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

NASA and ESA Collaboration on Hexavalent Chrome Alternatives

Pretreatments Only Interim Test Report

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Preface

This test report was prepared by ITB, Inc., through the National Aeronautics and Space Administration (NASA) Technology Evaluation for Environmental Risk Mitigation (TEERM) Principal Center, under Contract Number NNH09CF09B. The structure, format, and depth of technical content of the report was determined by NASA TEERM in response to the specific requirements of this project.

NASA TEERM has partnered with the European Space Agency (ESA) for this project. NASA and ESA share common risks related to material obsolescence associated with hexavalent chromium used in corrosion-resistant coatings.

NASA TEERM works closely with the John F. Kennedy Space Center (KSC) Corrosion Technology Laboratory to ensure the quality of the test data, including the review and analysis within this report. Similarly, ESA works with the European Space Research and Technology Centre (ESTEC).

Acknowledgements

ITB, Inc. would like to thank Mr. Thomas Rohr with ESA ESTEC for continued project support and contribution. ITB, Inc. would also like to thank Mr. Jerry Curran for coordination and oversight of the KSC Corrosion Technology Laboratory and the KSC Beachside Atmospheric Test Facility, as well as Ms. Fatema Janjali for coordination and testing support at ESA ESTEC. In addition, ITB, Inc. would like to thank Mr. Teddy Back for test panel preparation and coating application at KSC and Mr. Guillaume Sierra for coating application at MAP.

Executive Summary

Hexavalent chromium (hex chrome or Cr(VI)) is a widely used element within applied coating systems because of its self-healing and corrosion-resistant properties. The replacement of hex chrome in the processing of aluminum for aviation and aerospace applications remains a goal of great significance. Aluminum is the major manufacturing material of structures and components in the space flight arena. The National Aeronautics and Space Administration (NASA) and the European Space Agency (ESA) are engaged in a collaborative effort to test and evaluate alternatives to hexavalent chromium containing corrosion coating systems. NASA and ESA share common risks related to material obsolescence associated with hexavalent chromium used in corrosion-resistant coatings.

In the United States, Occupational Safety and Health Administration (OSHA) studies have concluded that hexavalent chromium is carcinogenic and poses significant risk to human health. On May 5, 2011, amendments to the Defense Federal Acquisition Regulation Supplement (DFARS) were issued in the Federal Register. Subpart 223.73 prohibits contracts from requiring hexavalent chromium in deliverables unless certain exceptions apply. Subpart 252.223-7008 provides the contract clause prohibiting contractors and subcontractors from using or delivering hexavalent chromium in a concentration greater than 0.1 percent by weight for all new contracts associated with supplies, maintenance and repair services, and construction materials.

ESA faces its own increasingly stringent regulations within European directives such as Registration, Evaluation, Authorisation and Restriction of Chemical (REACH) substances and the Restriction of Hazardous Substances Directive (RoHS) which have set a mid-2017 sunset date for hexavalent chromium.

NASA and ESA continue to search for an alternative to hexavalent chromium in coatings applications that meet their performance requirements in corrosion protection, cost, operability, and health and safety, while typically specifying that performance must be equal to or greater than existing systems.

The overall objective of the collaborative effort between NASA TEERM and ESA is to test and evaluate coating systems (pretreatments, pretreatments with primer, and pretreatments with primer and topcoat) as replacements for hexavalent chrome coatings in aerospace applications. This objective will be accomplished by testing promising coatings identified from previous NASA, ESA, Department of Defense (DOD), and other project experience. Additionally, new materials will be analyzed according to ESA-identified specifications.

Testing is in progress at NASA John F. Kennedy Space Center (KSC) and ESA European Space Research and Technology Centre (ESTEC).

1 Introduction

1.1 Background

Hexavalent chromium (hex chrome or Cr(VI)) is a widely used element within applied coating systems because of its self-healing and corrosion-resistant properties. The replacement of hex chrome in the processing of aluminum for aviation and aerospace applications remains a goal of great significance. Aluminum is the major manufacturing material of structures and components in the space flight arena. The processing and maintenance of this material against degradation and corrosion is of prime importance to the National Aeronautics and Space Administration (NASA) and the European Space Agency (ESA) in preserving space operation capabilities.

Key to the operability and preservation of aluminum has been the use of chromated systems (pretreatments and primers). With applied coatings, the high corrosion resistance offered by chromated films is attributed to the presence of both hexavalent and trivalent chromium in the coating. The trivalent chromium (tri chrome or Cr(III)) is present as an insoluble hydrated oxide. Hex chrome imparts a “self-healing” characteristic to the coating during an oxidative (corrosive) attack. Chromated coatings also play a critical role in supporting and enhancing the adhesion of the primer coating to the substrate.

In the United States, Occupational Safety and Health Administration (OSHA) studies have concluded that hexavalent chromium is carcinogenic and poses significant risk to human health. On May 5, 2011, amendments to the Defense Federal Acquisition Regulation Supplement (DFARS) were issued in the Federal Register. Subpart 223.73 prohibits contracts from requiring hexavalent chromium in deliverables unless certain exceptions apply. These exceptions include authorization from a level no lower than a general or flag officer or a member of the Senior Executive Service from the Program Executive Office or equivalent level, or unmodified legacy systems. Otherwise, Subpart 252.223-7008 provides the contract clause prohibiting contractors and subcontractors from using or delivering hexavalent chromium in a concentration greater than 0.1 percent by weight for all new contracts associated with supplies, maintenance and repair services, and construction materials.

ESA faces its own increasingly stringent regulations within European directives such as Registration, Evaluation, Authorisation and Restriction of Chemical (REACH) substances and the Restriction of Hazardous Substances Directive (RoHS) which have set a mid-2017 sunset date for hexavalent chromium.

NASA and ESA continue to search for an alternative to hexavalent chromium in coatings applications that meet their performance requirements in corrosion protection, cost, operability, and health and safety, while typically specifying that performance must be equal to or greater than existing systems.

Please note, this interim test report includes test data from NASA only. At the time of this report, testing at ESA ESTEC was in progress.

1.2 Objective

The overall objective of the collaborative effort between NASA and ESA is to test and evaluate coating systems (pretreatments, pretreatments with primer, and pretreatments with primer and topcoat) as replacements for hexavalent chrome coatings in aerospace applications. This objective will be accomplished by testing promising coatings identified from "Hexavalent Chrome Free Coatings for Electronics Applications, Refinement of Coating Processes Report July 31, 2013" and "GSDO Program Hexavalent Chrome Alternatives Final Pretreatments Test Report September 01, 2013" as well as several other coating systems that have shown acceptable performance in previous Department of Defense (DOD) laboratory and atmospheric testing. Additionally, new materials will be analyzed according to ESA-identified specifications.

1.3 Summary of Previous NASA TEERM Projects

Results from "Hexavalent Chrome Free Coatings for Electronics Applications, Refinement of Coating Processes Report July 31, 2013" and "GSDO Program Hexavalent Chrome Alternatives Final Pretreatments Test Report September 01, 2013" show that hexavalent chrome free pretreatments can perform as good as or better than hexavalent chrome containing pretreatments. In review of the data generated during these projects, hexavalent chrome free pretreatments should be considered for implementation. Based on the results of salt spray resistance testing, multiple hexavalent chrome free pretreatments met the requirements of MIL-DTL-5541 "Chemical Conversion Coatings on Aluminum and Aluminum Alloys".

Results from these previous efforts indicate there is a need to evaluate how primers and topcoats perform over hexavalent chrome free pretreatments. Unexpected interactions between hexavalent chrome free pretreatments, primers, and topcoats could produce a coating system that does not meet specifications. This collaborative effort between NASA and ESA aims to explore these unknowns.

2 Test Articles

2.1 Alloys

The aluminum alloys in this project were selected because of their relatively common use in avionics and aerospace applications and/or their ability to exhibit similar performance to other materials of interest. Two of the alloys, 2024-T8 and 7075-T73, were supplied by ESA. All test panels were procured mill finished without mill markings. Mill finish is as supplied from the mill (raw material manufacturer), not polished, and will most likely have a dull matte appearance. The aluminum alloys selected for this project include:

- 2024-T3
- 2024-T8
- 6061-T6
- 7075-T6
- 7075-T73

2.2 Pretreatments

NASA and ESA selected the hexavalent chrome free pretreatments to be tested for this project. Two of the pretreatments, Bonderite M-NT 65000 and MAPSIL® SILICo (thin), were supplied by ESA. MAPSIL® SILICo (thin) is approximately 3 micrometers (µm) to 5 µm thick when applied to a panel. The selected pretreatments are listed in Table 1.

Table 1 – Pretreatments

Pretreatment	Manufacturer
Metalast TCP HF	Metalast
SurTec 650 V	SurTec
Bonderite M-NT 65000	Henkel
MAPSIL® SILICo (3–5 µm)	MAP

3 Pretreatment Testing

3.1 Method

Test panels with pretreatment only are currently being tested and evaluated by NASA and ESA. Table 2 provides test methods, evaluation criteria, and the location of where tests are being conducted.

Table 2 – Pretreatment Testing

Test	Test Method	Evaluation Criteria	Location
Salt Spray Resistance	ASTM B 117	MIL-DTL-5541	NASA KSC and ESA ESTEC
Humidity Testing	N/A	MIL-DTL-5541	ESA ESTEC
Thermal Cycle Testing	N/A	MIL-DTL-5541	ESA ESTEC

3.2 Salt Spray Resistance

This test is used to rapidly evaluate the performance of a coating or coating system and how well it prevents corrosion. Salt spray exposure and corrosion resistance is a requirement of MIL-DTL-5541.

3.2.1 Test Procedure

Test panels are 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). Test panels are inspected for the formation of corrosion every 168 hours.

Salt spray resistance testing was performed at the NASA KSC Corrosion Technology Laboratory and at ESA ESTEC. This report only provides data from NASA testing at this time.

3.2.2 Evaluation Procedure

Test panels were evaluated for pitting every 168 hours; spots or pits were circled with a marker. Pit count was recorded weekly with test panels being pulled from testing at the discretion of the lab technician. Areas within 0.25 inch from the edges, identification markings, and holding points during processing or salt spray exposure shall be excluded. Loss of color shall not be cause for rejection.

3.2.3 Test Results

Pretreatment only testing was divided into two batches. Batch one includes SurTec 650V and Bonderite M-NT 65000 pretreated test panels. Batch two includes Metalast TCP-HF and MAPSIL® SILICo thin (3–5 µm). Photographs of the test panels are located in Appendix A.

Test results from salt spray testing for SurTec 650V pretreatment are contained in Table 3. SurTec 650V on 6061-T6, 7075-T-6, and 7075-T73 alloy test panels outperformed the requirements of MIL-DTL-5541. Even after 672 hours of salt spray testing, the 6061-T6, 7075-T6, and 7075-T73 alloy test panels had very limited pitting when analyzed per MIL-DTL-5541. As noted in the tables below, magnification was required to identify pitting and pretreatment breakdown in discolored areas observed on the test panels. SurTec 650V on 2024-T3 alloy test panels had mixed results with one of the 2024-T3 test panels (5003) outperforming the requirements of MIL-DTL-5541 after 672 hours of testing. For the other two 2024-T3 test panels, one panel (5002) met the requirements of MIL-DTL-5541 after 504 hours but had pitting that exceeded the requirement after 672 hours. The final 2024-T3 test panel (5001) only met the requirements of MIL-DTL-5541 after 168 hours of testing. From that point forward, pitting increased until the test panels was pulled from testing after 504 hours. SurTec 650V on 2024-T8 alloy test panels did not perform well. All three test panels did not meet the requirements of MIL-DTL-5541 after 168 hours of testing. The test panels were left in the test chamber for further monitoring. As expected, pitting continued until the test panels were removed from testing.

Table 3 – Salt Spray Testing Results; SurTec 650V

Unique Panel #	Substrate	Test Location	Pit Count				
			168 hrs.	336 hrs.	504 hrs.	672 hrs.	TOTAL
5001	2024-T3	NASA	0	7	28		35
5002	2024-T3	NASA	0	1	3	9	13
5003*b	2024-T3	NASA	0	0	0	2	2
5007	2024-T8	NASA	24	10			34
5008	2024-T8	NASA	6	1	7		14
5009	2024-T8	NASA	17	5	12		34
5013*b	6061-T6	NASA	0	0	0	0	0
5014*a	6061-T6	NASA	0	0	0	5	5
5015*a	6061-T6	NASA	0	0	0	0	0
5019*a	7075-T6	NASA	0	0	0	0	0
5020*a	7075-T6	NASA	0	0	0	1	1
5021*a	7075-T6	NASA	0	0	0	3	3
5025*b	7075-T73	NASA	0	0	0	0	0
5026*b	7075-T73	NASA	0	0	0	0	0
5027*b	7075-T73	NASA	0	0	0	0	0

Pulled from testing

*a - areas of discoloration on the panel, numerous very small pits seen under magnification, evidence of pretreatment breakdown

*b - Test panels appear dull/grainy, numerous very small pits seen under magnification, evidence of pretreatment breakdown

Test results from salt spray testing for Bonderite M-NT 65000 pretreatment are contained in Table 4. Bonderite M-NT 65000 on 6061-T6, 7075-T6, and 7075-T73 alloy test panels outperformed the requirements of MIL-DTL-5541. After 672 hours of salt spray testing, the 6061-T6 alloy test panels had no pitting when analyzed per MIL-DTL-5541. For the 7075-T6 alloy test panels, one test panel had zero pitting observed after 672 hours of testing, the other 2 test panels met the requirements of MIL-DTL-5541 after 504 hours with heavy pitting being observed after 672 hours of testing. For the 7075-T73 alloy test panels, two test panels had no pitting observed after 672 hours of testing, the other test panel met the requirements of MIL-DTL-5541 after 336 hours with only 2 additional pits being observed after 672 hours of testing. As noted in the tables below, magnification was required to identify pitting and pretreatment breakdown in discolored areas observed on the test panels. Bonderite M-NT 65000 on 2024-T3 and 2024-T8 alloy test panels performed poorly. None of the test panels passed the requirements of MIL-DTL-5541 after 168 hours of testing. The test panels were left in the test chamber for further monitoring. As expected, significant pitting continued until the test panels were removed from testing after 336 hours of testing.

Table 4 – Salt Spray Testing Results; Bonderite M-NT 65000

Unique Panel #	Substrate	Test Location	Pit Count				
			168 hrs.	336 hrs.	504 hrs.	672 hrs.	TOTAL
6001	2024-T3	NASA	50	27			77
6002	2024-T3	NASA	8	28			36
6003	2024-T3	NASA	17	38			55
6007	2024-T8	NASA	51	42			93
6008	2024-T8	NASA	54	37			91
6009	2024-T8	NASA	87	38			125
6013*b	6061-T6	NASA	0	0	0	0	0
6014*b	6061-T6	NASA	0	0	0	0	0
6015*b	6061-T6	NASA	0	0	0	0	0
6019*a	7075-T6	NASA	0	0	0	0	0
6020*a	7075-T6	NASA	0	0	0	30	30
6021*a	7075-T6	NASA	0	0	3	30	33
6025*b	7075-T73	NASA	0	0	0	0	0
6026*b	7075-T73	NASA	0	4	2	0	6
6027*b	7075-T73	NASA	0	0	0	0	0

Pulled from testing

*a - areas of discoloration on the panel, numerous very small pits seen under magnification, evidence of pretreatment breakdown

*b - Test panels appear dull/grainy, numerous very small pits seen under magnification, evidence of pretreatment breakdown

Test results from salt spray testing for Metalast TCP HF pretreatment are contained in Table 5. Metalast TCP HF on 6061-T6 alloy test panels outperformed the requirements of MIL-DTL-5541. After 672 hours of salt spray testing, the 6061-T6 alloy test panels had no pitting when analyzed per MIL-DTL-5541. The 7075-T6, and 7075-T73 alloy test panels did not perform as expected. The 7075-T6 test panels were deemed failed after 336 hours of salt spray testing and removed from testing. The 7075-T73 test panels showed signs of corrosion formation after 336 hours of salt spray testing as noted below. The test panels remained in the salt spray chamber and after 504 hours of testing pitting was observed on all test panels. As noted in the tables below, magnification was required to identify pitting and pretreatment breakdown in discolored areas observed on the test panels. The 7075-T73 test panels were left in the salt spray chamber. After 672 hours of testing, severe pitting, more than 30 pits, were observed on all of the 7075-T73 test panels. Metalast TCP HF on 2024-T3 and 2024-T8 alloy test panels performed poorly. None of the 2024-T8 test panels passed the requirements of MIL-DTL-5541 after 168 hours of testing. For the 2024-T3 panels following 168 hours of salt spray testing two of the three test panels had limited pitting and were left in the salt spray chamber. After 336 hours, the 2024-T3 panels had severe pitting on all areas of the panels.

Table 5 – Salt Spray Testing Results; Metalast TCP HF

Unique Panel #	Substrate	Test Location	Pit Count				
			168 hrs.	336 hrs.	504 hrs.	672 hrs.	TOTAL
4007	2024-T8	NASA	50+				50+
4008	2024-T8	NASA	50+				50+
4009	2024-T8	NASA	50+				50+
4001	2024-T3	NASA	50+				50+
4002	2024-T3	NASA	6	30+			30+
4003	2024-T3	NASA	2	50+			50+
4013	6061-T6	NASA	0	0	0	0	0
4014	6061-T6	NASA	0	0	0	0	0
4015	6061-T6	NASA	0	0	0	0	0
4019	7075-T6	NASA	0	*			0
4020	7075-T6	NASA	0	*			0
4021	7075-T6	NASA	0	*			0
4025	7075-T73	NASA	0	**	7	30+	30+
4026	7075-T73	NASA	0	**	4	30+	30+
4027	7075-T73	NASA	0	**	4	30+	30+

Pulled from testing

* Pitting concentrated in area of discoloration; pulled from testing

** Black spots observed in the grain of the panel, expected pitting with no tails

Pits starting to form. Very small pits, observed under magnification are forming and there is an increase in the number of black spots in the grain

Test results from salt spray testing for MAPSIL® SILICo (3–5 µm) pretreatment are contained in Table 6. Results for MAPSIL® SILICo (3–5 µm) on 2024-T8, 2024-T3, 6061-T6, 7075-T6, and 7075-T73 were inconsistent. MAPSIL® SILICo (3–5 µm) on 6061-T6 test panels met the requirements of MIL-DTL-5541 after 168 hours of salt spray testing. After 336 hours of salt spray testing, only one 6061-T6 panel exceeded the requirements of MIL-DTL-5541. MAPSIL® SILICo (3–5 µm) on 7075-T6 test panels met the requirements of MIL-DTL-5541 after 168 hours of salt spray testing. After 336 hours of salt spray testing, only one 7075-T6 panel exceeded the requirements of MIL-DTL-5541. For 7075-T73, only two test panels met the requirements of MIL-DTL-5541 after 168 hours of salt spray testing. After 336 hours of salt spray testing, only one 7075-T73 panel exceeded the requirements of MIL-DTL-5541. For 2024-T8, only one test panel met the requirements of MIL-DTL-5541 after 168 hours of salt spray testing. For 2024-T3, two test panels met the requirements of MIL-DTL-5541 after 168 hours of salt spray testing. For MAPSIL® SILICo (3–5 µm) the failed test panels had heavy corrosion formation and showed signs of coating breakdown.

Table 6 – Salt Spray Testing Results; MAPSIL® SILICo (3–5 µm)

Unique Panel #	Substrate	Test Location	Pit Count				
			168 hrs.	336 hrs.	504 hrs.	672 hrs.	TOTAL
3007	2024-T8	NASA	*				
3008	2024-T8	NASA	0	*			
3009	2024-T8	NASA	*				
3001	2024-T3	NASA	*				
3002	2024-T3	NASA	0	*			
3003	2024-T3	NASA	0	*			
3013	6061-T6	NASA	0	*			
3014	6061-T6	NASA	0	*			
3015	6061-T6	NASA	0	0	0	**	
3019	7075-T6	NASA	0	*			
3020	7075-T6	NASA	0	0	0	**	
3021	7075-T6	NASA	0	*			
3025	7075-T73	NASA	*				
3026	7075-T73	NASA	0	0	0	**	
3027	7075-T73	NASA	0	*			
* Heavy corrosion - pulled from testing							
** Coating breakdown, pitting over entire panel							

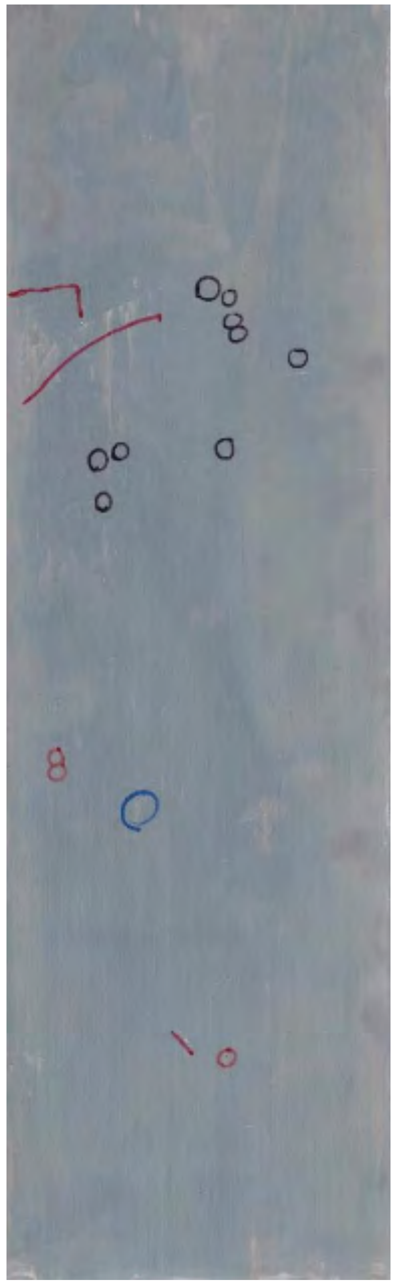
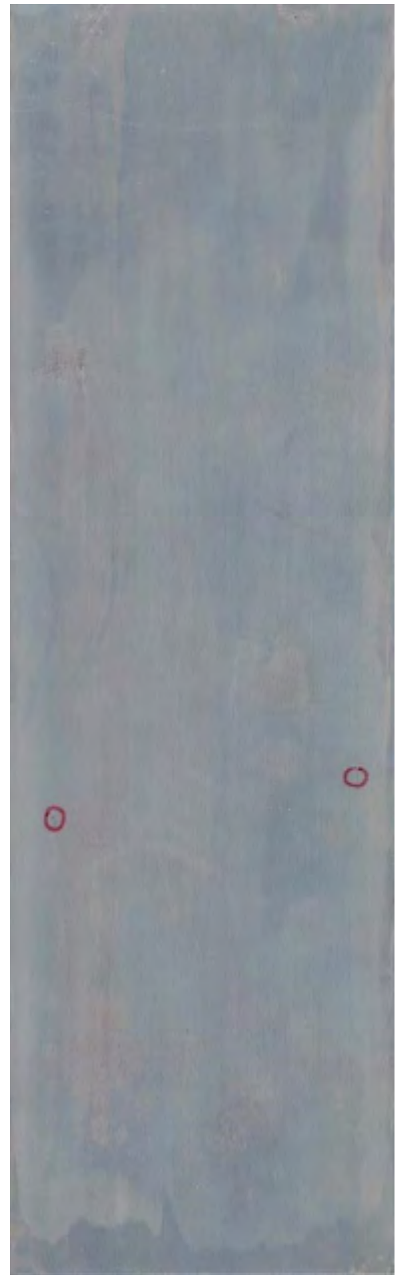
4 Conclusions

To be completed when testing and evaluation is finished.

5 Recommendations

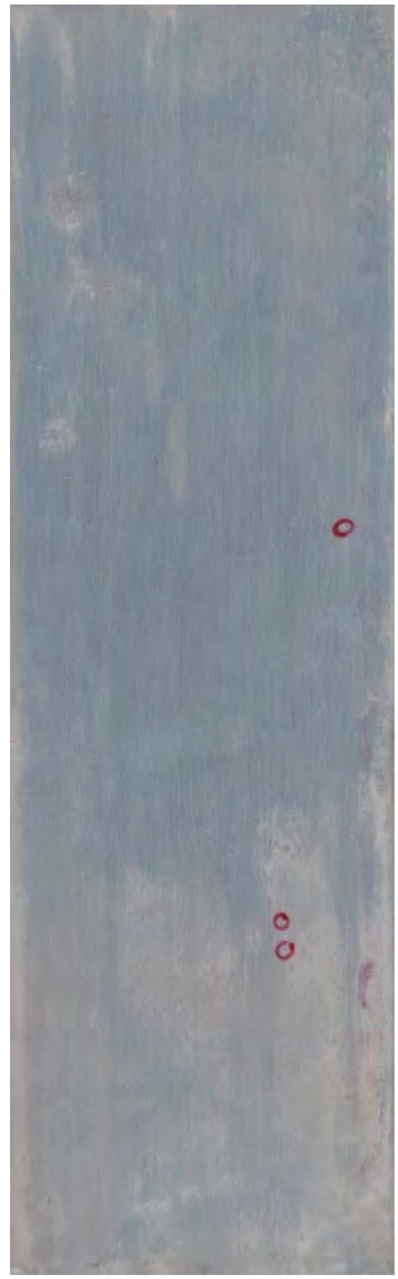
To be completed when testing and evaluation is finished.

Appendix A - Pretreatment Only Salt Spray Resistance Test Panel Photographs

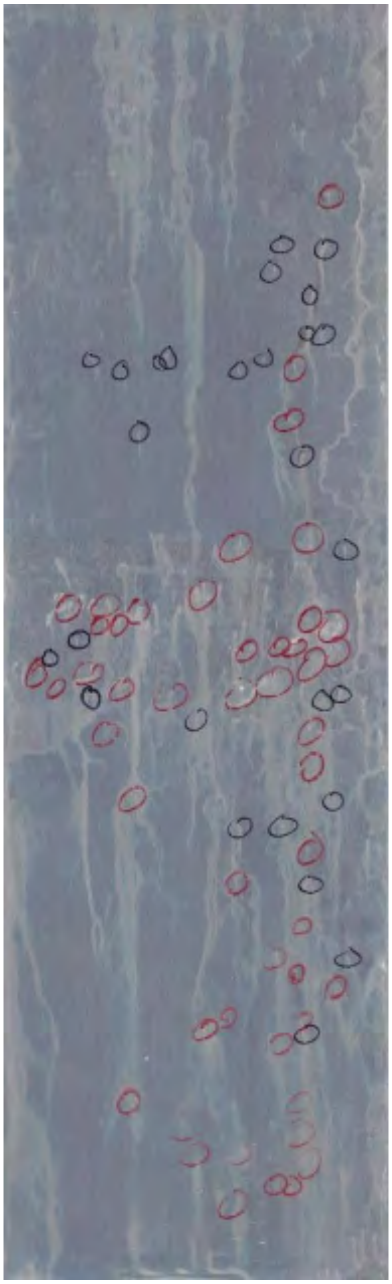
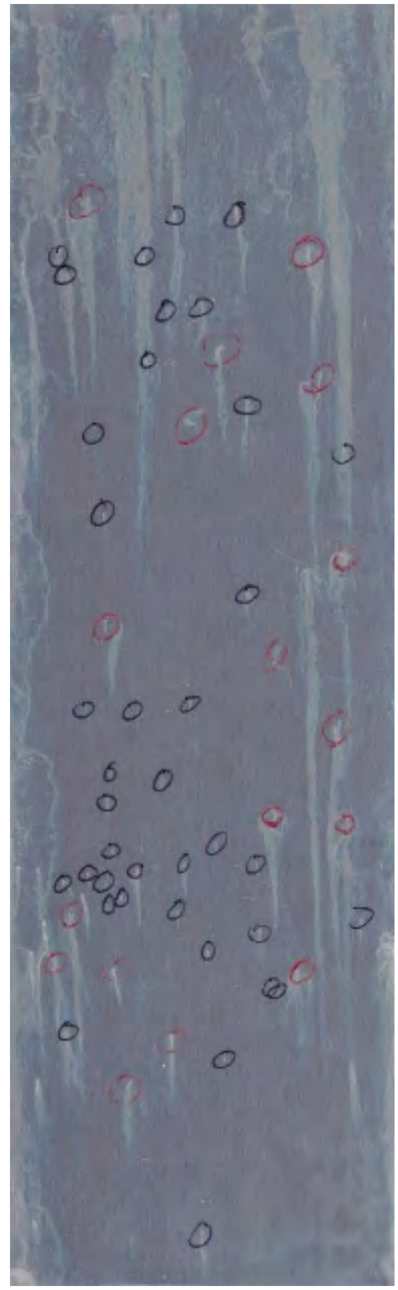
SurTec 650 V		
2024-T3 (5001)	2024-T3 (5002)	2024-T3 (5003)
504 Hours	672 Hours	672 Hours
		

SurTec 650 V		
2024-T8 (5007)	2024-T8 (5008)	2024-T8 (5009)
336 Hours	504 Hours	504 Hours
		

SurTec 650 V		
6061-T6 (5013)	6061-T6 (5014)	6061-T6 (5015)
672 Hours	672 Hours	672 Hours
		

SurTec 650 V		
7075-T6 (5019)	7075-T6 (5020)	7075-T6 (5021)
672 Hours	672 Hours	672 Hours
		

SurTec 650 V		
7075-T73 (5025)	7075-T73 (5026)	7075-T73 (5027)
672 Hours	672 Hours	672 Hours
		

Bonderite M-NT 65000		
2024-T3 (6001)	2024-T3 (6002)	2024-T3 (6003)
336 Hours	336 Hours	336 Hours
		

<table>
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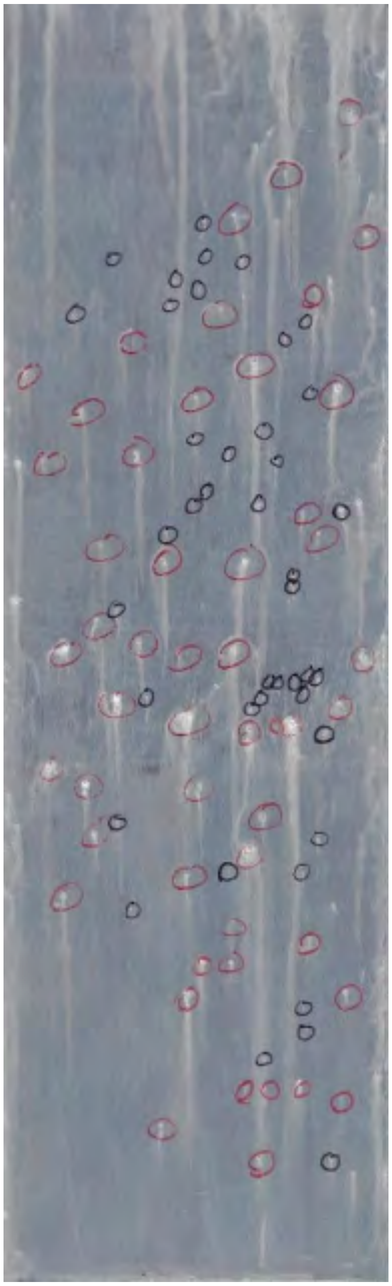
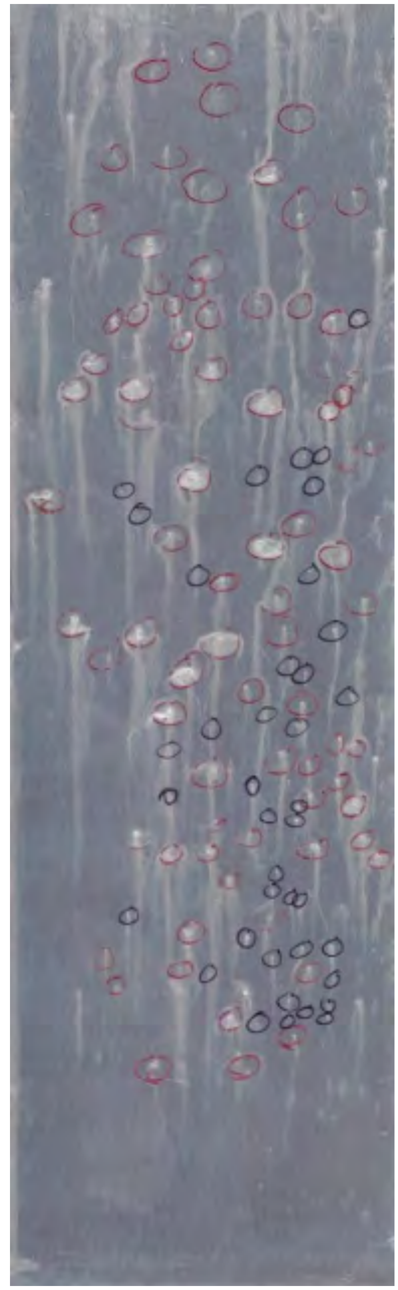
<tr><td>2024-T8 (6007)</td><td>2024-T8 (6008)</td><td>2024-T8 (6009)</td></tr>
<tr><td>336 Hours</td><td>336 Hours</td><td>336 Hours</td></tr>
<tr><td></td><td></td><td></td></tr>
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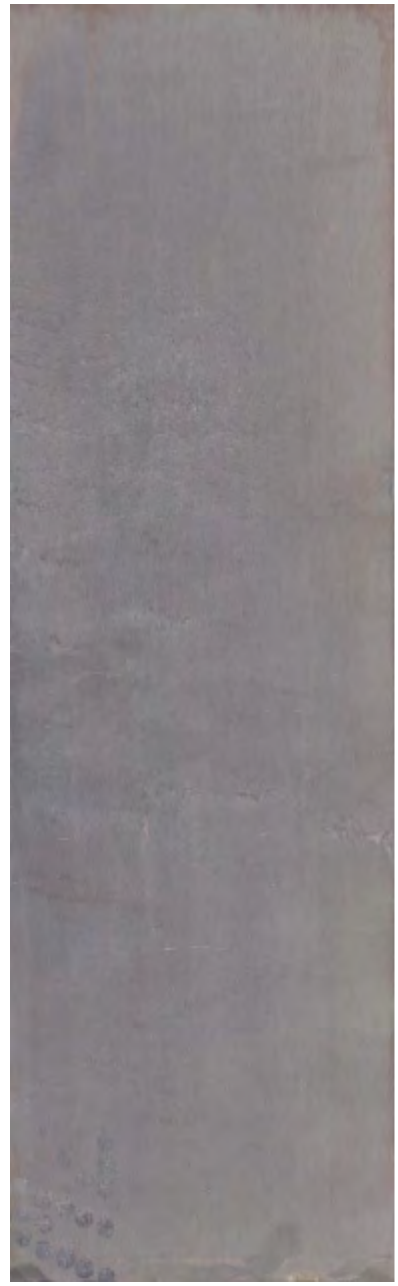
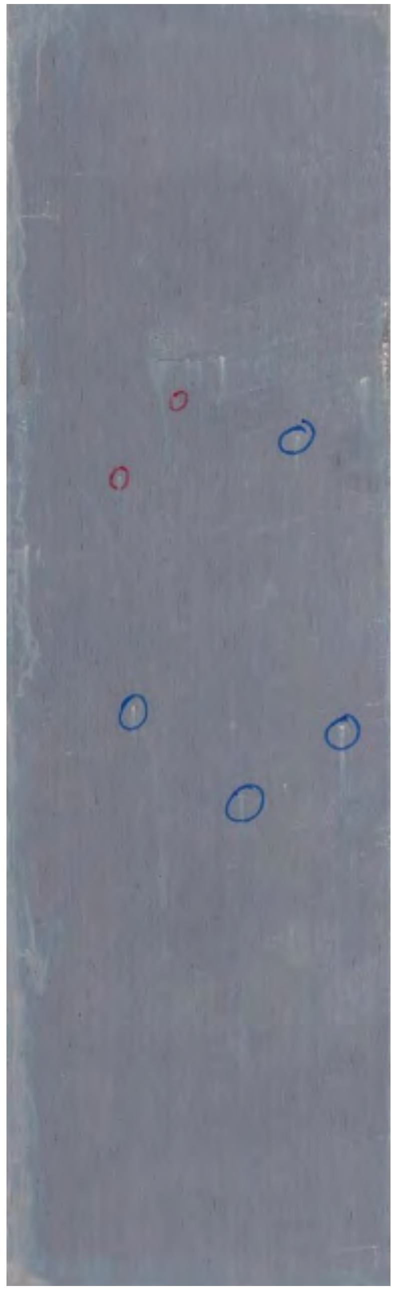
Bonderite M-NT 65000		
6061-T6 (6013)	6061-T6 (6014)	6061-T6 (6015)
672 Hours	672 Hours	672 Hours
		

Bonderite M-NT 65000		
7075-T6 (6019)	7075-T6 (6020)	7075-T6 (6021)
672 Hours	672 Hours	672 Hours
		

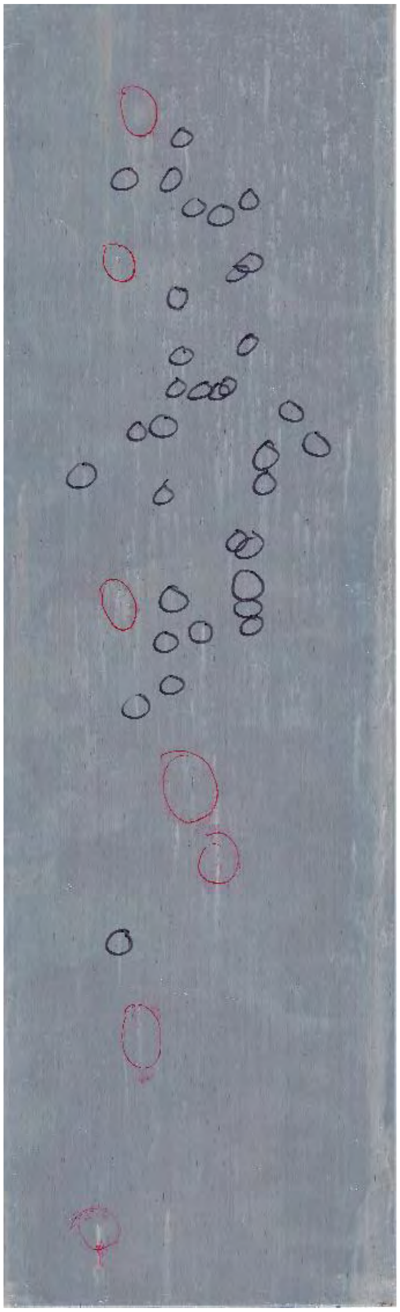
Bonderite M-NT 65000		
7075-T73 (6025)	7075-T73 (6026)	7075-T73 (6027)
672 Hours	672 Hours	672 Hours
		

Metalast TCP HF		
2024-T8 (4007)	2024-T8 (4008)	2024-T8 (4009)
168 Hours	168 Hours	168 Hours
		

Metalast TCP HF		
2024-T3 (4001)	2024-T3 (4002)	2024-T3 (4003)
168 Hours	336 Hours	336 Hours
		

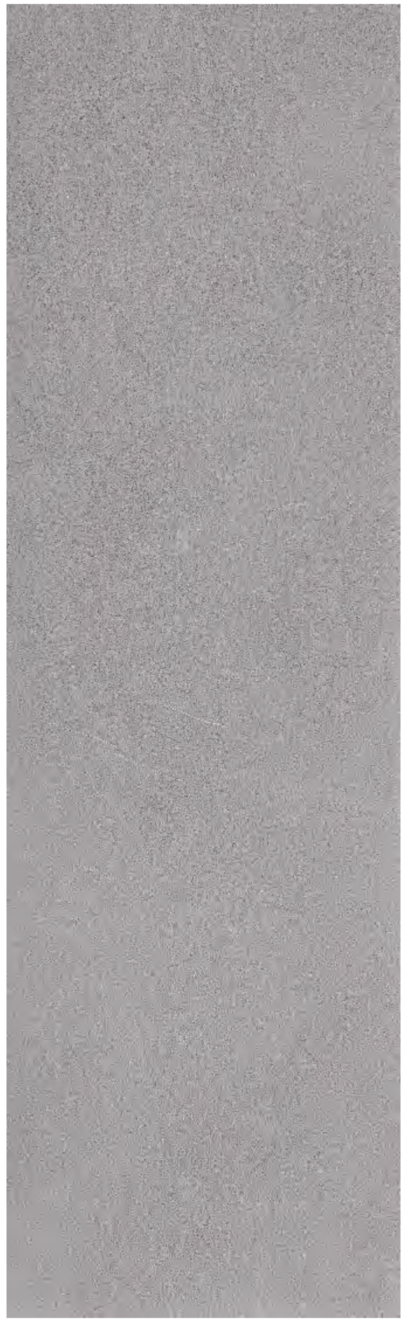
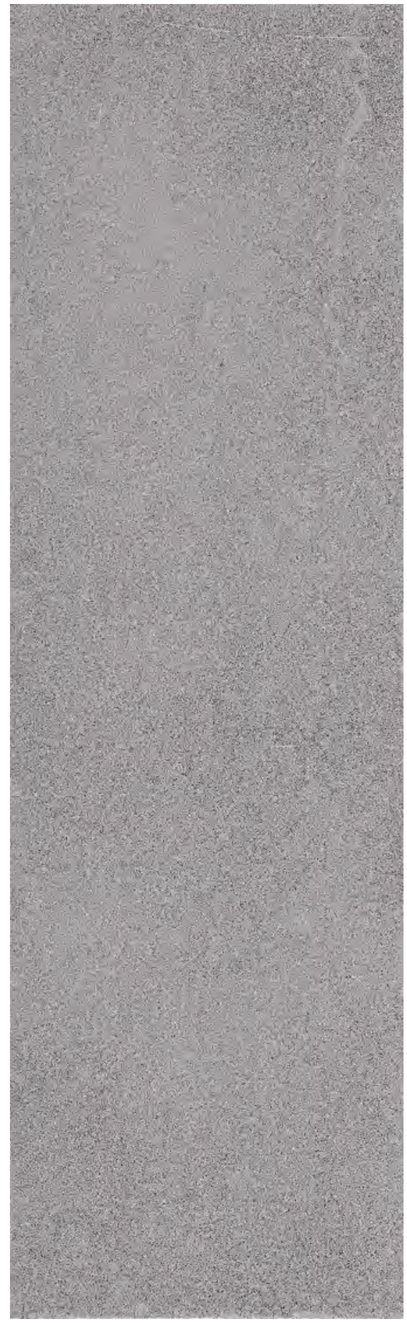
Metalast TCP HF		
6061-T6 (4013)	6061-T6 (4014)	6061-T6 (4015)
672 Hours	672 Hours	672 Hours
		

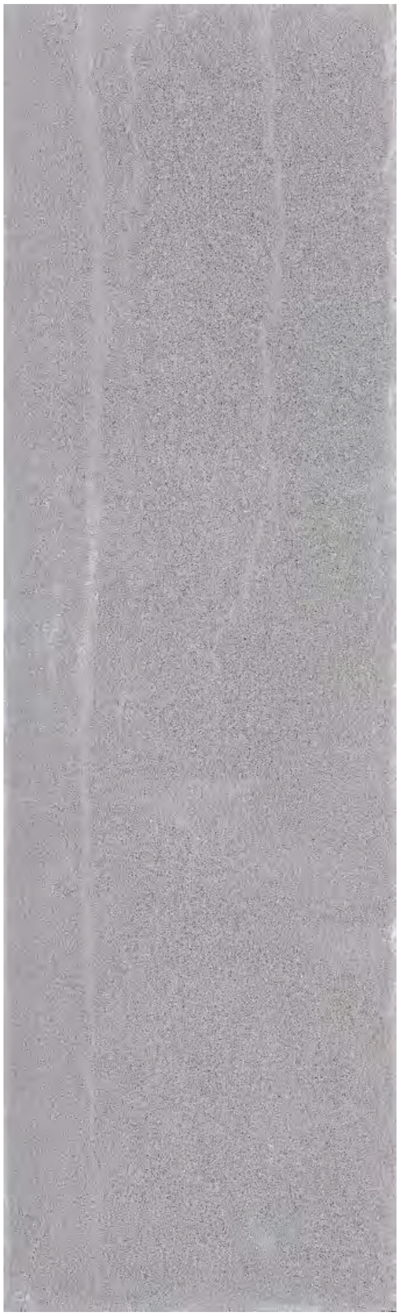
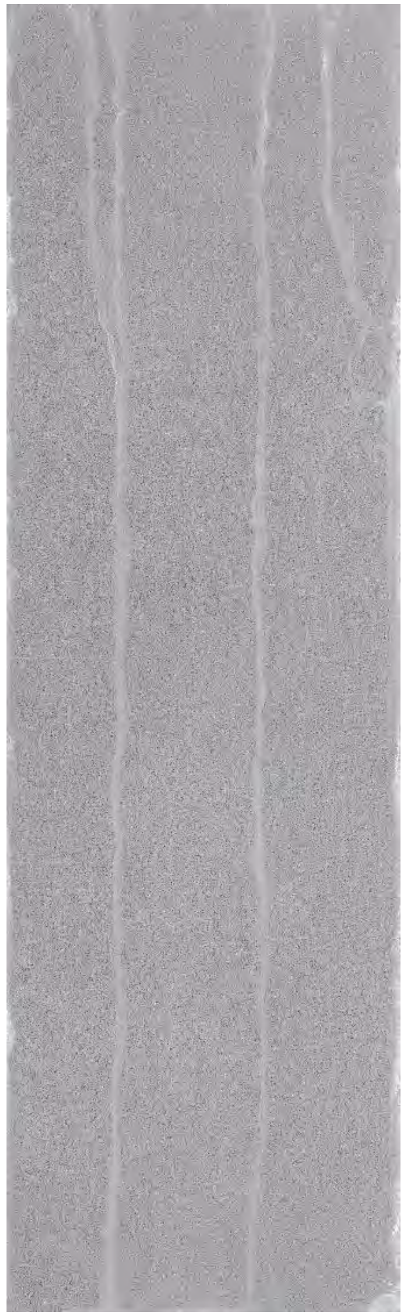
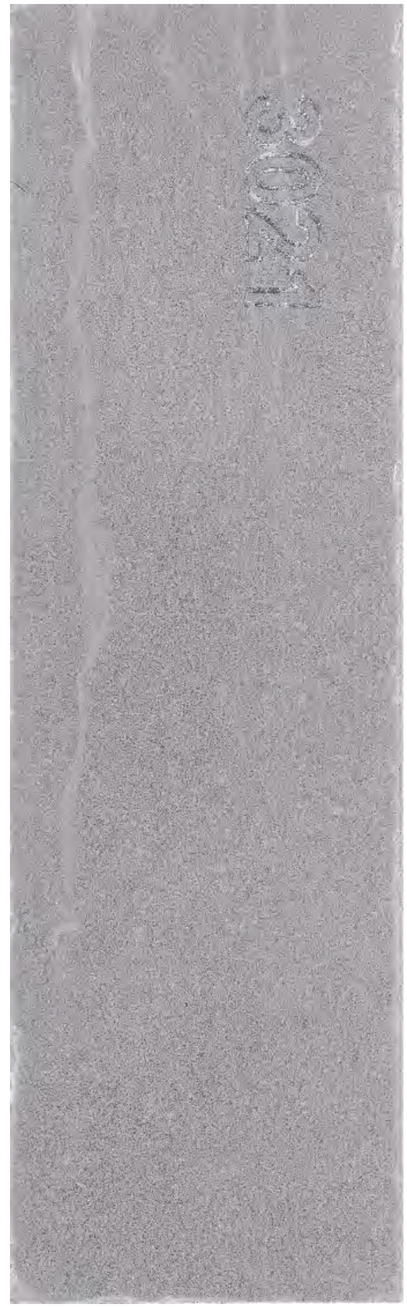
Metalast TCP HF		
7075-T6 (4019)	7075-T6 (4020)	7075-T6 (4021)
336 Hours	336 Hours	336 Hours
		

Metalast TCP HF		
7075-T73 (4025)	7075-T73 (4026)	7075-T73 (4027)
672 Hours	672 Hours	672 Hours
		

MAP Silico {3–5 µm}		
2024-T8 (3007)	2024-T8 (3008)	2024-T8 (3009)
168 Hours	336 Hours	168 Hours
		

MAP Silico {3–5 µm}		
2024-T3 (3001)	2024-T3 (3002)	2024-T3 (3003)
168 Hours	336 Hours	336 Hours
		

MAP Silico {3–5 µm}		
6061-T6 (3013)	6061-T6 (3014)	6061-T6 (3015)
336 Hours	336 Hours	672 Hours
		

MAP Silico {3–5 µm}		
7075-T6 (3019)	7075-T6 (3020)	7075-T6 (3021)
336 Hours	672 Hours	336 Hours
		

MAP Silico {3–5 µm}		
7075-T73 (3025)	7075-T73 (3026)	7075-T73 (3027)
168 Hours	672 Hours	336 Hours