



# DEPARTMENT OF THE NAVY

NAVAL SEA SYSTEMS COMMAND  
1333 ISAAC HULL AVE SE  
WASHINGTON NAVY YARD DC 20376-0001

IN REPLY TO:

4123  
Ser 05M2T/2009-119  
13 May 2009

Warren Environmental, Inc.  
Attn: Jane Warren, President  
P.O. Box 1206  
Carver, MA 02330

Dear Ms. Warren:

SUBJ: QUALIFICATION OF S-301-01 AS A SINGLE-COAT AND T-301-03/S-301-01  
AS A TWO-COAT SYSTEM TO MIL-PRF-23236C - COATING SYSTEMS FOR  
SHIP STRUCTURES

Based on data provided to the Navy by Warren Environmental Inc., NAVSEA has determined that Warren Environmental's "S-301-01" coating conforms to the requirements of MIL-PRF-23236C for Type VII, Class 5/18, Grade C, single-coat, fuel-tank coating; Type VII, Class 7/18, Grade C, single-coat, ballast-tank coating; and Type VII, Class 11/18, Grade C, single-coat, boiler-feedwater tank coating. In addition, NAVSEA has determined that Warren Environmental's "T-301-03/S-301-01" coating system conforms to the requirements of MIL-PRF-23236C for Type VII, Class 5, Grade C, two-coat, fuel-tank coating; Type VII, Class 7, Grade C, two-coat, ballast-tank coating; and Type VII, Class 11, Grade C, two-coat, boiler-feedwater tank coating.

Qualification is hereby granted to your products to be manufactured at Warren Environmental's plant located at 137 Pine Street, Middleboro, MA (CAGE: 356X6). Qualification is granted to your product subject to the conditions stated in enclosure (1) of this letter. Electronic Qualified Products List QPL-23236 will be updated accordingly to add "S-301-01" and "T-301-03/S-301-01" as shown below.

## SINGLE-COAT SYSTEM

GOVERNMENT DESIGNATION	MANUFACTURER'S DESIGNATION	TOTAL DRY FILM THICKNESS (mils)	TEST OR QUALIFICATION REFERENCE	MANUFACTURER'S NAME AND ADDRESS
Type VII Class 5/18 Grade C	S-301-01	System: 20-30 mils DFT in Single Coat	NEHC Ltrs 6262 Ser IH9/000755 thru 000760 of 4/11/05; NRL 5700 Ser 6130-298 of 4/8/08; DL Lab Rpt 14981 of 2/6/07	Warren Environmental, Inc. 137 Pine Street Middleboro, MA 02346 CAGE: 356X6 Plant: Same Address
Type VII Class 7/18 Grade C	S-301-01	System: 20-30 mils DFT in Single Coat	NEHC Ltrs 6262 Ser IH9/000755 thru 000760 of 4/11/05; NRL 5700 Ser 6130-298 of 4/8/08; DL Lab Rpt 14981 of 2/6/07	Warren Environmental, Inc. 137 Pine Street Middleboro, MA 02346 CAGE: 356X6 Plant: Same Address

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**SINGLE-COAT SYSTEM (cont'd)**

GOVERNMENT DESIGNATION	MANUFACTURER'S DESIGNATION	TOTAL DRY FILM THICKNESS (mils)	TEST OR QUALIFICATION REFERENCE	MANUFACTURER'S NAME AND ADDRESSES
Type VII Class 11/18 Grade C	S-301-01	System: 20-30 mils DFT in Single Coat	NEHC Ltrs 6262 Ser IH9/000755 thru 000760 of 4/11/05; NRL 5700 Ser 6130-298 of 4/8/08; DL Lab Rpt 14981 of 2/6/07	Warren Environmental, Inc. 137 Pine Street Middleboro, MA 02346 CAGE: 356X6 Plant: Same Address
<b>TWO-COAT SYSTEM</b>				
Type VII Class 5 Grade C	T-301-03/S-301-01	System: 25-50 mils DFT in Single Coat	NEHC Ltrs 6262 Ser IH9/000755 thru 000760 of 4/11/05; NRL 5700 Ser 6130-298 of 4/8/08; DL Lab Rpt 14981 of 2/6/07	Warren Environmental, Inc. 137 Pine Street Middleboro, MA 02346 CAGE: 356X6 Plant: Same Address
Type VII Class 7 Grade C	T-301-03/S-301-01	System: 25-50 mils DFT in Single Coat	NEHC Ltrs 6262 Ser IH9/000755 thru 000760 of 4/11/05; NRL 5700 Ser 6130-298 of 4/8/08; DL Lab Rpt 14981 of 2/6/07	Warren Environmental, Inc. 137 Pine Street Middleboro, MA 02346 CAGE: 356X6 Plant: Same Address
Type VII Class 11 Grade C	T-301-03/S-301-01	System: 25-50 mils DFT in Single Coat	NEHC Ltrs 6262 Ser IH9/000755 thru 000760 of 4/11/05; NRL 5700 Ser 6130-298 of 4/8/08; DL Lab Rpt 14981 of 2/6/07	Warren Environmental, Inc. 137 Pine Street Middleboro, MA 02346 CAGE: 356X6 Plant: Same Address

Unfortunately, the qualification data package is still incomplete for Warren Environmental's coating systems as a Type VII, Class 9/18, Grade C. Therefore, NAVSEA cannot approve these coatings for potable-water service at this time.

If you have any questions regarding today's letter, please contact Mr. Beau Brinckerhoff by calling (202)781-3659 or via email at [beau.brinckerhoff@navy.mil](mailto:beau.brinckerhoff@navy.mil).

Sincerely,



CHERYL A. TURNER  
Technical Policy and  
Standards Division

Encl:  
(1) Conditions

Copy to:  
GSA, Randall Schoeber

## CONDITIONS UNDER WHICH QUALIFICATION IS GRANTED

The listing of your product on the Qualified Products List does not guarantee acceptance of the product in any future purchase nor does it constitute a waiver of the requirements of the specification or the provisions of any contract.

Any use of the listing for publicity, advertising, or sales will not state or imply that the product or the process is the only one of that type so qualified, or that the government in any way recommends or endorses the manufacturer's product in preference to other qualified products. (Violation cause for removal from the list.)

Listing applies only to products produced in, or process used in, the plant specified in the letter of notification. The listing is effective at 8:00 a.m. (Local time of the qualifying activity) as of the date of the letter of notification.

Listing applies to amendments or revisions of the specification, unless otherwise notified.

Listing applies only to products or processes identical to those qualified (or to products defined in the family of products granted qualification coverage). The qualifying activity must be advised in advance of any intended change to a qualified product or process and must be provided with complete description of the change. Failure to notify the qualifying activity of any change is cause for removal from the listing regardless of the extent of the change.

**Jane Warren**

**From:** Turner, Cheryl A CIV NAVSEA HQ, SEA 05 [cheryl.a.turner@navy.mil]  
**Sent:** Thursday, March 24, 2011 3:00 PM  
**To:** Jane Warren  
**Cc:** Best, Scott; Ingle, Mark W CIV NAVSEA HQ, SEA 05; John Tock; james.martin@nrl.navy.mil; Garrett, Megan - IS; Buckley, KellyAnn - IS; randall.schober@gsa.gov  
**Subject:** RE: WARREN ENVIRONMENTAL'S REQUEST FOR QPL APPROVAL FOR TYPE VII, CLASS 13 BASED ON NRL DATA UNDER MIL-PRF-23236D  
**Signed By:** cheryl.a.turner@navy.mil  
**Attachments:** Warren-05S-2010-263-Q-2010-260.pdf; Warren-05M2T-2009-119-23236-051309MI.pdf

Ms. Warren,

This is in response to the below email which was submitted on Warren's behalf by Mr. Scott Best (PPG). NAVSEA has reviewed the NRL test data report and in accordance with the recommendations of the report, the Warren product, "Safe-T Plus" (former name S-301-01), does meet the requirements as a MIL-PRF-23236D, Type VII, Class 13 and Class 13/18, Grade C coating and should be listed as such on the QPL.

QPL-23236 will be updated accordingly to add your "Safe-T Plus" coating system under the Class 13 and Class 13/18 government designations. These classes will be identified as a single-coat and two-coat system with the same DFT and test references as identified on the attached QPL approval letter, which approved other classes.

The single coat product will be shown on QPL-23236 as "Saf-T Plus", and the two-coat product will be shown as "T-301-03/Saf-T Plus". Accept this email as an addendum to the attached NAVSEA letter Ser 05M2T/2009-119 of 13 May 2009.

If you have questions, let me know.

Cheryl Turner, SEA 05S1  
 QPL Program Manager  
 Technical Specs & Stds  
 (202)781-3734 - office phone  
 (202)781-4554 - office fax

Q-2011-062

**From:** Best, Scott [mailto:best@ppg.com]  
**Sent:** Thursday, February 24, 2011 7:08  
**To:** Turner, Cheryl A CIV NAVSEA HQ, SEA 05  
**Cc:** Choi, Mike; Jane Warren  
**Subject:** Warren Safe-T plus class 18 approvals

Hi Mrs. Turner,

I talked with Jane Warren about this subject that we discussed last week. I'm attaching the test data that shows the product has passed the 23236 type VII

3/25/2011

class 13/18 requirements. When I look in the QPL, it is not listed. Would you need any additional information to get it on the QPL? Is there any chance we can get a letter stating that it meets the requirements to help tide us over until it reaches the QPL?

Please let me know if you have any other questions.

Thank you for your help with this.

Very Respectfully,

Scott Best

Manager Government Contracts

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-----Original Message-----

From: Best, Scott [mailto:best@ppg.com]

Sent: Friday, January 14, 2011 13:55

To: Turner, Cheryl A CIV NAVSEA HQ, SEA 05

Subject: FW: Name Change

FYI.

Regards,

Scott

From: Jane Warren [mailto:jane@warrenenviro.com]

Sent: Tuesday, January 11, 2011 10:37 AM

To: Best, Scott

Subject: RE: Name Change

According to this test report, we passed class 13, but the other tests were not performed.

3/25/2011

Jane Warren

Warren Environmental Inc

A & W Maintenance Inc

P.O. Box 1206

Carver, Ma. 02330

Ph:508-947-8539

Fax: 508-947-3220

<http://www.warrenenviro.com>

6130/6012  
July 7, 2007



**Naval Research Laboratory**  
4555 Overlook Ave, S.W.  
Washington, DC 20375-5320



**Center for Corrosion  
Science and Engineering**

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## **EVALUATION OF WARREN ENVIRONMENTAL S-301 EPOXY COATING USING MIL-PRF-23236C & THE SINGLE COAT/RAPID CURE COATINGS PROGRAM**

Sandra Ambris – SAIC, Inc. Key West, FL  
James R. Martin – NRL, Code 6138, Washington, D.C.  
James Zakrzewski – SAIC, Inc. Key West, FL  
Jan Bergh – NRL, Code 6138, Key West, FL  
Arthur A. Webb – NRL, Code 6138, Washington, D.C.  
Keith E. Lucas – NRL, Code 6130, Washington, D.C.  
Edward E. Lemieux – NRL, Code 6136, Key West, FL  
Tiffanee Donowick-SET, Inc. Washington, D.C.

Encl (1) to NRL Ltr  
3900  
6130/XXXX

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## 1.0 INTRODUCTION

The Naval Research Laboratory (NRL) under direction of the Office of Naval Research (ONR) in partnership with the corrosion and coatings Technical Authority, NAVSEA 05M1, fund an Future Naval Capabilities (FNC) / Total Owner Ship Coat (TOC) study to investigate commercially off the shelf (COTS) and new technology for single coat/rapid cure coatings for corrosion control of shipboard ballast, fuel, combined holding, and potable water tanks. This report provides detailed information on the test and evaluation a COTS product Warren Environmental S-301 epoxy coating manufactured by Warren Environmental.

As of this writing, the Warren Environmental S-301 has been NSF-61 certified for potable water tanks. It was also given NEHC approval for use in Navy vessels on April 11, 2005. The Part A-Base received NEHC document number 000757 and Part B-Activator, document number 000758.

The coating was obtained, applied, tested, and evaluated in accordance with the requirements of MIL-PRF-23236C<sup>1</sup>, Type VII, Class 5, 7, 9, 11, 13, & 18, Grade C. The application was done at the customer's facility in Carver, MA. The testing and evaluation was conducted at the NRL Coatings Research Laboratory, Key West, Florida. The test and evaluation period covered in this report is July 13, 2004 through August 30, 2005.

The Warren Environmental S-301 coating was evaluated for critical basic coatings properties, general exposure performance and certain class specific tests required under MIL-PRF-23236C qualification. Class specific tests were for type VII, Class 5, 7, 9, 11 and 18, Grade C coatings. Class distinctions were as follows:

*Type VII* - A coating system having a maximum VOC content of 150 g/L (1.25 lb/gal) of coating. Coatings proposed for qualification testing to this Type have no solvent added to either the base resin component or the hardener component.

*Class 5* - A coating system without a shop primer (see 6.6.5) for use in fuel tanks, seawater ballasted fuel tanks, seawater tanks, and bilges and other ship structures interior and exterior. Class 5 coatings are not for use in tanks that may be used for fresh or potable water.

*Class 7* - A coating system without a shop primer (see 6.6.5) for use in dedicated seawater ballast tanks. Class 7 coatings are not for use in tanks that may be used for freshwater, potable water, fuels, or other hydrocarbons.

*Class 9* - A coating system without a shop primer (see 6.6.5) for use in dedicated potable or freshwater tanks. Class 9 coatings are not for use in tanks that may be used for seawater, fuels, or other hydrocarbons.

*Class 11* - A coating system without a shop primer (see 6.6.5) for use in dedicated boiler feed water (pure water) tanks. Class 11 coatings may not be used for seawater, potable water, freshwater, fuels or other hydrocarbons.

*Class 13* - A coating system without a shop primer (see 6.6.5) for use in dedicated chemical holding (ship sewage/waste) tanks (CHT tanks). Class 13 coatings may not be used for seawater, potable water, freshwater, fuels or other hydrocarbons.

*Class 18* - A single-coat coating system without a shop primer.

*Grade C* - A coating system which is to be applied and cured at a temperature above 50°F (11°C).

Results of the single coat evaluation include program overview, experimental techniques, test methodology testing procedures and results. Testing was broken into three significant parts: 1) General Single Coat Evaluation,

2) MIL-PRF-23236C Class Specific Coatings Evaluation and 3) Coatings Physical Properties Testing. Table 1 reflects the performance and summary of testing and evaluation.

**Table 1- Performance Summary Sheet**

Test Requirement	MIL-PRF-23236C Section/Spec	Pass/Fail Evaluation Criteria	Test Results	Pass/Fail Evaluation
<b>Description of Coating</b>	<b>3.1.1</b>	<b>Type VII Grade C</b>		
VOC Content	3.2.2.2	Type: VII, 150g/L – 1.25 lb/gal or less	116.2.g/L	PASS
<b>Gloss Topcoat</b>	3.3	≥ 30 @ 60 degrees	81.6	PASS
<b>Potable &amp; Freshwater Class 9 (500 gallon)</b>	3.4		Passed 1000 gal	INCOMPLETE
Color in Water	3.4.2/4.5.11.1	Color units <10	<1	PASS
Taste in Water	3.4.3/4.5.11.2	Threshold ≤2	5.0	FAIL Requesting a retest
Odor in Water	3.4.4/4.5.11.3	Threshold ≤2	1.8	PASS
Chlorine Residual	3.4.5/4.5.11.4	Not less than 50% decrease	<2%	PASS
Phenol Residual	3.4.6/4.5.11.5	< 1ppm	0	PASS
Immersion Resistance	3.4.7 / 4.5.2.3			ACCEPTABLE
Adhesion D4541	3.4.7 / 4.5.2.3	≥ 50% original	1664psi,BY100 1279psi,YZ85, 75%,YB15	PASS
Blisters D714	3.4.7 / 4.5.2.3	Blisters <4, few	No blistering	PASS
Edge Rusting	3.4.7 / 4.5.2.3	< 0.1% total edge	No edge rusting	PASS
Pin Hole Rusting	3.4.7 / 4.5.2.3	No pinhole rusting	No pinhole rusting	PASS
<b>Cathodic Disbondment All classes except 16, 17, &amp; 19</b>	3.5/4.5.16	Not >4% undercutting	No undercutting	PASS
<b>Dry Time or Cure Time All Classes</b>	3.6	<23 hours dry < 7 days cure	DRY THRU 3.5 HRS	PASS
<b>Application Characteristics</b>	3.8	Acceptable Application	Applied in a single coat with 2-3 passes.	ACCEPTABLE
Edge Retention Type VII only, all classes & grades	3.8.1	70% minimum on 1.0 mm radius	73%	PASS
Sag Resistance D4400	3.8.2	Sag <2X max WFT	0 Sag	PASS
<b>Immersion Resistance</b>	3.9			ACCEPTABLE
<b>Class 5 - Fuel &amp; Seawater</b>	3.9.1 / 4.5.2.1			PASS
Adhesion D4541	3.9.1 / 4.5.2.1	≥ 50% of original	1996psi, BY100/1871psi BY100 95%	PASS
Blisters D714		Blisters <4, few	No blistering	PASS
Pin Hole Rusting		No Pin holes	No pinhole rusting	PASS
<b>Class 7 - Seawater only</b>	3.9.3 Cycle A			ACCEPTABLE
Adhesion D4541	3.9.3/4.5.2.2.1	Overcoat ≥ 50% original	1916 psi OR 84% of original	PASS
Blisters D714	3.9.3/4.5.2.2.1	Blisters <4, few	No blistering (10)	PASS
Pin hole Rusting	3.9.3/4.5.2.2.1	No Pin Holes	No surface or edge rusting	PASS
<b>Condition in Container – All Classes except VIII &amp; VIIa</b>	3.10	See Specification	Coating easily dispersed and used in brushing, spraying and rolling.	PASS
<b>JP-5 Aviation Fuel Compatibility, Class 5 only</b>	3.13		All results within limits	PASS
Fuel Color	3.13.1/4.5.4.2	Saybolt color change ≤ 2	No color change	PASS
Corrosion	3.13.2/4.5.4.3	No increase	No increase	PASS
Existent Gum	3.13.3/4.5.4.4	Increase ≤4 mg/100ml	<1mg/100ml	PASS
Solids Contamination	3.13.4/4.5.4.5	Increase ≤2 mg/l	≤ .30	PASS
Bromine	3.13.5/4.5.4.6	<10%		NOT TESTED
<b>Aviation Gasoline (Mogas) Compatibility, Class 5 only</b>	3.13		All results within limits	ACCEPTABLE
Fuel Color	3.13.1/4.5.4.2	Saybolt color change ≤ 2	≤-1 color change	PASS
Corrosion	3.13.2/4.5.4.3	No increase	No increase	PASS

Test Requirement	MIL-PRF-23236C Section/Spec	Pass/Fail Evaluation Criteria	Test Results	Pass/Fail Evaluation
Existent Gum	3.13.3/4.5.4.4	Increase ≤4 mg/100ml	≤2	PASS
Solids Contamination	3.13.4/4.5.4.5	Increase ≤2 mg/l	0.02 increase	PASS
Bromine	3.13.5/4.5.4.6	<10%	No increase	PASS
<b>Resistance to Boiler feed water, 500 hrs. @ 180F, Class 11</b>	3.15			ACCEPTABLE
Adhesion D4541	3.15/4.5.12	≥ 50% of original	1779psi, YZ100 1641psi, YZ100 R 95%	PASS
Blistering D714	3.15/4.5.12	< #4 few	No blistering 10	PASS
Rusting	3.15/4.5.12	No edge rusting Type VII	No edge or pinhole rusting	PASS
<b>CHT Testing – Class 13</b>	3.16			ACCEPTABLE
Adhesion D4541	3.16/4.5.13	≥ 50% of original	2236psi, BY100 1868psi, BY70-YZ 30 90%	PASS
Blistering D714	3.16/4.5.13	No blisters in excess of #8 few	No blistering 10	PASS
Pin hole Rusting	3.16/4.5.13		No edge or pinhole rusting	PASS
<b>Condensing Humidity – 2000 hours @ 100F (38C) ASTM D4585</b>	3.17			ACCEPTABLE
Adhesion D4541	3.17/4.5.14	≥ 50% of original	1668psi BY100 1897psi BY100 100%	PASS
Blistering D714	3.17/4.5.14	Blisters <4, few	No blistering 10	PASS
Pin Hole Rusting	3.17/4.5.14	No pin holes	No edge or pinhole rusting 10	PASS
<b>Single Coat System Class 18</b>	3.22	Conform when tested to 4.5.20 & 3.2.1		PASS
Mix Ratio must be	3.2.1	1:1, 2:1, 3:1, 4:1	2:1	PASS
Single coat requirement	4.5.20	Test to class requested by manufacturer on QPL application but test using a single coat application in all the applicable tests.		
Class 5 Fuel, SW	3.22	4.5.20	Tested for 1 year.	INCOMPLETE
Class 7 Ded. SW	3.22	4.5.20	Tested for 1 year.	ACCEPTABLE
Class 9 PW	3.22	4.5.20	Tested for 1 year.	INCOMPLETE
Class 13 CHT	3.22	4.5.20	Tested for 1 year.	ACCEPTABLE
<b>Additional Non-MIL-PRF-23236C Testing Performed</b>				
<b>Accelerated Corrosion</b>	ASTM B117	0 – 10 rating	No damage 10	ACCEPTABLE
Adhesion Test	ASTM D 4541	Overcoat ≥ 50% original	2152psi,BY90 YZ10 1985psi,BY100 95%	PASS
<b>Alternate Immersion</b>	NRL Test #2 year exposure	0 – 10 rating ASTM D1654, D610, D714	.5mm creepage @scribe=10 No blistering	ACCEPTABLE
Adhesion Test	ASTM D 4541	Overcoat ≥ 50% original	2264psi,BY100 2003psi,BY100 90%	PASS
<b>Atmospheric Exposure</b>	NRL Test #1 year exposure	Retain 50% of original gloss/D1654 scribe evaluation	Gloss post exposure:84.1 1 year:6.8 Color post exposure 1 year: No blistering or creepage 10.	Failed color & gloss- fading. Passed scribe evaluation.
Adhesion Test	ASTM D 4541	Overcoat ≥ 50% original	2236psi,BY100 1858psi,BY80 YZ20 90%	PASS
<b>NRL Touch up &amp; Repair</b>	NRL TEST #3	Adhesion equal to that of a new coating	1741psi, BY100 1653psi, CB10, BY90	PASS

## 2.0 MATERIALS AND METHODS

Testing utilized in this report generally conforms to procedures and methodology for single coat coatings as defined in MIL-PRF-23236C. This test program investigates and evaluates coating technologies that have the following general properties:

- Applications by patented plural component spray equipment.
- Application using standard industrial surface preparation and coating practices currently present in the shipbuilding and ship repair environment
- Able to meet or exceed the performance requirements of MIL-PRF-23236C
- Cure to walk-on times of 4 hours at 75 degrees Fahrenheit
- Completely cured and ready for service in 24 hours at 75 degrees Fahrenheit
- Overcoat window greater than 45 minutes at a substrate temperature of 90 degrees Fahrenheit
- 100% volume solids, solvent free
- Free of aromatic amines (methylenedianiline)
- 1:1, 2:1, 3:1, 4:1 mix ratio
- Self-priming
- Provide optimum performance when applied to 20 to 30 mils DFT in a single application

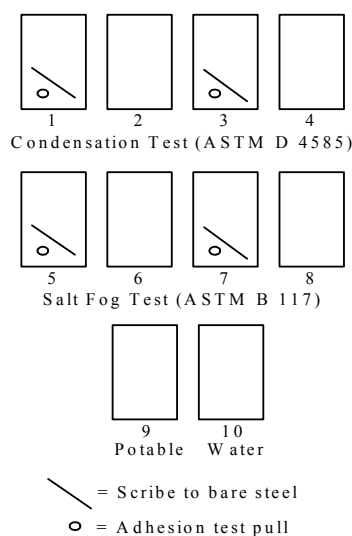
## 2.1 Panel Testing Matrix, Testing Documentation and Panel Evaluation

### 2.1.1 Photographic Documentation

Initial photographs of each panel were taken to record initial conditions and for comparison to exposed panels after testing. Correspondingly, final photographs of each panel were taken to document panel condition, damage and for comparison to initial photographs.

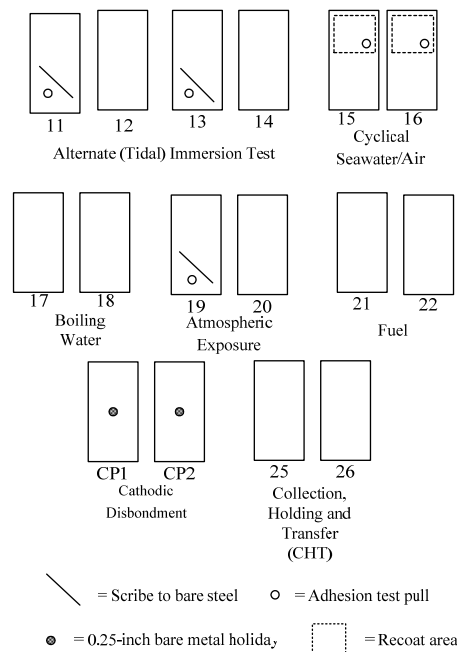
### 2.1.2 Panel Exposure Test Matrix

Figure 1 and Figure 2 list the sample number, size, and test conditions for each candidate system. In addition, the substrate preparations are listed. Twenty-six panels were utilized, including controls. The panels, not having environmental testing and exposure, were kept in reserve for possible future use and/or comparison to exposed panels. The control test panels were stored at ambient laboratory conditions in an area shaded from direct lighting.



**Figure 1 - Configuration of 4 x 6 Test Panel**

Exposed panels were evaluated at intervals during the testing and/or at the end of the exposure test duration as appropriate. Panel evaluations were performed in accordance with ASTM D 1654<sup>2</sup> (scribe), ASTM D 610<sup>3</sup> (rust) and ASTM D 714<sup>4</sup> (blister). Any noticeable film defects were noted and described in the test results.



**Figure 2 - Configuration of 6 x 12 Test Panel**

## 2.2 Substrate Preparation

Test panels were fabricated using 1/8-inch thick cold rolled carbon steel sheared into two sizes: 4" x 6" and 6" x 12". For each coating system, twenty-four 4" x 6" panels and twenty-four 6" x 12" panels were prepared.

### 2.2.1 Surface Preparation

All edges and corners of each panel were ground lightly to remove any sharp edges and corners. The panels were then washed using MIL-D-16791 degreaser and allowed to dry. Both sides of each panel were grit blasted using 36-grit aluminum oxide blast media to achieve SSPC SP-10<sup>5</sup> near-white metal finish. Using TESTEX tape to verify a surface profile on several panels, the average readings were 2.4 mils. In addition, two conductivity measurements were taken using a Horiba B-173 Twin Cond. conductivity meter, with the average readings being 9. Care was taken not to contaminate the surfaces after blast cleaning with water or oils including fingerprints. Table 2 lists the sample number, size and test conditions for each test system. In addition, the pretreatment of each test system is listed.

**Table 2- Test System Configuration and Pretreatment**

<b>SAMPLE</b>	<b>SIZE</b>	<b>TEST</b>	<b>PRETREATMENT</b>
1	4×6	Condensation	Scribe & Pull
2	4×6	Condensation	None
3	4×6	Condensation	Scribe & Pull
4	4×6	Condensation	None
5	4×6	Salt Fog	Scribe & Pull
6	4×6	Salt Fog	None
7	4×6	Salt Fog	Scribe & Pull
8	4×6	Salt Fog	None
9	4×6	Potable Water	None
10	4×6	Potable Water	None
11	6×12	Alternate Immersion	Scribe & Pull
12	6×12	Alternate Immersion	None
13	6×12	Alternate Immersion	Scribe & Pull
14	6×12	Alternate Immersion	None
15	6×12	Cyclical Immersion Recoat	None
16	6×12	Cyclical Immersion Recoat	None
17	6×12	Boiling Water	None
18	6×12	Boiling Water	None
19	6×12	Atmospheric Exposure	Scribe & Pull
20	6×12	Atmospheric Exposure	None
21	6×12	Fuel	None
22	6×12	Fuel	None
CP1	6×12	Cathodic Disbondment	None
CP2	6×12	Cathodic Disbondment	None
25	6×12	CHT	None
26	6×12	CHT	None

In some cases, alternatively, the edge coat may have been applied on top of the candidate coating system after all coats have been applied, however the previous procedures apply.

## 2.3 Coating Application

The candidate single coat coating was prepared according to MIL-SPEC 23236C instructions. The coating was applied in one spray coat at the manufacturer's site in Carver, MA. The manufacturer's recommendations were strictly followed regarding film thickness, overcoat time, cure time, etc. A wet film thickness of 18-25 mils was applied in a single coat. Panels were cured for the recommended time of 7 days and then shipped to the NRL lab in Key West, Florida.

### 2.3.1 Panel Edge Coating

An edge coat was applied to specific panels that were in need, since they had been edge coated at the Warren facility and shipped to Key West. Edge coating is applied in order to reduce the possibility of edge related failure of the candidate coating. After the panels were cleaned, the edges of each panel were coated with an immersion service, edge retentive epoxy (ideally the same topcoat used as the back coat). The edge coat was applied by brush, and completely covered all edges on the panels, including any holes in the panels. The edge coat did not extend more than approximately ½ inch from the edges.

## 2.4 Coated Panel Test Preparation

### 2.4.1 Scribe



The test panels were scribed by milling a 1/8-inch wide cut to bare metal diagonally across the lower half of the test panel. The scribe started one inch from the bottom edge of the panel, 1/2 inch from the left edge of the panel, and terminated half way up the panel 1/2 inch from the right edge. A 1/8-inch spiral mill bit was used in an Enco Milling & Drilling Machine (Enco Manufacturing Co., Chicago, Illinois, USA) in all cases.

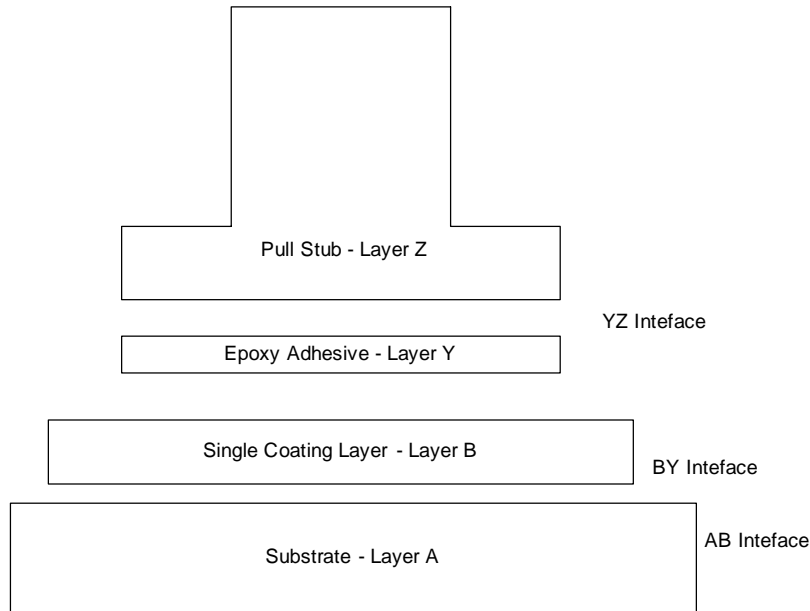
#### *2.4.2 Adhesion Test Pull*

This test was a determination of the adhesive strength of the candidate coating as adhered to the steel panel. A 1/2-inch round pull stub was rigidly glued and attached to the surface of the panel near the lower right corner of the panel, approximately 1/2 inch from both the bottom edge and the right edge. The pull stub was attached using J-B Weld Epoxy Steel (J-B Weld Co., Sulphur Springs, Texas, USA) directly to the topcoat. The epoxy was allowed to cure fully, per manufacturer instruction.

The pull stub is pulled off the panel using a Pneumatic Adhesion Tensile Testing Instrument (PATTI). The PATTI Adhesion Coatings Tester Model 2A (SEMicro Corp., Rockville, Maryland, USA) pneumatically extracts the pull stub off the panel and provides a readout of the line pressure required. The pressure reading from the instrument is converted to the tensile strength of the coating adhesion using a table calibrated to the piston utilized. The tensile strength and the mode of failure are recorded. The mode of failure observed visually, identifies the actual coating layer at which failure occurred and whether the failure was cohesive (within the layer) or adhesive (between the layers). Adhesive strength or Pull-Off Tensile Strength (POTS) is measured in pounds per square inch (psi) for each coating system and is determined in accordance with ASTM D 4541<sup>6</sup>

Adhesion data were collected from all of the panels pretreated with a pull. In addition, the mode of failure was recorded. The average pull-off tensile strength of the coating system was calculated from the individual panel data. This average value was used as a baseline for comparisons of recoat adhesion strength, as required by exposure tests. To effectively report data, a special identification system was employed. Figure 1 gives a schematic diagram to illustrate the coating layers and their letter designations utilized for a two coat coating system. The mode of failure is described using a series of letters or letter combinations, each followed by a number. Each layer of the test system, from the substrate to the pull stub, is assigned a letter. The test system substrate (steel panel) is denoted A. The first coating system layer is denoted B, and subsequent coating layers are assigned successive letters (C, D, etc.) as applicable. In addition, letters are assigned to the pull stub and the epoxy adhesive used to affix it to the test system. The letter Z always denotes the pull stub, and the letter Y always denotes the epoxy adhesive. Single letters (such as B or Y) represent the layer at which failure occurred, and denote internal cohesive failure within that layer. Letter combinations (such as AB, BC, CY, etc.) represent inter-coat adhesive failure between the two layers. The number following each letter code represents the estimated percentage of the failure that occurred in or between the specified coats. Adhesion tests were performed prior too and at the conclusion of the testing.

As an example, the code AB 20, B 50, BC 20, YZ 10 represented the following. Adhesive failure was observed between the substrate and the first coat of the coating system (layers A and B), and it accounted for 20% of the total area of failure. Cohesive (intracoat) failure was observed within the first coat of the coating system (layer B), and it accounted for 50% of the total area of failure. Intercoat adhesive failure was observed between the first and second coats of the coating system (layers B and C), and it accounted for 20% of the total area of failure. Finally, adhesive failure was observed between the epoxy adhesive and the pull stub (layers Y and Z) and it accounted for 10% of the total area of failure.



**Figure 3 - Coating layer diagram for adhesion test mode of failure evaluation**

### **3.0 EVALUATION USING ENVIRONMENTAL TESTING AND MIL-PRF-23236C**

#### **3.1 Coating Physical Properties**

Five physical properties tests were performed on the mixed product at the time of application and included: Dry Time, Resistance to Sag, Application Characteristics, Edge Retention Characterization, and Volatile Content (VOC).

##### *3.1.1 Dry Time or Cure Time*

Drying time testing is performed in accordance with ASTM D 5895-01<sup>7</sup>. The drying time is determined by placing a sample of the coating onto the top portion of a Form 1B – Penopac chart (The Leneta Company, Mahwah, New Jersey, USA) immediately after mixing (or after the induction time specified by the manufacturer if applicable). A calibrated draw down blade is used to form a film of a uniform thickness appropriate for the coating. The form is then placed in an environmental control chamber at 25°C and 40% relative humidity. A circular drying time recorder (Paul N. Gardner Company, Inc., Pompano Beach, Florida, USA) is placed over the film and started. The timer is set to 6 or 12 hours, depending on the manufacturer’s stated drying time for the coating. The drying time is calculated from the degrees of arc of the impression made by the drying time recorder using the following calculation:

$$\text{drying time} = \frac{X}{360} \times T$$

Where: X= degrees of arc of the impression, and T= set time of the recorder to complete a full circle.

### 3.1.2 Sag Resistance

The sag resistance test is not included in the MIL-PRF-23236C specification. It was added to help determine the application characteristics of the coating systems. The sag resistance of the coating is determined in accordance with Method 4494.1 of FED-STD-141<sup>8</sup>.

A sample of the coating is dropped onto the top portion of a Form 1B – Penopac chart (The Leneta Company, Mahwah, New Jersey, USA) immediately after mixing. A notched blade is drawn down the form in one smooth continuous motion, creating bands of coating approximately ¼-inch wide and 1/16-inch apart. The blade was chosen so that the thickness of the middle notch is near the recommended application thickness, thereby bracketing the recommended thickness with some bands thinner and some bands thicker. Leneta Anti-sag Meter blades ASM-1 and ASM-4 (The Leneta Company, Mahwah, New Jersey, USA) were used for the coatings in this test. Blade ASM-4 has ten calibrated notches that range in thickness from 3 mils to 12 mils in 1-mil steps. Blade ASM-4 has eleven calibrated notches that range in thickness from 4 mils to 24 mils in 2-mil steps.

Immediately after the bands of coating were drawn down the chart, the chart was hung vertically such that the bands run horizontally on the chart with the thickest band at the bottom. The coating was allowed to dry in an environmental control chamber at 25 °C and 40% relative humidity.

After drying, the chart was examined to determine the sag resistance of the coating. The bands are examined for the thinnest band where the coating has crossed into the strip below it. The thinnest band of coating where this occurs is considered the thickness at which the coating's sag resistance has failed. The recommended coating thickness and the failure thickness were noted. The criteria for sag resistance, was not specified under MIL-PRF-23236C section 3.12 as noted by section 3.8.2. The criteria, given by Naval Sea Systems Command on 22 Jan 2004, was to be 2 times the target wet film thickness (WFT), required by the manufacturers data sheet at ambient temperatures. This means that if the target thickness was 5 mils WFT, sag shall not exceed 15 mils WFT.

### 3.1.3 Application Characteristics

This portion of the testing is a comparative and somewhat subjective commentary on the ease and efficacy of the manual application of the coating to the various panel conditions. The Warren Environmental S-301 was applied using patented spray equipment (patent # 5,645,217). Model 629,913. The Warren equipment utilizes GRACO 46:1 pumps, along with Binks parts. The 2:1 mix ratio is heated to a temperature of approximately 112°F for the activator and 181°F for the base so that the viscosity can be controlled. Environmental conditions at the time of application were: 71.7°F air temperature, 61.9%RH, and 57.3°F dew point. 10 gallons of base (batch# 40831-2) and 5 gallons of hardener (batch# 40831-1) were placed in the appropriate containers and circulated through the equipment for approximately 30 minutes to allow for proper heating. The S-301 was then applied to successive panel groups.

### 3.1.4 Edge Retention Characterization

The edge retention specimen should use an Aluminum Alloy 6061, 1 inch structural angle (90°) section approximately 6 inches long. Preparation of the specimen for test required three basic steps: surface cleaning, radius grinding, and grit blasting to provide acceptable anchor tooth prior to coatings application. Procedures for preparation were as follows:

Cleaning: Specimens were cleaned thoroughly with acetone or appropriate solvent to remove all traces of grease prior to abrasive blasting and coating application.

Radius Grinding: The 90° edge of the specimen was ground to a radius of 1 mm by lengthwise passes on 60 grit sandpaper. The sample was gradually tilted over toward the flat side with each successive pass, and then

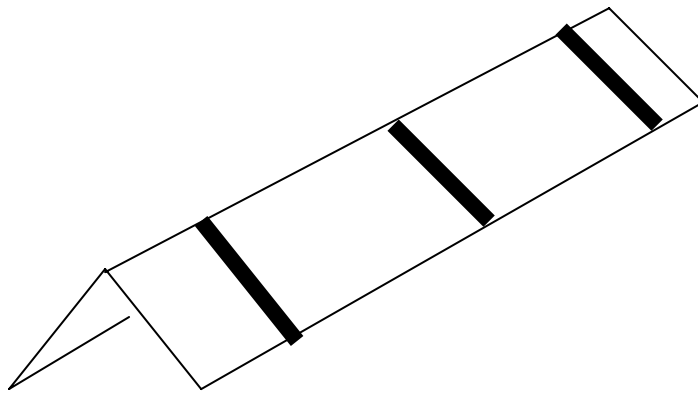
reversed to achieve a symmetrical grind. A 1 mm machinist's radius gage was used to confirm that the correct radius has been achieved across the entire length of the angle.

Grit Blasting: Specimen was grit blasted using  $\text{Al}_2\text{O}_3$  media to achieve an anchor tooth profile of 1-3 mils. Care was taken to minimize rounding of the edge with excess blasting.

The candidate coating was then brush applied on the specimen angle to the wet film thickness recommended by the manufacturer. After application, the specimens were cured at ambient laboratory conditions for a minimum of 72 hours before sectioning.

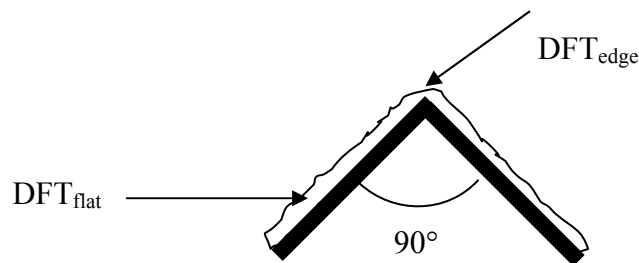
The cured test sample was sectioned at three representative locations by removing a ~5 mm slice of the angle. The dry film thickness of each section was measured from a photomicrograph from an optical microscope, or directly from a magnified digital image on the computer monitor. Measurements were made at sufficient distance from the 90 degree angle to ensure there is no wrap around edge effect-- this is designated *DFT (flat)* in Figure 4. The minimum thickness at the apex is also measured to determine DFT (edge).

Pass/fail criteria: A minimum of three separate samples were prepared and measured. Three sections were further cut from each sample, with the coating thickness measured and edge retention calculated for the 9 sections. For acceptance of the pass/fail criteria, the average of all readings shall not be less than 70%. Figure 5 shows an example of an edge retention failure while Figure 6 shows a specimen with an acceptable value above 70%.

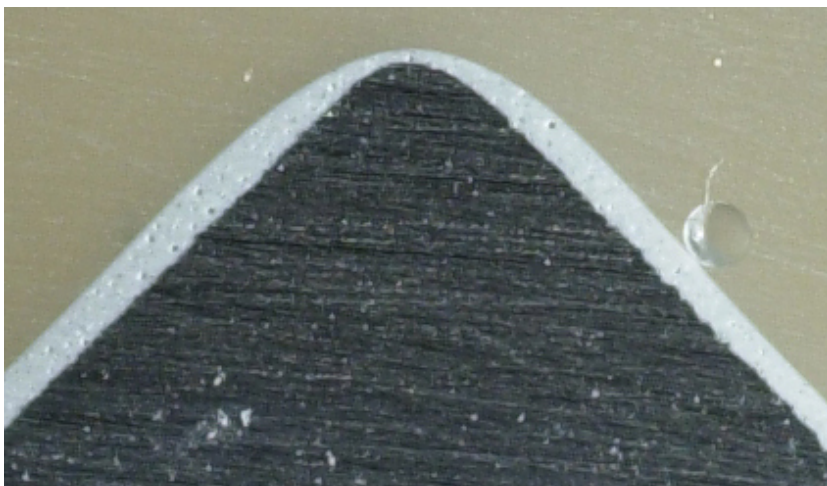


The edge retention is calculated from:

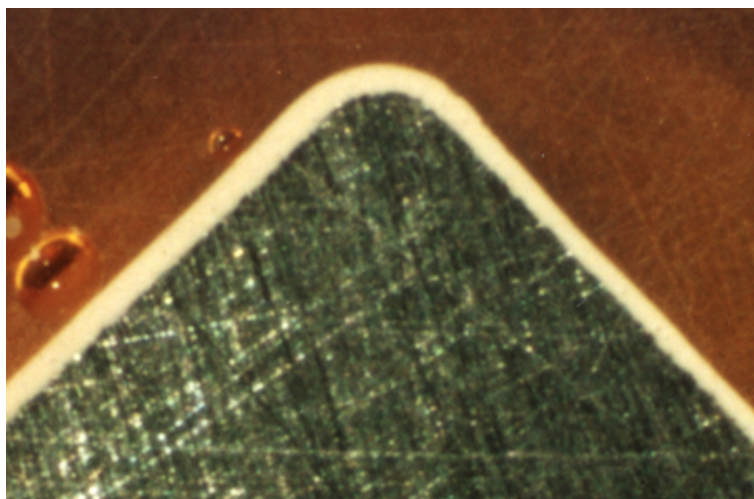
$$\% \text{ Retention} = \text{DFT (edge)} / \text{DFT (flat)} \times 100$$



**Figure 4 - Specimen angle diagram for determining edge retention**



**Figure 5 - Example of a coating that fails the edge retention test (~30x magnification)**



**Figure 6 - Example of a coating that passes the edge retention test (~30x magnification)**

### *3.1.5 Volatile Content (VOC) Determination*

The VOC of the test coating was determined in accordance with USEPA 40 CFR Ch.1, part 60, Appendix A, Method 24. This Method references ASTM D 2369<sup>9</sup>, Standard Test Method for Volatile Content of Coatings as the required procedure. NRL has modified and expanded this method to accommodate the high-solids epoxy formulations that did not test accurately in the standard procedure. In addition to the original solvent dispersal and 110°C one hour oven bake, three additional treatments have been added: solvent/24 hour ambient cure; no solvent/bake; no solvent/24 hour ambient cure. These additional conditions are intended to obviate concerns that the solvent dispersal stage could interfere with the intended chemical reaction of the components.

Results are reported as both the standard method and as an average of the four different treatments. If the percent volatile content of the coating is less than the VOC limit for the coating type, a pass rating is indicated for the VOC qualification requirement. If the percent volatile content is greater than the VOC limit for the type of coating being tested, a fail rating is indicated. This coating system was submitted for testing as a type VII coating, which means the VOC content of each layer of the coating system must be less than 1.25 lb/gal (150 g/L).

### 3.2 Environmental Exposure Testing

Designated prepared test panels were evaluated in various test regimes including: *Condensing Humidity; B-117 Salt Fog; Alternate immersion and Constant Immersion.*

#### 3.2.1 ASTM D4585 Condensation Testing

Four 4 x 6 x 1/8-inch test panels were prepared in accordance with the identified stages, labeled and inserted into the ASTM D 4585<sup>10</sup> Condensing Humidity tester for 2000 hours. The condensation test was performed in accordance with ASTM D 4585. Water vapor was generated by heating a pan of tap water to 50°C at the bottom of a Q-C-T Cleveland Condensation Tester (Model QCT/ADO, The Q-Panel Company, Cleveland, Ohio, USA). Figure 7 shows an example of the condensation tester used in this study. The designated test panels were placed as tiles above the heated water, such that the backsides of the panels were exposed to the cooling effects of the ambient temperature air of the laboratory. The resulting heat transfer causes water vapor to condense on the test panels as water saturated with air. The test panels are inclined so that as condensate runs off the test surface by gravity it is continuously replaced by fresh condensate. The test panels were continuously exposed to these conditions for 2000 hours.

The test panels were removed immediately upon conclusion of the test period. The test panels were wiped dry with a soft paper towel or soft cloth and examined between 5 and 10 minutes after removal from the apparatus to ensure that any water effects were still evident. For all of the test panels, the protection rating of the coating system is determined according to the 0 to 10 scale described in ASTM D 1654 (scribe), 10 representing no creepage, ASTM D 610 (rust), 10 representing no rust and ASTM D 714 (blister), 10 representing no blistering. Any noticeable film defects were noted and described in the test results. For those panels that were pretreated with a scribe and pull, the extent of blistering, rusting, undercutting or other damage near the pretreated areas was carefully noted and included in the final report.



**Figure 7 - Condensing humidity tester**

#### 3.2.2 ASTM B-117 Environmental Exposure

Four 4 x 6 x 1/8-inch test panels were prepared in accordance with the identified stages, labeled and inserted into the ASTM B117<sup>11</sup> test apparatus for 1000 hours. Figure 1 identifies the panel test configuration used in these

tests. The alternate immersion test was not included in the MIL-PRF-23236C specification, but was utilized in the coating evaluation to help predict the service life of the coating systems in a tidal environment.

The salt fog test was conducted in accordance with ASTM B-117. A Q-FOG Cyclic Corrosion Tester (Model Q-FOG/CCT1100, manufactured by The Q-Panel Company, Cleveland, Ohio, USA) was used as the test chamber. Figure 8 shows an example of the cyclic corrosion tester used in this study. The Q-FOG comes from the factory with an internal program to follow the ASTM B-117 test method. This program was used to create the test conditions for the specified test panels.

The B-117 test apparatus utilizes a 5% sodium chloride solution prepared for use in the test chamber by adding 50 grams sodium chloride (Sigma Chemical Company, St. Louis, Missouri, USA) per liter of laboratory prepared de-ionized water. The pH of the salt solution is adjusted to 6.5-7.2 using dilute NaOH or HCl solutions. A clean, oil-free air supply is used to supply the nozzles in the test chamber. The temperature is maintained at  $35 \pm 1.7^\circ\text{C}$  for the duration of the test.

The test panels were placed in fiberglass racks such that they are parallel to the flow of fog through the chamber and were inclined approximately  $15^\circ$  from vertical. Two clean fog collectors were placed in the chamber with the test panels periodically during the test. The quantity of fog, salt concentration in the collected solution and pH of the collected solution were monitored using the salt solution from the fog collectors throughout the test period. The concentration and pH of the prepared salt solution could be adjusted, if necessary, to achieve a  $5 \pm 1$  weight percent sodium chloride and pH of 6.5 – 7.2 in the collected fog solution. The fog generator was adjusted as necessary to achieve between 1.0 to 2.0 mL/hour in the fog collectors. The test conditions were maintained continuously and not interrupted except to place and retrieve the fog collectors necessary for monitoring the test conditions.



**Figure 8 - Salt fog test apparatus**

Upon completion of the 1000 hour test duration, the test panels were carefully removed, rinsed with clean water to remove salt deposits and examined carefully. For all of the test panels, the basic corrosion performance of the coating system after exposure to corrosive environment was determined according to the 0 to 10 scale described in ASTM D 1654 for scribe evaluation, ASTM D 610 for degree of rusting and ASTM D 714 for degree of blistering. For those panels that were pretreated with a scribe and pull, the extent of blistering, rusting, under cutting, or other damage near the pretreated areas was carefully noted.



### 3.2.3 Alternate Immersion Exposure

Twelve 6 x 12 x 1/8-inch test panels were prepared in accordance with the identified stages, labeled and inserted into alternate immersion for one year. Figure 2 identifies the panel test configuration used in these tests.

The alternate immersion test was not included in the MIL-PRF-23236C specification, but was utilized in the coating evaluation to help predict the service life of the coating systems in a tidal environment. The designated panels were placed in a tank and subjected to a 24-hour test cycle involving the following four steps in the order specified:

1. The tank is filled with fresh natural seawater to a level that submerges all of the test panels,
2. The tank is kept full for 12 hours with the seawater constantly being refreshed;
3. The tank is drained to a level where only approximately one inch of each test panel is immersed. The rest of the panels are above the water line and might dry naturally;
4. The tank is kept at the low level for 12 hours.

Operations 1 to 4 constituted one complete cycle. This cycle was repeated continuously for one year. Figure 9 shows an example of the tank system used in the alternate immersion test.



**Figure 9 - Alternate immersion test immersion tanks**

Upon completion of the test duration, the test panels were carefully removed, rinsed with clean water to remove salt deposits and examined carefully. For all of the test panels, the protection rating of the coating system was determined according to the 0 to 10 scale described in ASTM D 1654. For those panels that were pretreated with a scribe and pull, the extent of blistering, rusting, under cutting, or other damage near the pretreated areas were carefully noted.

## 3.3 MIL-PRF 23236C Qualification Tests

### 3.3.1 Cathodic Disbondment Testing

Cathodic disbondment panels were tested in accordance with MIL-PRF-23236C section 3.5/4.5.22 and consisted of the following procedure. Panels for this evaluation required that a 10 gage copper wire attached for later connection to the magnesium test anode. Testing was performed at NRL Marine Corrosion facility in Key West, FL. in 4' diameter by 3' deep circular fiberglass tanks. The tanks, shown in Figure 10, contained continually refreshed natural seawater.





**Figure 10 - Cathodic disbondment test tank with anode**

A high potential magnesium anode was suspended in the center of the tank and was electrically connected to each panel. The test panels consisted of coated 6" x 12" x 1/8" cold rolled mild steel plates that were mounted along the perimeter of the tank at a distance of 2 feet from the anode. Directly in the center of each panel, a 1/4" hole was drilled through the coating to the metal substrate, but not through the steel. The test was run for a period of 90 days, (3 months) and evaluated per MIL-PRF-23236C section 3.5/4.5.16. Pass/fail criteria stated that there shall be no more than 4% damage at the holiday.

### *3.3.2 Cyclical Seawater/Air Immersion Resistance (Cycle A)*

Natural seawater replaces synthetic seawater (3% salt in distilled water) in these procedures. Some materials that perform well in synthetic seawater tests might not perform as well in natural seawater with all of its inherent organisms and additional dissolved substances. Therefore, natural seawater is used to better predict actual performance in the tank environment. The designated panels are subjected to a test cycle involving the following three steps in the order specified:

1. Salt water immersion for 5 days: panels are immersed totally for 5 days in an immersion tank (see Figure 11),
2. Air dry at ambient laboratory temperature for 2 days: the immersion tank is drained and the panels are allowed to dry at ambient temperature,
3. Hot water immersion for two hours: panels are immersed totally in hot, deionized water for two hours at 80 °C. (This is an added step to simulate cleaning of the ballast tank with a heated pressure washer.)



**Figure 11 - Immersion tank used for cyclical testing**

Operations 1 to 3 constitute one complete test cycle. This cycle is repeated and coating deterioration is noted after each complete cycle. If the coating is still satisfactory after 25 cycles, the panels are wiped lightly with a soft cloth and fresh water. The panels are allowed to dry for 48 hours. The central upper third of one side of each panel (13 mm (½ inch) inward from the edges) is recoated with one coat of the finish coat of the coating system. The recoated panels are allowed to dry for one week. Adhesion is determined in the recoated area of one of the panels in the same manner that it is determined for panels pretreated with the pull procedure. Both panels are subject to 25 additional test cycles. The total coating system must meet the following requirements to achieve an acceptable (pass) result.

1. It must show no pinhole rusting, loss of adhesion either between coats or to the substrate, or blisters larger than 1.5 mm (1/16 inch) in diameter.
2. Any blisters less than 1.5mm in diameter must not increase in size after the tenth cycle and must not exceed 3% of the test panel surface area.
3. It must show no edge rusting.
4. On the recoated area, adhesion of the added coating must be at least half of the adhesion of the original coats.

All coating systems that do not meet one or more of the above criteria receive an unacceptable (fail) result.

### *3.3.3 Fuel and Seawater*

Immersion test for fuel and seawater ballast exposure (all types, classes 5 and 6, all grades). Two coated panels shall be subjected to 25 cycles or to prior failure, whichever comes first, of the following test cycle. The cycle comprises three operations carried out in the order specified with panels prepared as specified in 4.5.2(a) and (b):

- a. Salt water immersion for 1 week (7 days): Immerse panels totally for 1 week in natural seawater, at a temperature of  $27 \pm 6$  °C ( $80 \pm 10$  °F). There shall be no more than 8 hours between completion of step (a) and the beginning of step (b).
- b. Aromatic fuel immersion for 1 week (7 days): Following salt water immersion, the test Warren S-301 test panels were immersed totally in JP-5 at a temperature of  $27 \pm 6$  °C ( $80 \pm 10$  °F). There shall be no more than 8 hours between completion of step (b) and the beginning of step (c).

c. Hot water immersion for two hours: This operation is intended to simulate conditions encountered in the use of tank cleaning equipment. Following fuel immersion, the panels were immersed in natural seawater or natural seawater, for 2 hours at 80 °C (175 °F) (nominal).

Operations (a) to (c) constitute one complete test cycle.

The total coating system must meet the following requirements to achieve an acceptable (pass) result.

1. It must show no pinhole rusting, loss of adhesion either between coats or to the substrate, or blisters larger than 1.5 mm (1/16 inch) in diameter.
2. Any blisters less than 1.5mm in diameter must not increase in size after the tenth cycle and must not exceed 3% of the test panel surface area.
3. It must show no edge rusting.
4. On the recoated area, adhesion of the added coating must be at least half of the adhesion of the original coats.

#### *3.3.4 Atmospheric Exposure*

The atmospheric exposure test is not included in the MIL-PRF-23236C specification. It was added to help predict the service life of the coating systems. The atmospheric exposure test is conducted in accordance with ASTM D 1014-95<sup>12</sup>. The designated panels are subjected to exposure out of doors.

The test panels are placed in aluminum racks facing south at a 45° angle from horizontal as shown in Figure 12. The racks are set outside the laboratory approximately 18 to 40 feet from the Eastern Shore of Fleming Key. The test panels are approximately 5 to 12 feet above sea level when in the racks. The test panels are insulated from the aluminum by plastic supports below the panels and PVC strips on the clamps holding the panels in the racks. The test panels are mounted such that no shadows are cast on other test panels, and so that any run off from one test panel does not flow to any other test panel.



**Figure 12 - Atmospheric exposure rack in Key West, Florida**

The test duration is indeterminate, and may continue indefinitely. Every three months for at least one year, the test panels are examined carefully and observations are recorded. In addition, the protection rating of the coating system is determined for each test panel according to the 0 to 10 scale described in ASTM D1654. For those panels that were pretreated with a scribe and pull, the extent of blistering, rusting, undercutting or other damage near the pretreated areas is carefully noted. If the test is continued beyond one year, the test panels are examined every 6 months and evaluated as above. The test is terminated at the discretion of the scientist and final examination of the test panels is performed at termination.

### *3.3.5 Aviation Fuel Compatibility*

Two sets of fuel compatibility tests are conducted with one set using JP-5 fuel and one set using AVGAS. The preparation of the two types of fuels for testing is described in separate sections below. The aviation fuel compatibility test procedures below apply to both types of fuel with the exception of the bromine test which is only conducted with AVGAS. Any differences in test procedures for the two types of fuel are noted in the text.

JP-5 Fuel Test Sample Preparation - Approximately 5 liters of fuel is prepared by repeated filtration through type AA Millipore filters until the fuel is particle free. The JP-5 fuel used for each segment of the fuel compatibility test conforms to MIL-T-5624<sup>13</sup>. Two of the coated test rods are chosen for the test, based on visual examination of the quality and evenness of the coating. The chosen test rods are washed thoroughly with hot water at  $82 \pm 6$  °C for 15 to 30 minutes, drained and dried. The test rods are mounted on 6-mm by 180-mm square plastic beaker covers that have been center drilled for mounting the test rods. Four clean 2-L beakers are filled with 1000 – 2000 mL of prepared JP-5 fuel. Two of the beakers are covered with a rod-mounted cover as test samples and two beakers are covered with standard plastic covers as control samples. All of the beakers are stored in the dark at  $27 \pm 6$  °C for 30 days. The fuel is swirled by rotating the beakers 3 times daily. On the 31<sup>st</sup> exposure day, samples of the JP-5 fuel test and control samples are bottled and sent to Herguth Laboratories, Inc. 101 Corporate Place, Vallejo, CA 94590. Fuel color, corrosion, existent gum and solids contamination determinations were performed by Herguth Laboratories at the request of NRL.

AVGAS Test Sample Preparation - Commercial aviation gasoline (low lead) is filtered twice through two layers of 1.2 micron glass micro fiber 9.0 cm diameter filter paper (Cole Parmer U-0664874) through a 4 inch diameter Buchner funnel into a 2 Liter Pyrex vacuum flask. Approximately three-fourths of a gallon is added to each of four one-gallon wide-neck clear glass jars (with a shatter/leak resistant PVC exterior coating (Cole Parmer U-34604-50)). The jars are sealed for six days and kept in a light-proof flammable solvent cabinet until the immersion of the rods. Two fuel rods are added to each jar. Two additional jars are preserved as control samples which remain unexposed to any coating sample. The jars are gently swirled twice daily, and maintained in the dark at 82°F for 30 days. The fuel rods are then removed and the jars are visually inspected and photos are taken. Five days later, the AVGAS test and control samples are bottled and sent to Spectrum Laboratories, Inc. 1460 W. McNab Road, Ft. Lauderdale, FL 33309. Fuel color, corrosion, existent gum, solids contamination, and bromine determinations are performed by the laboratory and the results are noted.

### *3.3.6 Fuel Color Test*

The color is determined for both the test and control samples in accordance with ASTM D 156<sup>14</sup>. For JP-5 fuel, the Saybolt color numbers of the test sample and the control sample are reported. A difference of two or less indicates an acceptable (pass) result. A Saybolt color difference between the test and control samples of greater than two indicates an unacceptable (fail) result.

### *3.3.7 Corrosion Test*

Corrosiveness is determined for both the test and control samples in accordance with ASTM D 130<sup>15</sup>. For JP-5 fuel, the copper strip is incubated in the presence of the fuel samples for 3 hours at 50 °C (per ASTM D 1655-05<sup>16</sup>). For AVGAS, the copper strip is exposed to the sample for 2 hours at 100 °C per ASTM D 910-04<sup>17</sup>. The classification of the test strips and the exposure times are reported for both the control and the test sample. An acceptable (pass) result is indicated if the test sample shows no more corrosiveness than the control sample. If there is any increase in corrosiveness in the test sample relative to the control, an unacceptable (fail) result is indicated.

### *3.3.8 Existent Gum Content*

Existent gum is determined for both the test and control samples in accordance with ASTM D 381<sup>18</sup>. For JP-5 fuel, unwashed existent gum only is determined. The existent gum is reported in mg/100 mL. An acceptable (pass) result is indicated if the difference between the existent gum in the test sample and the existent gum in the control sample is 4 mg/100 mL or less or the washed existent gum in the test sample and the existent gum in the control sample is 2 mg/100 mL or less. An unacceptable (fail) result is indicated if the difference between the unwashed existent gum in the test sample and the existent gum in the control sample is greater than 4 mg/100 mL or the difference between the washed existent gum in the test sample and the washed existent gum in the control sample is greater than 2 mg/100 mL.

### *3.3.9 Solids Contamination Test*

The solids contamination for both the test and control samples is determined in accordance with ASTM D 2276<sup>19</sup> for the JP-5 fuel and ASTM D 5452<sup>20</sup> for the AVGAS. The total sediment in mg/L and the volume of sample filtered in mL for both the test and control samples are reported. An acceptable (pass) result is indicated if the difference in total sediment between the test and control sample does not exceed 2 mg/L. An unacceptable (fail) result is indicated if the difference in total sediment between the test and control sample exceeds 2 mg/L.

### *3.3.10 Bromine*

The concentration of ethylene dibromide (bromine) is determined for the AVGAS sample only. The methodology is per EPA method 8260<sup>21</sup> which utilizes mass spectroscopy. This method is preferred over the process described in MIL-PRF-23236C because it is less hazardous, cumbersome, expensive, and time consuming to set up. The mass spectroscopy method is widely used and provides analysis within a few seconds. An acceptable (pass) test result is indicated if the reduction in the bromide content is less than 10%. An unacceptable (fail) result is indicated if the reduction in the bromide content is 10% or greater. A sample must reflect <21 ppm of total bromides to pass.

### *3.3.11 Collection, Holding and Transfer*

The tests are performed as specified in MIL-PRF-23236C. The test panels are immersed in each of the solutions listed below for a period of 14 days at a temperature of  $21 \pm 2.7^{\circ}\text{C}$  ( $70 \pm 5^{\circ}\text{F}$ ). Each operation involves removing the panel from a solution, recording the observations, and then placing the panel in the next solution. Operations using solutions (I) to (V) constitute one complete test cycle (70 days). This cycle is repeated for a total test time of 140 days (20 weeks) and coating deterioration is recorded after each complete cycle. Care is taken not to contaminate solutions with carry over of solution between immersions. Panels are rinsed and/or washed with soap to remove residue as needed. Solutions are tested, refreshed, or replaced as necessary to maintain solution composition.

### CHT Test Solutions:

- I. Acetic acid and natural ocean water adjusted to a pH of 3.5 – 4.0
- II. 10 parts urea concentrated ammonium hydroxide in 90 parts natural ocean water
- III. 7 parts concentrated sulfuric acid in 93 parts natural ocean water
- IV. 10 parts urea 90 parts natural ocean water
- V. 2 parts detergent and 98 parts natural ocean water

The coating system is evaluated for pinhole corrosion, ASTM D 4541 adhesion of the original and recoated surfaces, cohesion and adhesion values of the tested system to itself and the substrate shall be at least 50 percent of the values for cohesion and adhesion of the untested original coating to itself and the substrate before testing. It shall be evaluated for blistering and surface imperfections (includes peeling) larger than 1.5 mm (1/16) inch in diameter. It is also evaluated for color, gloss, and edge rusting.

### *3.3.12 Potable and Fresh Water Tests*

The test procedures for the potable water tests are based on the MIL-PRF-23236C. Minor modifications have been made to the specified procedures to facilitate testing. The American Public Health Association (APHA), American Water Works Association (AWWA), and the National Sanitation Foundation (NSF) industrial and municipal standards are referred to in the specification and are also used as references. The water utilized for the taste and odor testing was purified by sand filtration, carbon filtration, reverse osmosis, ion exchange, ultraviolet radiation, and ozonation. Conductivity of this water was measured at 1 microSiemen. Water utilized for all of the tests except taste and odor was 18.2 megohm resistivity DeIonized (DI) water, locally (within the same laboratory room) purified by a continually circulating reverse osmosis, Ion Exchange, and carbon filtration system.

The Warren S-301 coating was applied to a 24" x 24" x 1/4" sheet of ultra high molecular weight polyethylene at a thickness of 25 mils, allowed to dry for one day, and then recoated. The system is then allowed a final cure of at least one week. This procedure was completed during the initial application of test panels as stated in section 2.3 Coatings Application.

MIL-PRF-23236C specifies many tests to qualify a coating system for an application type, this section specifically addresses those tests for Potable Water Tanks (4.5.11.1 through 5), specifically:

- 4.5.11.1 – Color in water: The extent to which a coating imparts a change in color to water stored in a coated tank.
- 4.5.11.2 – Taste in Water: The extent to which a coating imparts a change in taste to water stored in a coated tank.
- 4.5.11.3 – Odor in Water: The extent to which a coating imparts a change in odor to water stored in a coated tank.
- 4.5.11.4 – Chlorine Residual: The extent to which a coating reacts with chlorine (as hypochlorite) during disinfection of the tank.
- 4.5.11.5 – Phenol Contamination: The extent to which a coating leaches phenolic compounds into water stored in a coated tank.

The acceptability requirements for the tests above are listed below per MIL-PRF-23236C, Section 3.4:

- 3.4.2 – Color in Water: When tested as specified in 4.5.11.1, color shall not be greater than 10 (units).
- 3.4.3 – Taste in Water: When tested as specified in 4.5.11.2, threshold taste values (FTN) shall not be greater than 2.
- 3.4.4 – Odor in Water: When tested as specified in 4.5.11.3, threshold odor values (TON) shall not be greater than 2.

- 3.4.5 – Chlorine Residual: When tested as specified in 4.5.11.4, chlorine residual shall not decrease in excess of 50 percent of concentration.
- 3.4.6 – Phenol Contamination: When tested as specified in 4.5.11.5, the cured film of each coating of the coating system shall not leach phenolic compounds in concentrations greater than 1 part per million (ppm).

### 3.3.12.1 Test Procedure

The procedure for applying the bell jar wax ring is as follows:

The following steps (2-9) are repeated to seal two 1.4liter Pyrex bell jars on the coating. A pen is used to make a circle on the paint surface that is very slightly larger than the OD of the bell jar lip. (Outline of Bell Jar Lip).

The 1.4 Liter Bell Jar is heated in a convection oven at 95°C for one hour. At same time, about 100 grams of paraffin (Gulf canning wax) is melted in an open container in the same oven. The wax is removed from the oven. A flux brush is used to paint a wax ring (3/8" wide – same annular thickness as the bell jar lip) around the interior of the pen circle drawn on the coating (step 1 above). Three layers of this wax ring (trace outline of circle three times with flux brush) are done. The wax ring on paint is allowed to cool and solidify (5-10 minutes).

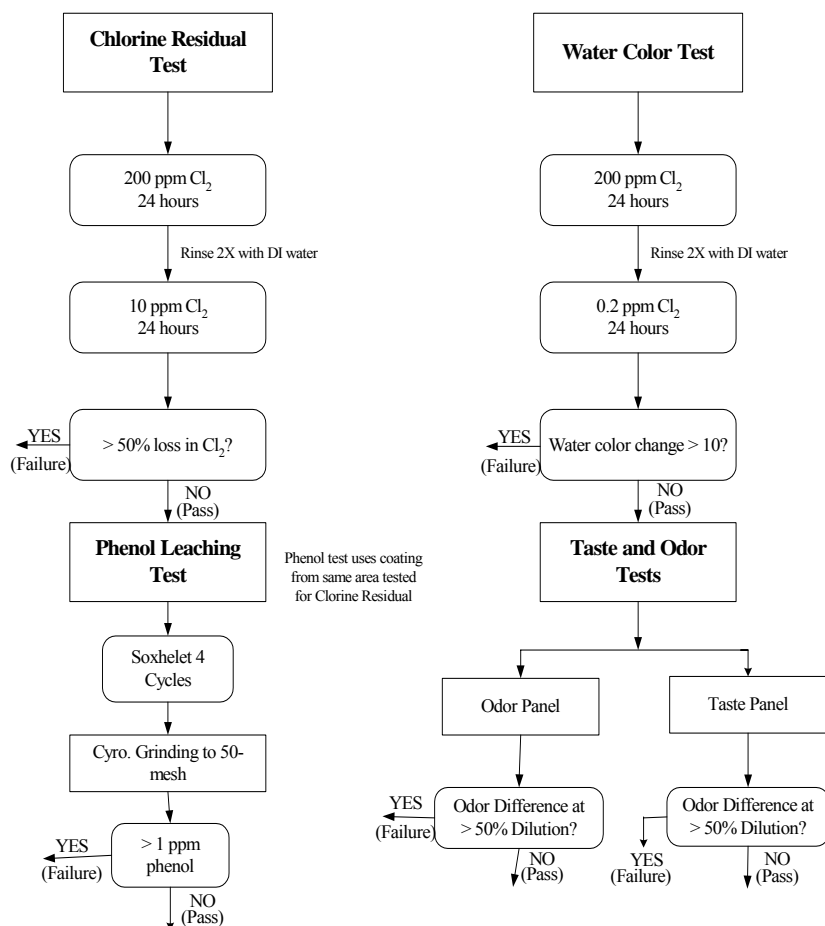
The Pyrex bell jar is removed from the convection oven. The lip of the bell jar is carefully heated for about one further minute utilizing an 1800-watt forced air hot air gun. Caution must be taken to rotate the bell jar quickly while utilizing the heat gun as Pyrex object could be shattered by thermal stresses from a heat gun, if too much of a hot spot builds up. The hot bell jar lip is pressed down onto the wax ring on the coating. 1-3 pounds of downward force pressure is applied on the bell jar, which is turned slightly but promptly (approximately 15°) in order to ensure that wax is uniformly melted under the bell jar. Force is continually applied on bell jar for about 2 minutes. The bell jar is allowed to cool for 1-2 hours. The top of the bell jar is not sealed. (Vacuum created by the cooling bell jar can break the wax seal). The bell jar is sealed until the 200ppm chlorine (color or chlorine residual tests) solution is added.

All glassware utilized is Pyrex, washed with Sparkleen, rinsed with tap water, rinsed three times with 18.2 MΩ DI water, and air-dried before use in testing. This procedure varies slightly from MIL-PRF-23236C draft revision 12 August 2003 in the following ways:

Two 1.4 bell jars are used simultaneously instead of one 500 mL bell jar. The procedure as written does not provide sufficient water for the taste and odor tests. Taste and odor testing is performed simultaneously. This minimizes the manpower and scheduling required for the 5-person taste and odor panel. Figure 13 represents the sequence of tests.

The following Chemetrics Kits were utilized for the chlorine and phenol extractions.

Available Chlorine 0.1 – 5 ppm Chemetrics K2505 kit  
 Available Chlorine 5.0 – 250 ppm Chemetrics 2505A kit  
 Available Chlorine 2.5 – 25 ppm Chemetrics K2505D kit  
 Phenol 0.1 – 12 ppm Chemetrics K8012 kit



**Figure 13 - Illustration of the sequence of tests**

### 3.3.13 Taste and Odor (Flavor) in Water

Sample preparation for evaluation by the taste and odor panel participants was performed according to MIL-PRF-23236C, APHA 2150B<sup>22</sup> (Threshold Odor Test) and APHA 2160B<sup>23</sup> (Flavor Threshold Test). The extractions were held in 2 L Erlenmeyer flasks and sealed with a ground glass or PTFE stopper. In accordance with the MIL spec, two separate extractions were taken for the taste and odor tests. The procedures state that separate extractions be performed at the same location on the test panel per 4.5.11.2 and 4.5.11.3. The first extraction is used for taste threshold and color change tests while the second is for odor threshold testing. Some individuals may consider this test subjective and not necessarily a failed result.

The taste and odor panel consisted of five individuals, including some having consumed waters produced and stored aboard ships in the past through service in the Navy. A range of sensitivity to taste and odor detection from dull to acute was reported by the individuals. However, all panelists received the same dilutions at the same time. Panelists were also instructed to refrain from discussing their opinions with other panelists during the taste and odor session.

### 3.3.14 Color Change Test

Prior to beginning the first extraction (for taste), 200 ml of the test water was retained in order to conduct color change tests per APHA 2120B<sup>24</sup>. After exposure of the remaining water was completed, a 50 ml sample was drawn and compared to the unexposed sample for any change in color using the platinum cobalt method, a stock solution



having known concentrations of potassium chloroplatinate ( $K_2PtCl_6$ ) and cobaltous chloride ( $CoCl_2 \cdot 6H_2O$ ) and assigned a color value of 500 Units. The stock solution is then diluted to the desired range of 1 to 10 units for comparison to the sample water.

### 3.3.15 Chlorine Residual

After the taste and odor extractions were completed, one of the bell jars was cleaned and repositioned over a fresh section of coating according to 4.5.11.4 of MIL-PRF-23236C. After the initial 24-hour exposure to 200 ppm water, a second 24-hour exposure to 10 ppm water was conducted. 200 ml of the 10 ppm water was retained as a control and compared to the exposed water for loss of chlorine into the coating. Table 14 reflects the chlorine test results for both exposed and control samples, corrected for the subsequent 1:10 dilution for the test kit range. The reported average of the exposed water indicates negligible residual chlorine, in compliance with the MIL spec requirement of 50% or greater of the original concentration.

### 3.3.16 Phenol Contamination

Phenol extraction tests were performed on the coating in compliance with MIL-PRF-23236C using a Soxhlet extractor. A portion of the coating was removed from the PE substrate and pulverized in a liquid-nitrogen cooled reciprocating mill then passed through a #50 mesh (0.0117 in) standard test sieve. Then, 300 mL of UHP water was refluxed through 1.0 g of the sieved coating for a 6-hour period. After completion, the water was tested using for phenol contamination using a CHEMetrics Phenols Test Kit, Model K-8012 having a range of 0-1 ppm using the C-1008 visual standard. This kit closely resembles APHA test method 5530D, but eliminates the handling of toxic phenol to mix standard comparison solutions.

### 3.3.17 Simulated Boiler Feed Water

All conditions were tested in accordance with MIL-PRF-23236C section 3.15/4.5.12 panels will be used to determine the acceptable performance for dedicated reserve feed water tanks. The designated 6 x 12 inch test panels were constantly immersed in 180°F de-ionized water in a bath for 500 hours. After the prescribed exposure, the panels were removed from the hot water. They were immediately examined for any change in appearance, chalking, color change or film failure, such as blistering and rusting by visual observation.

## 4.0 RESULTS

### 4.1 Candidate Coating Physical Properties

#### 4.1.1 Dry Time

The dry time test was run for a 4 hour duration cycle. The results of the Warren S-301 for dry time are provided in Table 3 and *pass* as satisfactory.

**Table 3- Dry Time Milestones**

Set To Touch	.5 HRS
Tack Free	1.0 HRS
Dry Hard	2.0 HRS
Dry Through	3.5 HRS

#### 4.1.2 Resistance to Sag

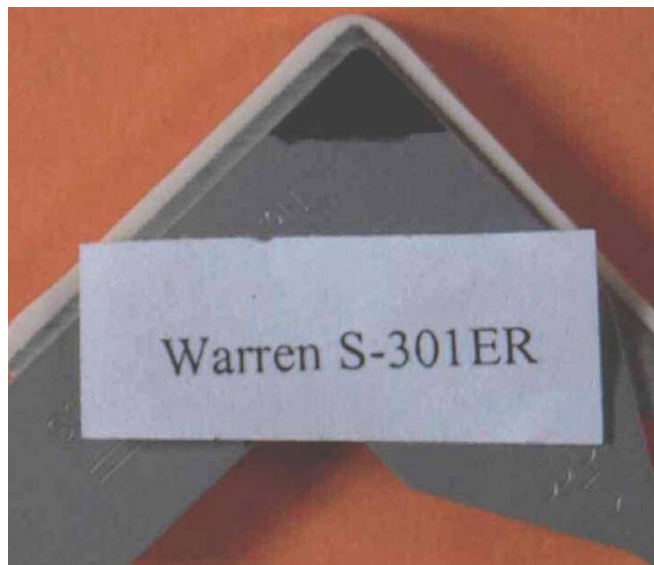
The sag resistance of the coating was determined in accordance with Method 4494.1 of FED-STD-141. The test was performed with a 4-24 mil ASM-4 Leneta drawdown blade. The resistance to sagging test was performed with a 0-24 mil ASM-4 Leneta drawdown blade and examination of the completed card after cure indicated no sag up to 24 mils. Coating thickness applied was approximately 7 mils. Results show acceptable performance with no sag and *pass* as satisfactory.

#### 4.1.3 Application Characteristics

This portion of the testing is a comparative and somewhat subjective commentary on the ease and efficiency of the manual application of the coating to the various panel conditions. The ease of application to the panels was notably good and film build was easy to achieve therefore giving it an acceptable result.

#### 4.1.4 Edge Retention

A minimum of three separate samples were prepared and measured. Three sections were further cut from each sample, with the coating thickness measure and edge retention calculated for the 9 sections. For acceptance the pass/fail criteria, the average of all readings shall not be less than 70%. Figure 14 shows a photograph of the Warren S-301 test sample. Results of the Edge Retention analysis of nine sections from three specimen angles indicated an average value of 73.72% edge retention. These results *pass* edge retention requirements.



**Figure 14 - Representative edge retention sample**

#### 4.1.5 VOC

This coating system was submitted for testing as a type VII coating, which means the VOC content of each layer of the coating system must be less than 1.25 lb/gal (150 g/L). The VOC content of the topcoat for this coating system this coating receives a *pass* rating as a type VII coating. Table 4 shows the results from the VOC testing. The standard VOC content was taken from averaging the results of dish 1-5, thus, the VOC content of the candidate coating was reported as .970 lb/gal or 116.2 g/L. In accordance with MIL-PRF 23236C 1.2.1 type VII is defined as containing less than 1.25 lb/gal or 150 g/L, this coating system receives a rating as a type VII coating.

**Table 4 - VOC Testing Results, ASTM D-2369-01 Modified**

Dish 1 - Solvent	Dish 2 - Solvent	Dish 3 - No Solvent	Dish 4 - No Solvent	Dish 5 Solvent	Average of
110C 1 hour	Air Cure 24 hours	110C 1 hour	Air Cure 24 hours	24 hours then 1 hr@110C	All Methods
1.871 lb/gal	0.069 lb/gal	0.410 lb/gal	0.102 lb/gal	0.069 lb/gal	0.970 lb/gal
224.2 g/L	8.3g/L	49.1 g/L	12.3 g/L	30.6 g/L	<b>116.2 g/L</b>

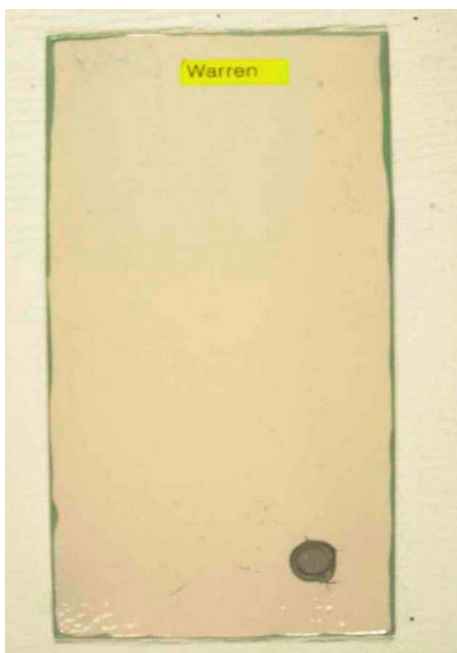
## 4.2 Environmental Exposure Testing Results

### 4.2.1 Condensation Testing

Panels 1-4 were prepared as shown in Table 5. The panels were then tested for water resistance using controlled condensation, ASTM D 4585 for 2000 hours shown in Figure 7. ASTM standards, D 610 for degree of rusting, D 714 for degree of blistering and D 1654 for scribe evaluation were used to define the performance of the test panels. The ratings are assigned on a scale from 0-10, with 10 indicating no damage and 0 meaning complete failure. Figure 15 shows a representative panel and the results are summarized in Table 5 reflecting a pass for all tested panels.

**Table 5 - Condensation Testing Results**

<u>SAMPLE</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<b>Size</b>	4x6	4x6	4x6	4x6
<b>Test</b>	Condensation	Condensation	Condensation	Condensation
<b>Pretreatment</b>	Scribe & Pull	None	Scribe & Pull	None
<b>POTS (psi)</b>	1652	N/A	1668	N/A
<b>Pre</b>	BY 100		BY 100	
<b>POTS (psi)</b>	1721	N/A	1897	N/A
	BY 70, YZ 30		BY 100	
<b>Percentage of adhesion</b>	100	100	100	100
<b>Mode of failure</b>	None	None	None	None
<b>Rust Grade (D610)</b>	9S	10	10	10
<b>Blister Rating (D714)</b>	10	10	10	10
<b>D1654 scribe min.</b>	0	N/A	0	N/A
<b>D1654 max</b>	0.5	N/A	0	N/A
<b>Average</b>	0.5	N/A	0	N/A
<b>Scribe Rating</b>	10	N/A	10	N/A



**Figure 15 - Representative panel after 2000 hour condensing humidity test**

Degree of rusting was defined in accordance with ASTM D 610. MIL-PRF 23236C does not specify this method as pass/fail criteria but, constitutes pinhole rusting as a failure. All panels were assigned a rating of 9 or better, indicating a *pass*. Degree of blistering was evaluated using the standards defined in ASTM D 714 and the Warren S-301 sample showed no blistering. Basic corrosion performance of the coating system was evaluated using ASTM D 1654, noted in Table 5 as scribe is not specified in MIL-PRF-23236C. The panels received a rating of 9-S or better. The odd numbered panels were also subject to ASTM D 4541, adhesion test and the results are summarized above in Table 5. According to MIL-PRF-23236C which states at the completion of the test the adhesion shall be at least 50% of the original giving the panels a *pass* according to this criteria.

#### **4.2.2 ASTM B-117**

Four panels were inserted into the salt fog test apparatus according to ASTM B-117 for 1000 hours. For all of the test panels, the protection rating of the coating system was determined according to the 0 to 10 scale described in ASTM D 1654 (scribe), ASTM D 610 (rust) and ASTM D 714 (blister). The results can be found in Table 6 and a representative panel can be seen in Figure 16. Degree of rusting was defined in accordance with ASTM D 610. MIL-PRF 23236C does not specify this method as pass/fail criteria but, constitutes pinhole rusting as a failure. All panels were assigned a rating of 9-G or better, indicating a pass. Degree of blistering was evaluated using the standards defined in ASTM D 714. The four panels showed no degree of blistering, indicating a pass per MIL-PRF 23236C. Basic corrosion performance of the coating system was evaluated using ASTM D 1654, noted in Table 6 as scribe. The remaining panels showed zero to 0.5 mm creepage from the scribe reflecting a rating of 10 and 9 respectively. ASTM D 1654 is not defined in MIL-PRF 23236C, therefore a pass/fail criteria is not defined.

**Table 6 - B117 Environmental Exposure Testing Results**

<b>SAMPLE</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>
<b>Size</b>	4x6	4x6	4x6	4x6
<b>Test</b>	Salt Fog	Salt Fog	Salt Fog	Salt Fog
<b>Pretreatment</b>	Scribe & Pull	None	Scribe & Pull	None
<b>POTS (psi) pre</b>	2152 BY 90 YZ 10	N/A	1794 BY 95 YZ 5	N/A
<b>POTS (psi)</b>	1985 BY100	N/A	1520 BY100	N/A
<b>Mode of failure</b>	None	None	None	None
<b>Rust Grade</b>	10	10	10	10
<b>Blister Rating</b>	10	10	10	10
<b>Min. Undercut (mm)</b>	0	N/A	0	N/A
<b>Max. Undercut (mm)</b>	0	N/A	0	N/A
<b>Average</b>	0	N/A	0	N/A
<b>Scribe Rating</b>	10	N/A	10	N/A



**Figure 16 - Representative panel after 1000 hour B-117 test**

#### *4.2.3 Alternate Immersion Exposure*

Upon completion of the test, the panels were carefully removed, rinsed with clean water to remove salt deposits and examined carefully. For all of the test panels, the protection rating of the coating system was determined according to the 0 to 10 scale described in ASTM D 1654 (scribe), ASTM D 610 (rust) and ASTM D 714 (blister). For those panels that were pretreated with a scribe and pull, the extent of blistering, rusting, undercutting, or other damage near the pretreated areas were carefully noted. This coating showed no pinhole rusting or blistering and received a *pass*. Test results are summarized in Table 7 and a test panel can be seen in Figure 17. ASTM 1654 is not defined in MIL-PRF 23236C, therefore a pass/fail criteria is not defined.

**Table 7 - Environmental Exposure Testing Results**

<b>SAMPLE</b>	<b>11</b>	<b>12</b>	<b>13</b>	<b>14</b>
<b>Size</b>	6x12	6x12	6x12	6x12
<b>Test</b>	AI	AI	AI	AI
<b>Pretreatment</b>	Scribe & Pull	None	Scribe & Pull	None
<b>POTS (psi) pre</b>	2264 BY 100	N/A	1983 BY 95 YZ 5	N/A
<b>POTS(psi) post</b>	2003 BY 100 90%	N/A	1920 BY 100 95%	N/A
<b>Mode of failure</b>	None	None	None	None
<b>Rust Grade</b>	10	10	9S	10
<b>Blister Rating</b>	10	10	10	10
<b>Min. Undercut (mm)</b>	0	N/A	0	N/A
<b>Max. Undercut (mm)</b>	0	N/A	0.5	N/A
<b>Average</b>	0	N/A	0	N/A
<b>Scribe Rating</b>	10	N/A	10	N/A



**Figure 17 - Representative panels from alternate immersion testing**

### **4.3 MIL-PRF- 23236C Qualification Testing Results**

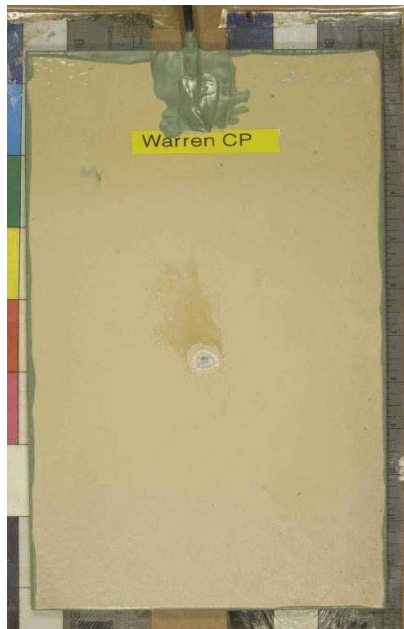
Table 19 below summarizes the results of the MIL-PRF-23236C Qualification Testing identified in Table 2. The ratings are assigned on a scale from 0-10 with 10 indicating no damage and 0 meaning complete failure (refer to the corresponding ASTM standards for further details, D 610 for degree of rusting, D 714 for degree of blistering and D 1654 for scribe evaluation).

#### **4.3.1 Cathodic Disbondment**

Cathodic Disbondment was evaluated per MIL-PRF-23236C section 3.5/4.5.16. Pass/fail criteria stated that there shall be no more than 4% damage at the holiday. The 90 day cathodic disbondment test results received a *pass* and results are reflected in Table 8 and shown in Figure 18.

**Table 8 - Cathodic Disbondment Test Results**

PANEL EVALUATIONS			
PANEL#	D610 RUST	D714 BLISTER	D1654 SCRIBE
CP1	10	10	10
CP2	10	10	10



**Figure 18 - Representative cathodic protection test panel**

#### *4.3.2 Cyclical Seawater/Air Immersion Resistance (Cycle A)*

All coating systems that do not meet one or more of the above criteria, as stated in section 3.3.2 of this document, receive an unacceptable (fail) result. There was no adhesion failure to recoated area, no blistering or rusting therefore this Warren S-301 test sample received a *pass*. The test results are listed in Table 9 and a test panel can be seen in Figure 19.

**Table 9 - Cyclical Seawater/Air Immersion Test Results**

<b>SAMPLE</b>	<b>15</b>	<b>16</b>
<b>Size</b>	6x12	6x12
<b>Test</b>	Seawater/Air	Seawater/Air
<b>Pretreatment</b>	None	None
<b>POTS pre</b>	2312 - BY 85, YZ 15	2144 - BY 100
<b>POTS Recoat</b>	2027- BY 100	1862- BY 80, YZ 20
<b>Mode of Failure</b>	None	None
<b>Rust Grade</b>	10	10
<b>Blister Rating</b>	10	10



**Figure 19 - Representative seawater/air test panel**

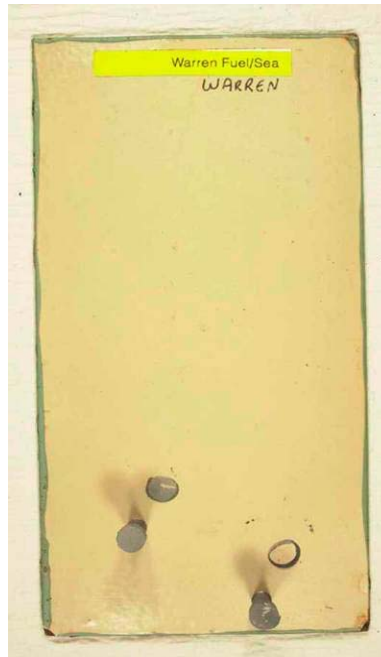
#### *4.3.3 Fuel and Seawater*

For this coating system, the cyclical seawater/fuel test was initiated on 25 July 2004. The test was completed and the final panel evaluations performed on 22 August 2005. The total coating system must meet the requirements stated in section 3.3.3 of this document to achieve an acceptable (pass) result. The test panels were exposed to 25 cycles and showed no pinhole rusting or blistering. Test results reflect a pass and are listed in Table 10. Figure 20 is a Warren S-301 seawater/fuel test panel.



**Table 10 - Cyclical Seawater/Fuel Test Results**

<u>SAMPLE</u>	<u>21</u>	<u>22</u>
Size	6x12	6x12
Test	Seawater/Fuel	Seawater/Fuel
Pretreatment	None	None
POTS (psi) pre	1861 BY 100	2017 BY 100
POTS recoat (psi)	1996 BY 90 YZ 10 100%	1843 BY 100 90%
Mode of failure	None	None
Rust Grade	10	10
Blister Rating	10	10



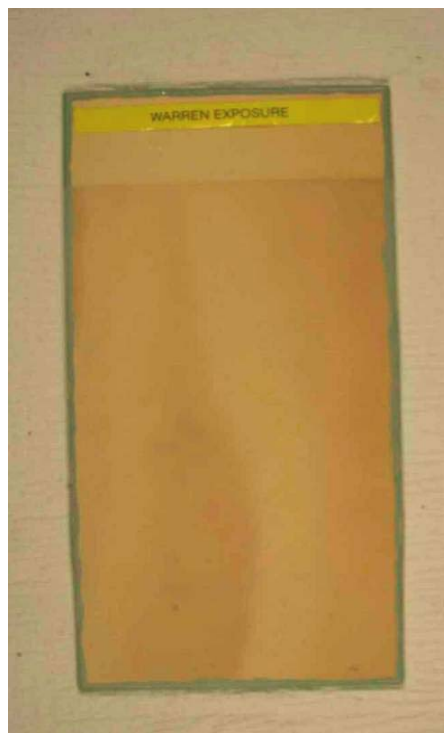
**Figure 20 -Representative seawater/fuel test panel**

#### *4.3.4 Atmospheric Exposure*

The test duration is indeterminate, and may continue indefinitely. Every three months for at least one year, the test panels were examined carefully and observations are recorded. In addition, the protection rating of the coating system is determined for each test panel according to the 0 to 10 scale described in ASTM D1654. For those panels that were pretreated with a scribe and pull, the extent of blistering, rusting, undercutting or other damage near the pretreated areas was carefully noted. If the test is continued beyond one year, the test panels are examined every 6 months and evaluated as above. The test results reflected in this report document a one year cycle. The panels did reflect some chalking and fading of color. Initial gloss was 84.1 and the final gloss was a 6.8 giving this sample a *failed* result. Test results are summarized in Table 11. Figure 21 reflects a Warren S-301 atmospheric exposure test panel after 1 year.

**Table 11- Exposure Test Results**

<b>SAMPLE</b>	<b>19</b>	<b>20</b>
<b>Size</b>	6x12	6x12
<b>Test</b>	Exposure	Exposure
<b>Pretreatment</b>	None	Scribe & Pull
<b>POTS (psi) pre</b>	N/A	2236 BY 100
<b>POTS (psi)</b>	N/A	1858 BY 80 YZ 20 80%
<b>Mode of Failure</b>	None	None
<b>Rust Grade</b>	10	10
<b>Min. Undercut (mm)</b>	N/A	0
<b>Max. Undercut (mm)</b>	N/A	0
<b>Scribe Rating</b>	10	10
<b>Blister Rating</b>	10	10
<b>Gloss</b>	84.1/6.8	74.9/7.6
<b>Color</b>	83.3/9.9	82.5/11.4



**Figure 21- Representative atmospheric exposure test panel**

#### *4.3.5 Aviation Fuel Compatibility*

Two sets of fuel compatibility tests are conducted with one set using JP-5 fuel and one set using AVGAS. The preparation of the two types of fuels for testing is described in section 3.3.5 of this report. The aviation fuel compatibility test procedures apply to both types of fuel with the exception of the bromine test which is only conducted with AVGAS. Any differences in test procedures for the two types of fuel are noted in the text. Test results are listed in Table 12 and reflect a *pass*.

**Table 12 - JP-5/AVGAS Fuel Test Results**

<b><u>SAMPLE</u></b>	<b><u>A JP-5</u> (724674)</b>	<b><u>B JP-5</u> (724675) CONTROL</b>	<b><u>C JP-5</u> (724676)</b>	<b><u>D AVGAS</u> (725795)</b>	<b><u>E AVGAS</u> (725796) CONTROL</b>	<b><u>F AVGAS</u> (725797)</b>
<b>Saybolt Color</b>	16	16	16	-13	-13	-14
<b>Copper Corrosion</b>	1A 3 Hours	1A 3 Hours	1A 3 Hours	1A 3 Hours	1A 3 Hours	1A 3 Hours
<b>Particulate Contaminant</b>	.24 mg/L	.30 mg/L	.12 mg/L	.20 mg/L	.40 mg/L	.34 mg/L
<b>Volume Filtered</b>	500mL	500mL	500mL	500mL	500mL	500mL
<b>Existent Gum</b>	<1 mg/100mL	1 mg/100mL	<1 mg/100mL	1 mg/100mL	2 mg/100mL	2mg/100mL
<b>Total Halogens</b>	N/A	N/A	N/A	<191 ppm	<187 ppm	<174 ppm
<b>Total Bromides</b>	N/A	N/A	N/A	<21 ppm	<21 ppm	<19 ppm

#### 4.3.6 Fuel Color Test

The fuel color test is described in section 3.3.6 of this report. There was no change in color of the Warren S-301 sample and it received a *pass* result. Test results noted in Table 12.

#### 4.3.7 Corrosion Test

The corrosion test is described in section 3.3.7 of this report. The Warren S-301 sample received a *pass* result due to no increase in corrosion. Test results in Table 12.

#### 4.3.8 Existent Gum Content

An unacceptable (fail) result is indicated if the difference between the unwashed existent gum in the test sample and the existent gum in the control sample is greater than 4 mg/100 mL or the difference between the washed existent gum in the test sample and the washed existent gum in the control sample is greater than 2 mg/100 mL. The Warren S-301 sample received a *pass* result and test results are noted in Table 12.

#### 4.3.9 Solids Contamination Test

The solids contamination test is described in section 3.3.9 of this report. The Warren S-301 sample received a *pass* result due to there being <.30mg/L of solids contamination. Test results noted in Table 12.

#### 4.3.10 Bromine Test

The concentration of ethylene dibromide (bromine) is determined for the AVGAS sample only. This test procedure is described in section 3.3.10 of this report. The Warren S-301 sample received a *pass* result due to their being <21 ppm of total bromides. Test results noted in Table 12.

#### 4.3.11 Collection, Holding and Transfer

The Warren S-301 samples showed no blistering or surface imperfections giving it a *pass* result. Test results are reported in Table 13. Figure 22 is a Warren S-301 CHT test panel.

**Table 13 - CHT Test Results**

<b>SAMPLE</b>	<b>25</b>	<b>26</b>
<b>Size</b>	6x12	6x12
<b>Test</b>	CHT	CHT
<b>Pretreatment</b>	None	None
<b>POTS (psi) pre</b>	N/A	2236 BY 100
<b>POTS (psi)</b>	N/A	1868 BY 70 YZ 30 80%
<b>Mode of failure</b>	None	None
<b>Rust Grade</b>	10	10
<b>Blister Rating</b>	10	10
<b>Gloss</b>	83.1/80.6	75.7/62.4
<b>Color</b>	84.1/82.3	83.4/81.49



**Figure 22 - Representative CHT test panel**

#### *4.3.12 Potable and Fresh Water Tests*

The test procedures for the potable water tests are based on the MIL-PRF-23236C and are described in section 3.3.12 of this report. Minor modifications have been made to the specified procedures to facilitate testing. The values for FTN (Flavor Threshold Number) and TON (Threshold Odor Number) were not to be greater than 2.0, a unit-less value based on the methodology of APHA 2150B, TON and 2160B, FTN.

**Table 14 - Potable Water Odor Test Results**

	ODOR											
	<u>OP1</u>	<u>OP2</u>	<u>OP3</u>	<u>OP4</u>	<u>OP5</u>	<u>OP6</u>	<u>OP7</u>	<u>OP8</u>	<u>OP9</u>	<u>OP10</u>	<u>OP11</u>	
Panelist	0/200	17/200	25/200	35/200	0/200	50/200	70/200	0/200	100/200	140/200	200/200	TON
1	0	0	0	1	1	1	0	0	0	0	1	1.0
2	0	0	1	0	1	0	0	0	0	0	1	1.0
3	0	0	1	1	0	1	0	1	0	0	1	1.0
4	0	0	0	1	0	1	1	1	1	0	1	1.0
5	0	0	0	0	0	1	0	0	0	0	1	1.0
6	0	0	0	1	0	1	1	0	1	1	1	5.7
											AVG TON=	1.8

**Table 15 - Potable Water Taste Results**

	TASTE											
	<u>TP1</u>	<u>TP2</u>	<u>TP3</u>	<u>TP4</u>	<u>TP5</u>	<u>TP6</u>	<u>TP7</u>	<u>TP8</u>	<u>TP9</u>	<u>TP10</u>	<u>TP11</u>	
Panelist	0/200	17/200	25/200	35/200	0/200	50/200	70/200	0/200	100/200	140/200	200/200	FTN
1	0	1	0	1	0	1	1	0	1	1	1	5.7
2	1	1	1	1	1	1	1	0	1	1	1	4.0
3	0	0	1	1	0	1	1	0	1	1	1	8.0
4	0	0	1	1	0	1	0	0	1	1	1	2.0
5	1	1	1	1	0	1	1	1	1	1	1	2.0
6	0	0	1	1	0	1	1	0	1	1	1	8.0
											AVG FTN=	5.0

#### 4.3.13 Taste and Odor (Flavor) in Water

Odor results are listed in 14 and reflect a *passing* result for the Warren S-301 sample because the test threshold is <2. Taste results are listed in Table 15 and reflect a *fail* result due to the test threshold requirement being <2. The fail result could be due to an incorrect volume to surface area ratio used with this test sample. Some individuals may consider this test subjective and not necessarily a failed result. As of this writing, a new taste test has been requested.

#### 4.3.14 Color Change Test

The color change test procedure is described in section 3.3.14 of this report. The Warren S-301 sample received a *pass* result which is noted in Table 16.

**Table 16 - Color Test Results**

<u>COLOR CHANGE</u>		
Unexposed:	<1	
Exposed:	<1	
	0 Units Change	

#### 4.3.15 Chlorine Residual Test

The chlorine residual test is described in section 3.3.15 of this report. The reported average of the exposed water indicates negligible residual chlorine, in compliance with the MIL spec requirement of 50% or greater of the original concentration. The Warren S-301 sample received a *pass* result which is listed in **Error! Reference source not found.**

#### 4.3.16 Phenol Contamination Test

The phenol contamination test is described in section 3.3.16 of this report. Phenol extraction tests were performed on the coating in compliance with MIL-PRF-23236C using a Soxhlet extractor. An average phenol contamination value of 0.10 ppm was obtained, well within the MIL spec requirement of 1.0 ppm or lower. The Warren S-301 sample received a passing result which is listed in Table 17.

**Table 17 - Phenol/Chlorine Residual Test Results**

	<u>CONTROL</u>			<u>EXPOSED</u>			<u>RES. AVG.</u>
<b>Free Cl</b>	11.0	10.0	10.0	10.5	10.0	10.5	<b>100.0%</b>
<b>Total Cl</b>	11.0	11.0	11.0	11.0	11.0	10.5	<b>98.5%</b>
<b>Phenol</b>	0.0	0.0	0.0	0.0	0.0	0.0	<b>0.0</b>

#### 4.3.17 Simulated Boiler Feed Water Test

All conditions were tested in accordance with MIL-PRF-23236C section 3.15/4.5.12 and are described in section 3.3.17 of this report. The Warren S-301 samples all *passed* with no visible damage. Test results are listed in Table 18. Figure 23 is a Warren S-301 boiler feed test panel.

**Table 18 - Simulated Boiler Feed Test Results**

<u>SAMPLE</u>	<u>17</u>	<u>18</u>
<b>Size</b>	6x12	6x12
<b>Test</b>	Boiler Feed	Boiler Feed
<b>Pretreatment</b>	None	None
<b>POTS (psi) pre</b>	N/A	1874 BY 100
<b>POTS recoat (psi)</b>	N/A	1843 BY 100 99%
<b>Mode of failure</b>	None	None
<b>Rust Grade</b>	10	10
<b>Blister Rating</b>	10	10



**Figure 23 - Warren S-301 simulate boiler feed test panel**

## 5.0 CONCLUSIONS

The Warren Environmental S-301 coating demonstrated acceptable performance in test protocols and conditions relating to Type VII, Classes 5, 7, 9, 11 & 18, Grade C of MIL-PRF-23236C, with the exception of the potable water taste test and exposure testing. The taste test is subjective and at the time of this writing a request has been made for a retesting of the Warren S-301. It is the opinion of the authors that the fail rating received by the Warren Environmental S-301 is not critical to the performance of the coating system. The testing performed here does not conclude all requirements the vendor must achieve for a qualified products list. A thorough investigation of the requirements for these classes must be investigated by the manufacture to determine the information they are responsible for.

The Warren Environmental S-301 was applied to a demonstration ballast tank on the USS OAK HILL in July of 2005 (LSD-51) in seawater ballast tank 3-121-1W. The 1 year inspection was completed in September 06 but a final report has not been published as of the writing of this report.

Table 19 below details the general test results provided by NRL.

**Table 19 - Summary of Testing Results**

Test Requirement	MIL-PRF-23236C Section/Spec	Pass/Fail Evaluation Criteria	Test Results	Pass/Fail Evaluation
<b>Description of Coating</b>	<b>3.1.1</b>	<b>Type VII Grade C</b>		
VOC Content	3.2.2.2	Type: VII, 150g/L – 1.25 lb/gal or less	116.2.g/L	PASS
<b>Gloss Topcoat</b>	3.3	≥ 30 @ 60 degrees	81.6	PASS
<b>Potable &amp; Freshwater Class 9 (500 gallon)</b>	3.4			INCOMPLETE
Color in Water	3.4.2/4.5.11.1	Color units <10	<1	PASS
Taste in Water	3.4.3/4.5.11.2	Threshold ≤2	5.0	FAIL Requesting a retest
Odor in Water	3.4.4/4.5.11.3	Threshold ≤2	1.8	PASS

Test Requirement	MIL-PRF-23236C Section/Spec	Pass/Fail Evaluation Criteria	Test Results	Pass/Fail Evaluation
Chlorine Residual	3.4.5/4.5.11.4	Not less than 50% decrease	<2%	PASS
Phenol Residual	3.4.6/4.5.11.5	< 1ppm	0	PASS
Immersion Resistance	3.4.7 / 4.5.2.3			ACCEPTABLE
Adhesion D4541	3.4.7 / 4.5.2.3	≥ 50% original	1664psi,BY100 1279psi,YZ85, 75%,YB15	PASS
Blisters D714	3.4.7 / 4.5.2.3	Blisters <4, few	No blistering	PASS
Edge Rusting	3.4.7 / 4.5.2.3	< 0.1% total edge	No edge rusting	PASS
Pin Hole Rusting	3.4.7 / 4.5.2.3	No pinhole rusting	No pinhole rusting	PASS
<b>Cathodic Disbondment All classes except 16, 17, &amp; 19</b>	3.5/4.5.16	Not >4% undercutting	No undercutting	PASS
<b>Dry Time or Cure Time All Classes</b>	3.6	<23 hours dry < 7 days cure	DRY THRU 3.5 HRS	PASS
<b>Application Characteristics</b>	3.8	Acceptable Application	Applied in a single coat with 2-3 passes.	ACCEPTABLE
Edge Retention Type VII only, all classes & grades	3.8.1	70% minimum on 1.0 mm radius	73%	PASS
Sag Resistance D4400	3.8.2	Sag <2X max WFT	0 Sag	PASS
<b>Immersion Resistance</b>	3.9			ACCEPTABLE
<b>Class 5 - Fuel &amp; Seawater</b>	3.9.1 / 4.5.2.1			PASS
Adhesion D4541	3.9.1 / 4.5.2.1	≥ 50% of original	1996psi, BY 100/1871psi BY100 95%	PASS
Blisters D714		Blisters <4, few	No blistering	PASS
Pin Hole Rusting		No Pin holes	No pinhole rusting	PASS
<b>Class 7 - Seawater only</b>	3.9.3 Cycle A			ACCEPTABLE
Adhesion D4541	3.9.3/4.5.2.2.1	Overcoat ≥ 50% original	1916 psi OR 84% of original	PASS
Blisters D714	3.9.3/4.5.2.2.1	Blisters <4, few	No blistering (10)	PASS
Pin hole Rusting	3.9.3/4.5.2.2.1	No Pin Holes	No surface or edge rusting	PASS
<b>Condition in Container – All Classes except VIII &amp; VIIa</b>	3.10	See Specification	Coating easily dispersed and used in brushing, spraying and rolling.	PASS
<b>JP-5 Aviation Fuel Compatibility, Class 5 only</b>	3.13		ALL RESULTS WITHIN LIMITS	INCOMPLETE
Fuel Color	3.13.1/4.5.4.2	Saybolt color change ≤ 2	No color change	PASS
Corrosion	3.13.2/4.5.4.3	No increase	No increase	PASS
Existent Gum	3.13.3/4.5.4.4	Increase ≤4 mg/100ml	≤1mg/100ml	PASS
Solids Contamination	3.13.4/4.5.4.5	Increase ≤2 mg/l	≤.30	PASS
Bromine	3.13.5/4.5.4.6	<10%		NOT TESTED
<b>Aviation Gasoline (Mogas) Compatibility, Class 5 only</b>	3.13		All results within limits	ACCEPTABLE
Fuel Color	3.13.1/4.5.4.2	Saybolt color change ≤ 2	≤-1 color change	PASS
Corrosion	3.13.2/4.5.4.3	No increase	No increase	PASS
Existent Gum	3.13.3/4.5.4.4	Increase ≤4 mg/100ml	≤.2	PASS
Solids Contamination	3.13.4/4.5.4.5	Increase ≤2 mg/l	0.02 increase	PASS
Bromine	3.13.5/4.5.4.6	<10%	No increase	PASS
<b>Resistance to Boiler feed water, 500 hrs. @ 180F, Class 11</b>	3.15			ACCEPTABLE
Adhesion D4541	3.15/4.5.12	≥ 50% of original	1779psi, YZ100 1641psi, YZ100 R 95%	PASS
Blistering D714	3.15/4.5.12	< #4 few	No blistering 10	PASS
Rusting	3.15/4.5.12	No edge rusting Type VII	No edge or pinhole rusting	PASS
<b>CHT Testing – Class 13</b>	3.16			ACCEPTABLE
Adhesion D4541	3.16/4.5.13	≥ 50% of original	2236psi, BY100 1868psi, BY70-YZ 30 90%	PASS
Blistering D714	3.16/4.5.13	No blisters in excess of #8 few	No blistering 10	PASS
Pin hole Rusting	3.16/4.5.13		No edge or pinhole rusting	PASS



Test Requirement	MIL-PRF-23236C Section/Spec	Pass/Fail Evaluation Criteria	Test Results	Pass/Fail Evaluation
<b>Condensing Humidity – 2000 hours @ 100F (38C) ASTM D4585</b>	3.17			ACCEPTABLE
Adhesion D4541	3.17/4.5.14	≥ 50% of original	1668psi BY100 1897psi BY100 100%	PASS
Blistering D714	3.17/4.5.14	Blisters <4, few	No blistering 10	PASS
Pin Hole Rusting	3.17/4.5.14	No pin holes	No edge or pinhole rusting 10	PASS
<b>Single Coat System Class 18</b>	3.22	Conform when tested to 4.5.20 & 3.2.1		PASS
Mix Ratio must be	3.2.1	1:1, 2:1, 3:1, 4:1	2:1	PASS
Single coat requirement	4.5.20	Test to class requested by manufacturer on QPL application but test using a single coat application in all the applicable tests.		
Class 5 Fuel, SW	3.22	4.5.20	Tested for 1 year.	INCOMPLETE
Class 7 Ded. SW	3.22	4.5.20	Tested for 1 year.	ACCEPTABLE
Class 9 PW	3.22	4.5.20	Tested for 1 year.	INCOMPLETE
Class 13 CHT	3.22	4.5.20	Tested for 1 year.	ACCEPTABLE
<b>Additional Non-MIL-PRF-23236C Testing Performed</b>				
<b>Accelerated Corrosion</b>	ASTM B117	0 – 10 rating	No damage 10	ACCEPTABLE
Adhesion Test	ASTM D 4541	Overcoat ≥ 50% original	2152psi,BY90 YZ10 1985psi,BY100 95%	PASS
<b>Alternate Immersion</b>	NRL Test #2 year exposure	0 – 10 rating ASTM D1654, D610, D714	.5mm creepage @scribe=10 No blistering	ACCEPTABLE
Adhesion Test	ASTM D 4541	Overcoat ≥ 50% original	2264psi,BY100 2003psi,BY100 90%	PASS
<b>Atmospheric Exposure</b>	NRL Test #1 year exposure	Retain 50% of original gloss/D1654 scribe evaluation	Gloss post exposure:84.1 1 year:6.8 Color post exposure 1 year: No blistering or creepage 10.	Failed color & gloss fading. Passed scribe evaluation.
Adhesion Test	ASTM D 4541	Overcoat ≥ 50% original	2236psi,BY100 1858psi,BY80 YZ20 90%	PASS
<b>NRL Touch up &amp; Repair</b>	NRL TEST #3	Adhesion equal to that of a new coating	1741psi, BY100 1653psi, CB10, BY90	PASS

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IN REPLY REFER TO

3900  
Ser 6130/6053  
25 JAN 2007

From: Commanding Officer, Naval Research Laboratory  
To: Commander, Naval Sea Systems Command (05M1, Brinkerhoff)

Subj: USS OAK HILL LSD-51: 15-MONTH FOLLOW-UP REPORT: ONR/NRL/NAVSEA  
RAPID CURE UHS EPOXY IN SALT WATER BALLAST SERVICE TEST REPORT:  
WARREN ENVIRONMENTAL S-301 RAPID CURE EPOXY APPLIED IN  
SEAWATER BALLAST TANK 3-121-1-W JULY 2005

Encl: (1) Two copies of subject report

1. Enclosure (1) entitled "USS OAK HILL LSD-51: 15-Month Follow-Up Report: ONR/NRL/NAVSEA Rapid Cure UHS Epoxy in Salt Water Ballast Service Test Report: Warren Environmental S-301 Rapid Cure Epoxy Applied in Seawater Ballast Tank 3-121-1-W July 2005" provides the summary results of Rapid Cure coatings application and demonstration aboard naval combatants.
2. This work was funded by the Office of Naval Research (ONR) with technical oversight by the Naval Sea Systems Command, Code 05M1 to study the feasibility and applicability of utilizing Rapid Cure coatings in tanks & voids.
3. The NRL points of contact are Keith Lucas, Code 6130, 202-767-0833, e-mail: keith.lucas@nrl.navy.mil; and Arthur Webb, Code 6138, 202-404-2888, e-mail: art.webb@nrl.navy.mil.

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January 17, 2007



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**Center for Corrosion  
Science and Engineering**

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## **USS OAK HILL LSD-51**

### **15-Month Follow-up Report**

#### **ONR/NRL/NAVSEA Rapid Cure UHS Epoxy in Salt Water Ballast Service Test Report: Warren Environmental S-301 Rapid Cure Epoxy Applied in Salt Water Ballast Tanks 3-121-1-W July 2005**

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## INTRODUCTION

This report covers the 15-month follow-up inspection and documentation of the application of one Rapid Cure coating system in one tank as part of the ONR Rapid Cure Coating System demonstration program. The initiative involves the implementation of proven commercial coating products which are new to the U.S. Navy's Tank and Void preservation process. Table 1 summarizes the tank where the demonstration was successfully performed and the coating system that was applied. The application was accomplished during a shipyard availability. The follow-up inspection was accomplished at the home port of USS Oak Hill at Little Creek NAB in Norfolk, VA.

**Table 1**

<b>Coating</b>	<b>Tanks</b>
Warren Environmental S-301	SWB Tank Number 3-121-1-W

The tank is referred to as a Third Deck wing wall ballast tank. The tank's inboard bulkhead is the well deck wing wall and the outboard bulkhead is the hull. The tank is located below the Ship's laundry. There are ballast tanks located below the tank (5-121-1-W and 5-125-1-W). The tank is filled by the Ship's Salt Water Fire Main System and empties by way of a ballast valve.

The application was accomplished July 2005 and this follow-up inspection was accomplished late October 2006. The original application report follows this follow-up inspection report.

## 15-MONTH FOLLOW-UP SURVEY RESULTS

The Warren Environmental S-301 coating system in this tank, SWB 3-121-1-W, was in excellent condition, with far less than 1/10 of 1% corrosion noted. The sacrificial zincs that were installed following the application were like new with very little deterioration, reinforcing the observed condition of the coating systems. Most corrosion observed was running rust originating from the interior surface of ferrous pipe brackets that were not disassembled for preservation. There were a few small areas of mechanical damage noted. Two deep pits were bleeding rust and it appeared that the coating did not flow into the pitted steel. The coating presents a slightly chalked out appearance which is typical of many epoxy coatings and not detrimental. The deck of the tank appeared more slippery than typically encounter, but it appears that the tank remains continually damp, which probably explains the sensation. Overall, the coating was rated excellent.



**USS Oak Hill LSD-51 SWB Tk 3-121-1-W with Warren Environmental S-301 Rapid Cure coating system applied 15-months earlier. Looking Aft**



**USS Oak Hill LSD-51 SWB Tk 3-121-1-W with Warren Environmental S-301 Rapid Cure coating system applied 15-months earlier. Looking Forward**





**USS Oak Hill LSD-51 SWB Tk 3-121-1-W with Warren Environmental S-301 Rapid Cure coating system applied 15-months earlier. Looking Aft at the Overhead. All corrosion seen is rust stain originating from interior of pipe brackets.**



**Note the two spots of corrosion. These are deep pits where the coating did not flow into and seal. Note the overall excellent film build on all edges and outside corners.**



**USS Oak Hill LSD-51 SWB Tk 3-121-1-W with Warren Environmental S-301 Rapid Cure coating system applied 15-months earlier. Looking Forward at the Overhead. All corrosion seen is rust stain originating from interior of pipe brackets.**



**Note the mildew that has formed on various surfaces. This tank appears to stay continually damp. All corrosion noted originates from pipe bracket interiors.**





**Overhead at Forward end. Corrosion seen on the Forward Bulkhead in way of the Tank Level Indicators (TLI's) originates with uncoated mating surface of the mounting brackets.**



**Note the black mildew on various surfaces**

## **ORIGINAL INSTALLATION REPORT**



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IN REPLY REFER TO

3900  
Ser 6130/6023  
05 Sep 2006

From: Commanding Officer, Naval Research Laboratory  
To: Commander, Naval Sea Systems Command (05M1, Brinkerhoff)

Subj: USS OAK HILL (LSD-51) TEST APPLICATION OF WARREN  
ENVIRONMENTAL S-301 COATING RAPID CURE UHS EDGE  
RETENTIVE EPOXY IN SEAWATER BALLAST TANK 3-121-1-W

Encl: (1) Two copies of subject report

1. Enclosure (1) entitled "USS OAK HILL (LSD-51) test application of Warren Environmental S-301 Coating Rapid Cure UHS Edge Retentive Epoxy in Seawater Ballast Tank 3-121-1-W" provides the summary results of Rapid Cure coatings application and demonstration aboard naval combatants.
2. This work was funded by the Office of Naval Research (ONR) with technical oversight by the Naval Sea Systems Command, Code 05M1 to study the feasibility and applicability of utilizing Rapid Cure coatings in tanks & voids.
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**USS OAK HILL (LSD-51) TEST APPLICTION OF WARREN  
ENVIORNMENTAL S-301 COATING RAPID CURE UHS EDGE  
RETENTIVE EPOXY IN SEAWATER BALLAST TANK 3-121-1-W**

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# **Tank Demonstration Coating Applications USS OAK HILL (LSD 51)**

## **INTRODUCTION**

This report covers the application of Warren Environmental S-301 Rapid Cure coating system in one tank as part of the ONR Rapid Cure Coating System demonstration program. The initiative involves the implementation of proven commercial coating products which are new to the U.S. Navy's Tank and Void preservation process. Table 1 summarizes the tanks where the demonstrations were successfully performed and the coating system that was applied. The application was completed during a Docked Phased Maintenance Availability (DPMA) at BAE's commercial repair facility in Norfolk, Virginia by Mid Atlantic Coatings.

**Table 1**  
**Rapid Cure Tank Coatings Installation on**  
**USS OAK HILL (LSD-51)**

<b>Coating</b>	<b>Tanks</b>
Warren Environmental S-301	3-121-1-W

## **BACKGROUND**

### *Coatings and Application Technology Trends*

Recently the trend for industrial and marine corrosion control coatings has transitioned from coatings with a high solvent content to coatings with lower solvent content (High Solids) and solvent free (100% solids) systems. As part of this process, the coatings industry has actively pursued the development and fielding of high solids and solvent free coatings for a wide variety of services. The key features of these coatings have been the reduction and or elimination of solvents which contain volatile organic compounds (VOC). Other advances include institution of Rapid Cure technology, cold temperature (<50F) and high temperature (>95F) application and curing capabilities, and the extension of service life. In support of the application of these products, technological advances were necessary in plural component spray equipment suitable for field use. The development of plural component equipment includes: plural component pumps, dual feed spray guns, heated spray lines, mixing blocks, and product heaters. These advances have become widely utilized in many facets of the heavy industrial coatings market. Because the U.S. Navy has been actively involved in the identification of improved materials and processes aimed at reducing maintenance and operating costs, the utilization of both high solids/solvent free coatings and plural component application equipment has been actively pursued.



### *ONR Rapid Cure Coating Demonstration Initiatives*

Since 2002 the Office of Naval Research has been installing Rapid Cure coatings in shipboard tanks as a part of the ONR Rapid Cure Program. These coatings are designed to meet increasingly stringent preservation requirements, including shortened repair availabilities, extended service life due to lengthened periods between docking cycles, increased deployment requirements, and ever increasing environmental regulations. As part of this effort, the Naval Research Laboratory (NRL) and Naval Surface Warfare Center (NSWC) have coordinated the field demonstration of an epoxy based Rapid Cure coating system for one seawater ballast tanks on board the USS OAK HILL.

### *Coatings Evaluation and Qualification*

The identification and performance qualification testing of commercially available Rapid Cure coating systems is conducted for NAVSEA 05M by NRL as a part of a comprehensive cooperative agreement. As such, NRL is responsible for screening candidate coatings, long term laboratory testing, and shipboard demonstrations.

All candidate Tank and Void coating systems are subjected to laboratory qualification testing in accordance with the most recent revision of MIL-PRF-23236, *Performance Specification, Coating Systems for Ship Structures*. During the qualification laboratory tests, which last approximately one year, the performance of each candidate coating is closely monitored. The performance of the coating based on the test results is reported to NAVSEA. In addition to laboratory performance evaluations, a shipboard demonstration is necessary to fully assess the coating's performance. During this demonstration, the feasibility of utilizing these coatings in an industrial environment on NAVY platforms is evaluated. Lessons learned from applications are evaluated and recommendations for future applications and processes are considered. The in-service demonstrations consist of a closely monitored application during regularly scheduled maintenance availabilities. During this application commercial contractors, blasters and painters, are utilized and the coating is installed using testing and evaluation PPI's. Comprehensive application review and documentation is completed for each demonstration. Coatings that pass the laboratory qualification process and exhibit acceptable shipboard in-service performance are submitted for qualification under the requirements of the Qualified Products List (QPL) under MIL-PRF-23236.

## **Rapid Cure Tank Coatings Demonstrations on USS OAK HILL**

### *Tank Selection*

One seawater ballast tank was selected for this demonstration application. The tank and coating system applied was selected based on experience with the coating material, tank complexity, the proximity of the tanks to other work, the length of availability, and product availability, which all contribute to the risk associated with the application. The application of Warren Environmental S-301 had not been previously attempted on a Navy ship. Additionally the applicator did not have previous experience with the Warren Environmental S-301 in tanks. For this reason, it was decided to install this coating in a tank with low complexity of surface geometries to mitigate the risk associated with the coating application.

### *Previous Coating System*

The previous tank coatings were reported to be installed in 1996 using MIL-P-24441 coating systems. Inspections during previous availabilities in 2002 indicated the coating was in condition 2 to condition 3 and required replacing. The performance life of the MIL-P-24441 system was less than 10 years.

All of the coatings employed in shipboard demonstrations are commercially available coatings which have been previously employed in other industrial applications. Demonstration coating applications are completed using the guidance for coating application in the Preservation Process Instruction (PPI) Core, PPI NBR: 63101-000 (Rev 16). Each coating applied under the demonstration program has unique application requirements and recommended parameters; therefore a Draft PPI tailored to the specific requirements of each coating system demonstrated under this program has been developed.

### *Application Demonstration*

The Rapid Cure seawater ballast tank coating installation demonstration on USS OAK HILL (LSD 51) took place during scheduled maintenance and repair availability, also known as a Docked Phased Maintenance Availability (DPMA) at BAE's Norfolk, Virginia ship repair yard. The DPMA began in July 2005 and was completed in September, 2005. The purpose of the DPMA was to conduct regularly scheduled ship maintenance, including the blasting and painting of several seawater ballast tanks. Tank work was conducted under the supervision of MARMC. Surface preparation and coating application was performed by Mid Atlantic Coatings Company under subcontract to and direct supervision of BAE. This was the first time for Mid Atlantic Coatings to apply the Warren Environmental S-301 Rapid Cure coating system onboard a US Navy Ship. Mid Atlantic Coatings provided assurances that they had significant experience with the use of

plural component equipment in the application of high solids coatings. Mid Atlantic Coatings also conducted a brief test application of the coating at their industrial facility prior to conducting application at the waterfront onboard the ship. The work item covering the preservation of the tank for this availability included one seawater ballast tank that was selected for this demonstration application. The tanks selected and the coating applied is listed in Table 1. The Mid-Atlantic Regional Maintenance Command (MARMC) provided government oversight for this application. NRL and NSWC provided technical support to the applicators and MARMC for the coating applications.

### *Surface Preparation*

Surface preparation for the tank was funded as part of the repair availability and was accomplished under the supervision of MARMC. The work was performed by Mid Atlantic Coatings under subcontract to BAE. Requirements for the surface preparation work item were in accordance with the appropriate sea water ballast tank and potable water tank preservation requirements of the CORE PPI (rev 16) and NAVSEA Standard Item 009-32. These requirements included:

- Initial cleaning and pressure wash to remove loose surface contaminants
- Maintaining humidity below 50% and keeping the temperature 5 degrees above the dew point to minimize condensation.
- Abrasive blasting in accordance with Steel Structures Painting Council (SSPC) SP-10, Near White Metal and the establishment of a surface profile of 2-4 mils.
- Ensure that conductivity levels were below 30 microsiemens/cm<sup>2</sup>.

Mid Atlantic conducted abrasive blasting in the tank to remove coating and loose rust. Upon completion of the abrasive blasting the tanks were inspected for corrosion damage needing repair. The tanks were then washed to remove chloride contamination and abrasive blasted again to achieve a level of cleanliness as specified in SSPC SP-10, Near White Metal Blast. The tanks were inspected to ensure compliance with surface preparation checkpoint requirements by MARMC Shipbuilding Specialist. A representative from NRL attended the surface preparation check point in the tank to provide technical input for any questions regarding the coating and its interaction with the surface preparation.

### *Product Applied*

All of the coatings employed in shipboard demonstrations are commercially available coatings which have been previously employed in other industrial applications. Demonstration coating applications are completed using the guidance for coating application in the Preservation Process Instruction (PPI) Core, PPI NBR: 63101-000 (Rev 16). Each coating applied under the demonstration program has unique application requirements and recommended parameters; therefore a Draft PPI tailored to the specific requirements of each coating system demonstrated under this program has been developed.

The Warren Environmental S-301 coating system is a two component, epoxy based high solid, Rapid Cure coating system. This coating must be applied with plural component spray equipment as specified by the manufacturer. The paint hopper is designed to maintain the correct temperature ratio for each component of the paint. The PPI containing the specific requirements for the application of this coating is **PPI NBR: 63101-001J Test and Evaluate Warren Environ (REV 00)**. The tank coating application took place over the course of four days and consisted of three spray applied coats of S-301 (two coats of blue on one white) and three brush and roll touch up coats. Touchup consisted of a brush and roll application following each spray application using the S-301 material applied to holidays and areas inaccessible to spray or that did not meet minimum coating thickness requirements. Surface preparation of the areas around the access inserts was completed using a portable blast pot and abrasive blasting. Warren Environmental S-301 was used to paint these areas after successful completion of the surface preparation. The product batch numbers for the material applied in this application are listed in table 2 below.

**Table 2**  
**Warren Environmental S-301 Coating System**

Product Name	Component	Batch Number	Color
301 Prime Coat	A (resin)	50706-2	Blue
301 Top Coat	A (resin)	50706-1	White
301	B (hardener)	50706-3	Clear

### *Coating Application*

The average high August temperatures in Norfolk are around 85 Fahrenheit. Unseasonably warm temperatures resulted in tank temperatures in the upper 90s with higher temperatures encountered for bulkheads and overhead areas that were exposed to direct sunlight on the opposite side. Temperatures in this range would affect the performance or application characteristics of this coating system by reducing the overcoat window to no more than 48 hours.

Application was accomplished employing a plural component spray pump which was equipped with pump mounted pre-heaters, heated lines, a remote mix block and single feed spray guns. Using the plural component equipment, the prime coat application and top coat was completed without incident.

The inspection of the top coat of the system for adequate dry film thickness (DFT) coverage was conducted in accordance with the requirements of SSPC PA-2 and NAVSEA's controlling documents. The controlling documents allowed DFT readings between 18 and 36 mils. Up to 10% of the complex structures may have up to 50 mils. Once DFT measurements were confirmed to be within specification the staging was removed and the access cutouts were welded back in place. Appropriate surface

preparation and touchup coating application were effected to repair the areas disturbed by the hot work and the staging removal. Touchup was performed using the products listed in the table for each individual coating.

## **CONCLUSIONS**

This project was determined to be successful in the application demonstration of the Warren Environmental coating systems in one tank. Lessons learned during this demonstration project were addressed and should be incorporated into future applications of these types of coating system as follows:

- Extended overcoat windows actually improve production time when outside influences require paint application to be delayed between coats.
- Using the Warren Environmental plural component pumps, mixing blocks, and product heating tanks to apply the Rapid Cure products in a high volume application is feasible for suitably trained applicators.
- Training to use plural component spray equipment for high solid materials is applicable for this Rapid Cure material but additional training is required in the use of the Warren product heating tanks.
- Blasting, completing structural repairs, washing the surfaces, and finally blasting again to meet SSPC SP-10 surface preparation requirements does eliminate high surface chloride levels and improves overall surface preparation.
- DFT requirements for Single Coat applications should be reviewed so as to ensure the most benefits of this type of application are realized.
- Using abrasive blasting to prepare the access insert touch up areas is feasible in a production type environment.
- The touch up and repair material needs to be assessed for a way to shorten the amount of time required to complete these touch up application.

## **APPENDIX A**

USS OAK HILL (LSD 51)  
Warren Environmental S-301



**Figure 1**  
**USS OAK HILL (LSD-51)**  
**INITIAL “as found” CONDITIONS 3-121-1-W**



**Figure 2**  
**USS OAK HILL (LSD-51)**  
**INITIAL “as found” CONDITIONS 3-121-1-W**



**Figure 3**  
**USS OAK HILL (LSD-51)**  
**INITIAL “as found” CONDITIONS 3-121-1-W**



**Figure 4**  
**USS OAK HILL (LSD-51)**  
**Final Coat Applied 3-121-1-W**





Figure 5  
**USS OAK HILL (LSD-51)**  
**Final Coat Applied 3-121-1-W**



Figure 6  
**USS OAK HILL (LSD-51)**  
**Final Coat Applied 3-121-1-W**