as follows: C > A > B.
4. The LPR data shows that only products A and C were able to achieve a steady state general corrosion rate below 2mpy. The fluctuations seen in the corrosion potential from cell B were also seen in the LPR data from that cell, making this the least preferred product as judged by LPR. The overall ranking was C > A > B.
5. The gravimetric data suggested the following ranking of the inhibitors: C > A > B.
6. Visual inspection supported the ranking given in points 3, 4 and 5: C > A > B.
7. All four assessment techniques (corrosion potential, LPR, gravimetric measurements and visual observation of the coupons) suggest the following ranking be given to the three inhibitors tested. Product C is the most effective, followed by Product A and then Product B.