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## PROTECTIVE COATING ANALYSIS

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### ABSTRACT

As a National Center for Energetics, the Indian Head Division, Naval Surface Warfare Center (IHDI, NSWC) is committed to providing quality products in support of the Fleet. In concert with this effort, the AEPS/PAD Engineering and Production Divisions provide life-cycle support for various Navy Aircrew Escape Propulsion Systems (AEPS) devices used in aircraft. This cradle-to-grave philosophy has led to a strive for technical excellence as well as environmental responsibility. To support these goals, the AEPS/PAD Engineering and Production Divisions recently performed extensive testing of various low Volatile Organic Compound (VOC) coatings for use on its various (AEPS) devices. Testing was performed to identify an environmentally compliant coating with improved durability.

Coating application and evaluations conformed to American Society for Testing and Materials (ASTM) and Military specifications and were accomplished in two phases. Phase I subjected twenty-seven coating systems to a battery of qualitative and resistance properties testing. The coatings were derived from an exhaustive family of epoxies, latex, urethanes, calcium sulfonates, vinyls, and alkyds. Upon completion of Phase I, it was determined that polyamide epoxies and polyurethanes exhibited the qualities sought. The compliant coatings' performance exceeded that of the baseline significantly, application was easier, and results correlated well with published field testing. Initial difficulties were overcome concerning the application, clean-up, testing, and health hazards of the low VOC paints. Complicated air sampling for the determination of isocyanates and polyisocyanates was conducted to certify that levels were below all established limits in this country in an effort to protect the worker. Six of the best performing primers and paints were selected for more complex and stringent testing in Phase II. The six systems were selected on the basis of their ratings after evaluation using a weighted scoring system. Heavily weighted characteristics were those that exhibited the greatest potential to impact worker health, paint performance, and the environment. Phase II resulted in the selection of a polyurethane topcoat and reduced the field of primers to three. The testing demonstrated that the polyurethane coating exhibited excellent adhesion and great resistance to lifting and chipping in addition to polyurethanes' well-known attributes of chemical and UV resistance, flexibility, and high gloss retention. This paper discusses the development, test methods, results, and lessons learned from Phases I and II of the Protective Coating Analysis performed at the Indian Head Division, Naval Surface Warfare Center.

## 1. INTRODUCTION

Over the years, environmental regulations have become more stringent and expansive in the areas that they cover. To control air pollution, restrictive environmental regulations encouraged the development of paint formulations that generate less air emissions. These coatings were entering the market with little research to support manufacturer's claims and there were limited studies for comparisons. While the Maryland Department of the Environment (MDE) proposed further restrictions for metal coatings, IHDIV, NSWC identified a need to establish a more durable coating due to discrepancy reports received concerning corrosion on AEPS units. These factors contributed to the decision to conduct an evaluation prior to selecting a replacement coating. The primary objective for these assessments was to compare new coating technologies that meet proposed environmental regulations in Maryland and exhibit performance exceeding that of the current coating system.

## 2. BACKGROUND

The purpose of the Protective Coating Analysis program stems from two concerns: environmental compliance and durability. Impending environmental regulations stipulated that all paints utilized for metal protection have low Volatile Organic Compound (VOC) levels by May 1996, with the exception of maintenance coatings. The maximum allowable VOC content in the Maryland legislature's draft was 420 g/L (3.5 lb/gal). The paints that IHDIV, NSWC were using on AEPS devices exceeded the allowable VOC level. If a replacement coating was not found, production of AEPS devices could not be maintained. Corrosion reports from the Fleet further compounded the issue by adding a durability concern.

AEPS devices are typically found on ejection seats of various aircraft supporting the national defense. These units have various amounts of explosives and propellants in them and are critical in the ejection sequence. AEPS items are "man-rated" which means that failure may cause a loss of life. Thus, when AEPS devices show extensive corrosion, they are rejected and disposed. AEPS coatings must maintain their integrity in rugged environments for up to seventeen years. These conditions consist of temperature extremes from -65°F to 165°F, extreme humidity, and corrosive environments found on aircraft carriers.

## 3. EVALUATION PROGRAM

Phase I consists of applying the selected low VOC coating systems to test panels for both qualitative and resistance testing and evaluation. The objectives of Phase I were to identify environmentally compliant coatings that exceed the performance of the currently used system, identify alternate coating systems to utilize for production, and select six systems for further validation. Phase II was developed to further reduce the field of candidates while validating Phase I test results. A weighted scoring system devised to meet the above-mentioned objectives was employed for all test panel evaluations. Phase III testing shall subject the three best coating systems from Phase II to identical tests utilizing various substrates found on AEPS devices. Finally, these panels shall be exposed to weathering tests.

## 4. PHASE I

Prior to any testing, an extensive literature search and a review of various government and industry testing was performed. The intent of this research was to identify coatings that could work well in the unique AEPS operating environment and meet program concerns. These efforts identified twenty-seven coating systems that claimed to fit the criteria. The coatings were from various urethane, epoxy, vinyl, alkyd, latex, and calcium sulfonate families. The original coating system that served as the baseline for comparison consisted of a polyamide epoxy primer topcoated with an acrylic lacquer complying with MIL-P-23377F and MIL-L-81352A, respectively.

### 4.1 METHODS

A flat 4130 steel substrate was utilized because it is the dominant material used on AEPS motors. Since the original application does not allow any alteration to the plated surface (i.e., sanding or blasting), each panel was cleaned and wiped down with solvent per ASTM D609-95. The current coating system served as the control sample. All coatings were applied with High Volume Low Pressure (HVLP) spray guns. Application, recoat time, and dry times were per the individual manufacturer's recommendations. Since one of the objectives was to meet or exceed current performance levels, the majority of the tests were derived from the acrylic lacquer's test specification (MIL-L-81352A). The remainder of the tests were taken from MIL-P-83126A, the specification for the design of AEPS devices, in order to ensure that the coatings could withstand the AEPS operating environment. Each of the coating systems were subjected to qualitative and resistance properties testing. All tests were performed in triplicate, such that three panels were utilized per paint panel, per test. The following gives a brief description of each test and its purpose:

#### 4.1.1 QUALITATIVE TESTS

**Working Properties - MIL-L-81352A.** Primer is applied to each panel and allowed to dry. Two coats of paint are then applied and allowed to air dry for twenty-four hours. After the drying period, the paint is visually inspected for cobwebbing, running, sagging, streaking, blushing, orange peeling, and hazing. The purpose of the working properties test is to evaluate surface appearance and working (spraying and drying) properties of the paint.

**Self-lifting Properties - MIL-L-81352A.** Primer is applied to each panel and allowed to dry fully. Two coats of paint are then applied to the primer with different drying intervals between each coat. Hence, the purpose of this test is to evaluate intercoat adhesion of the system's topcoat and evaluate its tolerance to drying interval deviations from the recommended interval.

**Surface Appearance - MIL-L-81352A.** Panels from the working properties test are utilized for this test. This test is similar to the working properties test but more stringent in that the surface of the coating is examined under a magnification of 10 to 15 diameters.

**Primer Lifting - MIL-L-81352A.** Primer is applied to panels and allowed to dry at different intervals prior to application of two coats of paint. The purpose of this test is to evaluate adhesion of the primer to the substrate and rate its tolerance to drying interval variations.

**Coating Anchorage -** Primer and paint are applied to each panel. After air-drying for one week, the coating is subjected to a knife test (FED-STD-141C, Method 6304.1). This test indicates the flexibility of the coating, the ribboning ability of the coating, and dry-knife adhesion.

Adhesion (wet) Tape Test - FED-STD-141C, Method 6301.2. Primer and paint are applied on panels. Each panel is air-dried for one week and then partially immersed in distilled water for twenty-four hours. After removal from the water, panels are wiped dry. A stylus is used to impart two straight lines one inch apart along the width of the immersed portion. Subsequently, a one inch strip of masking tape is applied perpendicular to the lines and removed in one brisk stroke. The purpose of this test is to evaluate intercoat and substrate adhesion.

#### 4.1.2 RESISTANCE PROPERTIES TESTS

Water Immersion - FED-STD-141C, Method 6011. Primer and paint are applied to each panel and allowed to air-dry for one week. Panels are then immersed in distilled water for twenty-four hours. This test was performed in conjunction with the tape test. The purpose of this test is to evaluate the coating's integrity when subjected to prolonged immersion.

Temperature Cycling - MIL-P-83126A. Primer and paint are applied to each panel and allowed to air-dry for one week. Panels are then cycled three times from -65°F to 160°F. The purpose of this test is to evaluate performance of the coating during temperature transitions and at prolonged temperature extremes.

Humidity Test - MIL-P-83126A. Primer and paint are applied to each panel and allowed to air-dry for one week. Panels are then stored at a temperature of 120° ± 5°F and 100% humidity for ten days. The purpose of this test is to evaluate the coating's ability to withstand humid environments.

Salt-spray Test - MIL-STD-810, Method 509. Panels from the humidity test are utilized for this test. Panels are subjected to 168 hours of salt-spray in a 20% salt solution. The purpose of this test is to demonstrate the coating's ability to withstand corrosive environments.

#### 4.2 PHASE I RESULTS

Three main objectives were accomplished during Phase I:

- (1) Coatings were identified that exceeded the performance of the baseline sample.
- (2) Six systems were selected for further validation as shown in Table 1.
- (3) Viable alternatives were put into place to maintain production of AEPS units. These coating systems, marked with an asterisk in Table 1, were chosen from the top six because they performed most consistently during testing.

TABLE 1. PHASE I SELECTED COATINGS

PRIMER	TOPCOAT
MIL-P-53030 *	MIL-C-85285B *
MIL-P-85582 *	MIL-C-85285B
Zinc-rich epoxy	TT-P-2756 *
MIL-P-53030	Flouropolyurethane
Moisture-cure (MC) MIOZinc	MC Urethane
Calcium sulfonate	MIL-C-85285B

In comparing results with other government and private agencies, it was discovered that Phase I results balanced well with previously performed testing and actual use by the New Hampshire Department of Transportation (DOT), North Carolina's DOT, Naval Aviation Depots at Cherry Point, NC and Jacksonville, FL, Alberta Transportation and Utilities, and Aerojet Propulsion. Also, results correlated well with MIL-F-7179G which was developed for the protection of aerospace weapons systems.

## 5. PHASE II

A complete test plan, similar to that used for Phase I, was developed and executed. The testing in Phase II was designed to narrow the field of top performers from paints selected from Phase I and validate Phase I test results. Additional testing introduced here includes resistance to cyclic corrosion testing, chipping, and solvents.

### 5.1 METHODS

A recommended practice is to configure accelerated test models so they imitate the actual objects to be coated. Test results utilizing this emulation of actual shapes have been shown to more closely replicate actual field performance. This reasoning led to the use of non-flat test panels for this phase: Each panel had a 1/2" x 1" "U"-shaped channel tack welded near the bottom. This channel simulates the curves found on AEPS devices while the edges serve to test the relative ability of the coating to adhere to non-continuous surfaces.

### 5.2 ADDITIONAL TESTS

The humidity test was replaced with a method corresponding to ASTM 2247 because of a wider acceptance in industry. The panels subjected to the humidity test were photographed when removed from the chamber, however, they were placed back into the chamber within the hour to subsequently undergo the cyclic salt-fog test. This quick transition is required per MIL-P-83126A.

A cyclic corrosion test replaced the continuous exposure salt spray test performed in Phase I. This decision was based on simulation logic and past research. Since actual atmospheric exposures include both wet and dry conditions, it seems natural to model accelerated laboratory tests after a cyclic environment. A dry-off period makes this test more strenuous because it gives oxygen an opportunity for adsorption, resulting in the formation of oxide layers<sup>1</sup>. This action occurs after the humidity has deposited a condensed film that sets up an electrical potential between the iron and other materials in the metal, thus causing corrosion. Research indicates that with cyclic corrosion tests, the relative corrosion rates, structure and morphology are more similar to those seen outdoors<sup>2</sup>. Consequently, cyclic corrosion tests usually provide a closer correlation to outdoor exposure than conventional salt spray tests. This has been demonstrated in numerous tests and is becoming more widely accepted<sup>3</sup>. A cyclic test corresponding to ASTM G85 ANNEX 5 was used in Phase II because it simulated the conditions AEPS devices are exposed to. This test consists of salt-fog, humidity, and dry-off cycles.

Chip resistance was a primary concern for this effort because chipping was reported along with the corrosion in discrepancy reports. Chipping occurs when AEPS devices are placed on top of sharp objects during handling and routine maintenance operations. Phase II incorporated ASTM D3170 which provided a method to test and evaluate test panels for chip resistance. This test projects road gravel at the painted surface at a pressure of seventy psi for ten seconds.

### 5.3 PHASE II RESULTS

Two primary objectives were achieved during Phase II: (1) Phase I test results were validated, and (2) the field of candidates was reduced to one topcoat and three primers. The difference between all six coatings' performance was imperceptible after temperature cycling, tape, and primer lifting tests. The best performing paints correlated again with MIL-F-7179G. The topcoat corresponding to TT-P-2756 was eliminated due to poor chipping performance and pinholing. The fluoropolyurethane was removed from further consideration due to poor chip and cyclic salt-fog resistance and the need for a highly volatile thinner. The moisture-cure topcoat and primer also required a highly volatile thinner and there were application problems related to their viscosity. The calcium-sulfonate primer performed superbly in the cyclic salt-fog, however, failure to maintain intercoat adhesion with an ideal topcoat caused its removal from further testing.

The topcoat, MIL-C-85285B, was selected as a permanent replacement coating for AEPS devices following these failures. Reducing the number of topcoats to one shall reduce the variables in Phase III and ensure the validity of the final primer selection. The remaining primers to be further evaluated are water-reducible polyamide epoxies. The MIL-P-53030 is lead and chromate free while primer meeting MIL-P-85582 uses barium chromate in its formulation. The zinc-rich epoxy primer performed very well after initial difficulty was met reducing its viscosity.

### 6.0 ANALYSIS

Since the majority of results were qualitative versus quantitative, a weighted scoring system was utilized in evaluating results. This method involves the defining of classifications for various types of "defects" and then weighting each type. Weights were assigned based on the impact each characteristic would have on AEPS application, service life, maintenance, environmental concerns, and worker safety. This system is based on a modified control chart used to evaluate quality. Table 2 details the weighting scheme for each characteristic evaluated as seen below:

TABLE 2. Q-CHART WEIGHTING SCHEME

QUALITATIVE TESTS (Phase I)		QUALITATIVE TESTS (Phase II)	
<u>Primer</u>	<u>Weight</u>	<u>Primer</u>	<u>Weight</u>
Intercoat adhesion	1.00	Intercoat adhesion	1.00
Coating anchorage	2.00	Coating anchorage	2.00
Tape test	2.50	Dry tape test	2.50
Primer lifting	3.00	Tape test	3.00
Thinning	3.00	Primer lifting	4.00
<u>Top Coat</u>		<u>Top Coat</u>	
Working properties	0.50	Working properties	0.50
Surface appearance	1.00	Surface appearance	1.00
Water resistance	2.00	Water resistance	2.00
Self-lifting	2.50	Resists self lift	2.50
Thinning	3.00	Thinning	3.00
RESISTANCE TESTS (Phase I)		RESISTANCE TESTS (Phase II)	
<u>System</u>	<u>Weight</u>	<u>System</u>	<u>Weight</u>
Water immersion	2.00	Water immersion	2.00
Temperature cycling	2.00	Temperature cycling	2.00
Humidity	3.00	Salt-fog/Humidity	5.00
Salt-spray	5.00	Chipping	4.00
		Solvent	3.00

The weighting scheme above was used to create a Quality Score Chart or Q-Chart.<sup>4</sup> The Q-Charts for Phases I and II are shown in Figures 1 and 2 respectively. To arrive at a final score for each coating system, each characteristic weight was multiplied by 0.5, 1.0, 2.0, or 3.0 signifying poor, fair, good, or excellent performance respectively. For example, the medium used for thinning corresponded to these ratings based on whether the coating used a solvent (0.5), other (1.0), water (2.0), or no thinner (3.0). This value was then multiplied by the weight given to the characteristic "Thinning". Of the qualitative properties, thinning was given the most weight due to worker safety and environmental regulations. The next most important qualitative properties are primer lifting and self-lifting. These properties were weighted accordingly due to the important role that primer and intercoat adhesion play in a coating's service life and performance. Salt-spray was weighted the greatest under resistance properties since the most rigorous coating environment is a corrosive one. Additionally, it has been historically recognized that salt-spray tests are the most severe tests of coating integrity.



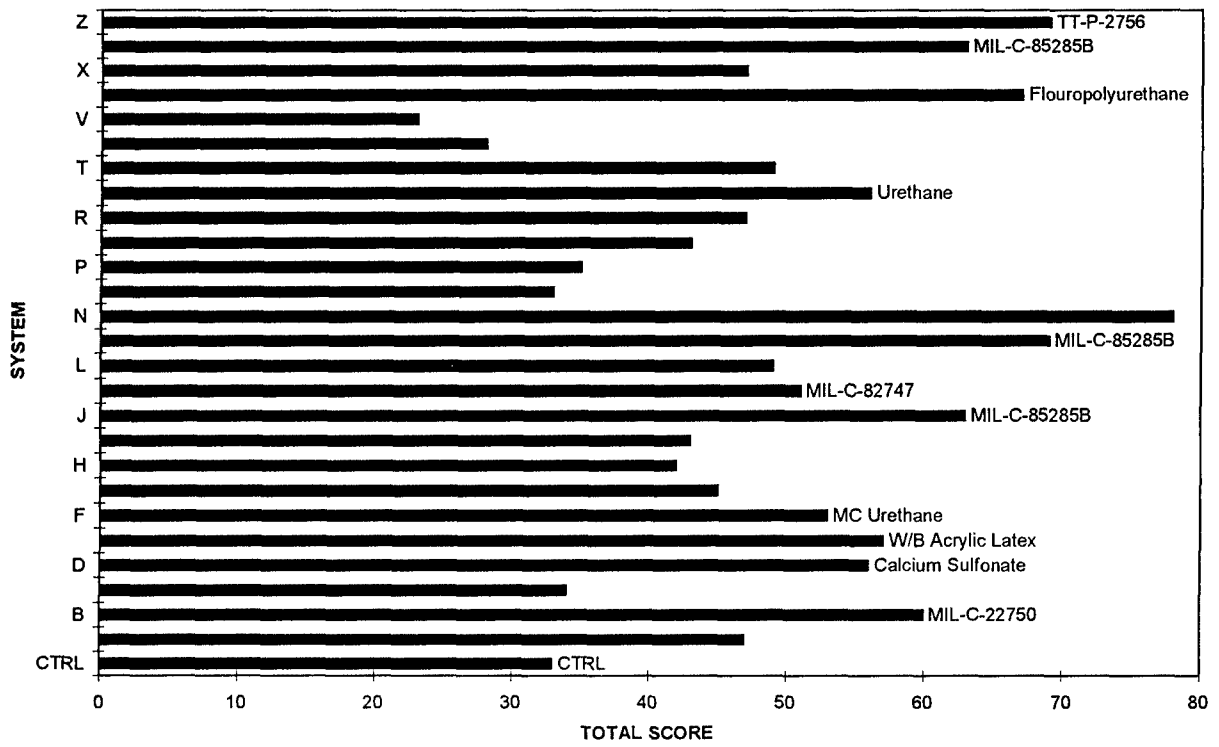


Figure 1. Phase I test results. Q-chart depicts weighted scores of each coating system.

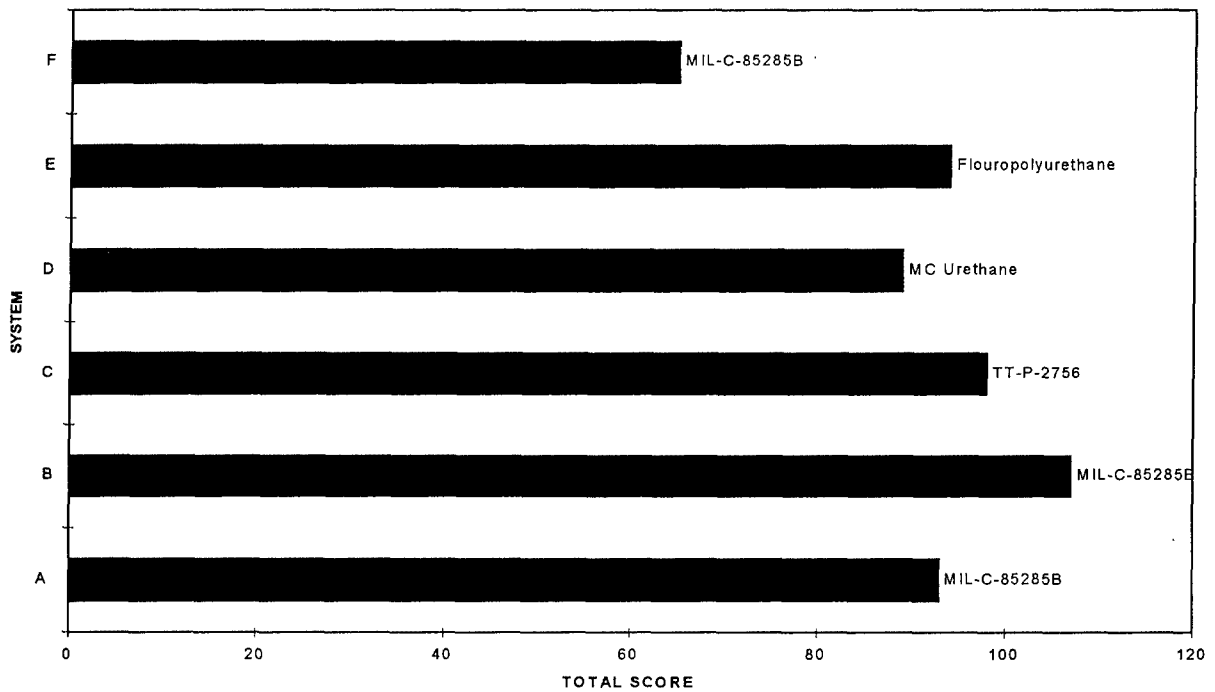


Figure 2. Phase II test results. Q-chart depicts weighted scores of each coating system.

## 7.0 LESSON LEARNED

Throughout execution of the program, various lessons were learned. Test plans were modified as necessary to incorporate these changes in order to refine the test methods. The following details the lessons learned throughout Phases I and II:

- (1) A procedure was provided for sealing the panel edges. This was done in order to prevent corrosion that formed at the edges during resistance tests from proliferating to the rest of the panel.
- (2) The temperature in the paint booth was recorded. Different temperatures can affect the application and drying of the coatings and this helped to control this variable.
- (3) The recommended cleaner, thinner, mixing ratio, viscosity, dry times, and thickness were recorded on the evaluation sheets. Recording this information helped the application of paints run much more efficiently and provided a reference during analysis.
- (4) When the paint sprayed inadequately, the first panels were not covered uniformly. Therefore, the applicator sprayed a section of scrap metal until it applied consistently.
- (5) Dry film thickness was measured in order to ensure coatings were applied per manufacturer's recommendations.
- (6) The engineer was required to be present during mixing and first application. This resulted from an incident during which too much solvent was added to the coating, thus causing the coating's VOC level to exceed the maximum allowable level. One of the criteria examined in Phase I was how simple the coating was to work with out of the containers. Applicators were instructed to make adjustments as necessary to get an adequate spray while staying below the VOC limit, however, oversight was not in place for three days during Phase I.
- (7) When urethane coatings first showed positive results, the isocyanate issue was examined. This examination indicated that safe isocyanate exposure levels were neither well documented nor fully understood. One of the primary manufacturers, Miles Inc., has established guidelines and the Occupational Safety and Health Administration (OSHA) has set a very conservative limit for the hexamethylene diisocyanate (HDI) monomer. Industrial hygienists from the National Naval Medical Center in Bethesda, Maryland conducted complex air sampling for HDI-Polyisocyanate and the HDI-Monomer in a spray paint booth at IHDI, NSWC. The levels for each were found to be below the Miles Guideline Limit of 1 part per million (PPM) and the OSHA's Short Term Exposure Limit (STEL) of 0.18 PPM respectively. Therefore, approval was granted to operate with a combination respirator as described in Table 3 along with other related test equipment.

TABLE 3. TEST EQUIPMENT

EQUIPMENT	MANUFACTURER	MODEL
Paint spray guns	Air-Verter	AV-001MG (#5cap,#12noz)
Viscosity cup	Zahn	Signature Series S90, #2
Masking tape	3M	Code No. 250
Gloves, protective	Ansell Edmont	Neolatex
Combination filter/respirator	Mine Safety Appliances Co.	Comfo II w/GMA-H Cart.
Rubber covered roller	Paul N. Gardner Company	Meets Fed. Test Std. 6301.2
Chip resistance test chamber	Q-Panel Company	Gravelometer ASTM D3170
Coating thickness gage	DeFelsco	6000-2
Standard salt-fog chamber	Singleton	32
Temperature recorder	Honeywell	AR100
Humidity chamber	Envirotronics	EH125-2
Temperature recorder	Honeywell	455X21
Thermal cycling chamber	Webber	WF40-100
Temperature recorder	Honeywell	AR100
Environmental test chamber	Atotech USA Inc.	CCT-P20

### CONCLUSION

The development and execution of a customized test plan is an effective method to evaluate coatings for specific applications. The actual testing of products gave results that often conflicted with the coating manufacturer's claims, but also was essential to making a replacement decision. That decision, based on the research and experimental results herein, was also bolstered by applying, testing, and evaluating the products on-site. This enabled the applicators and test personnel to become intimate with the coatings while providing a high level of control over the exposure conditions endured by the test panels. These factors contributed greatly to a high level of confidence in this analysis and the replacement coating selected for AEPS devices.

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