NASA Technology Evaluation for Environmental Risk Mitigation Kennedy Space Center, FL 32899

NASA and ESA Collaboration on Alternative to Nitric Acid Passivation FINAL Test Report

August 31, 2016

NASA Contract: NNH15CM58Z Task Order: NNH16CP29D





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Final Test Report: Parameter Optimization of Citric Acid Passivation for Stainless Steel Alloys

August 31, 2016

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1 Introduction

1.1 Background

National Aeronautics and Space Administration (NASA) Headquarters chartered the Technology Evaluation for Environmental Risk Mitigation Principal Center (TEERM) to coordinate agency activities affecting pollution prevention issues identified during system and component acquisition and sustainment processes. The primary objectives of NASA TEERM are to:

- Reduce or eliminate the use of hazardous materials or hazardous processes at manufacturing, remanufacturing, and sustainment locations.
- Avoid duplication of effort in actions required to reduce or eliminate hazardous materials through joint center cooperation and technology sharing.

Corrosion is an extensive problem that affects the National Aeronautics and Space Administration (NASA) and the European Space Agency (ESA). The damaging effects of corrosion result in steep costs, asset downtime affecting mission readiness, and safety risks to personnel. Consequently, it is vital to reduce corrosion costs and risks in a sustainable manner.

NASA and ESA have numerous structures and equipment that are fabricated from stainless steel. The standard practice for protection of stainless steel is a process called passivation. Passivation is defined by The American Heritage Dictionary of the English Language as "to treat or coat (a metal) in order to reduce the chemical reactivity of its surface." Passivation works by forming a shielding outer (metal oxide) layer that reduces the impact of destructive environmental factors such as air or water. Consequently, this process necessitates a final product that is very clean and free of iron and other contaminants.

Typical passivation procedures call for the use of nitric acid; however, there are a number of environmental, worker safety, and operational issues associated with its use. Citric acid is an alternative to nitric acid for the passivation of stainless steels. Citric acid offers a variety of benefits including increased safety for personnel, reduced environmental impact, and reduced operational cost.

American Society for Testing and Materials (ASTM) A 967 "Standard Specification for Chemical Passivation Treatments for Stainless Steel Parts" and Aerospace Material Specifications (AMS) 2700 "Passivation Treatments for Corrosion-resistant Steel", allow for the use of citric acid in place of nitric acid for the passivation of stainless steel. Citric acid is similarly called out in the ASTM A 380 "Standard Practice for Cleaning, Descaling, and Passivation of Stainless Steel Parts, Equipment, and Systems" standard. Unfortunately, specific processing parameters are not specified in the standards.

Citric acid passivation is not a new technology; it was developed (many years ago) for the beverage industry in Germany to process containers that were free of iron which causes an unwanted taste to the beverage. It was determined that nitric acid passivation could not provide the degree of

passivation required. Other industries in the U.S. have only recently begun using citric acid. There are a variety of benefits from the substitution of citric acid for nitric acid for passivation.

This test plan "NASA and ESA Collaboration on Alternative to Nitric Acid Passivation, Project Plan; May 2016" contains the critical requirements and tests necessary to qualify citric acid as an alternative to nitric acid. These tests were derived from engineering, performance, and operational impact (supportability) requirements defined by a consensus of NASA and DoD participants.

1.2 Objective

The primary objective of this effort is to qualify citric acid as an environmentally-preferable alternative to nitric acid for the passivation of stainless steel alloys. While citric acid use has become more prominent in industry, there is little evidence that citric acid is a technically sound passivation agent, especially for the unique and critical applications encountered by NASA and ESA.

2 **Test Articles**

This section outlines the preparation of the test panels. Test panels will be 4" x 4" x 0.125" approximate. Some of the stainless alloys may not be available in 0.125" thickness. Three (3) test panel replicates per passivation procedure were used for all testing.

for this project the following stainless steel alloys were tested.												
Alloy	С	Mn	Cr	Mo	Ni	Fe	Si	Р	S	Al	Cu	Ti
304	0.08	2	18 - 20		8 - 10.5	BAL	0.75	0.04	0.03			
316	0.08	2	16 - 18	2 - 3	10 - 14	BAL	0.75	0.04	0.03			
321	0.08	2	17 - 19		9 - 12	BAL	0.75	0.04	0.03			0.7
13-8PH	0.05	0.1	12.25 - 13.25	2 - 2.5	7.5 - 8.5	BAL	0.1	0.01	0.008	.9 - 1.35		
15-5PH	0.07	1	14 - 15.5		3.5 - 5.5	BAL	1	0.04	0.03		2.2 - 4.50	
17-4PH	0.07	1	15 - 17.5		3 - 5	BAL	1	0.04	0.03		3 - 5	
17-7PH	0.09	1	16 - 18		6.5 - 7.5	BAL	1	0.04	0.03	0.75 - 1.5		
A286	0.08	2	13.5 - 16	1 - 1.5	24 - 27	BAL	1	0.025	0.025	0.35	0.5	1.9 - 2.35
AL6XN	0.03	2	20 - 22	6 - 7	23.5 - 25.5	BAL	1	0.04	0.03		0.75	

2.1 Stainless Steel Alloys

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2.2 Test Panel Preparation

The stainless steel test panels were prepared per Figure 1. This preparation process was used during a previously completed NASA TEERM Project, "Alternative to Nitric Acid Passivation; 2015". Test specimens were processed the same in all respects other than the actual passivation. Citric acid concentration/time/temperature passivation conditions were varied as part of this experimental effort.



Figure 1 – Preparation of Stainless Steel Test Panels

Prior to grit blasting, each panel was stamped with a unique alphanumeric descriptor to indicate alloy type. Each panel was grit blast with a fine steel media to impart contamination to the surface. Perfectly clean panels would be unlikely to show much corrosion within the testing period. The steel grit also removed the protective passivation layer provided by the manufacturer for storage and transport. The steel grit (ALC Item #S1587) used for the blasting process left a roughened finish on the surface of the metal.

After grit blasting, the samples were collated and organized by alloy and passivation treatment. The panels were carefully cleaned through the series of degreasing, cleaning, and rinsing steps as outlined in Figure 1.

Each panel was initially degreased with an acetone wipe of the surface. A second degreasing was performed with a detergent degreaser. The detergent degreaser consisted of a 15% by volume solution of Bruhlin 815 GD (30 minutes at 74 °C). The panels were removed from the degreaser and subsequently placed into a heated DI water bath to remove excess contaminants and the degreasing agent. The panels were then rinsed using an ambient temperature DI water spray.

Following the second rinse, a caustic (alkaline) cleaning was performed. This solution consisted of 7 ounces of Turco 4090 per gallon of DI water (20 minutes at 93 °C). After cleaning, the panels were rinsed in a heated DI water bath (5 minutes at 66 °C), followed by a DI spray-down with ambient temperature DI water. In order to verify that the degreasing and cleaning steps were performed successfully prior to passivation, the water-break of the final rinse was inspected to ensure appropriate removal of organic contaminants.

Cleaned panels were placed in passivation baths that contained citric acid solutions using the appropriate concentration, time and temperature conditions. Stainless steel tanks (using covers to limit evaporation (Figure 2)) were used. After each passivation was performed, rinses were conducted according to the USA (United Space Alliance) passivation process (Figure 1).



Figure 2 – Test Panels Soaking in Citric Acid Passivation

In order to determine the optimal citric acid passivation procedure for each alloy the following passivation parameters were employed.

- Time: 60 minutes, 90 minutes, and 120 minutes
- Temperature: 38 °C, 60 °C, and 82 °C
- Concentration: 4%, 7%, 10%

Parameters used for physical testing were determined by the stakeholders based upon the results of prior testing by the NASA TEERM office and USA.

After the rinsing process was performed, the pH of the water on the surface of each panel was checked to ensure that that it remained at a pH between 6.0 to 8.0. Ordinary pH paper was used to perform this function. The pH is typically of concern when a high volume of panels are being processed due to the increased possibility of residual passivation solution contaminating the rinse tanks. Although this study processed a relatively small number of panels, this step was included to remove any doubt that the acidic passivating solution was removed from the surface. Verifying that this parameter was met, the panels were subsequently dried with gaseous nitrogen. All panels were packaged in gaseous nitrogen prior to ASTM B117 salt spray exposure.

3 Parameter Optimization Testing

Previous testing by the NASA TEERM office and USA determined that the parameters for citric acid passivation that resulted in optimum performance varied by substrate. The studies found that passivation with elevated temperature and longer immersion times (temperature > 100 °F and time > 30 minutes) provide significantly better corrosion protection than treatments at ambient temperature or shorter immersion times (T \approx 100 °F and t \approx 30 minutes). The data obtained during the prior studies focused on a citric acid concentration of 4%. Parameter optimization testing, however, was necessary to determine the best parameters for other concentrations of 4%, 7% and 10%.

Table 1 outlines the parameters that were tested to determine the best passivation process for the alloys that were investigated in this project. For each stainless steel alloy previously tested, 54 test panels were required; two (2) concentrations X three (3) bath temperatures X three (3) dwell times X three (3) replicate panels = 54 panels. For each stainless steel alloy that has not yet been tested, 81 test panels will be required; three (3) concentrations X three (3) bath temperatures X three (3) dwell times X three (3) replicate panels = 81 panels.

Alloy	Passivation	Con	centration	u (%)	Bath T	Time (mi	minutes)						
304		4	7	10	38	60	82	60	90	120			
316		4*	7	10	38	60	82	60	90	120			
321		4*	7	10	38	60	82	60	90	120			
13-8PH		4	7	10	38	60	82	60	90	120			
15-5PH	Citric Acid	4*	7	10	38	60	82	60	90	120			
17-4PH		4	7	10	38	60	82	60	90	120			
17-7PH		4*	7	10	38	60	82	60	90	120			
A286		1			4	7	10	38	60	82	60	90	120
AL6XN		4	7	10	38	60	82	60	90	120			
* Outini-time	4	the state of the second											

 Table 1 – Parameter Optimization Test Outline

* Optimization testing completed in a previous project

3.1 Salt Spray Resistance

ASTM B117 salt spray testing was used to rapidly evaluate the ability of the passivation process to protect stainless steel alloys from corrosion. Table 2 outlines the parameters that were used for salt spray testing.

Table 2 – Salt Spray Testing Parameters

Test	Corrosion Protection	Requirement	Test Methodology	Evaluation	Acceptance Criteria	Location
Salt Spray	Passivation Only	SAE AMS 2700	ASTM B 117	ASTM D 610 @ 2 hours @ 168 hours	Alternative performs as well or better than control process	NASA Corrosion Technology Lab

3.1.1 Test Procedure

Test panels were sorted by their unique alphanumeric descriptor and further by their citric acid concentration. Figure 3 shows the sorting process.



Figure 3 – Sorting the Test Panels

Test panels were subjected to a 5 percent NaCl salt-spray, pH-adjusted to a range of 6.5 - 7.2, in accordance with ASTM B 117 (Standard Practice for Operating a Salt Spray (Fog) Apparatus). According to the testing standard requirement (SAE AMS 2700), test panels only had to be tested in the salt-spray chamber for 2 hours then evaluated for corrosion. In order to obtain the best possible test results, the test panels were further exposed for an additional 166 hours (168 hours total). After each test, the panels were rinsed with deionized (DI) water and were then dried using compressed air. The test panels were evaluated for signs of corrosion following 2 hours and 168 hours of salt spray testing.

One (1) color photograph of the test coupons for each substrate was taken before testing. One (1) color photograph of the test coupons was taken after two hours of salt spray exposure and at the conclusion of testing.

3.1.2 Evaluation Procedure

Per SAE AMS 2700, "Passivation of Corrosion Resistant" test panels were evaluated after two (2) hours of salt spray exposure. According to SAE AMS 2700, "Parts shall not show evidence of red rust following completion of the test".



Figure 4 – Evaluation of Test Panels after 168 hours

After completing the initial analysis per SAE AMS 2700, salt spray testing continued for an additional 166 hours. The additional testing gave an overall exposure duration of 168 hours, which was the exposure duration used during a previous NASA TEERM Project "Alternative to Nitric Acid Passivation; 2015".

4 Laboratory Results

Each panel was assessed on a pass/fail evaluation in accordance to SAE AMS 2700. Table 1 shows how the test panels preformed after 2 hours in the salt-spray chamber. Table 4 shows how the test panels preformed after 168 hours in the salt-spray chamber. Green indicates that, the panel showed no evidence of red rust following completion of the test, thus it passed. Red indicates that the panel showed signs of rust, thus it failed. Both tables depict how the panels were laid out for evaluation and pictures (found in Appendix A– Pictures from Each Evaluation Set). Some of the panels showed no overall corrosion but contained a single corrosion pit, thus they were considered a failure as well.

			Post 2 hour ASTM	B117 Evaluatio	ns						
Alloy	Passivation	Concentration (%)	Bath Temperature (°C)		1	Dwell T	ïme (m	inutes))		
				60			90			120	
			38C								
		4	60C								
			82C								
201		_	38C								
304	Citric Acid		60C								
			820								
			380								
		10	600								
		10	82C								
	1										
		4*									
			38C								
		7	60C								
316	Citric Acid		82C								
		10	38C								
		10	60C								ļ
			82C								
		4*									
			380								
		7	50C 60C								
321	Citric Acid		82C								
221											
			38C								
		10	60C								
			82C								
			38C								
		4	60C							Single pit	
			82C								
			290								
13-8PH	Citric Acid	7	500								
15-6111	Churc Acid	· ·	820								
			020								
			38C								
		10	60C				Single pit				
			82C								
	•										
		4*									
			38C								
		7	60C								
15-5PH	Citric Acid		82C								
			200								
		10	380		Single pit						
		10	60C								
	1	I	026								

Table 3 – 2 Hour ASTM D610 Evaluations

			380						Single nit	Single nit
		4	600						angle pit	single pit
		4	800							
			820							
		_	380							
17-4PH	Citric Acid	7	60C							
			82C							
			38C							
		10	60C							
			82C							
		4*								
			380							
		7	600							
17.701	Citria Aaid	,	820			<u> </u>				
1/=/FH	Citric Acid		020							
			390							
		10	380							
		10	60C							
			82C							
			38C							
		4	60C							
			82C							
			38C							
A286	Citric Acid	7	60C							
			82C							
			380							
		10	600							
		10	820							
			520							
			200							
			380	Single pit						
		4	60C							
			82C							
			38C							
AL6XN	Citric Acid	7	60C							
			82C							
			38C							
		10	60C							
			82C							
		8.0	ntimization testing com	mlatad		vione r	roject			
		÷ (pumization testing con	pieted	in a pre	vious p	roject			

			Post 168 ho	ur ASTM	B117 E	valuatio	ns					
Alloy	Passivation	Concentration	Bath Temp			1	Dwell T	ìme (m	inutes)			
					60			90			120	
			38C									
		4	60C									
			82C									
			38C									
304	Citric Acid	7	60C									
			82C									
			38C									
		10	60C									
			82C									
		4*										
			290									
		7	600									
316	Citric Acid	· '	820									
510	Child Acid		020									
			380									
		10	60C									
		10	82C									
		4*										
			38C									
		7	60C									
321	Citric Acid		82C									
			38C									
		10	60C									
			82C									
		1	200									
			500								et a sta	
		*	820								Single pit	
			020									
			380									
13-8PH	Citric Acid	7	600									
		-	82C									
			38C						Single pit			
		10	60C									
			82C									
		4*										
			38C									
	and a second	7	60C									
15-5PH	Citric Acid		82C									
			200									
		10	380		Single pit	Two pits						
		10	600							Single pit		
			82C									
1												

Table 4 – 168 Hour ASTM D610 Evaluations

$ A = \left[\begin{array}{cccccccccccccccccccccccccccccccccccc$				38C								Single pit	Single pit
17-4PH 6 82C 6 100			4	60C		Single pit					Single pit		
17-4PH Image: constraint of the sector of				82C			Two pits						
17-4PH 2 38C 38C </td <td></td>													
17-4PH Citric Acid 7 60C I I State pile State pile 82C 0 0 0 0 0 0 0 0 10 60C 0 0 0 0 0 0 0 10 83C 50C 0 0 0 0 0 0 10 83C 800 pile 0 0 0 0 0 0 10 83C 50C 0 0 0 0 0 0 0 10 83C 60C 0 0 0 0 0 0 0 10 83C 60C 0 0 0 0 0 0 0 10 82C 0 0 0 0 0 0 0 0 10 82C 0 0 0 0 0 0 0 0 0 10 60C 0 0 0 0 0 0 0 0 0 10 82C 0 0 0 0 0 0 0 0 0 10				38C					Single pit				
10 82C 0	17-4PH	Citric Acid	7	60C						Single pit			Single pit
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$				82C									
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$\begin{array}{ c c c c c c c c c c c c c c c c c c c$				38C					Single pit				
Alt Second pile Second pile Second pile Second pile 17-7PH 4* Image pile Image pile Image pile Image pile Image pile 17-7PH 7 38C Image pile Image pile Image pile Image pile 17-7PH 7 38C Image pile Image pile Image pile Image pile 17-7PH 7 38C Image pile Image pile Image pile Image pile 10 82C Image pile Image pile Image pile Image pile Image pile 10 60C Image pile Image pile Image pile Image pile Image pile 10 38C Image pile Image pile Image pile Image pile Image pile 10 38C Image pile Image pile Image pile Image pile Image pile A286 38C Image pile Image pile Image pile Image pile Image pile A286 7 38C Image pile Image pile Image pile Image pile Image pile A286 7 38C Image pile Image pile Image pile Image pile Image pile A286 1m			10	60C									
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$A^{*} = \begin{bmatrix} 4^{*} & 4^$								_					
17-7PH			4*										
17-7PH 7 38C 0<													
17-7PH Citric Acid 7 60C 0				38C									
17-7PH Citric Acid 82C 0			7	60C									
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	17-7PH	Citric Acid		82C									
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$													
10 60c 0			10	38C									
A286 38C A B A A B A A A A A A A A A A A A B C A A A A B A A B A <td></td> <td></td> <td>10</td> <td>60C</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>			10	60C									
A286 Citric Acid 38C Image: Constraint of the second secon				82C									
A286 38C 60C 60													
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$				38C									
A286 Citric Acid 382C Image: Construct on the second secon			4	60C									
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A286 Citric Acid 7 $\frac{38C}{60C}$ I I				300									
A286 Cline Acid 7 60C 82C 60C	1.296	Circle And I	-	380									
382C	A286	Citric Acid	/	60C									
38C 38C <td></td> <td></td> <td></td> <td>82C</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>				82C									
10 36C 60C 60C <td></td> <td></td> <td></td> <td>200</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>				200									
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4 60C 2 <th2< th=""> 2 2 <th2< th=""></th2<></th2<>				380	Single pit								
			4	820									
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ALGXN Citric Acid 7 50C	AL 6YN	Citric Acid	7	600									
ALOAN CHIR Acid 7 80C 82C	ALOAN	Citile Acid	ŕ	820									
				020									
380				38C									
10 60C			10	60C									
82C				82C									
* Optimization testing completed in a previous project			* Ontimiz	ration testin	ng com	pleted in	i a prev	ious pro	piect				

5 Conclusions

Overall, regardless of alloy, the higher citric acid concentration, temperature, and bath dwell time yielded the best results. Figure 5 gives a pass/fail percentage for the entire set of panels after 2 hours of salt spray testing and shows how the panels performed under the specific parameters. As the citric acid concentration increased, the percent of panels that passed also increased. When analyzing how the different temperatures performed, there is clear evidence that 38°C had a significantly greater number of failures than either 60°C or 82°C. When differentiating between 60°C and 82°C, there is not enough proof to signify that 82°C is better than 60°C because there is only a 1 percent difference in the failure data. Furthermore, the increased temperature increased difficulty in panel processing. When scaled to an industrial process, the 82°C baths would require constant replenishing.

An investigation of increasing immersion time reveals that an increased number of panels passed as immersion time increased. However, the difference between the percent passed for 90 and 120 minutes is approximately 2 percent. It was important to continue ASTM B117 testing for an additional 166 hours to clarify the results and draw the most accurate conclusions.

Figure 6 illustrates the performance of the test panels after 168 hours of exposure. With an increased exposure in the salt-spray chamber, the trend for citric acid concentrations changed slightly. The 4 percent pass rate exceeded the 7 percent pass rate by a very slim margin. For the series of panels investigated, a citric acid concentration of 10 percent showed an increased pass rate at 86.8%. The difference between 4 percent and 7 percent are very close, which makes it difficult to determine a clear trend. Following 168 hours of salt-spray exposure, the same trends following 2 hour of salt-spray testing regarding pass rates with respect to temperature persisted. 60°C and 82°C showed very similar pass rates with 82°C having a higher percentage of passing ratings. The failure rate of 38°C surpassed the pass rate by 12 percent, which is significant to determine that 38°C is not an ideal temperature for corrosion prevention by citric acid passivation. With the added time in the salt-spray chamber, the longer immersion times showed a clearer positive trend in pass rates. This strengthens the conclusion that 120 minutes may be the optimal immersion time.

The data for the differences between temperatures (60°C and 82°C) and immersion times (90 and 120 minutes) are less than 5 percent. But as the data shows, over time the differences between the two parameters increases. The data shows that the highest parameter for concentration, bath temperature and bathing time is the best for corrosion prevention of the stainless steel alloys tested. Overall, the parameters for the most ideal protection against corrosion using citric acid passivation are 10 percent citric acid concentration at 82° C for 120 minutes. Further, when analyzing Table 3 and Table 4, it is clear that every test panel with those specific parameters passed regardless of alloy.



Figure 5 – Percent Pass/Fail for Different Test Parameters for 2 Hours



Figure 6 – Percent Pass/Fail for Different Test Parameters for 168 Hours

6 Recommendations

The next step would be to expand the testing to include atmospheric exposure testing on the NASA Beachside Atmospheric Test Site at Kennedy Space Center. Testing the panels under coastal atmospheric conditions will provide performance data from exposure to very harsh "real world" environmental conditions. This will yield the greatest possible results to determine which citric acid passivation process provides the best protection against corrosion. Continued salt-spray testing for additional alloys and longer duration would further differentiate the test results, further clarifying which parameters perform best across the alloys.



Appendix A– Pictures from Each Evaluation Set













10% Citric Acid with Alloy 13-8 Post 2 hours	880 - 60 Min. 600 - 60 Min. 820 - 60 Min.	19% Dure And 193 Data 193 Data 196: 19 Min 196 196: 19 Min 400 197: 19 Min 197	120 Mm 120 Mm 120 Mm		











20 | P a g e

























26 | P a g e

27 | P a g e

