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DETERMINATION OF THE EFFECTIVENESS OF NONCHROMATED CONVERSION COATINGS FOR USE WITH IVD ALUMINUM COATINGS

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14. Abstract

The Air Force Air Logistics Centers (ALCs) have either recently eliminated or have plans to eliminate the usage of cadmium processing for the maintenance and overhaul of aircraft. The thrust of their effort is the elimination of the hazardous cadmium waste streams. The replacement process, ion vapor deposition (IVD) of aluminum, is free of the environmental problems associated with cadmium processing. However, as for cadmium processing, it still requires subsequent treatment with a chromate conversion coating which contains a known carcinogen. This program, then, addressed the effectiveness of non-chromated conversion coatings for use with IVD aluminum coatings. It concentrated on conversion coatings which were also being evaluated by the Aerospace Industry as replacements for chromated conversion coatings on bare aluminum alloys. IVD aluminum-coated steel and aluminum panels, and IVD aluminum-coated steel and titanium fasteners were treated with various candidate non-chromated conversion coatings and then subjected to various environmental exposures. The objective of this program was met in that a non-hazardous conversion coating, which could replace chromate conversion coating on IVD aluminum coatings, was identified and verified.

15. Subject Terms

CONVERSION COATING, NON-CHROMATED CONVERSION COATING, IVD ALUMINUM, SALT SPRAY, PRIMER ADHESION, CONTACT ELECTRICAL RESISTANCE

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PREFACE

This report was prepared by McDonnell Douglas Aerospace (MDA), McDonnell Douglas Corporation, St. Louis, Missouri under Contract No. F08635-94-C-0004, "Determination of the Effectiveness of Non-Chromated Conversion Coatings for Use with IVD Aluminum Coatings." The program was conducted by the Engineering Technology Group of the Environmental Assurance Division at MDA, St. Louis. The program was administered by the U.S. Air Force Armstrong Laboratory at Tyndall Air Force Base. Lt. Phil Brown, Lt. Ray Anthony Smith, and Lt. Carol Smith were the technical and administrative program managers. This final report summarizes the work performed between 29 October 1993 and 29 June 1995.

EXECUTIVE SUMMARY

A. OBJECTIVE

The objective of this program was to determine the effectiveness of nonchromated conversion coatings for use with Ion Vapor Deposited (IVD) aluminum coatings. The program began on 29 October 1993 and was successfully completed on 29 June 1995.

B. BACKGROUND

The Air Force Air Logistics Centers (ALCs) have either recently eliminated or have plans to eliminate the usage of cadmium processing for the maintenance and overhaul of aircraft. The thrust of their effort is the elimination of the hazardous cadmium waste streams. The replacement process, IVD of aluminum, is free of the environmental problems associated with cadmium processing. However, as for cadmium processing, it still requires subsequent treatment with a chromate conversion coating which contains a known carcinogen (i.e., hexavalent chrome). This program was conceived to identify and verify a replacement treatment for chromate conversion coating for use with IVD aluminum coatings.

C. SCOPE/METHODOLOGY/TEST DESCRIPTION

The objective of this program was completed under six tasks. Initially, candidate nonchromated conversion coatings were identified. Next, six of these candidates were subjected to screening tests using IVD aluminum-coated alloy steel panels. The screening tests included 5% neutral salt fog for 3000 hours, primer adhesion, and contact electrical resistance both before and after seven days exposure to 5% neutral salt fog. Next, the four most promising candidates determined from the screening tests were subjected to expanded environmental testing using IVD aluminum-coated alloy steel panels. Testing in this case involved 5% neutral salt fog for 8000 hours, sulfur dioxide salt fog for 500 hours, and outdoor exposure in St. Louis, MO for 58 weeks. Next, the performance of these same four treatments was evaluated in regard to aluminum alloys. More specifically, IVD aluminum-coated aluminum panels and IVD aluminum-coated alloy steel and titanium fasteners were treated and subjected to both 5% neutral salt fog and sulfur dioxide salt fog testing. Salt spray evaluation of the IVD aluminum-coated and treated fasteners involved installation of the fasteners in aluminum alloy bars with bare countersinks. Finally, a 20-gallon scale-up processing line was set up in the laboratory. Initially, it was used to conduct additional process optimization work for the two most promising treatments. After selection of the final optimized process, it was then used to verify this process. Process verification involved processing of IVD aluminum-coated panels over a 1-month period while monitoring the chemistry of the solutions daily.

D. RESULTS

Based on the testing described above, a nonchromated conversion coating, which could replace chromate conversion coating for IVD aluminum coatings, were identified and verified. Required functional performance and conformance to all applicable military specifications were demonstrated for this replacement conversion coating. The final process selected to replace chromate conversion coating for IVD aluminum coatings was a variation of the Sanchem-CC process. Basically, it involves immersion in a permanganate solution at 140°F for 3 minutes followed by immersion in a potassium silicate solution at 200°F for 1 minute.

E. CONCLUSIONS

The process consisting of Sealing Steps II and III of the Sanchem-CC process is a satisfactory corrosion resistant alternative to chromate conversion coating for IVD aluminum-coated alloy steel parts including fasteners. In addition, Sealing Steps II and III of the Sanchem-CC process appear to be a viable production process based on the process verification work performed under the final task of this program.

F. RECOMMENDATIONS

Proceed with set up of a pilot production line for alloy steel parts using the final process selected and verified under this program.

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SECTION I
INTRODUCTION

A. OBJECTIVE

The objective of this program was to determine the effectiveness of nonchromated conversion coatings for use with Ion Vapor Deposited (IVD) aluminum coatings. It was accomplished by first identifying candidate nonchromated conversion coatings, and then performing the testing necessary to verify required functional performance and conformance to all applicable military specifications.

B. BACKGROUND

IVD aluminum is a soft ductile coating whose properties are nearly identical to those of pure aluminum. Both the process which is a dry process performed in a vacuum chamber and the 1100 aluminum alloy evaporant are environmentally clean. MDA completed an Air Force Civil Engineering Support Agency (CESA) program to replace cadmium at the Air Logistics Centers (ALCs) with IVD aluminum in August 1992. The thrust of the program was elimination of cadmium hazardous wastestreams. The program included a demonstration phase at the Warner Robins ALC in which all parts that had been processed with cadmium were successfully changed to IVD aluminum.

All of the ALCs have now either closed or reduced usage with plans to close their cadmium processing lines. Most cadmium applications have been, or will be, changed to IVD aluminum.

Cadmium has been widely used as a corrosion-resistant finish on steel. Substitution of IVD aluminum for cadmium eliminates the hazardous waste produced by cadmium processes, eliminates work area exposure to toxic cadmium (which is OSHA-regulated), and eliminates the need to control toxic air emissions which will be EPA-regulated by the 1990 Clean Air Act Amendment (CAAA). In addition, electroplated cadmium processing introduces additional hazardous waste materials, such as cyanide in the plating bath, which also is eliminated.

The postcoat processing steps for IVD aluminum, structural aluminum, and the various cadmium processes are essentially the same. They all use a chromate conversion coating which provides additional corrosion protection and an improved base for paint adhesion.

However, chromate conversion coatings contain hexavalent chrome, a known carcinogen. The use of chromium compounds that are subsequently emitted to the atmosphere or enter wastewater or landfills is now under intense scrutiny by regulatory agencies.

The use of an effective conversion coating is essential to the functional performance of IVD aluminum. The corrosion resistance duration times required by the IVD aluminum military specification, MIL-C-83488, in a 5% neutral salt fog environment are shown in Table 1 as are actual duration times.

TABLE 1. REQUIRED AND ACTUAL CORROSION RESISTANCE DURATION TIMES FOR IVD ALUMINUM-COATED STEEL SUBJECTED TO A 5% NEUTRAL SALT FOG ENVIRONMENT

Class	Type I – No Conversion Coat		Type II – Chromate Conversion Coat	
	Required (Hrs)	Actual (Avg Hrs)	Required (Hrs)	Actual (Avg Hrs)
1	504	504	672	8,600
2	336	336	504	2,800
3	168	168	336	900

Actual salt fog duration times for Type II (chromated) coatings far exceed MIL-C-83488 requirements with averages of 8600, 2800, and 900 hours, respectively for Class 1, 2, and 3 coatings. Actual salt fog duration times for Type I (no conversion coat) coatings, however, do no better than meeting minimum requirements of 504, 336, and 168 hours, respectively for Class 1, 2, and 3 coatings. Actual duration times are derived from an extensive MDA database and are an average of all data sources.

C. SCOPE/APPROACH

To achieve the objective of this program, the program was broken down into six tasks. These tasks, including a brief description of each, are presented below.

1. Task 1 – Candidate Coating Survey

MDA conducted an industry/DOD survey to identify promising nonchromated conversion coatings for use with IVD aluminum coatings.

2. Task 2 – Candidate Coating Screening

MDA selected the most promising candidates identified under Task 1 and conducted screening tests using IVD aluminum-coated alloy steel panels. Testing included 5% neutral salt fog for 3000 hours, primer adhesion, and contact electrical resistance both before and after seven days exposure to 5% neutral salt fog. Task 2 also investigated materials for locally touching up or repairing a nonchromated conversion coating.

3. Task 3 – E-Coat Applied Primer

The intention of this task was to apply primer by electrodeposition to IVD aluminum-coated panels which had been treated with the nonchromated conversion coatings which passed the Task 2 screening tests. These E-Coat panels were then to be evaluated in regard to primer adhesion and corrosion resistance. This activity was proposed as an optional task under the program and was priced separately. The Air Force elected not to fund this portion of the program.

4. Task 4 – Extend Database With Accepted Candidate Coatings

MDA selected the four best nonchromated conversion coatings based on Task 2 testing, then conducted expanded corrosion resistance testing for those treatments using IVD aluminum-coated alloy steel panels. Testing included 5% neutral salt fog for 8000 hours, sulfur dioxide salt fog for 500 hours, and long-term outdoor exposure in St. Louis, MO.

5. Task 5 – Effect On Aluminum Alloys

The same four nonchromated conversion coatings selected and evaluated under Task 4 were next evaluated in regard to their ability to protect aluminum alloys. More specifically, IVD aluminum-coated 2024-T3 and 7075-T6 aluminum panels were treated with the various candidate treatments and then subjected to 5% neutral salt fog and sulfur dioxide salt spray testing. Also, IVD aluminum-coated titanium and alloy steel fasteners were treated with the four candidate nonchromated conversion coatings and then installed in 7075-T6 aluminum bars with bare countersinks. These fastener bars were then subjected to 5% neutral salt fog and sulfur dioxide salt fog testing, and the ability of the candidate nonchromated conversion coatings to protect the bare countersinks was determined.

6. Task 6 – Implement Pilot Production

MDA set up a 20-gallon processing line and conducted a process optimization study for the two treatments considered most promising at the conclusion of Task 5 testing. A final treatment was then selected, and this same 20-gallon scale-up line was used to verify the treatment selected. This process verification activity involved processing of IVD aluminum-coated alloy steel panels over a 1-month period and monitoring the chemistry of the solutions daily.

SECTION II
TASK 1 – CANDIDATE COATING SURVEY

A. OBJECTIVE

The objective of Task 1 was to conduct an industry/DOD survey to identify promising nonchromated conversion coatings for use with IVD aluminum coatings.

B. OVERVIEW

Twelve candidate nonchromated conversion coatings were identified for potential use with IVD aluminum coatings. A list of these twelve candidates is provided by Table 2. Important environmental factors were considered in regard to identifying these various treatments as candidates.

C. DISCUSSION

The twelve candidate nonchromated conversion coatings listed in Table 2 were identified by MDA in an industry/DOD survey conducted prior to contract award. At the time of contract award, an extensive review was conducted to identify additional nonchromated conversion coatings for investigation in the test program. The intention was to identify any treatments which had shown promising results in any industry/DOD test programs since selection of the initial twelve candidates. A review was made of all information presented at the Aerospace Chrome Elimination (ACE) team meetings which are held twice a year. Also, MDA attended the Third Annual Workshop on Chromate Replacements in Light Metal Finishing in September of 1993. The purpose of this workshop, hosted by the Sandia National Laboratories, was to provide a forum for the exchange of information related to developing and qualifying nonchromated conversion coatings for light metals (primarily aluminum). Approximately 45 individuals were in attendance with representation from the coating manufacturers (e.g., Parker+Amchem), metal suppliers (e.g., Alcoa), automotive industry, aerospace industry, and R&D community (e.g., Sandia Labs and several universities). MDA also contacted Mr. Jordan Rosengard at Hughes to discuss the status of their proprietary nonchromated conversion coating and Mr. John Jones at Boeing to discuss the performance of the latest version of the Australian CSIRO Laboratory's rare-earth treatment conversion coating. Finally, Mr. Glenn Moore of EG&G Idaho, Incorporated was contacted to discuss the status of the joint program between Boeing and the IDAHO National Engineering Laboratory to develop alternatives to chromium-based conversion coatings. It was learned that this proposed Air Force-funded program had been canceled. Based on the above investigation, it was determined that there were no other candidate nonchromated conversion coatings worthy of inclusion in the test program.

TABLE 2. CANDIDATE NONCHROMATED CONVERSION COATINGS IDENTIFIED

COATING	CHEMICAL DESCRIPTION	MANUFACTURER
Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I, II and III)	Permanganate Based	Sanchem, Inc.
Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I and II)	Permanganate Based	Sanchem, Inc.
Sanchem-CC (Sealing Step II only)	Permanganate Based	Sanchem, Inc.
Cobamine (Modified)	Cobalt Based	Parker+Amchem
Alodine NC 90/91	Organo-Metallic (Fluro-Metallic Acid Chelated with an Acrylate Polymer)	Parker+Amchem
PTD-3102-A	Organo-Metallic (Zirconia Reacted with an Organic Polymer)	Parker+Amchem
PTD-1323-CV	Iron Phosphate	Parker+Amchem
Bonderite 37	Zinc Phosphate	Parker+Amchem
Turcoat 6787	Zirconium Based (Contains an Organic)	Turco Products, Inc.
Turcoat 6788	Acrylic Emulsion with Corrosion Inhibitors	Turco Products, Inc.
Permatreat 1001	Organo-Metallic (Zirconia Based)	Betz Laboratories
Permatreat 1011	Organo-Metallic (Titanium Based)	Betz Laboratories

SECTION III
TASK 2 – CANDIDATE COATING SCREENING

A. OBJECTIVE

The primary objective of Task 2 was to select at least five candidate nonchromated conversion coatings and subject them to screening tests using IVD aluminum-coated alloy steel panels. A second objective was to evaluate various materials in regard to their ability to locally touch up or repair a nonchromated conversion coating.

B. OVERVIEW

Six candidate nonchromated conversion coatings were selected for screening. These candidates are listed in Table 3. Selection of these candidates was based primarily on the results of screening tests conducted under in-house funding prior to contract award. These tests included 5% neutral salt fog per ASTM B117 for 3000 hours, primer adhesion, and contact electrical resistance both before and after seven days exposure to 5% neutral salt fog. They were performed on the twelve candidate nonchromated conversion coatings of Table 2 using IVD aluminum-coated alloy steel panels. In addition to the results of screening tests performed under in-house funding, selection of the six candidate nonchromated conversion coatings for repeat screening tests was also based on environmental considerations. More specifically, they were all investigated and judged to be acceptable in regard to compliance with current regulations pertaining to air emissions, water or wastestreams, and landfill.

TABLE 3. LIST OF NONCHROMATED CONVERSION COATINGS SELECTED FOR TASK 2 SCREENING TESTS

NUMBER	NONCHROMATED CONVERSION COATING
1	Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I, II, and III)
2	Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I and II)
3	Alodine 2000 (Formerly Identified as Cobamine)
4	Sanchem-CC (Sealing Step II Only)
5	Permatreat 1001
6	Alodine NC 90/91

NOTE: Coatings are listed in order of preference based on 5% neutral salt fog testing for 3000 hours under in-house funding prior to contract award.

The six candidate nonchromated conversion coatings were subjected to the same screening tests conducted earlier under in-house funding for the original twelve candidates. Salt spray performance for this repeat testing, however, was not as good as the original results. Possible reasons for the poorer salt spray performance were postulated, and three supplemental test investigations were conducted. These supplemental investigations not only helped confirm the theories postulated, but were instrumental in downselecting to the required four candidate treatments under Task 4.

A touch-up investigation involving five different materials was also conducted. Initially, IVD aluminum-coated alloy steel panels were treated with the final, optimized process selected under Task 6 of this program. During conversion coating of the panels, the conversion coating was omitted from small selected areas on the panels by masking with small strips of tape. After conversion coating, 1 inch long scratches were made such that they were through the conversion coating and into the IVD aluminum coating. The non-conversion-coated areas and scratches were then touched up with the five different touch-up materials. The touched-up panels were then subjected to 5% neutral salt fog testing for two weeks.

C. DISCUSSION

1. Environmental Assessment

All of the six candidate nonchromated conversion coatings selected for repeat screening tests were investigated in regard to compliance with environmental regulations. More specifically, they were looked at in regard to current regulations pertaining to air emissions, water or wastestreams, and landfill. Along this line, it should be noted that manganese is a component of the Sanchem-CC process, cobalt is a component of the Alodine 2000 process, and zirconium is a component of the Permatreat 1001 process.

In regard to current landfill regulations, a review of the Code of Federal Regulations, Title 40, Part 261 revealed that neither manganese, cobalt or zirconium are regulated. Therefore, they are not considered hazardous waste.

In regard to current water or wastestream regulations, it was determined that there should be no problem with any of the six candidate nonchromated conversion coatings. More specifically, the six candidates would fall under the category of a metal finishing operation. Therefore, at the highest tier, they are regulated by the Code of Federal Regulations, Title 40, Part 433. A review of this document revealed no regulations for manganese, cobalt or zirconium. With this being the case, the candidate coatings would then be regulated by the General Pretreatment Regulations. Again, a review of these regulations revealed no restrictions on the components of the six candidate nonchromated conversion coatings. The final tier of control would be the local Metropolitan Sewer District. There are no regulations in St. Louis, MO pertaining to manganese, cobalt or zirconium in wastestreams.

In regard to air emissions, manganese and cobalt compounds, like chromium compounds, are considered air toxics or hazardous air pollutants (HAPs). This classification is based on the Air Toxics Amendment or Title III Program of the Clean Air Act Amendments of 1990. It should be noted that this law requires the EPA to write a MACT (Maximum Achievable Control Technology) standard for both manganese and cobalt compounds as well as for all of the other 187 chemicals or compounds identified as HAPs. Manganese and cobalt compounds are significantly less toxic than chromium compounds. At some point in the future they will probably be regulated. However, they will probably be lightly regulated when the required risk assessment is completed. Also, little or no air emissions of manganese or cobalt compounds would be expected when using the Sanchem-CC or Alodine 2000 processes, respectively.

2. Screening Tests

a. Panel Material and Preparation

The panels used for Task 2 testing were AISI 4130 steel, 4 X 6 X 0.050 inch thick, conforming to MIL-S-18729. This is the material required by the IVD aluminum military specification (MIL-C-83488) for corrosion resistance testing. Prior to IVD aluminum coating, the panels were vapor-degreased and grit-blasted using aluminum oxide abrasive conforming to McDonnell Material Specification MMS-411. The IVD aluminum coating on the side of the panels to be tested was applied to conform to MIL-C-83488, Class 2 (0.5-mil thick minimum). The actual average thickness of the IVD aluminum coating on the various panels ranged from 0.54 to 0.71 mil.

In accordance with standard practice, the IVD aluminum coating on all of the test panels was glass bead peened prior to application of the various nonchromated conversion coatings. The conversion coatings were applied to the panels within 24 hours after the peening operation.

Treatment of the IVD aluminum-coated steel panels in regard to the Alodine 2000, Alodine NC 90/91, and Sanchem-CC processes involved MDA hand-carrying the panels to Parker+Amchem in Madison Heights, MI and Sanchem, Inc. in Chicago, IL. This activity not only expedited treatment of the panels, but allowed MDA to witness treatment of the panels in the laboratory. The panels treated with Permatreat 1001 were sent by overnight delivery to Betz Laboratories, treated by Betz the day received, and then returned the day of treatment to MDA by overnight delivery. Chromate conversion-coated control panels were also prepared. They were treated in-house.

b. Contact Electrical Resistance

In most electrical and electronic applications, a part requires low electrical resistance as well as optimum corrosion resistance. MIL-C-83488 requires that Type II (i.e., chromate conversion coated) IVD aluminum coatings requiring low electrical resistance be treated with a solution which meets both the Class 1A corrosion resistance and Class 3 electrical resistance requirements of MIL-C-5541. Such solutions or conversion coatings require qualification initially to MIL-C-81706 (Chemical Conversion Materials for Coating Aluminum and Aluminum Alloys).

MDA has constructed a contact electrical resistance apparatus conforming to the requirements of MIL-C-81706. This apparatus is shown in Figure 1. Per the military specification, the contacting electrodes are copper with a finish not rougher than that obtained by the use of 000 metallographic abrasive paper. Also, the electrodes are sufficiently flat so that when the load is applied without a specimen between them, light is not visible through the contacting surfaces. The area of the upper electrode is one square inch and the area of the lower electrode is somewhat larger (i.e., 1.7 square inches for the MDA apparatus). Contact electrical resistance measurements are made using an applied electrode pressure of 200 pounds per square inch. Figure 2 shows a close up of the electrodes.

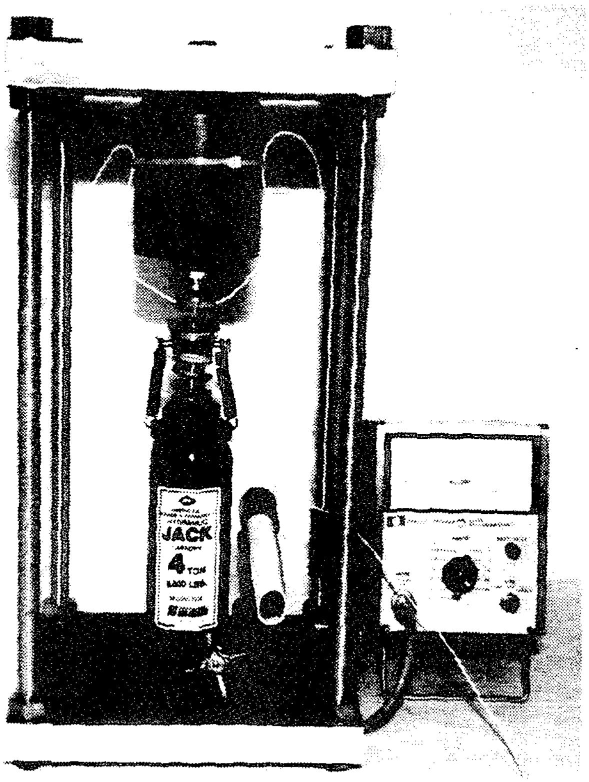

Figure 1. Contact Electrical Resistance Apparatus Conforming to MIL-C-81706

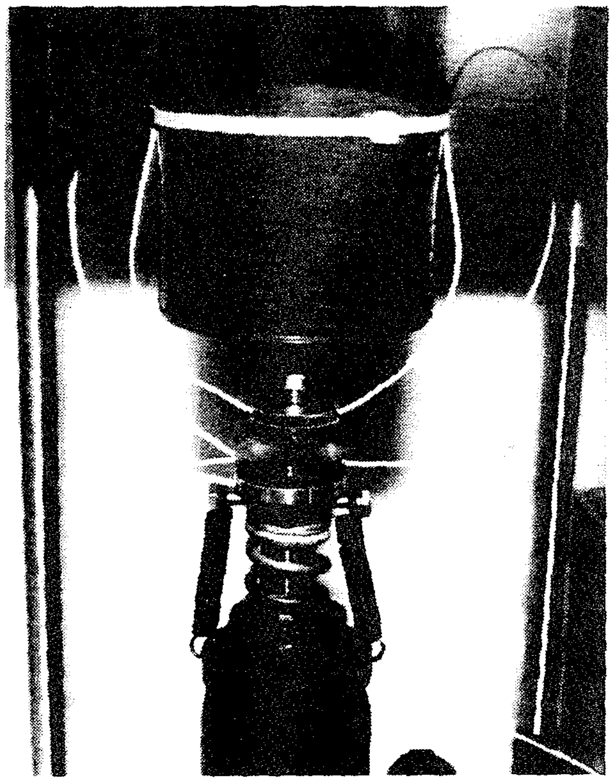

Figure 2. Close Up of Electrodes of Contact Electrical Resistance Apparatus

There are several noteworthy additional details in regard to the contact electrical resistance apparatus constructed by MDA. First, both the upper and lower electrodes are insulated from the load mechanism using a 0.2 inch thick piece of polycarbonate. Also, the upper electrode is attached to a self-aligning monoball to ensure parallel contact of the electrodes. Finally, two lead wires are soldered to the back side of each electrode. The current lead is located at the center of the electrode and the voltage lead is located 0.25 inch off-center. A manual switch has been incorporated into the circuitry to allow the voltage lead for the lower electrode to be attached directly to the panel via an alligator clip. This provision allows exact conformance to the electrical schematic of MIL-C-81706 in regard to the contact electrical resistance apparatus.

Electrical resistance measurements were made using a Hewlett Packard Model 4828A four lead milliohm meter. The required 200-pound load was applied to the panel using a hydraulic bottle jack which compressed a spring. As force was applied with the bottle jack, the spring would partially compress, maintaining a consistent 200-pound load. The applied force was electronically measured using a Model C62H-500-0200 load cell manufactured by Transducers, Inc. The output of the load cell was displayed digitally in pounds. Figure 3 shows a conversion-coated panel clamped-up between the electrodes.

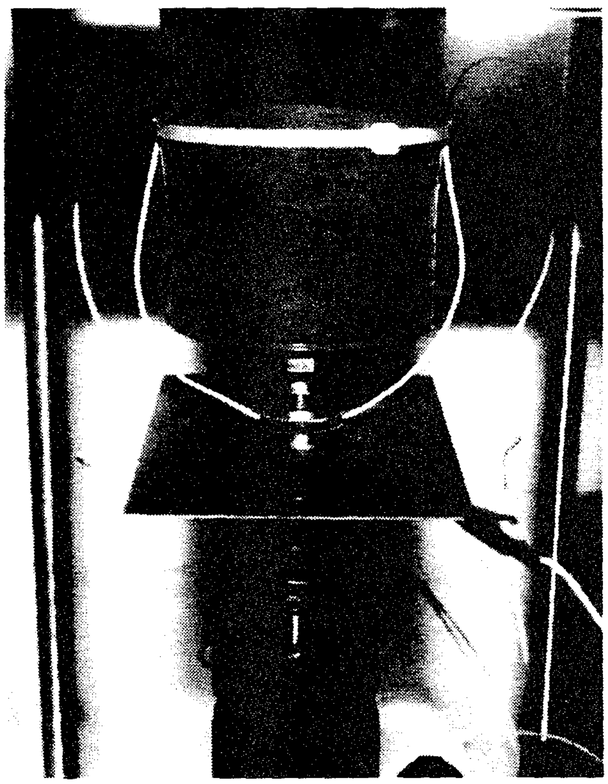

Figure 3. Conversion-Coated Panel Clamped Between Electrodes of Contact Electrical Resistance Apparatus

Two panels were evaluated in regard to contact electrical resistance for each of the six candidate nonchromated conversion coatings. Contact electrical resistance measurements were made both before and after 7 days of 5% neutral salt fog exposure to conform to the testing requirements of MIL-C-81706. Control specimens were also prepared and tested for Iridite 14-2 chromate conversion coating. Two panels were prepared and tested for each of the following immersion times in the Iridite 14-2: 30 seconds, 1 minute, and 2 minutes. The typical immersion time for IVD aluminum-coated parts per MDA process specification P.S. 13209 is 30 seconds.

The conversion coating was removed from one side of all the panels prior to contact electrical resistance testing. It was removed by sanding from the nontest side of the panel which is the side placed in contact with the lower electrode. Removal of the coating allowed measuring the resistance of the conversion coating on only one side of the panel which is the intent of MIL-C-81706.

The results of contact electrical resistance testing are presented in Table 4. MIL-C-81706 requires a contact electrical resistance of 5,000-microhms (5-milliohms) per square inch or less as treated, and 10,000-microhms (10-milliohms) per square inch or less after 7 days exposure to 5% neutral salt fog. In general, the resistance values were slightly higher after the salt spray exposure. Of greater significance, all of the candidate nonchromated conversion coatings, as well as the chromated conversion coating, had resistance values well below the upper limits of the military specification.

TABLE 4. RESULTS OF CONTACT ELECTRICAL RESISTANCE TESTING

CONVERSION COATING	DATE OF CONVERSION COATING	PANEL NO.	AVG. CONTACT ELECTRICAL RESISTANCE (MILLIOHMS) 1/		
			12/22/93 (NO EXPOSURE)	1/7/94 (NO EXPOSURE)	1/20/94 (AFTER 7 DAYS EXPOSURE TO 5% NEUTRAL SALT FOG)
Iridite 14-2 (Chromate Conversion Coating)					
30 Second Immersion	12/20/93	2-3	0.16	0.18	0.79
		2-4	0.26	0.20	1.08
1 Minute Immersion	12/20/93	2-11	0.30	0.30	1.00
		2-12	0.30	0.24	0.91
2 Minute Immersion	12/20/93	2-1	0.19	0.30	0.72
		2-2	0.29	0.31	0.86
Sanchem-CC (Sealing Step II Only)	12/16/93	3A-2	0.26	0.43	1.86
		3A-7	0.31	0.35	1.87
Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I & II)	12/16/93	3B-4	1.11	0.50	0.68
		3B-6	0.64	0.68	0.98
Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I, II, & III)	12/16/93	3C-3	1.21	0.62	1.44
		3C-8	1.21	1.12	0.51
Alodine 2000	12/14/93	4-5	0.97	0.84	1.07
		4-7	1.15	2.25	1.67
Alodine NC 90/91	12/14/93	5-5	0.09	0.13	1.36
		5-7	0.09	0.15	2.03
Permatreat 1001	12/14/93	6-5	2/	2/	2/
		6-8	0.14	0.30	0.71

NOTES: 1/ Average values noted are based on five measurements taken over the surface of the panel. The maximum allowable values per MIL-C-81706 are 5.00-milliohms as treated, and 10.00-milliohms after salt spray exposure.

2/ Conversion coating and IVD aluminum coating was inadvertently removed from the test side of the panel.

c. Primer Adhesion

Primer adhesion testing was conducted using three panels for each of the six candidate nonchromated conversion coatings, and three panels which were chromate-conversion-coated using Iridite 14-2. A compliant, water-borne, epoxy primer (i.e., Courtaulds Aerospace 513X408/910X831) conforming to MIL-P-85582, Type I and McDonnell Material Specification MMS-423 was applied to the various treated panels approximately 24 hours after treatment. Two coats of the primer were applied to provide a final dry film thickness of 0.0008 to 0.0012 inch. The primed panels were air-dried for approximately 30 minutes at room temperature and then oven dried for one hour at 150°F. Tape adhesion testing of the primer was conducted after a minimum of an additional air-dry time of 14 days at room temperature. Initially, a dry, tape adhesion test was performed on each panel. Then, a scribed, wet, tape adhesion test was performed on each panel per Method 6301 of Federal Test Method Standard 141. Testing per the Federal Standard involved immersion of the test panel in distilled water for 24 hours followed by scribing and tape testing.

Initially, the laboratory technician reported no primer adhesion failures for any of the conversion-coated panels after immersion in distilled water for 24 hours, scribing and then tape testing. Later, when the Principal Investigator of this program had an opportunity to closely examine all of the tape tested panels, it was discovered that two of the three panels treated with Sealing Step II of the Sanchem-CC process exhibited minor failure. More specifically, as shown in Figure 4, small pinpoints of primer were removed by the tape.

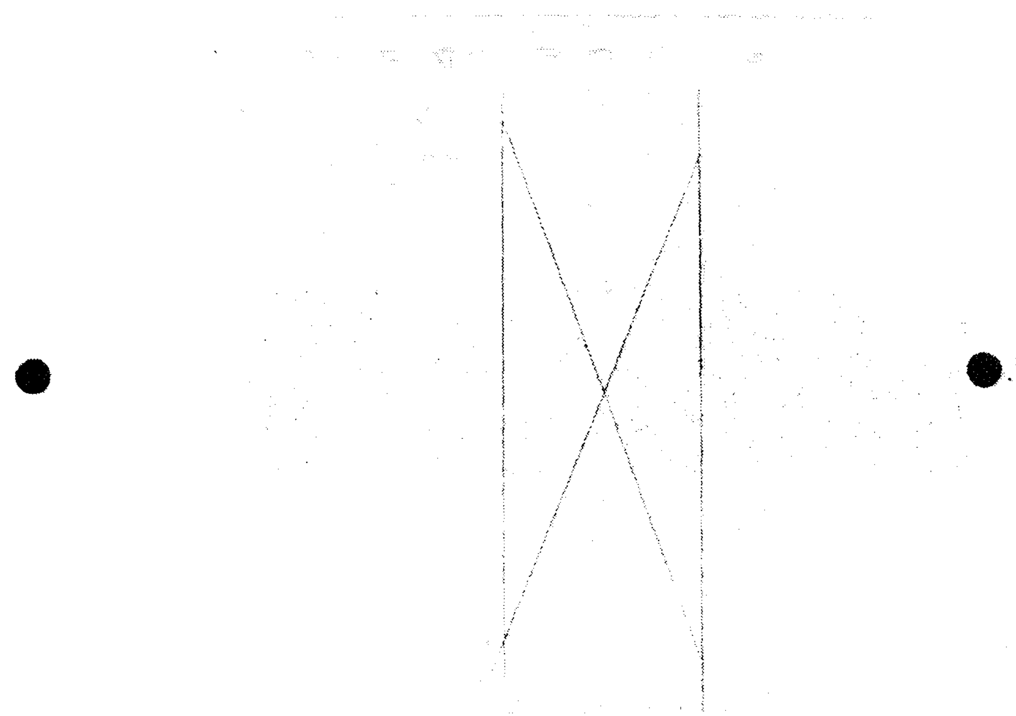

Figure 4. Panel 3A-1 After Scribed, Wet, Tape Adhesion Testing (Pinpoint Areas of Primer Pulled Off with Tape)

At the time the primer adhesion failure was discovered, Sealing Step II of the Sanchem-CC process had already been downselected for further evaluation under Task 4. As a result, tape testing was repeated for this treatment. In addition to again testing the Courtaulds water-borne primer which conforms to MIL-C-85582, Type I and MMS-423, a solvent based primer currently used in production (i.e., Courtaulds 519X303/910X357 conforming to MMS-425) was also tested. As before, two of the three panels primed with the MMS-423 water-borne material exhibited primer failure in a speckled or pinpoint pattern after water immersion. None of the three panels primed with MMS-425 primer failed. Figure 5 shows one of the failed MMS-423 primed panels and one of the acceptable MMS-425 primed panels.

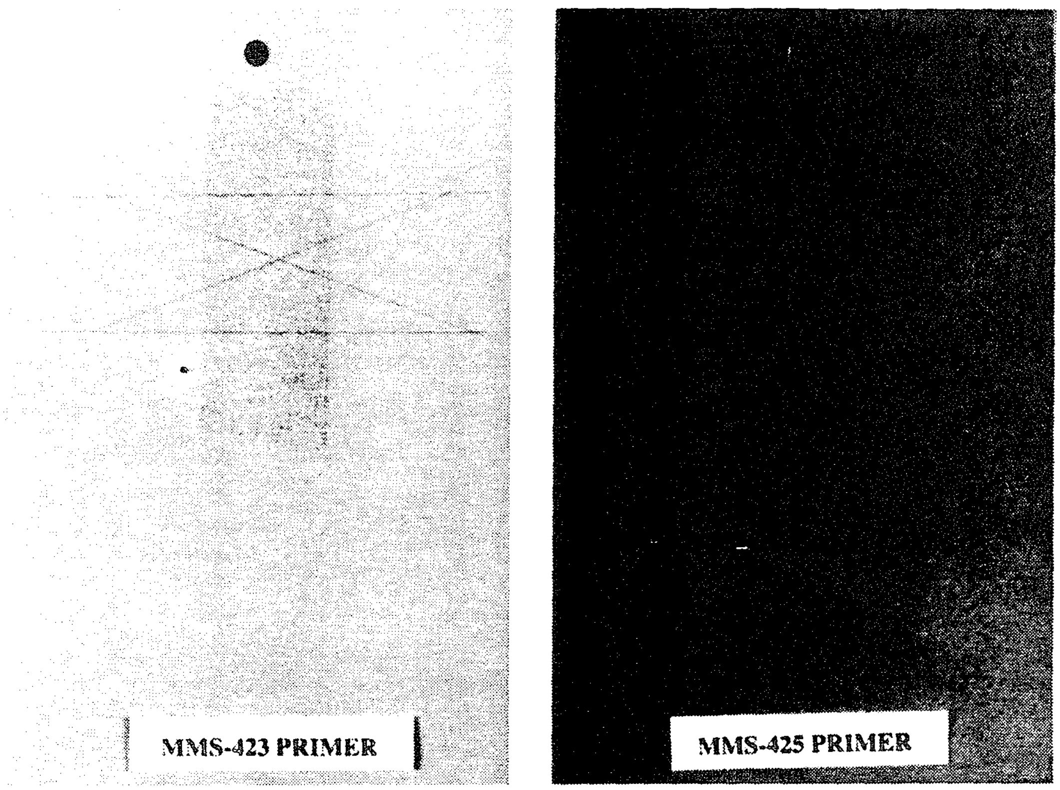

Figure 5. Primed Panels After Scribed, Wet, Tape Adhesion Testing Performed in Accordance with Method 6301 of Federal Test Method Standard 141

The MMS-423 water-borne primer currently used in production will be phased out in the future in favor of an improved water-borne primer. It is possible that this improved primer, or other second generation water-borne primers, will provide required primer adhesion in regard to Sealing Step II of the Sanchem-CC process.

d. Salt Spray (Five Percent Neutral Salt Fog)

Five percent neutral salt fog testing was conducted using three panels for each of the six candidate nonchromated conversion coatings, three panels which were chromate conversion-coated using Iridite 14-2, and three panels which were not conversion-coated. The intention was to expose the panels until failure or for 3000 hours maximum.

Figures 6 – 13 show all of the panels for each of the treatments after 504 hours of salt spray testing. This time period is noteworthy because the salt spray requirement of MIL-C-83488 for a Class 2 IVD aluminum coating (i.e., a 0.5-mil thick minimum coating) without failure is 504 hours. Failure of IVD aluminum-coated alloy steel is considered to occur at the first appearance of red rust. None of the panels showed any evidence of red rust after 504 hours of salt spray testing.

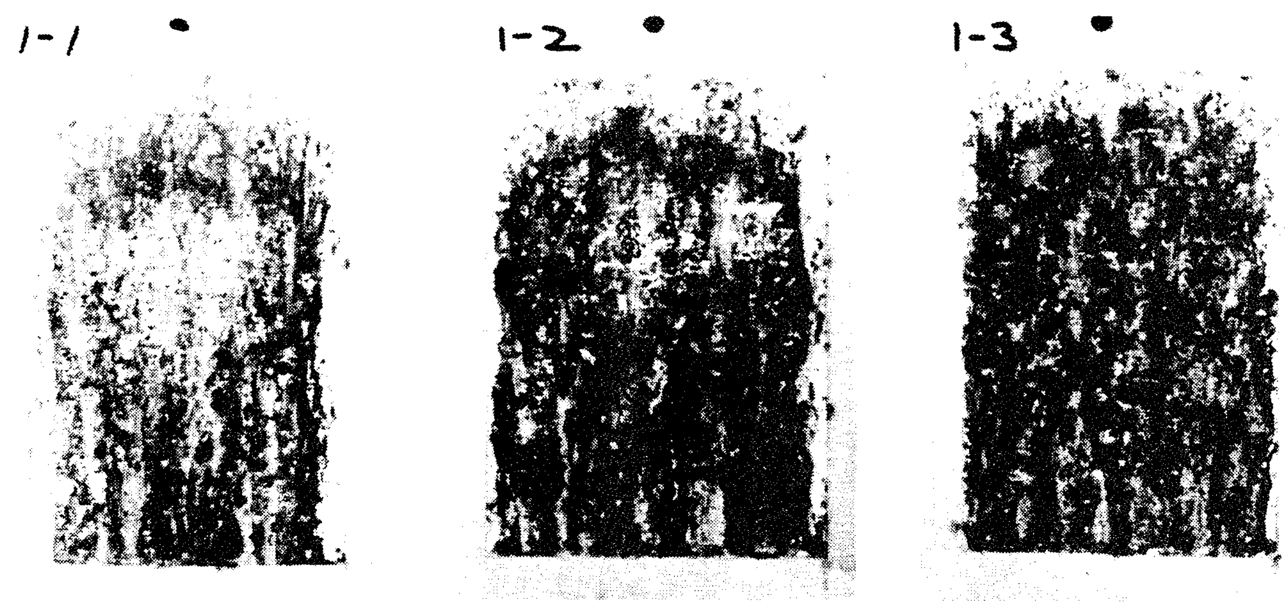

Figure 6. No Conversion Coating After 504 Hours of 5% Neutral Salt Fog Testing

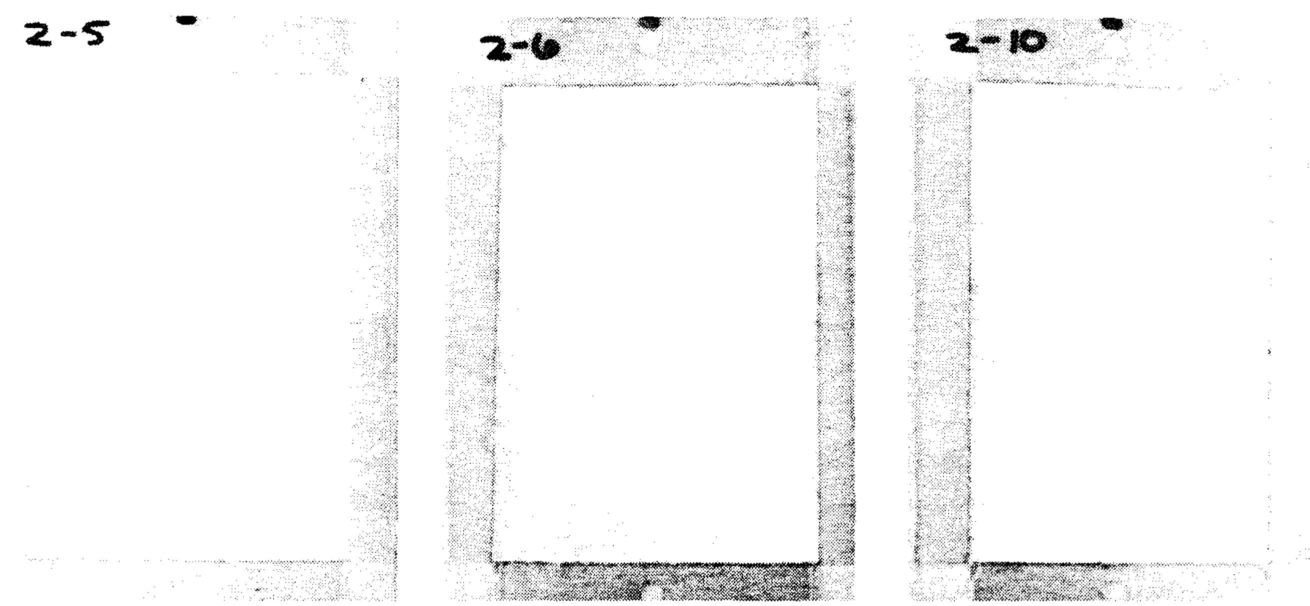

Figure 7. Chromate Treatment (Iridite 14-2) After 504 Hours of 5% Neutral Salt Fog Testing

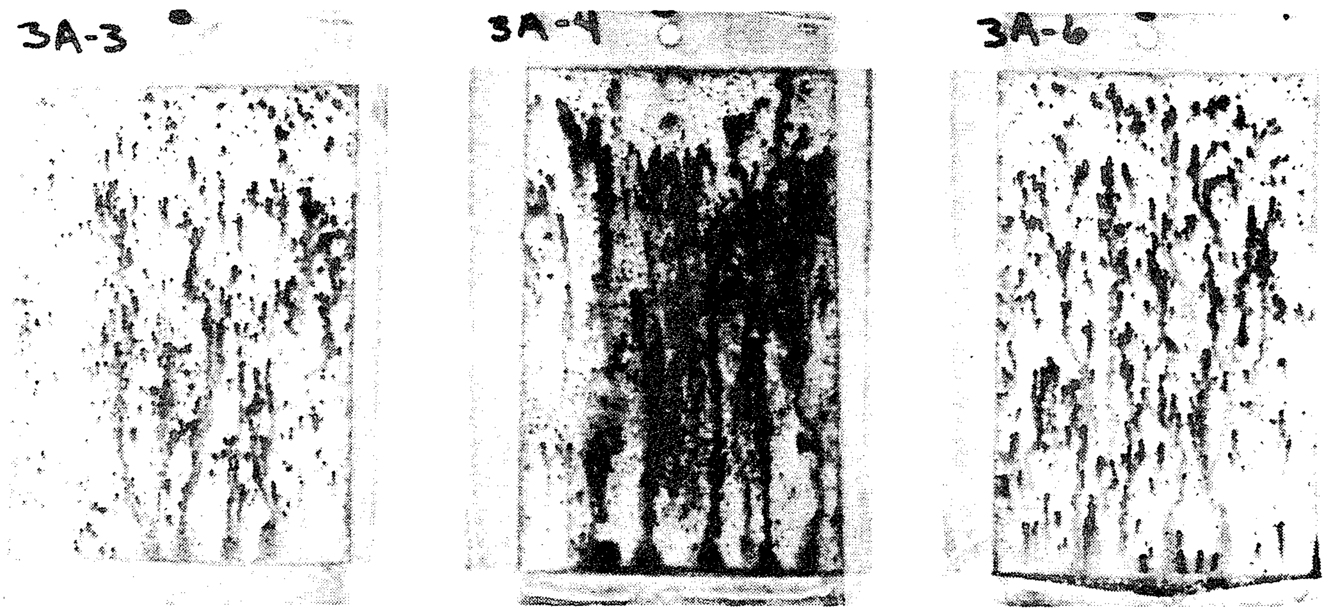

Figure 8. Sanchem-CC Treatment (Sealing Step II Only) After 504 Hours of 5% Neutral Salt Fog Testing

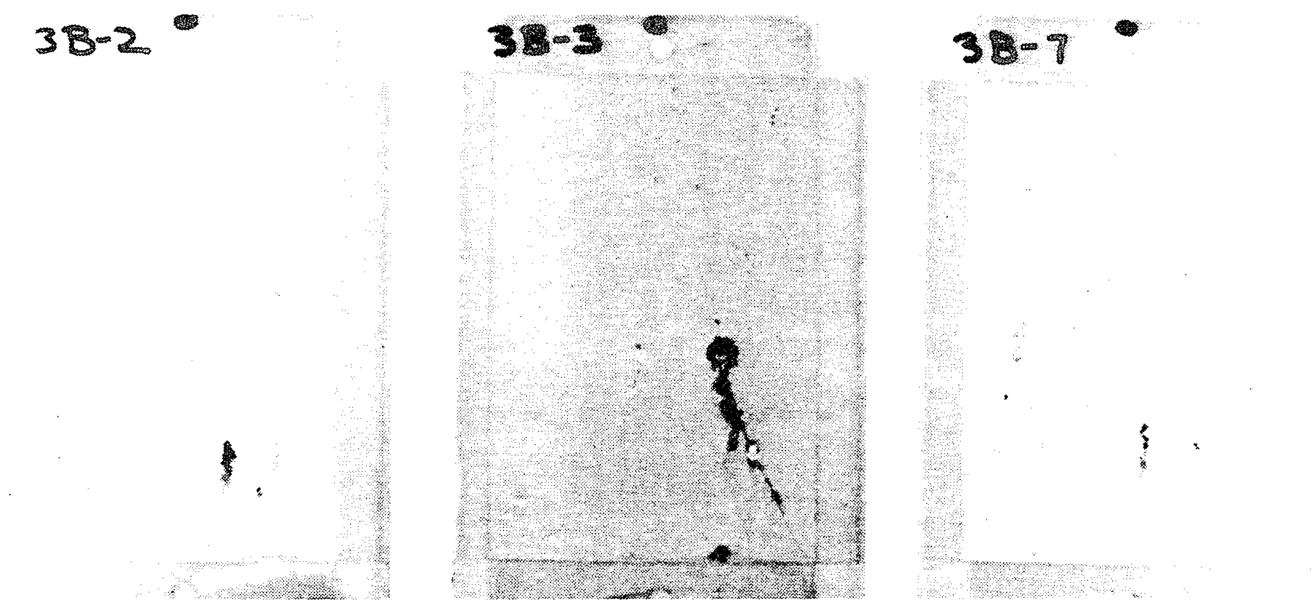

Figure 9. Sanchem-CC Treatment (Oxide Film Formation Step Followed By Sealing Steps I & II) After 504 Hours of 5% Neutral Salt Fog Testing

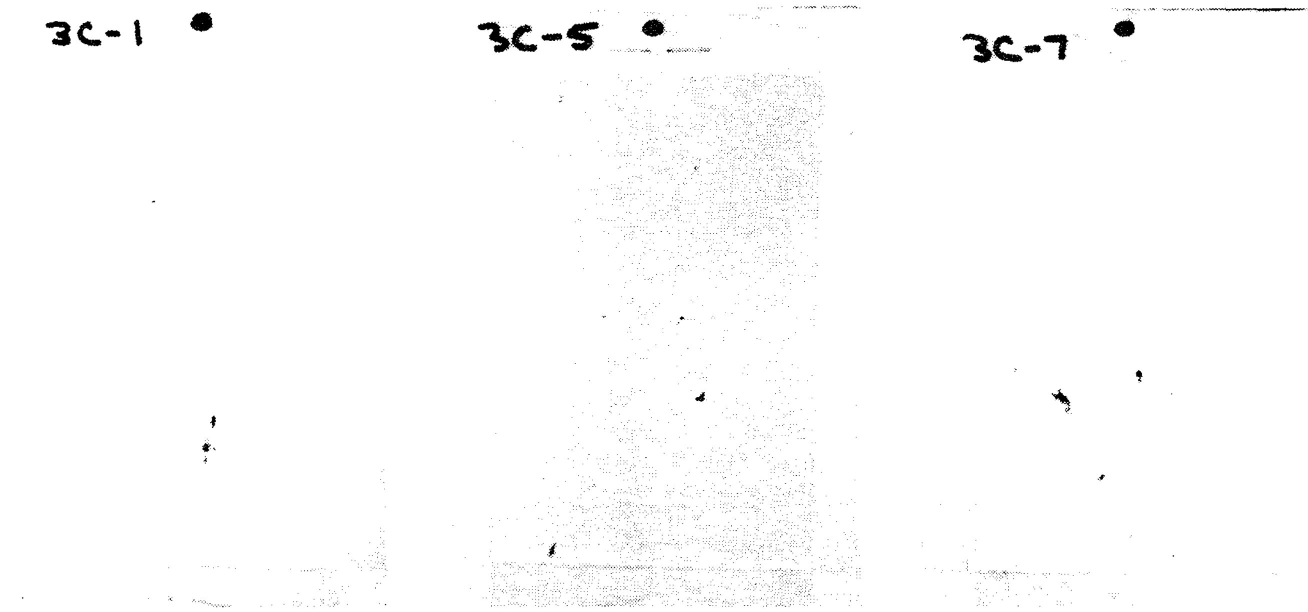

Figure 10. **Sanchem-CC Treatment (Oxide Film Formation Step Followed By Sealing Steps I, II, & III) After 504 Hours of 5% Neutral Salt Fog Testing**

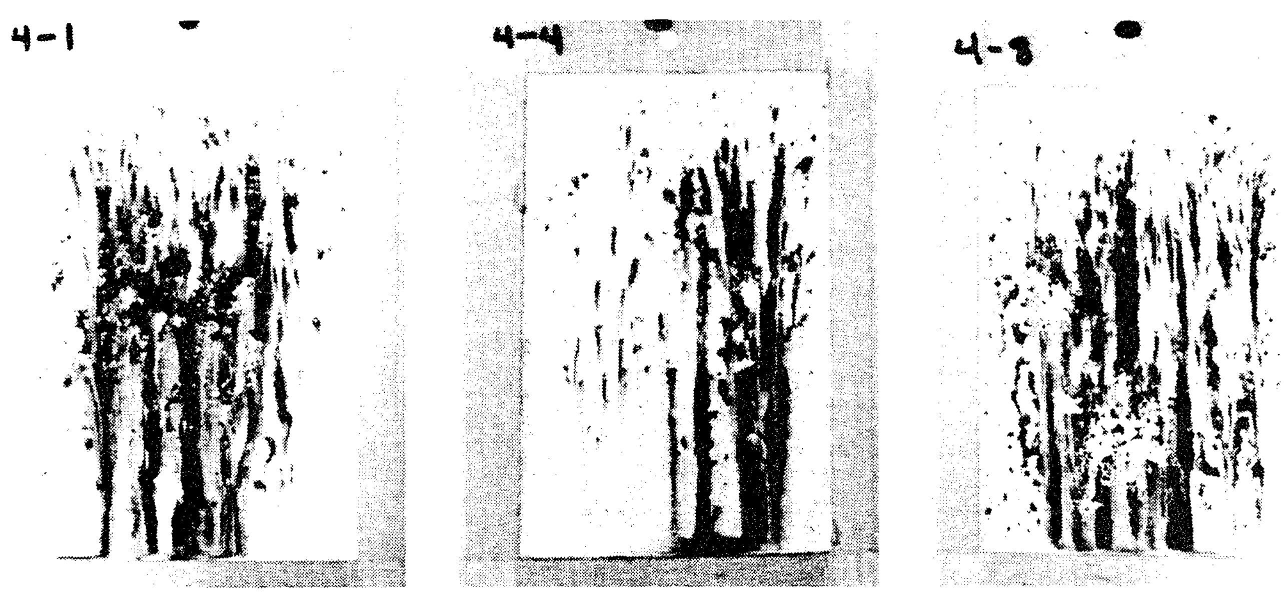

Figure 11. **Alodine 2000 Treatment After 504 Hours of 5% Neutral Salt Fog Testing**

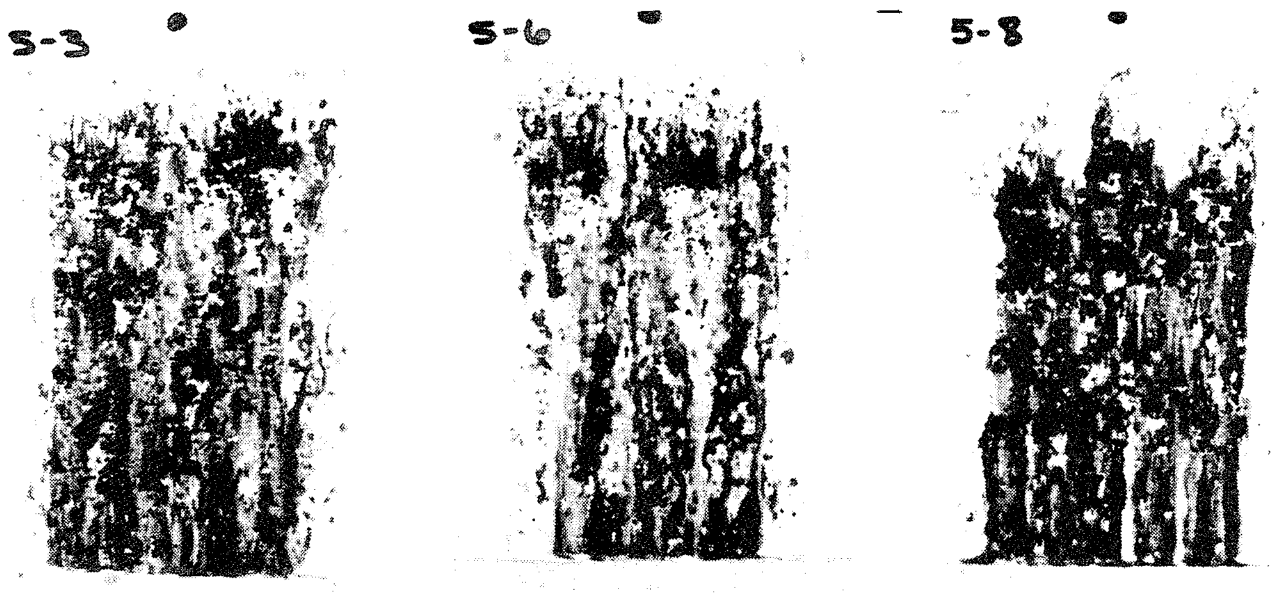

Figure 12. Alodine NC 90/91 Treatment After 504 Hours of 5% Neutral Salt Fog Testing

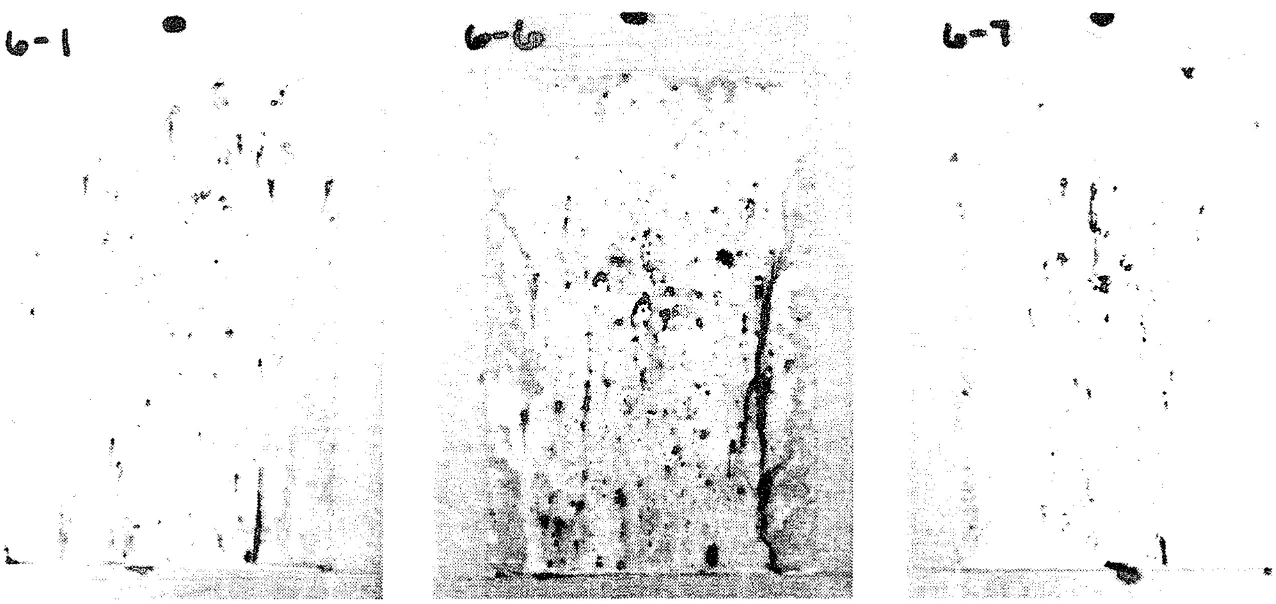

Figure 13. Permatreat 1001 Treatment After 504 Hours of 5% Neutral Salt Fog Testing

Compared to the corrosion resistance screening test performed earlier for these same treatments under in-house funding, pitting occurred sooner and was more pronounced in regard to the Task 2 screening test. The only exception was the chromate conversion-coated control panels which, as in the initial test, did not show evidence of pitting after 504 hours of salt spray testing.

At the conclusion of salt spray testing, the only panels which did not fail were those treated with Sealing Step II of the Sanchem-CC process (i.e., immersion for 1 minute in a permanganate solution at 140°F) and the chromate conversion-coated controls. In other words, the panels for all the other treatments showed evidence of red rust prior to 3000 hours of salt spray testing. Table 5 summarizes the time to failure for each of the treatments. Figures 14 – 21 show all of the panels for each of the treatments at the time of failure or after 3000 hours of salt spray testing, as applicable.

TABLE 5. TIME UNTIL SALT SPRAY FAILURE FOR CANDIDATE NONCHROMATED CONVERSION COATINGS

CONVERSION COATING	PANEL NO.	DATE OF FAILURE 1/	TIME UNTIL FAILURE HOURS (DAYS) 2/	RANKING 3/
No Conversion Coating	1-1	10 Feb	864 (36)	
	1-2	07 Feb	792 (33)	8
	1-3	02 Feb	672 (28)	
Chromate Conversion Coating (P.S. 13209)	2-5	4/	4/	1
	2-6	4/	4/	
	2-10	4/	4/	
Sanchem-CC (Sealing Step II Only)	3A-3	4/	4/	2
	3A-4	4/	4/	
	3A-6	4/	4/	
Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I & II)	3B-2	03 Mar	1,368 (57)	3
	3B-3	24 Feb	1,200 (50)	
	3B-7	03 Mar	1,368 (57)	
Sanchem-CC (Oxide Film Formation Step Followed by Sealing Steps I, II, & III)	3C-1	24 Feb	1,200 (50)	4
	3C-5	28 Feb	1,296 (54)	
	3C-7	21 Feb	1,128 (47)	
Alodine 2000	4-1	14 Feb	960 (40)	5
	4-4	21 Feb	1,128 (47)	
	4-8	16 Feb	1,008 (42)	
Alodine NC 90/91	5-3	04 Feb	720 (30)	7
	5-6	16 Feb	1,008 (42)	
	5-8	02 Feb	672 (28)	
Permatreat 1001	6-1	24 Feb	1,200 (50)	6
	6-6	09 Feb	840 (35)	
	6-7	14 Feb	960 (40)	

NOTES: 1/ Salt spray testing began 05 January 1994.
2/ Minimum requirement is 504 hours per MIL-C-83488.
3/ Based on average hours to failure for the three panels. Lowest number is best.
4/ Completed required 3000 hours of salt spray testing without failure. Testing concluded 10 May 1994.

Figure 14. No Conversion Coating At Time of Failure (1-1 864 Hours; 1-2 792 Hours; 1-3 672 Hours)

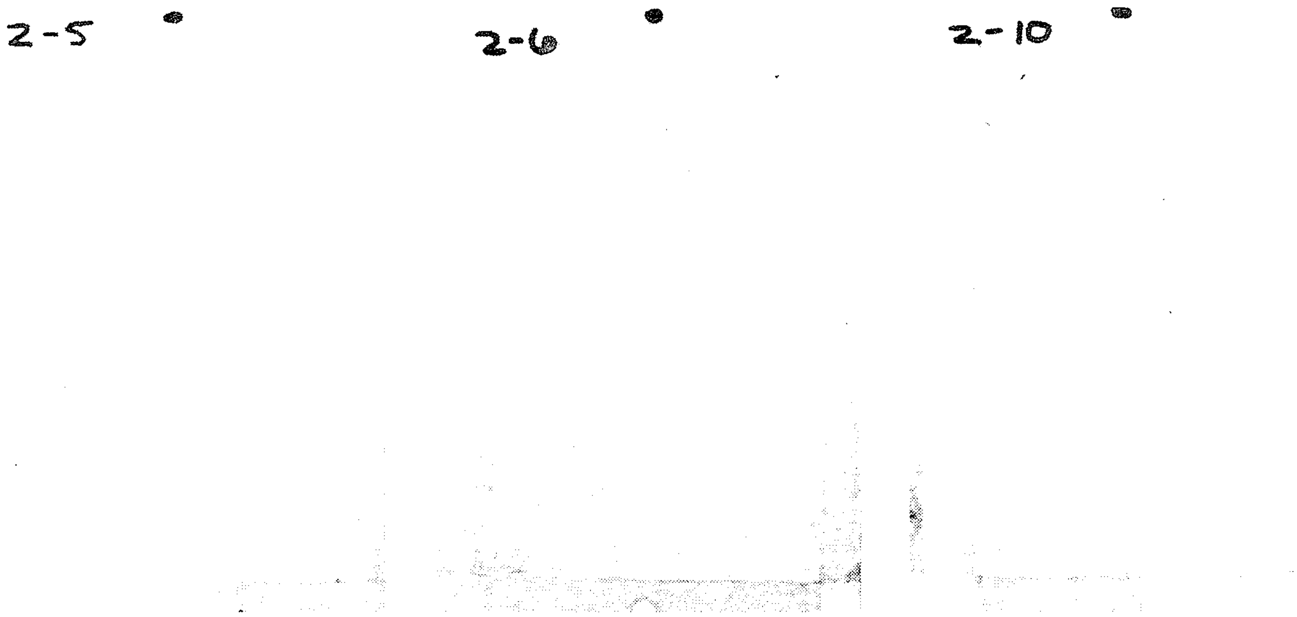

Figure 15. Chromate Treatment (Iridite 14-2) After 3000 Hours of 5% Neutral Salt Fog Testing

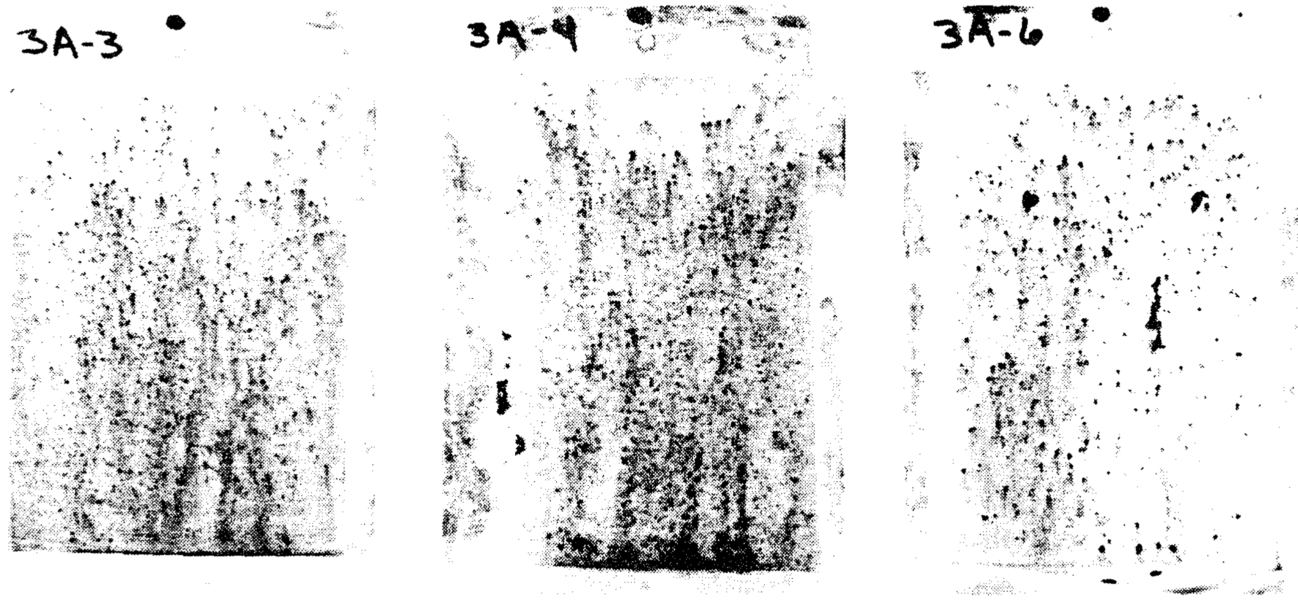

Figure 16. Sanchem-CC Treatment (Sealing Step II Only) After 3000 Hours of 5% Neutral Salt Fog Testing

Figure 17. Sanchem-CC Treatment (Oxide Film Formation Step Followed by Sealing Steps I & II) At Time of Failure (3B-2 1368 Hours; 3B-3 1200 Hours; 3B-7 1368 Hours)

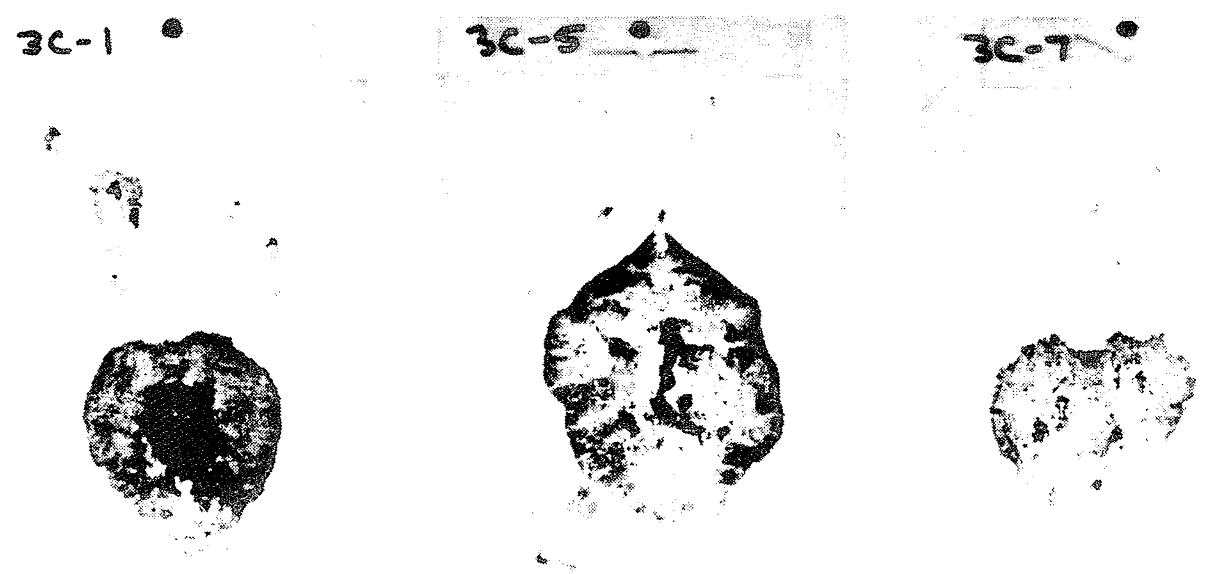

Figure 18. Sanchem-CC Treatment (Oxide Film Formation Step Followed by Sealing Steps I, II & III) At Time of Failure (3C-1 1200 Hours; 3C-5 1296 Hours; 3C-7 1128 Hours)

Figure 19. Alodine 2000 Treatment At Time of Failure (4-1 960 Hours; 4-4 1128 Hours; 4-8 1008 Hours)

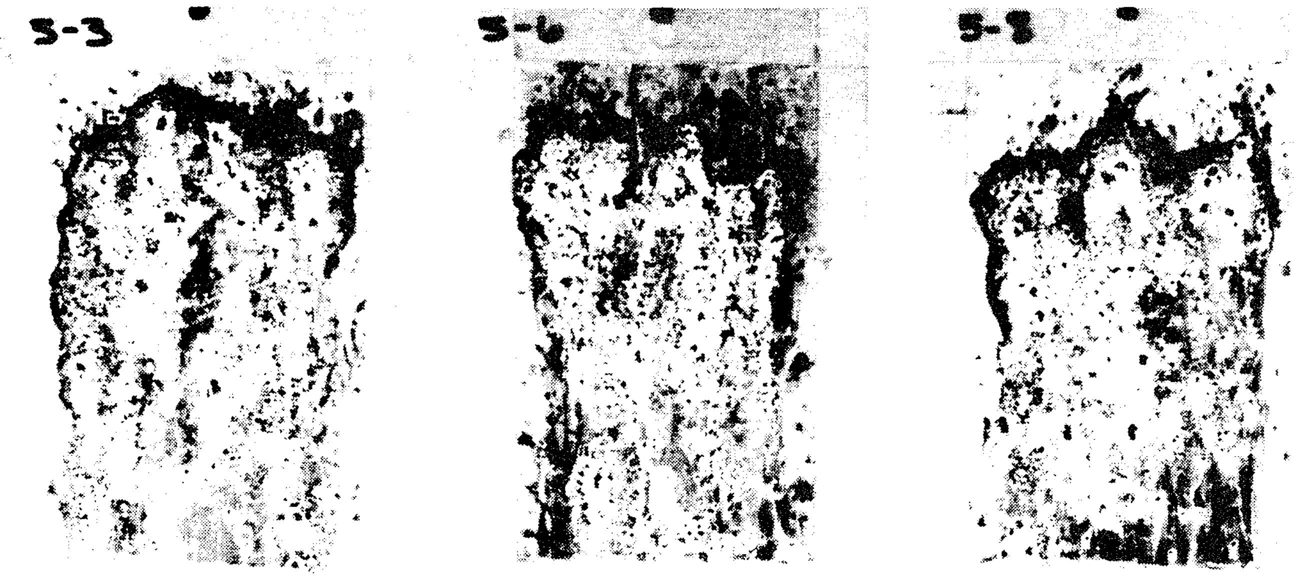

Figure 20. Alodine 90/91 Treatment At Time of Failure (5-3 720 Hours; 5-6 1008 Hours; 5-8 672 Hours)

Figure 21. Permatreat 1001 Treatment At Time of Failure (6-1 1200 Hours; 6-6 840 Hours; 6-7 960 Hours)

Once IVD aluminum depletion products appeared on the panels under Task 2 salt spray testing, depletion progressed much more rapidly compared to the earlier testing under in-house funding. In regard to the phenomenon of IVD aluminum depletion, substrate corrosion or failure, as evidenced by the appearance of red rust, occurs only after enough of the IVD aluminum coating has been depleted such that it will no longer provide galvanic protection. The salt spray performance of Sealing Step II of the Sanchem-CC process, which only involves immersion in a permanganate solution showed that it was much more effective in preventing IVD aluminum depletion than either the complete or near complete Sanchem-CC process (i.e., Oxide Film Formation Step followed by Sealing Steps I, II and III or Oxide Film Formation Step followed by Sealing Steps I and II).

For information purposes, under the previous in-house funded salt spray test, all six of the candidate nonchromated conversion coatings of Task 2 met the goal of 3000 hours of exposure without failure. The only exception was one of the three panels treated with Alodine 2000 which failed at 2184 hours.

The difference in corrosion resistance test results between the initial testing performed under in-house funding and the repeat testing under Task 2 is not without precedence. Previous testing of chromate conversion coatings on IVD aluminum has shown variations, some significant, from one salt spray test to another. Because of this variation, repeat corrosion resistance testing was conducted for the candidate nonchromated conversion coatings under this program.

The reason for the difference in salt spray performance between the two tests noted above is not clearly understood. It may be related to a variation in the roughness or density of the IVD aluminum coating. It may also be due to microscopic flaws in the IVD aluminum coating as a result of slight splattering of the aluminum or some other anomaly during vapor deposition. One possible explanation was that the sheet of AISI 4130 steel used for the salt spray panels had evidence of rust on one side. This rust was removed by grit blasting which is the standard deoxidation procedure prior to IVD aluminum coating. Both sides of each panel were grit-blasted and the side which did not show visible evidence of rust was used as the test side for corrosion resistance testing. Microscopic rust and resultant flaws, even after grit blasting, could have been present on the test side of the corrosion resistance test panels, and this could have caused salt spray performance to be poorer than expected.

3. Supplementary Test 1

A supplementary test was conducted to resolve the discrepancy in salt spray test results between the initial test performed under in-house funding and the Task 2 testing. More specifically, IVD aluminum-coated 4130 steel panels were prepared using steel sheet material totally free of any rust or surface imperfections. The IVD aluminum coating was applied to conform to MIL-C-83488, Class 2 (0.5-mil thick minimum). The actual average thicknesses on the panels ranged from 0.7 mil to 1.0 mil.

The above panels were treated with the Sanchem-CC process consisting of the Oxide Film Formation Step followed by Sealing Steps I and II. These panels, along with chromate conversion-coated control panels which were treated in-house, were then exposed to 5% neutral salt fog testing for 1872 hours. The salt spray performance of the Sanchem treated panels was significantly better than the equivalent panels tested initially under Task 2. Also, of interest, this supplementary test program revealed that the rate of depletion of the IVD aluminum coating was more rapid for the Sanchem treated panels compared to the chromate conversion-coated control panels. This conclusion was based on visually monitoring small areas on the test side of the panels corresponding to the location of hangars during IVD aluminum coating. These areas had little or no IVD aluminum coating present. A more complete description of the testing performed under Supplementary Test 1 is provided by Appendix A. This testing influenced the four candidate nonchromated conversion coatings downselected for testing under Task 4.

4. Supplementary Test 2

Salt spray testing under Task 2 indicated that Sealing Step II of the Sanchem-CC process did not prevent initial pitting, but was very effective in preventing IVD aluminum depletion and subsequent corrosion of the steel substrate. More specifically, dark spots or pits appeared in the coating on these panels after only one day of salt spray testing, but this did not result in failure (i.e., appearance of red rust) after 3000 hours of testing. Also, of significance, IVD aluminum depletion was limited to a few localized areas after 3000 hours of testing.

Based on the above performance of Sealing Step II of the Sanchem-CC process, a supplementary test was initiated aimed at reducing initial pitting of this treatment. IVD aluminum-coated 4130 steel panels were used for this testing. The steel sheet material used for these panels was totally free of any rust or surface imperfections. The IVD aluminum coating was applied to conform to MIL-C-83488, Class 2 (0.5-mil thick minimum). The actual average thickness of the IVD aluminum coating on the various panels ranged from 0.8 to 1.0 mil.

The IVD aluminum-coated steel panels were treated with eight variations of Sealing Step II of the Sanchem-CC process. Most of these variations involved increased temperature and/or immersion time. The different treatment variations, plus chromate conversion-coated control panels, were subsequently subjected to 1152 hours of 5% neutral salt fog testing. The objective of this supplementary testing was achieved in that most of the variations significantly reduced initial pitting. A more complete description of the testing performed under Supplementary Test 2 is provided by Appendix B. This testing influenced the four candidate nonchromated conversion coatings downselected for testing under Task 4.

5. Supplementary Test 3

Near the end of Task 2 salt spray testing for 3000 hours, it was learned that Parker+Amchem had made changes to their Alodine 2000 nonchromated conversion coating. These changes were the result of problems encountered in an 80 gallon scale-up evaluation performed at Boeing in Renton, WA. One change involved addition of an ingredient to the conversion coating solution to improve bath life stability. The other change, which was of greater significance, concerned the use of an organic/inorganic seal rather than the previous completely organic seal. The Parker+Amchem product designation for this improved seal was TD-3095-J.

The original version of Alodine 2000 was evaluated under Task 2 of this program in regard to salt spray performance, contact electrical resistance, and primer adhesion. Since this testing showed it was a promising candidate, a decision was made to repeat this testing for the improved Alodine 2000. The steel sheet material used for the required panels was totally free of any rust or surface imperfections. The IVD aluminum coating applied to the eight panels conformed to MIL-C-83488, Class 2 (0.5-mil thick minimum). The actual average thickness of the IVD aluminum coating on the various panels ranged from 0.84 to 0.98 mil.

After treatment with the improved Alodine 2000, the panels were subjected to 5% neutral salt fog, contact electrical resistance, and primer adhesion testing. All parameters related to this testing were identical to that performed earlier for the original version of Alodine 2000.

In summary, the improved Alodine 2000 treatment met contact electrical resistance and primer adhesion screening requirements. Also, and of significance, it demonstrated vastly improved salt spray performance compared to the original version tested earlier under Task 2. A more complete description of the testing performed under Supplementary Test 3 is provided by Appendix C. This testing influenced the four candidate nonchromated conversion coatings downselected for testing under Task 4.

6. Salt Spray Testing Conclusions

Based on corrosion resistance testing performed under Task 2, and Supplementary Tests 1, 2 and 3, a number of conclusions were made. These are summarized in the paragraphs which follow.

- The Sanchem-CC treated panels tested under Supplementary Test 1 (i.e., Oxide Film Formation Step followed by Sealing Steps I and II), performed significantly better than the equivalent panels tested initially under Task 2. This improved performance is most likely due to the fact that, unlike the Task 2 panels, the steel sheet material used for the Supplementary Test No. 1 panels was free of any rust or surface imperfections.

- Once panels treated with the complete or near complete Sanchem-CC process (i.e., Oxide Film Formation Step followed by Sealing Steps I, II and III or Oxide Film Formation Step followed by Sealing Steps I and II) show evidence of IVD aluminum depletion, the rate of depletion is much more rapid compared to chromate conversion-coated panels. This conclusion is supported by the results of testing under Task 2 and Supplementary Test 1.

- Pits appear in the coating much sooner (i.e., after 24 hours) for panels treated with only Sealing Step II of the Sanchem-CC process compared to panels treated with the complete or near complete Sanchem-CC process. However, although this pitting occurs sooner, Sealing Step II is vastly superior to the complete or near complete Sanchem-CC process in preventing IVD aluminum depletion. This conclusion is supported by the results of testing under Task 2 and Supplementary Tests 1 and 2.

- Modifications to Sealing Step II of the Sanchem-CC process involving increased time and/or temperature of the permanganate solution can significantly reduce the initiation of pits. This conclusion is supported by the results of testing under Supplementary Test 2.

- The improved Alodine 2000 treatment performed significantly better than the original version in regard to 5% neutral salt fog testing. This conclusion is supported by the results of testing under Supplementary Test 3.

7. Touch-Up Investigation

The ability to locally touch up or repair a nonchromated conversion coating and provide the same high level of corrosion protection as the original coating is important. The coating on aircraft parts is often removed during normal assembly operations involving trimming, drilling, and installation of countersinks. Also, the coating on aircraft parts can be scratched during normal aircraft manufacturing operations. Current chromate conversion coatings are available in brush application forms and are routinely used to rework localized areas. This same capability is required for nonchromated conversion coatings.

A test program was conducted to evaluate the effectiveness of various conversion coatings in regard to touch-up. The materials evaluated under this program are summarized below:

- Betz MetChem Permatreat 684 (non-rinse chromate conversion coating)
- Parker+Amchem Non-Rinse Chromate Conversion Coating (to be applied using special touch-up applicator)
- Parker+Amchem Bonderite 1455
- Permanganate Solution used for Sealing Step II of the Sanchem-CC Process, except at room temperature (double concentration used and Cab-O-Sil added as a thickening agent to provide retention on vertical surfaces).
- Chromate Conversion Coating Iridite 14-2 Suitable for Brush Application (Control)

Conversation with the manufacturers of the nonchromated conversion coatings evaluated under the Task 2 screening tests revealed that none of their products had been developed or optimized for touch-up. Along this line, the most promising Task 2 candidates (i.e., the Sanchem variations and Alodine 2000) require processing at elevated temperatures and it is unlikely that any of these would be practical for touch-up. A possible long shot exception, however, was the permanganate solution of Sealing Step II of the Sanchem-CC process. On the negative side, it would have to be used at room temperature rather than the recommended 140°F. On the positive side, the concentration could be doubled. Also, a thickening agent, such as Cab-O-Sil, could be added to promote retention on vertical surfaces. Based on the above, then, it would appear that chromate conversion coating type materials might be the best approach for coating touch-up, particularly since such a small amount of material is required. This approach is further strengthened by the fact that non-rinse chromate conversion coatings are available which would further minimize any hazardous waste. Bonderite 1455 is a combination organic/inorganic material with the consistency of water. According to Parker+Amchem, it has demonstrated the ability to provide corrosion protection when applied to cold rolled steel, aluminum and galvanize. Parker+Amchem suggested that it might be worthy of investigation as a touch-up material for IVD aluminum coatings.

IVD aluminum-coated alloy steel panels were used for the touch-up investigation. Initially, three small strips of a vinyl electroplating tape (i.e., 3M Tape No. 470), each 0.125 inch wide x 1.0 inch long, were applied at different locations on each of the IVD aluminum-coated steel panels to be used in the investigation. The panels were then treated using Sealing Steps II and III of the Sanchem-CC process. This treatment was selected as the final, best treatment under Task 6 of this development program. After treatment of the panels, the strips of tape were removed to provide three areas on each panel which were not conversion-coated. Next, three scratches were made at different locations on each of the panels. All were made such that they were through the conversion coating and into the IVD aluminum coating. The non-conversion-coated areas and scratches on each panel were then touched up with the candidate touch-up materials noted previously.

Two panels were touched up for each of the candidate touch-up materials, including the Iridite 14-2 control material. Figure 22 shows the panels touched up with Bonderite 1455 which are typical of all the panels touched-up.

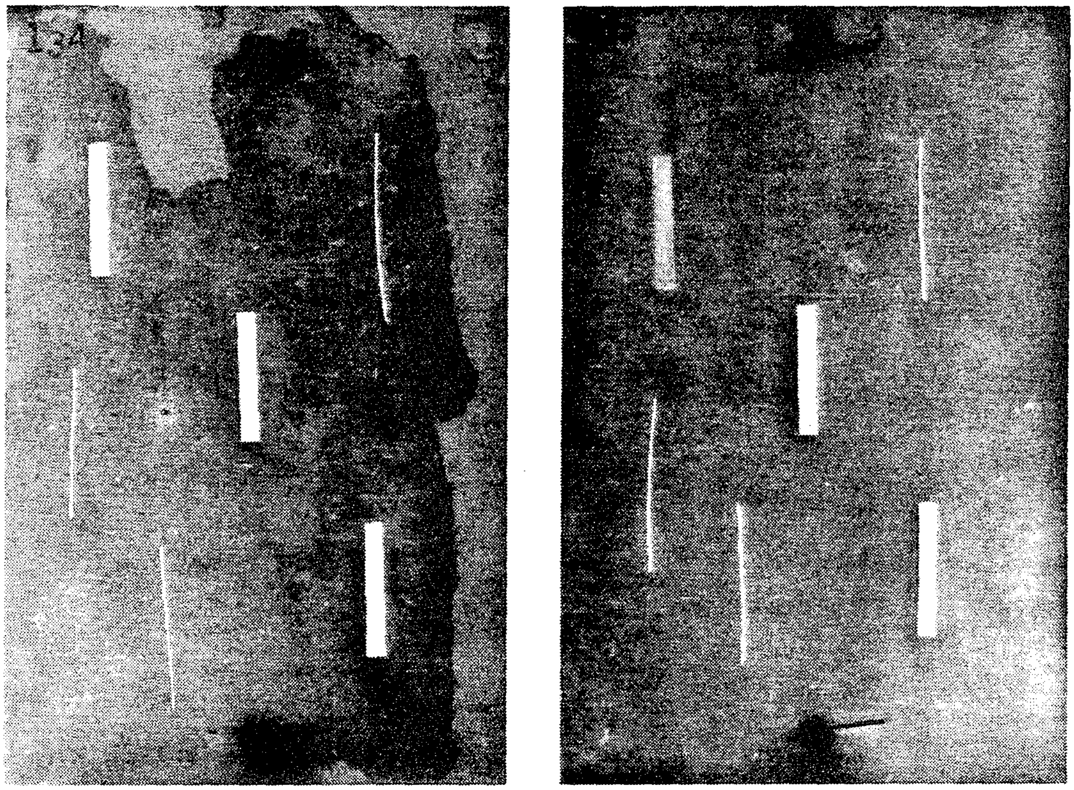

Figure 22. Panels Touched Up With Bonderite 1455

The touched-up panels were exposed to 5% neutral salt fog testing for 336 hours (two weeks). Basically, all of the touch-up materials provided acceptable performance, although some minor differences could be noted. As expected, the areas touched-up with Iridite 14-2 were totally free of any pits or other anomalies. This was also the case for the panels touched-up with Permatreat 684 non-rinse chromate conversion coating. The panels touched-up with Bonderite 1455, which are shown in Figure 23 at the conclusion of testing, also compared favorably with the chromate conversion-coated controls. The panels treated with Parker+Amchem's non-rinse chromate conversion coating, on the other hand, showed a few pits in the touched-up areas, but they were very small. The least acceptable, in regard to pitting, were the panels touched-up with the permanganate solution at double concentration. This performance was not totally surprising as the solution did not result in any visible change to the areas touched-up after it was wiped from the surface of the panel. Based on the above discussion, the candidate touch-up materials were ranked as noted below. The most desirable material is listed first.

- Iridite 14-2
  Permatreat 684
- Bonderite 1455
- Parker+Amchem Non-Rinse Chromate Conversion Coating
- Permanganate Solution

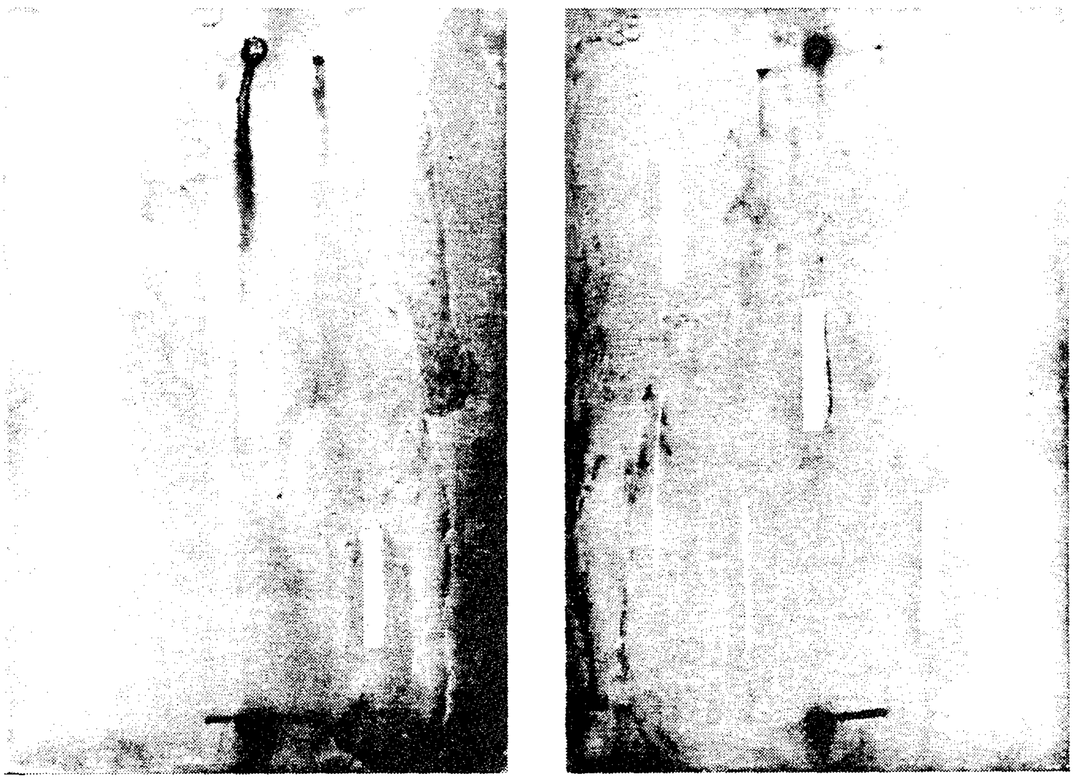

Figure 23. Panels Touched Up With Bonderite 1455 After 14 Days Exposure to 5% Neutral Salt Fog

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SECTION IV
TASK 3 – E-COAT APPLIED PRIMER

A. OBJECTIVE

The objective of Task 3 was to apply primer by electrodeposition to IVD aluminum-coated panels which had been treated with the nonchromated conversion coatings which passed the Task 2 screening tests. These E-coat panels were then to be evaluated in regard to primer adhesion and corrosion resistance.

B. OVERVIEW

The above activity was proposed as an optional task under the program and was priced separately. The Air Force elected not to fund this portion of the program.

SECTION V
TASK 4 – EXTEND DATABASE WITH ACCEPTED CANDIDATE COATINGS

A. OBJECTIVE

The objective of Task 4 was to select the four most promising nonchromated conversion coatings based on Task 2 screening tests, and then conduct expanded corrosion resistance testing for those treatments using IVD aluminum-coated alloy steel panels.

B. OVERVIEW

Using IVD aluminum-coated alloy steel panels, the four most promising candidate nonchromated conversion coatings based on Task 2 screening tests were subjected to 5% neutral salt fog testing per ASTM B117 for 8000 hours, sulfur dioxide salt fog testing for 500 hours, and outdoor exposure for 58 weeks. All of the candidate treatments completed 8064 hours of 5% neutral salt fog testing without failure (i.e., no appearance of red rust). Also, they all completed 504 hours of sulfur dioxide salt fog testing without failure, except for one panel for one of the treatments. Finally, all of the candidate nonchromated conversion coatings completed 58 weeks of outdoor exposure in St. Louis, MO without any evidence of any significant deterioration such as pitting, corrosion products, or IVD aluminum depletion.

C. DISCUSSION

1. Downselection to Four Candidate NonChromated Conversion Coatings

As specified in the Technical/Management Proposal for this development program, four nonchromated conversion coatings were selected for evaluation under Task 4 testing. Their selection was based on the results of Task 2 testing, and the corrosion resistance test results under Supplementary Tests 1, 2 and 3. The four candidates are listed below.

- Sealing Step II of the Sanchem-CC process, except at increased temperature (i.e., immersion in a permanganate solution at 170°F for three minutes).
- Sealing Steps II and III of the Sanchem-CC process (i.e., immersion in a permanganate solution at 140°F for three minutes followed by immersion in a potassium silicate solution at 200°F for one minute).
- PERMATREAT 1001
- Improved Alodine 2000

The Sanchem variations selected for continued testing under Task 4 do not include the Oxide Film Formation Step and Sealing Step I which are necessary steps for corrosion resistance when dealing with the bare aluminum alloys. These steps were not included based on salt spray test results under Task 2 and Supplementary Test 1. More specifically, the complete or near complete Sanchem-CC process (i.e., Oxide Film Formation Step followed by Sealing Steps I, II, and III or Oxide Film Formation Step followed by Sealing Steps I and II) did provide good resistance to initial pitting. However, more important, IVD aluminum depletion tended to begin sooner at anomalies such as pits and thin IVD aluminum-coated areas for the complete or near complete Sanchem-CC process. Furthermore, once IVD aluminum depletion began for the complete or near complete Sanchem-CC process, it progressed very rapidly with increased exposure time compared to the chromate conversion-coated panels. This behavior was not the case for panels treated with Sealing Steps II and III of the Sanchem-CC process or with only Sealing Step II of the Sanchem-CC process.

Sealing Step II of the Sanchem-CC process, except at increased temperature (i.e., 170°F), was selected for continued testing based on the results of salt spray testing under Supplementary Test 2. More specifically, testing under this supplementary test demonstrated that variations to Sealing Step II of the Sanchem-CC process, particularly those involving increased time and/or temperature, reduced the incidence of pitting compared to the standard Sealing Step II at 140°F.

The Sanchem treatment variation utilizing both Sealing Steps II and III was selected for further evaluation since this combination, under Supplementary Test 2, was very effective in reducing initial pitting. This performance supported the theory that the Sealing Step III potassium silicate solution offered enhanced sealing of the columnar or porous IVD aluminum coating.

2. Panel Material and Preparation

The panels used for Task 4 testing were totally free of any rust or surface imperfections. They were AISI 4130 steel, 4 x 6 x 0.040 inch thick, conforming to MIL-S-18729 which is the material required by the IVD aluminum military specification (MIL-C-83488) for corrosion resistance testing. Prior to IVD aluminum coating, the panels were vapor degreased and then grit blasted. The IVD aluminum coating on the side of the panels to be tested was applied to conform to MIL-C-83488, Class 1 (1.0-mil thick minimum).

The panels were IVD aluminum-coated in sets of thirty. One set of 30 panels was IVD aluminum-coated for each of the four downselected nonchromated conversion coatings and for the chromate conversion-coated controls. The average thickness of the IVD aluminum coating on each group of the 30 panels is noted below.

- Chromate Conversion Coating — 1.2 mil
- Sealing Step II of the Sanchem-CC process — 1.1 mil
- Sealing Steps II and III of the Sanchem-CC process — 1.1 mil
- PERMATREAT 1001 — 1.2 mil
- Improved Alodine 2000 — 1.1 mil

In accordance with standard practice, the IVD aluminum coating on all of the test panels was glass bead peened prior to application of the various conversion coatings. The conversion coatings were applied to the panels within 24 hours after the peening operation. Figure 24 shows a representative panel treated with Sealing Steps II and III of the Sanchem-CC process prior to testing. It is typical of the panels prepared for all of the conversion coatings tested.

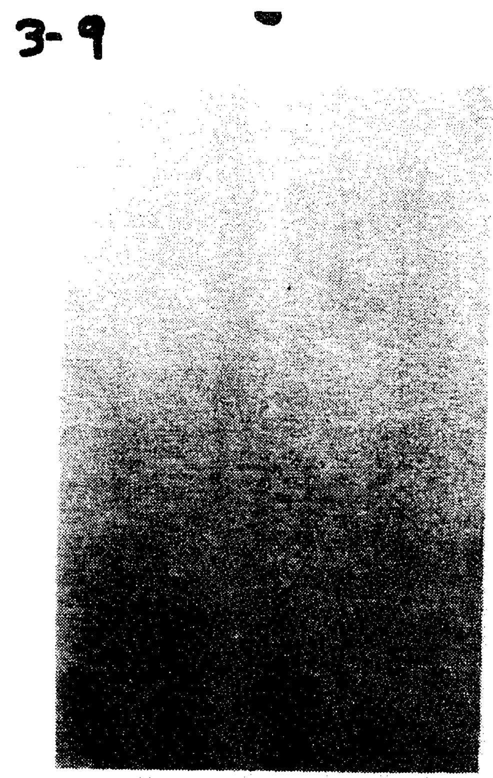

Figure 24. Sealing Steps II & III of the Sanchem-CC Process (Prior to Test)

3. Five Percent Neutral Salt Fog Testing for 8000 Hours

Ten panels were subjected to 5% neutral salt fog testing for each of the four candidate nonchromated conversion coatings and for the chromate conversion coating. Figures 25 – 29 show representative panels for each of these treatments after 672 hours of exposure. This time period is noteworthy because the salt spray requirement of MIL-C-83488 for a Class 1 IVD aluminum coating (i.e., a 1.0-mil thick minimum coating) without failure is 672 hours. Failure of IVD aluminum-coated steel is considered to occur at the first appearance of red rust. None of the panels showed any evidence of red rust after 672 hours of salt spray testing.

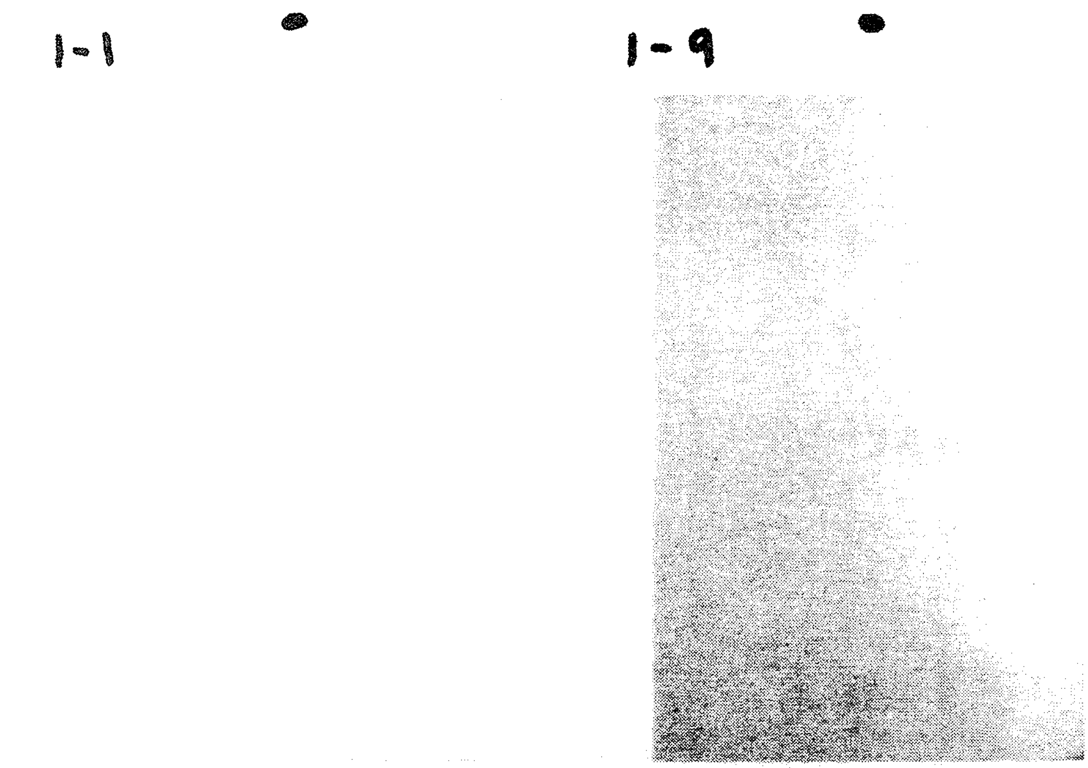

Figure 25. Chromate Conversion Coating (After 672 Hours of 5% Neutral Salt Fog Testing)

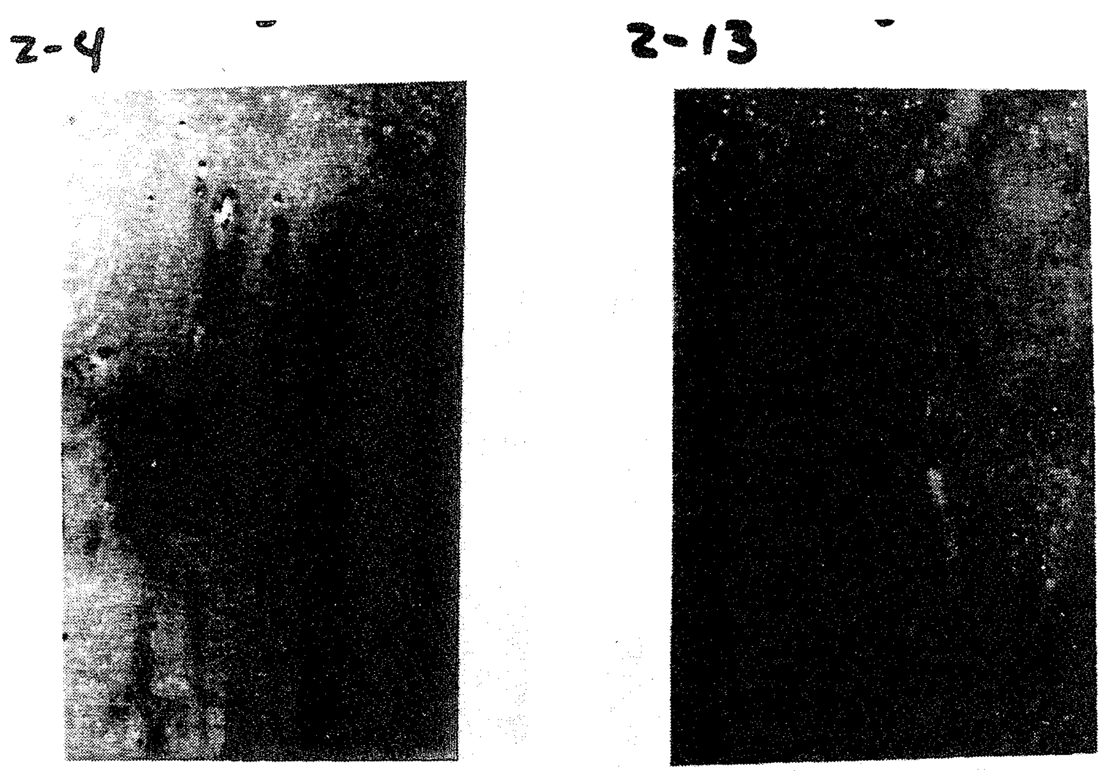

Figure 26. Sealing Step II of the Sanchem-CC Process (After 672 Hours of 5% Neutral Salt Fog Testing)

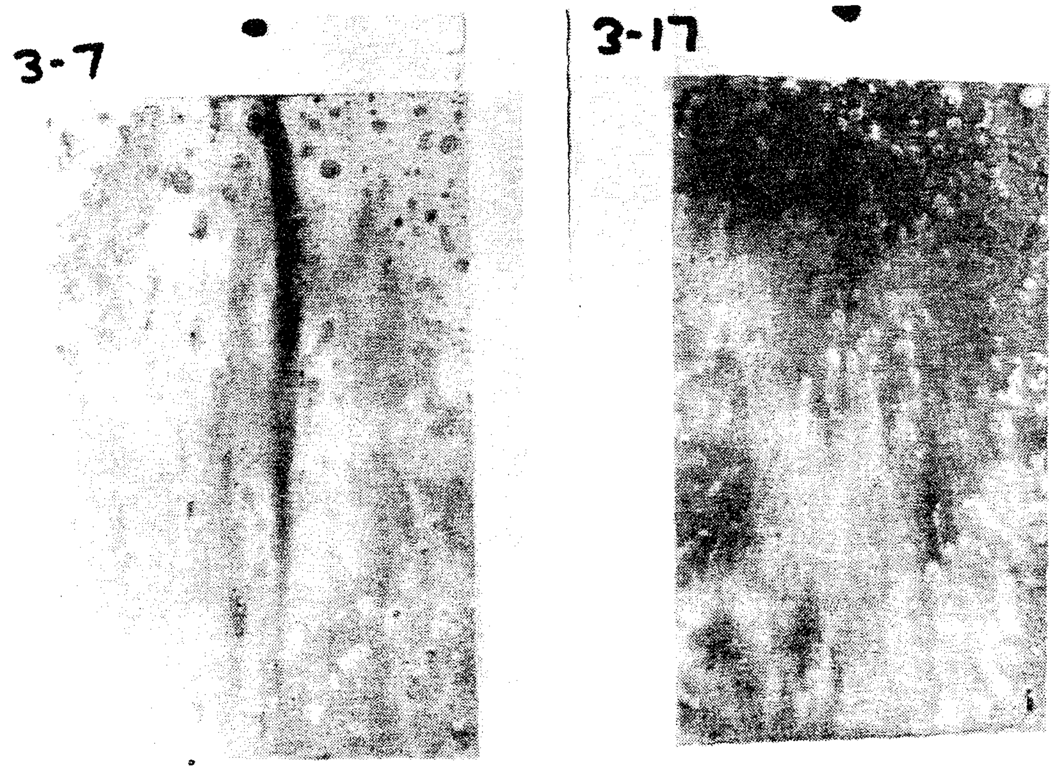

Figure 27. Sealing Steps II & III of the Sanchem-CC Process
(After 672 Hours of 5% Neutral Salt Fog Testing)

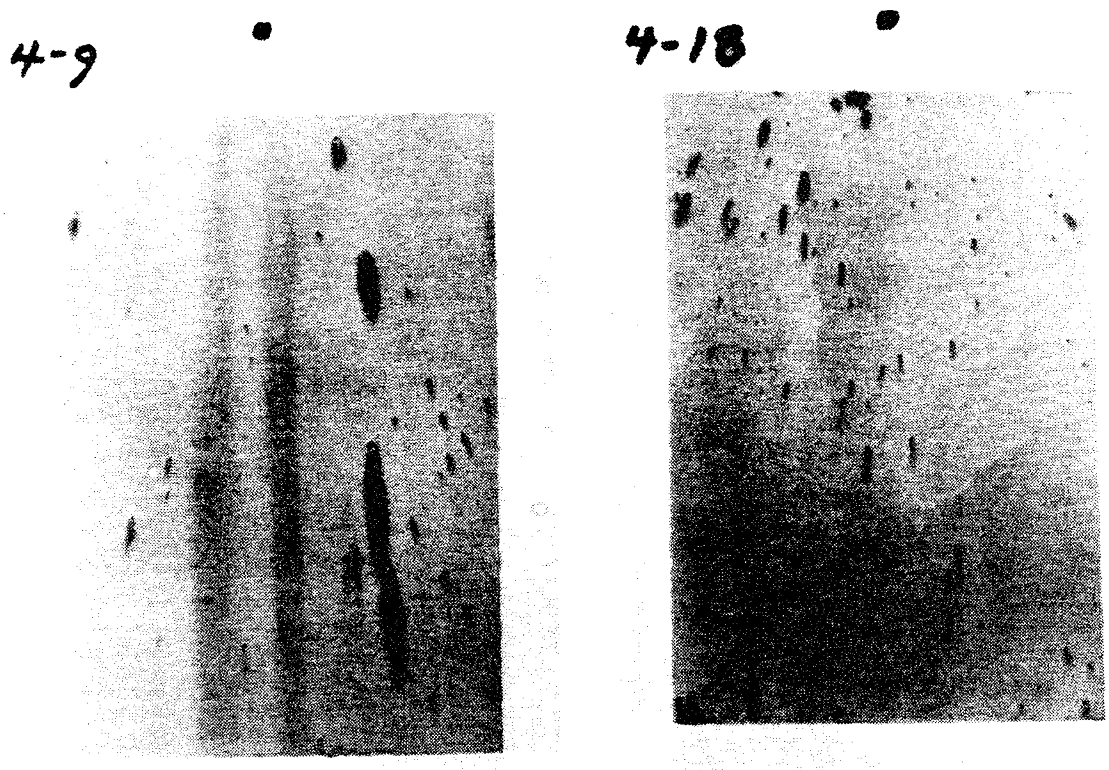

Figure 28. Permatreat 1001 (After 672 Hours of 5% Neutral Salt Fog Testing)

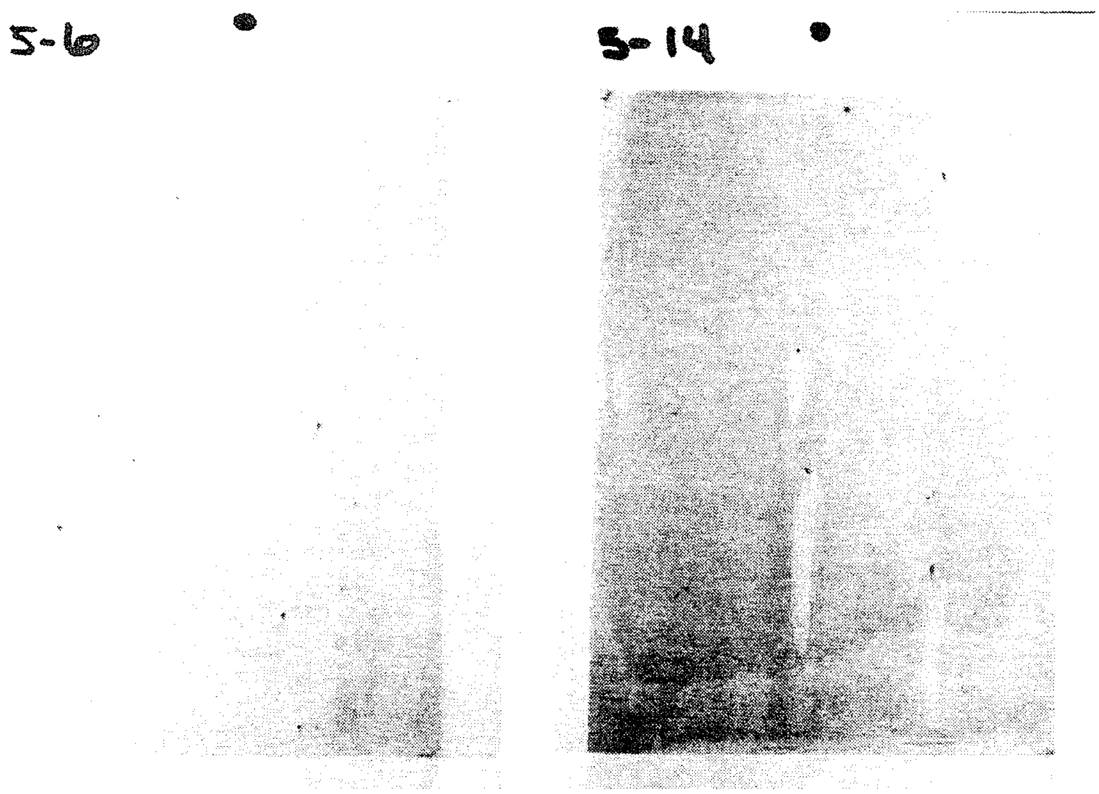

Figure 29. Alodine 2000 (After 672 Hours of 5% Neutral Salt Fog Testing)

The amount of pitting on some of the panels treated with Sealing Step II of the Sanchem-CC process (Reference Figure 26) after 672 hours of testing is somewhat surprising, based on the results of the Sealing Step II optimization studies conducted under Supplementary Test No. 2 of Task 2. The cause of this pronounced pitting on some of these later panels treated with Sealing Step II of the Sanchem-CC process is not known.

Figures 30 – 34 show the same representative test panels noted above, except after 4032 hours of 5% neutral salt fog testing. As was the case after 672 hours of testing, there was still no evidence of failure (i.e., no appearance of red rust) at this halfway point in testing.

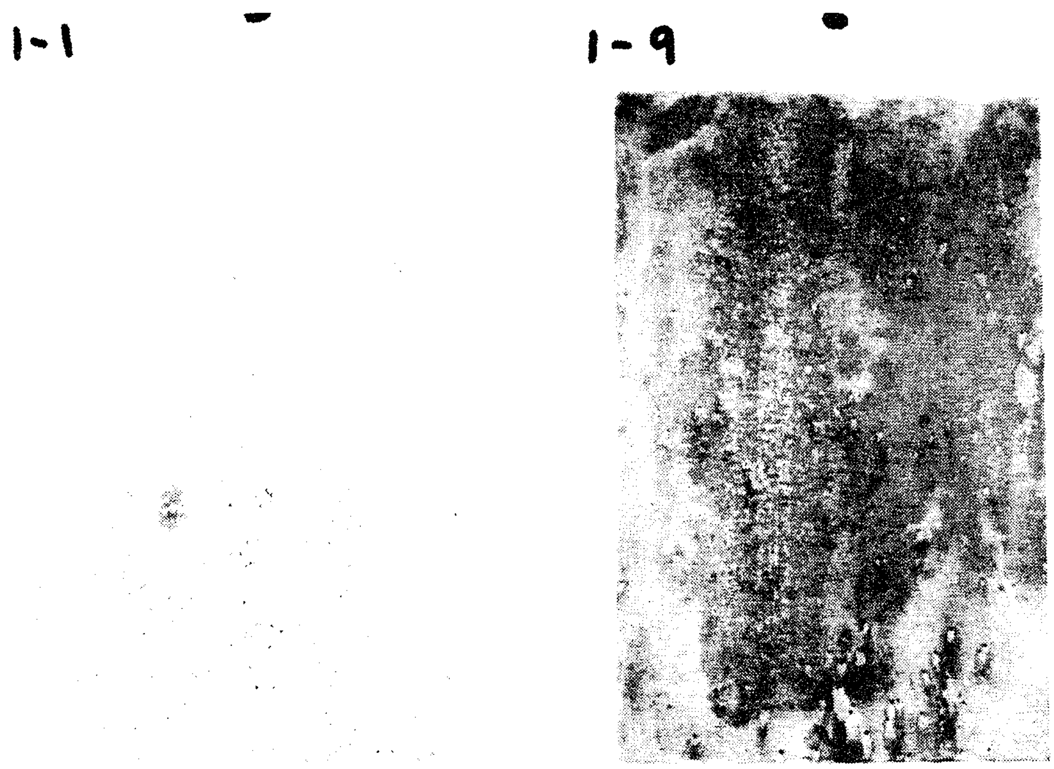

Figure 30. Chromate Conversion Coating (After 4032 Hours of 5% Neutral Salt Fog Testing)

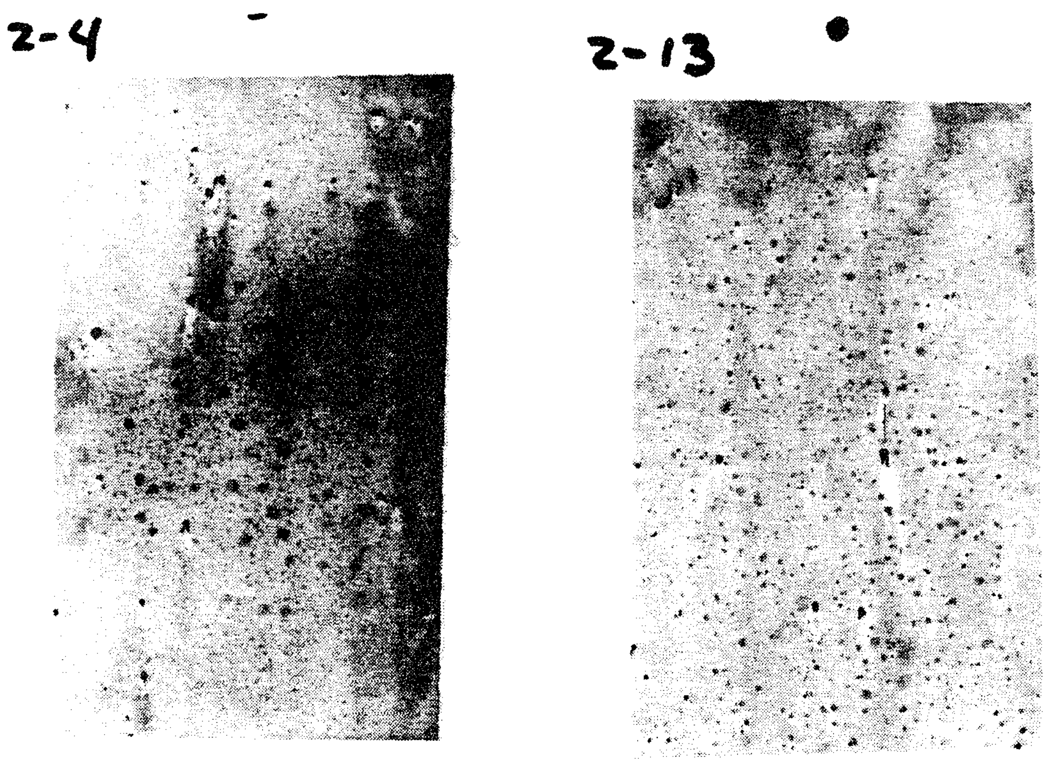

Figure 31. Sealing Step II of the Sanchem-CC Process (After 4032 Hours of 5% Neutral Salt Fog Testing)

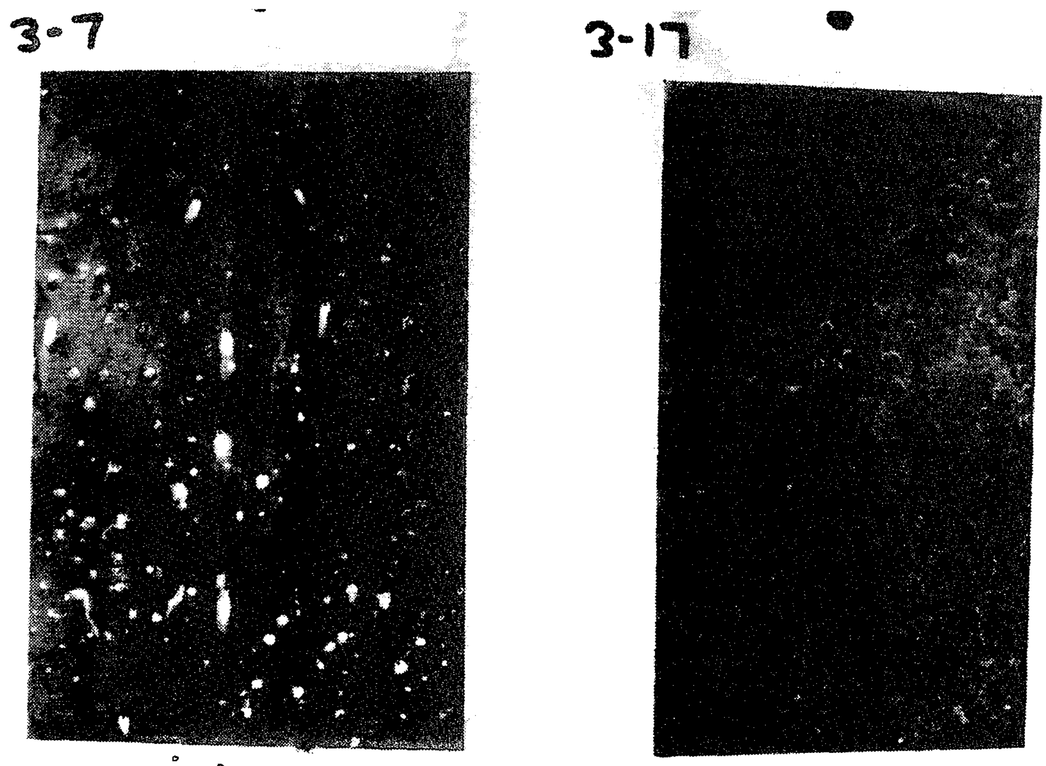

Figure 32. Sealing Steps II & III of the Sanchem-CC Process
(After 4032 Hours of 5% Neutral Salt Fog Testing)

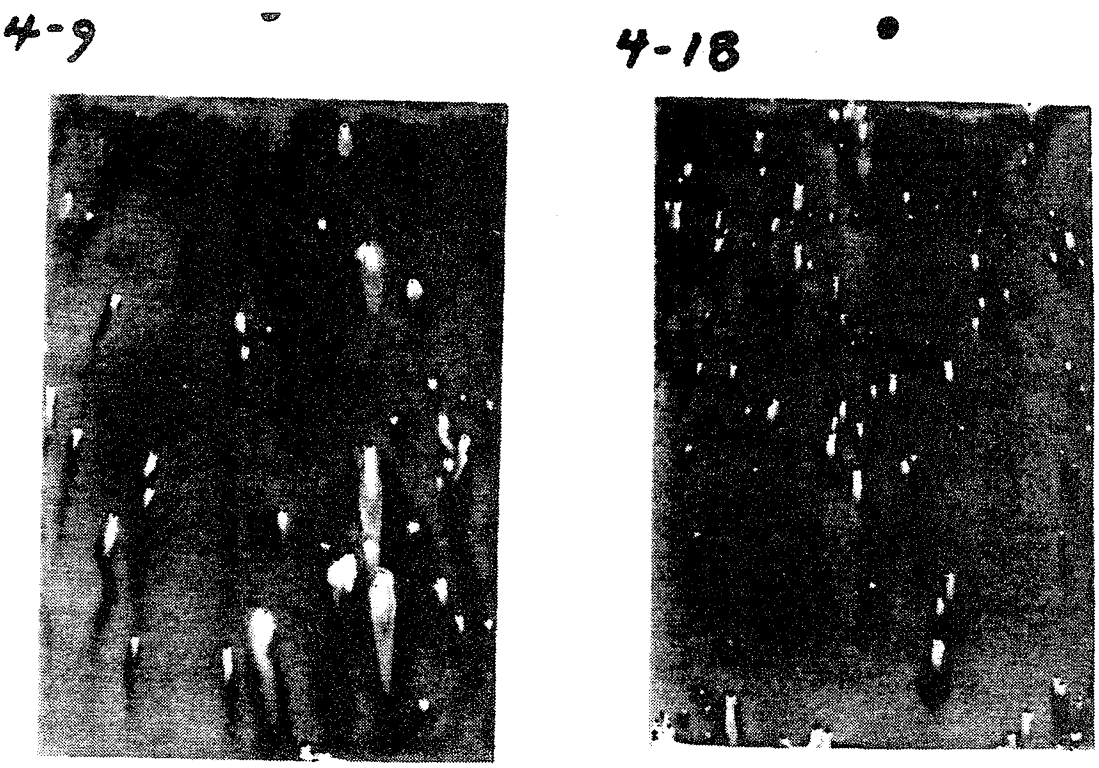

Figure 33. Permatreat 1001 (After 4032 Hours of 5% Neutral Salt Fog Testing)

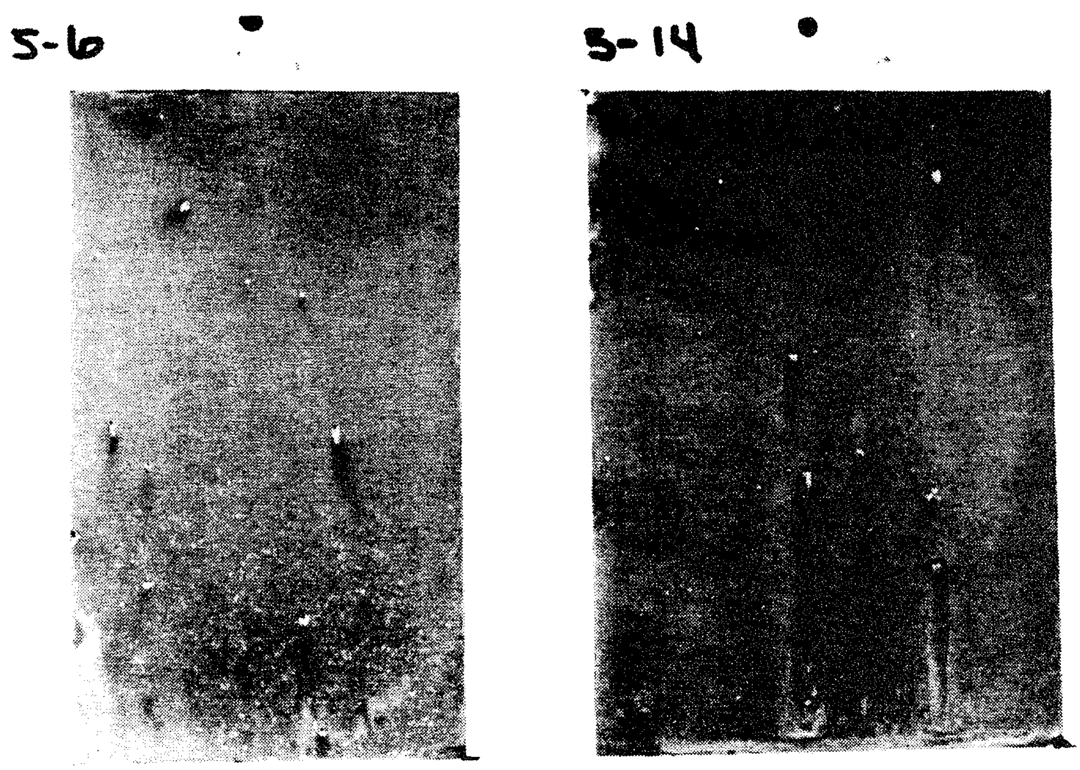

Figure 34. Alodine 2000 (After 4032 Hours of 5% Neutral Salt Fog Testing)

From the photographs of the panels after 4032 hours of 5% salt spray testing, one might surmise that there are significant differences in performance based on pit size and color changes associated with the pits. Along this line, the white color associated with the pits on many of the panels represents the beginning of IVD aluminum depletion. In spite of this and of significance, none of the panels had any gel type corrosion products and all were smooth to the touch.

Figures 35 – 59 show all of the panels at the end of 5% neutral salt fog testing after 8064 hours of exposure. None of the panels show any evidence of failure (i.e., no appearance of red rust). Also, none of the panels had any gel-type corrosion products and all were smooth to the touch.

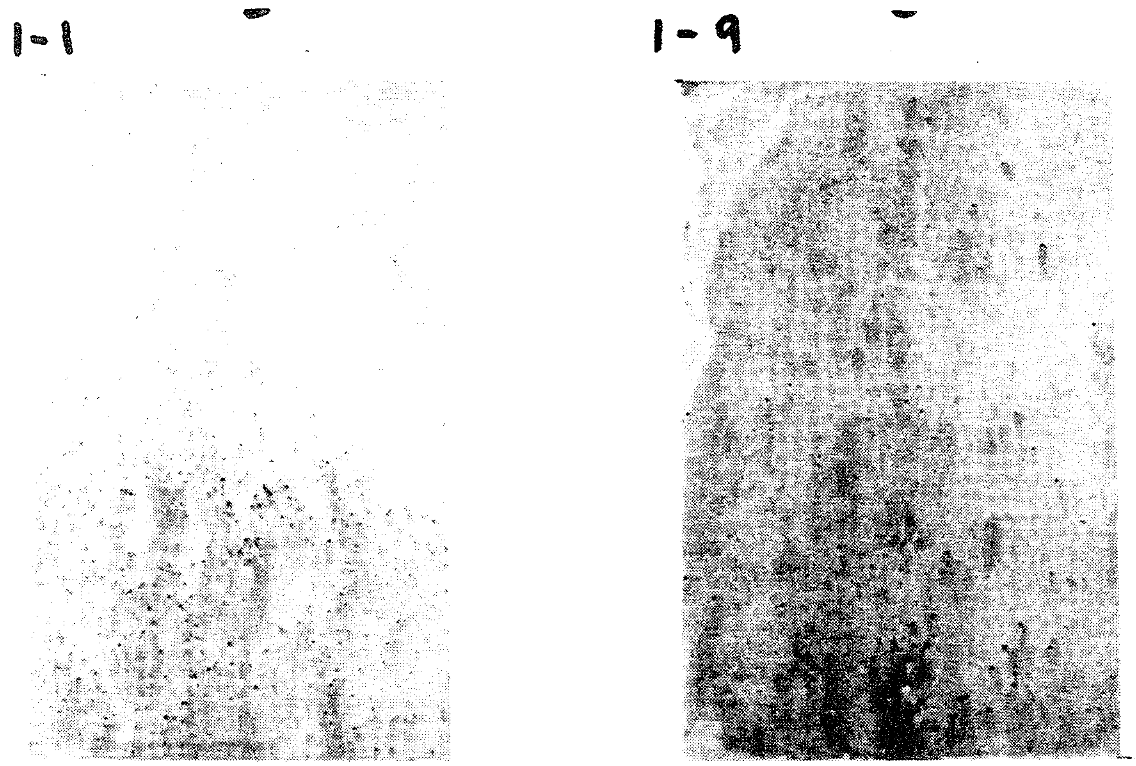

Figure 35. Chromate Conversion Coating (After 8064 Hours of 5% Neutral Salt Fog Testing)

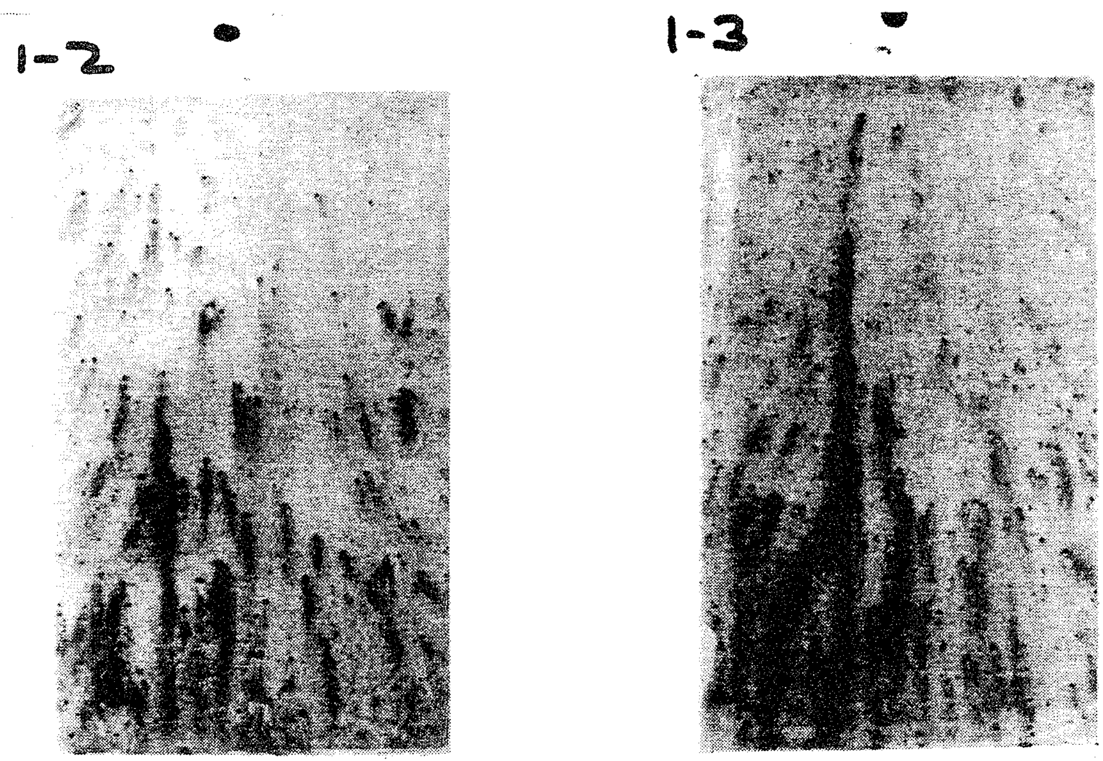

Figure 36. Chromate Conversion Coating (After 8064 Hours of 5% Neutral Salt Fog Testing)

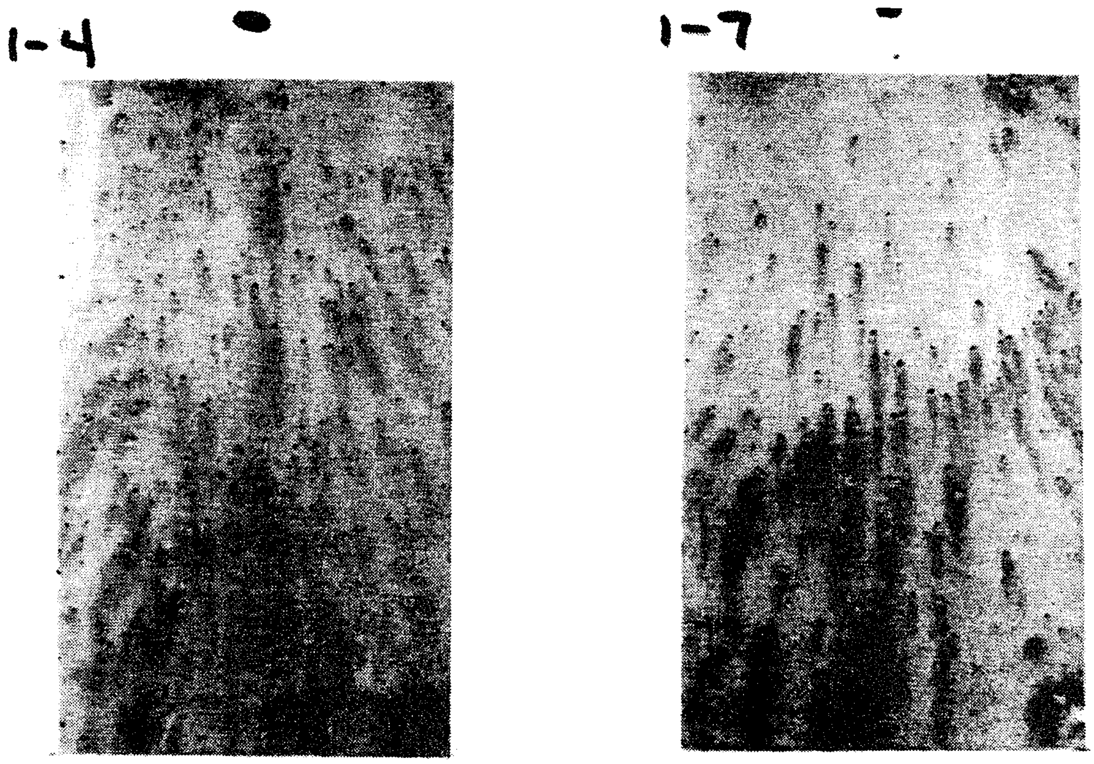

Figure 37. Chromate Conversion Coating (After 8064 Hours of 5% Neutral Salt Fog Testing)

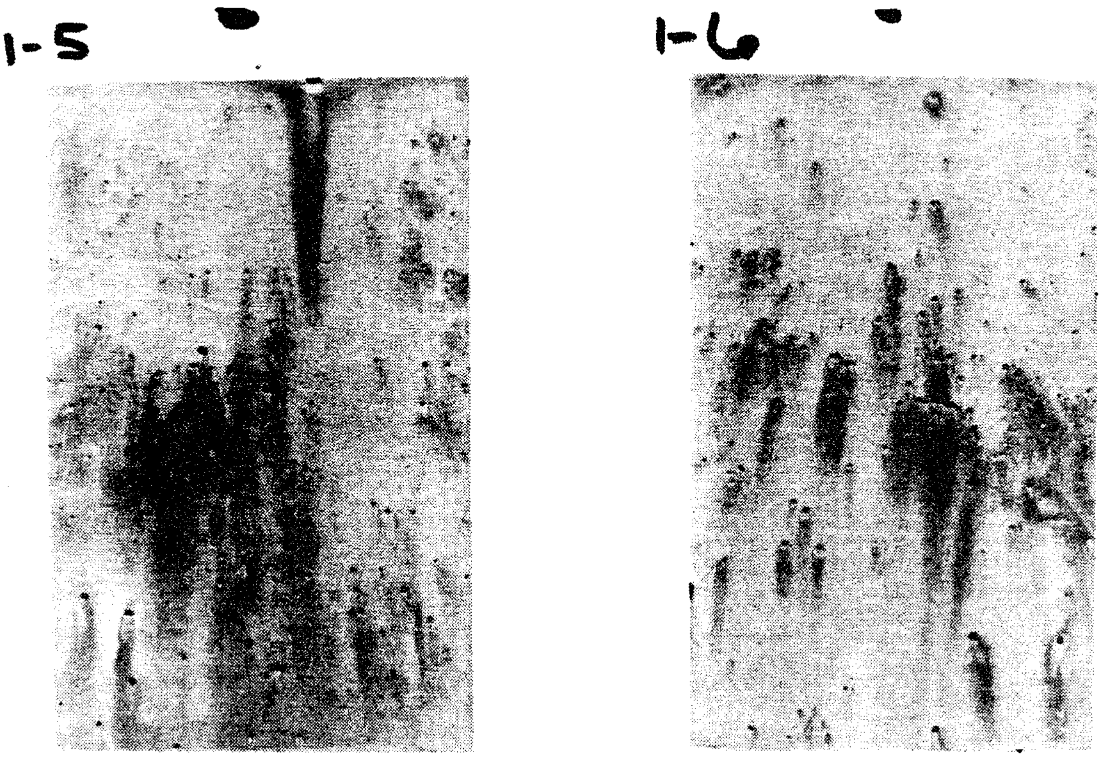

Figure 38. Chromate Conversion Coating (After 8064 Hours of 5% Neutral Salt Fog Testing)

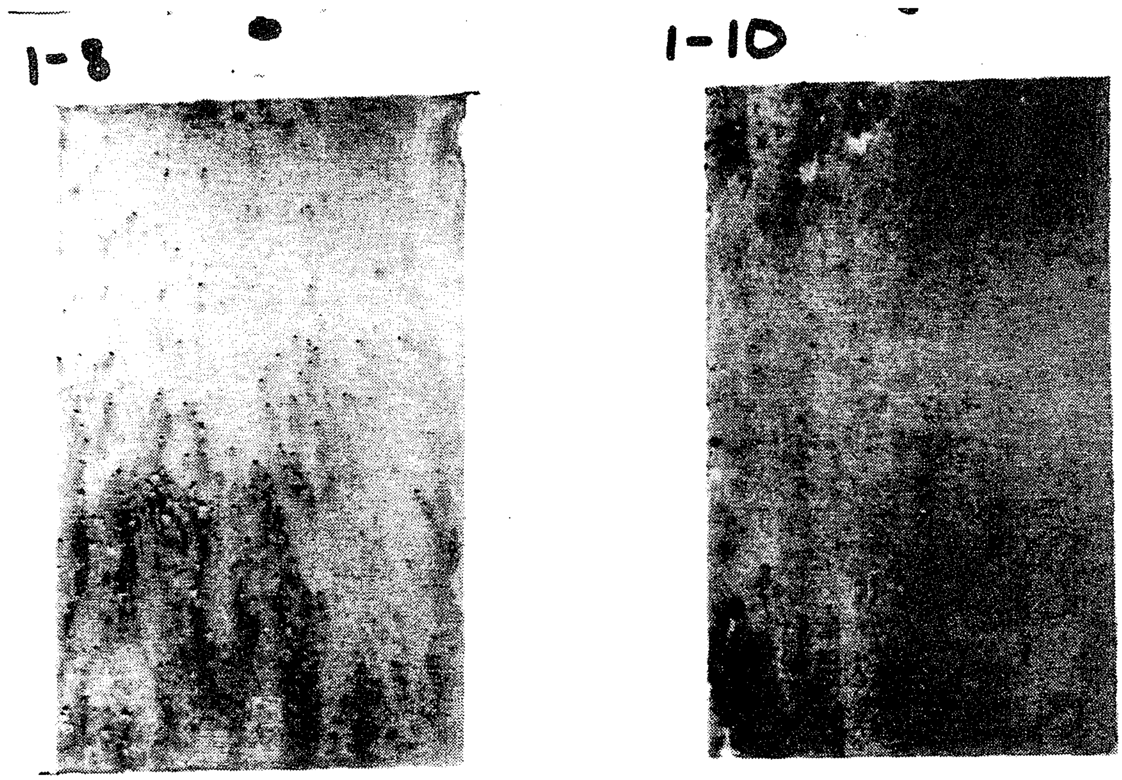

Figure 39. Chromate Conversion Coating (After 8064 Hours of 5% Neutral Salt Fog Testing)

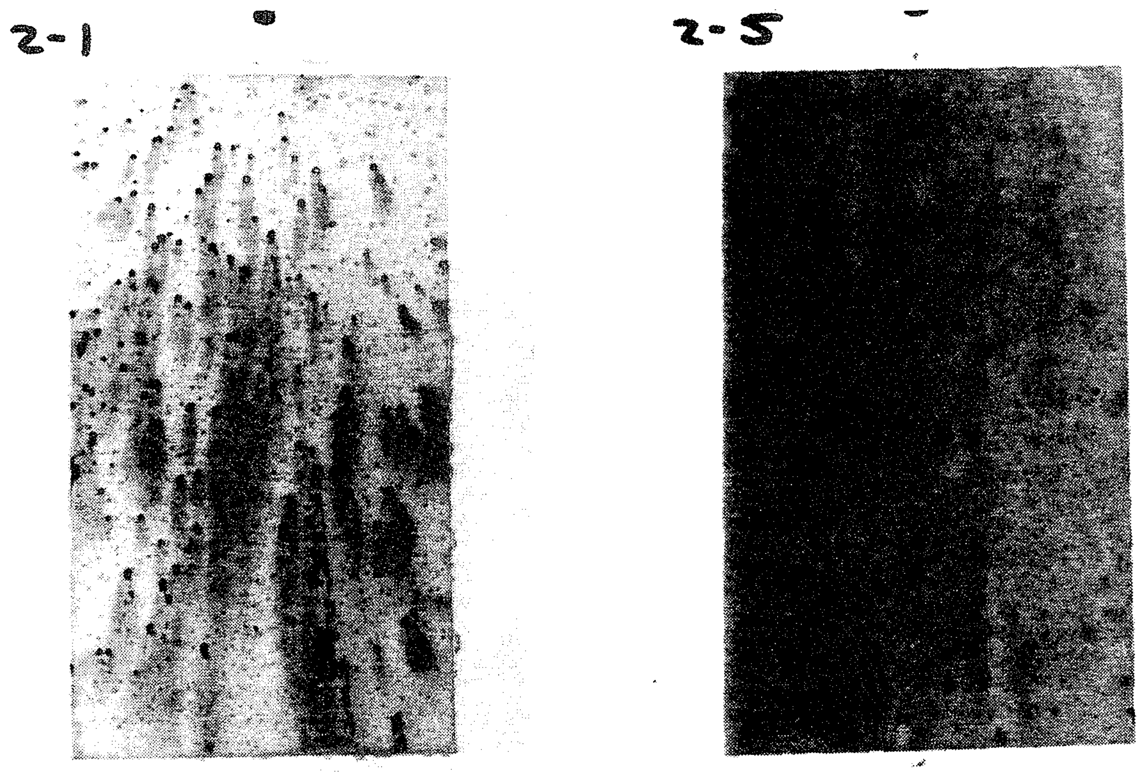

Figure 40. Sealing Step II of the Sanchem-CC Process
(After 8064 Hours of 5% Neutral Salt Fog Testing)

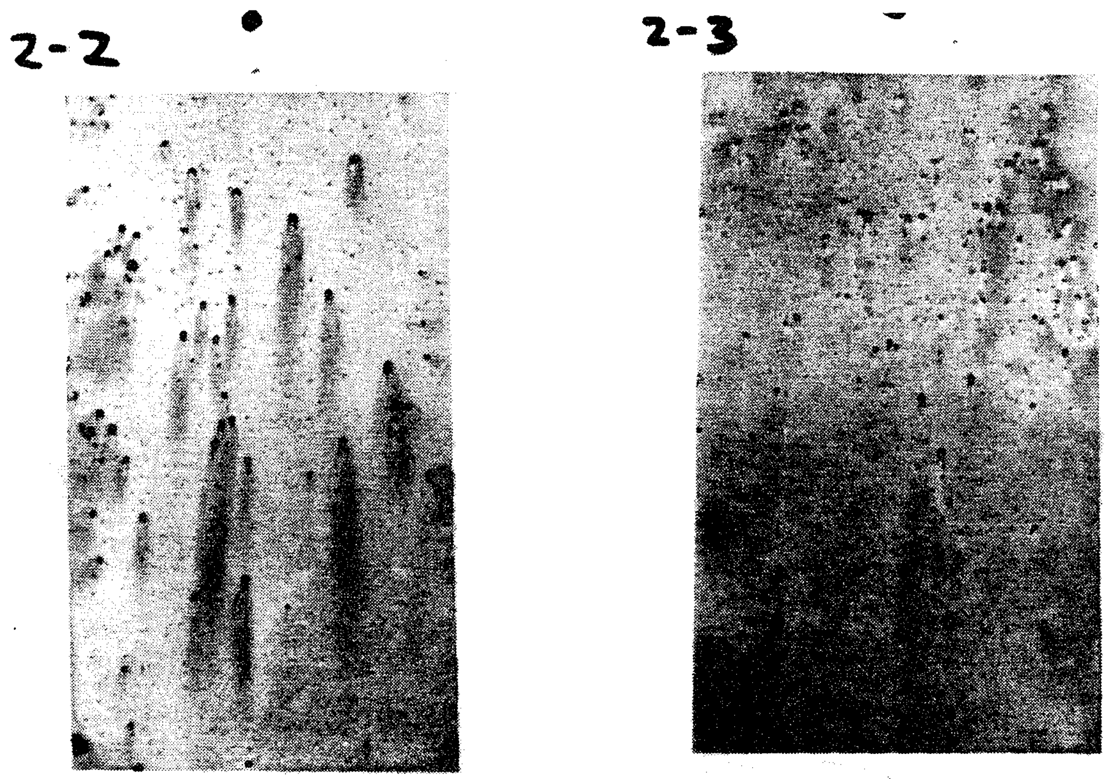

Figure 41. Sealing Step II of the Sanchem-CC Process (After 8064 Hours of 5% Neutral Salt Fog Testing)

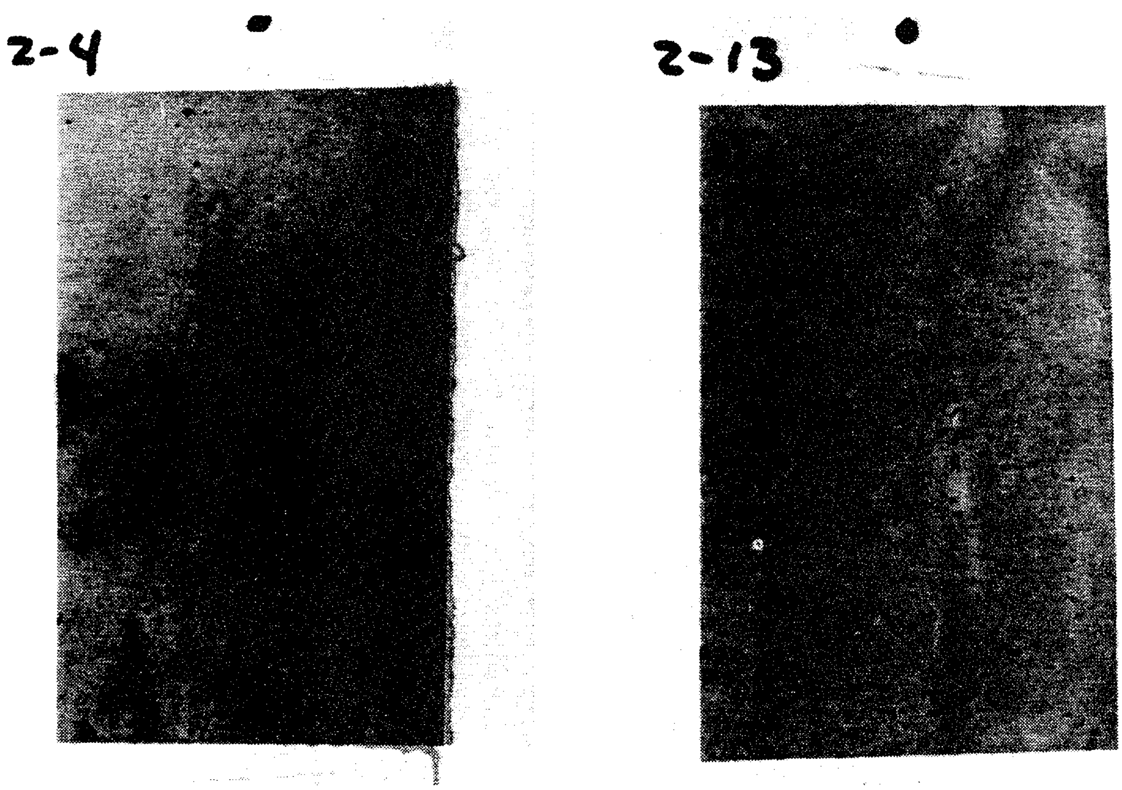

Figure 42. Sealing Step II of the Sanchem-CC Process (After 8064 Hours of 5% Neutral Salt Fog Testing)

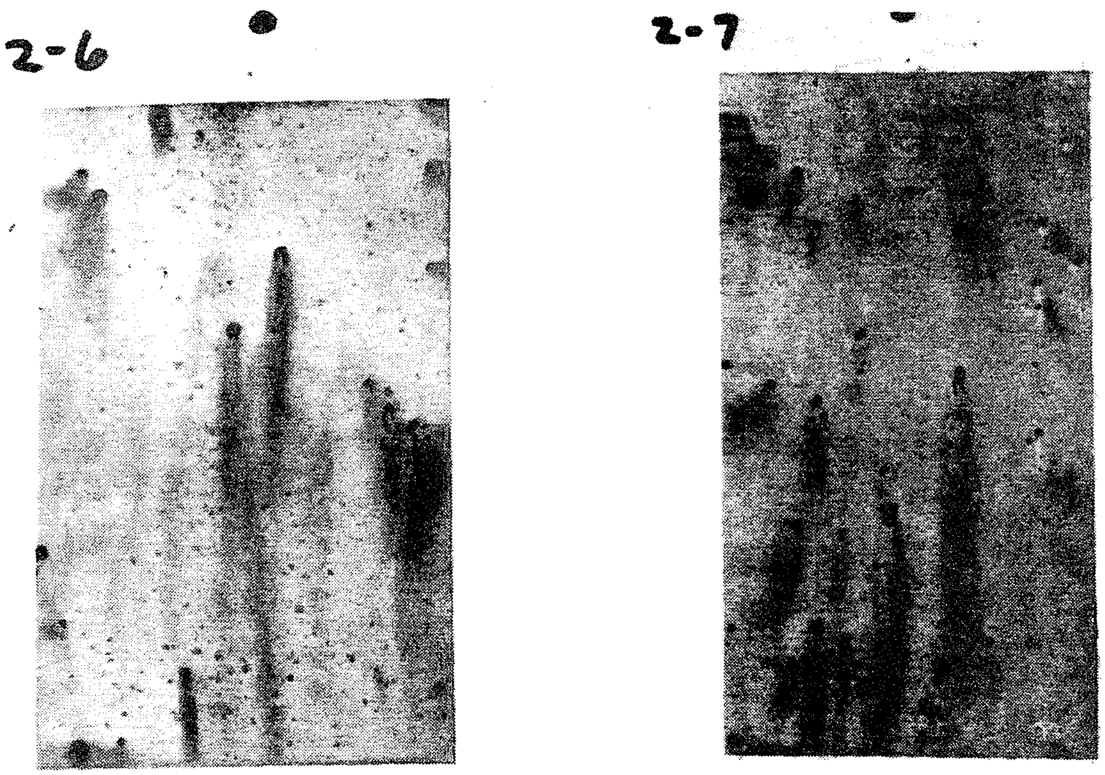

Figure 43. Sealing Step II of the Sanchem-CC Process
(After 8064 Hours of 5% Neutral Salt Fog Testing)

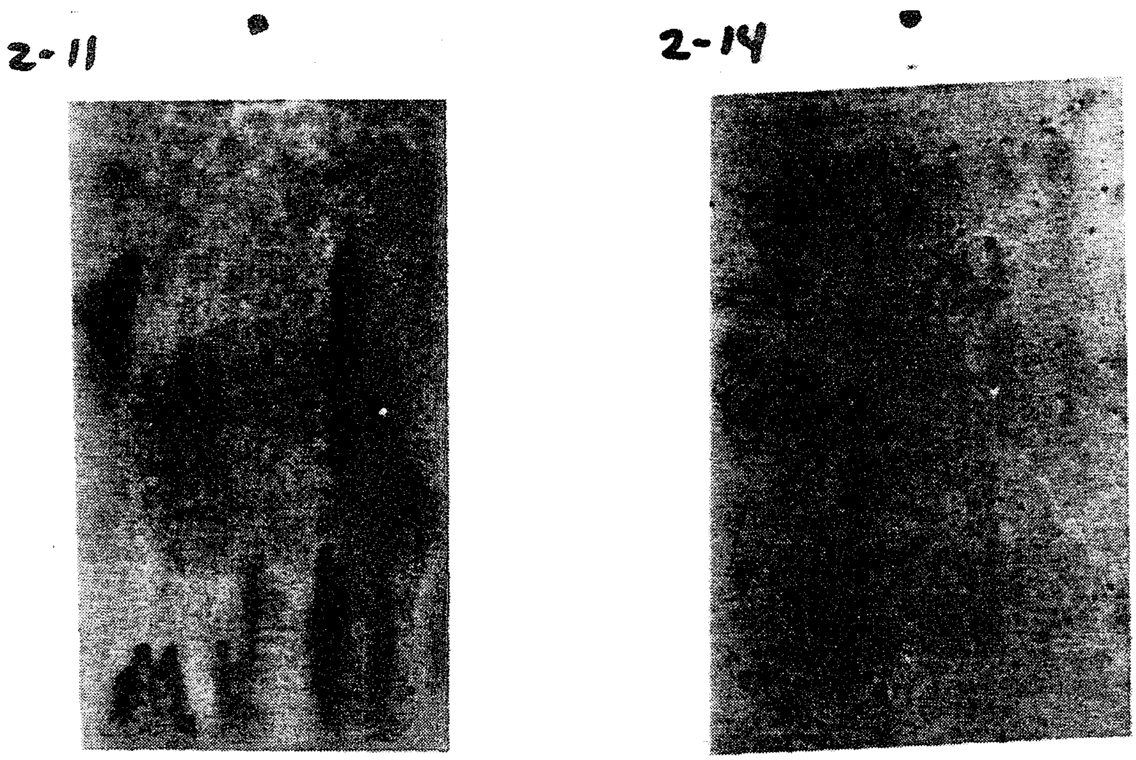

Figure 44. Sealing Step II of the Sanchem-CC Process
(After 8064 Hours of 5% Neutral Salt Fog Testing)

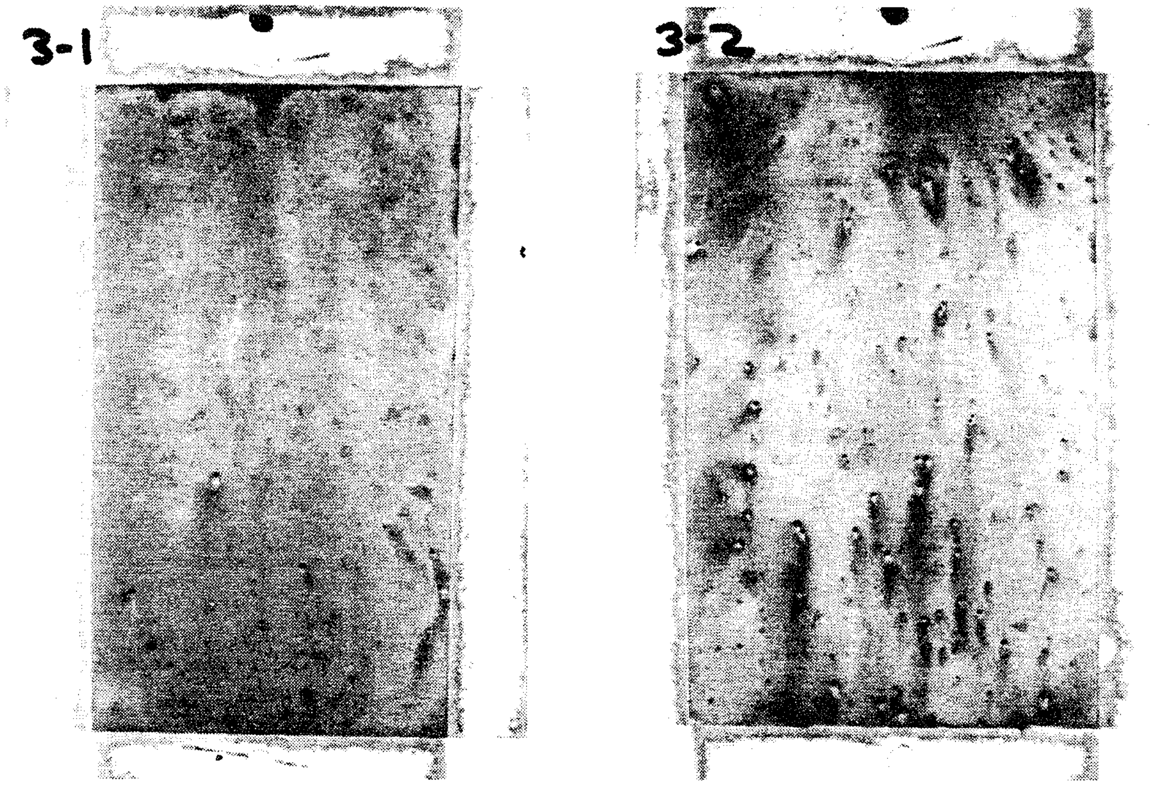

Figure 45. Sealing Steps II & III of the Sanchem-CC Process (After 8064 Hours of 5% Neutral Salt Fog Testing)

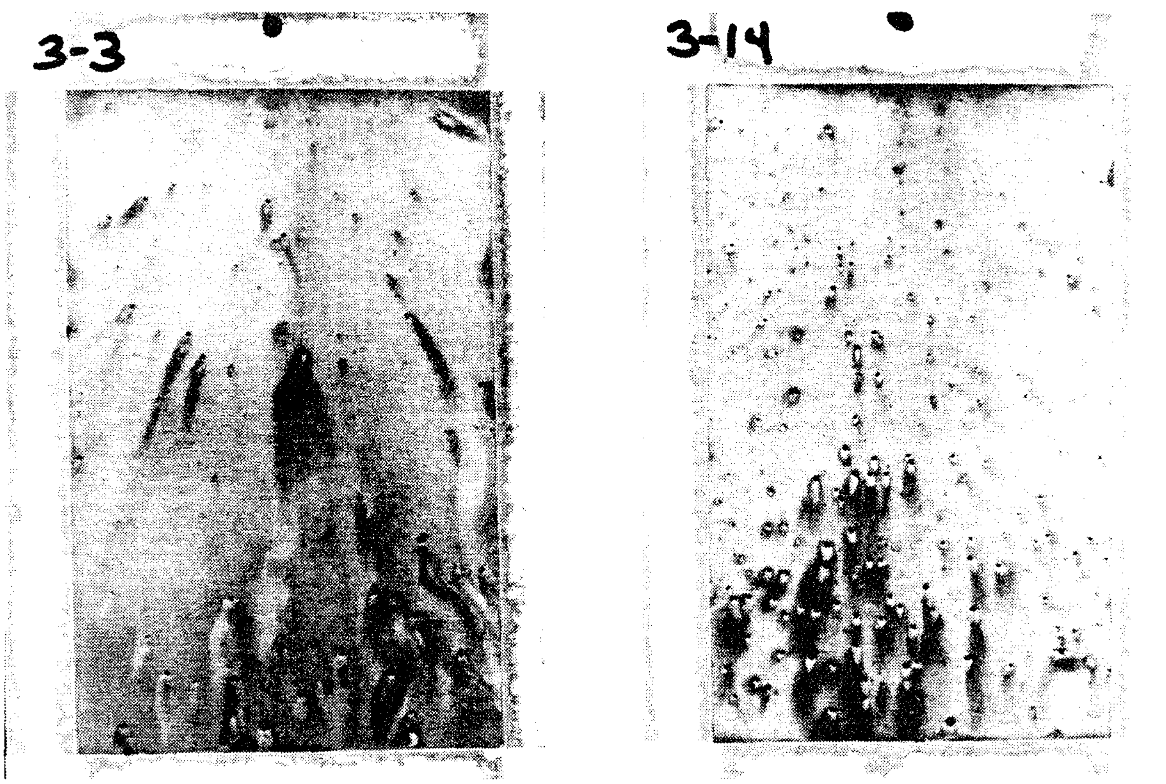

Figure 46. Sealing Steps II & III of the Sanchem-CC Process (After 8064 Hours of 5% Neutral Salt Fog Testing)

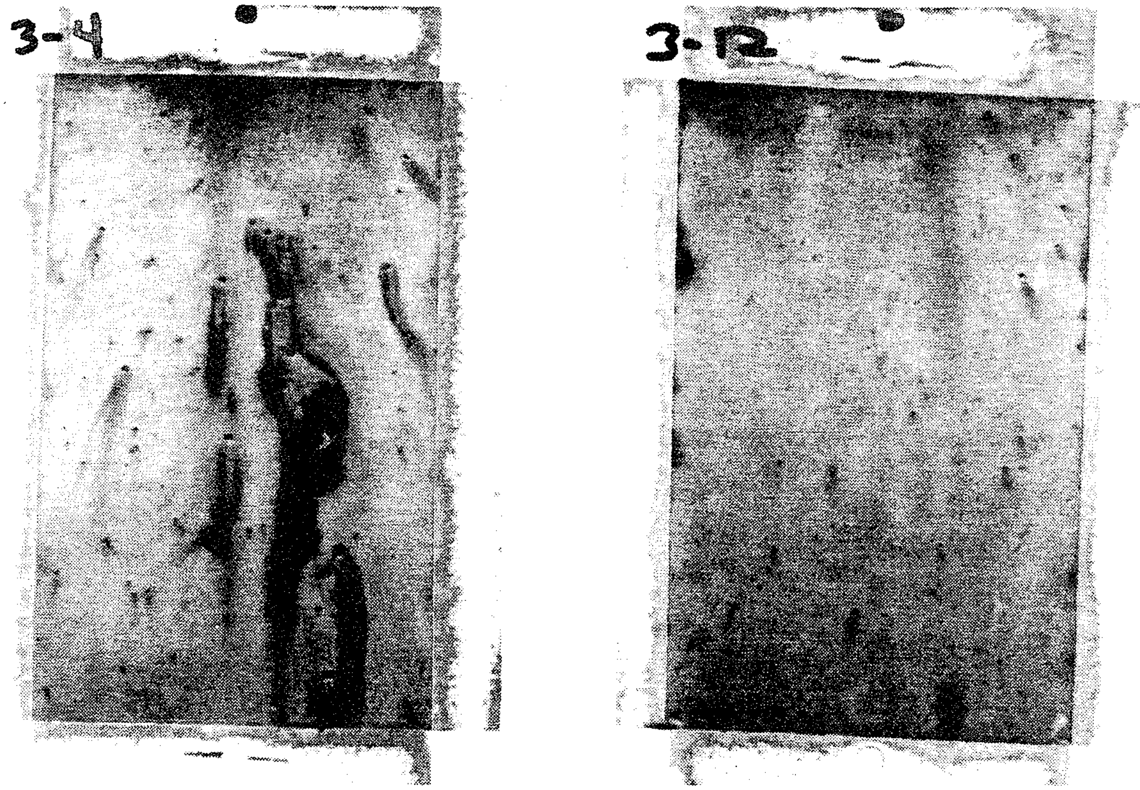

Figure 47. Sealing Steps II & III of the Sanchem-CC Process (After 8064 Hours of 5% Neutral Salt Fog Testing)

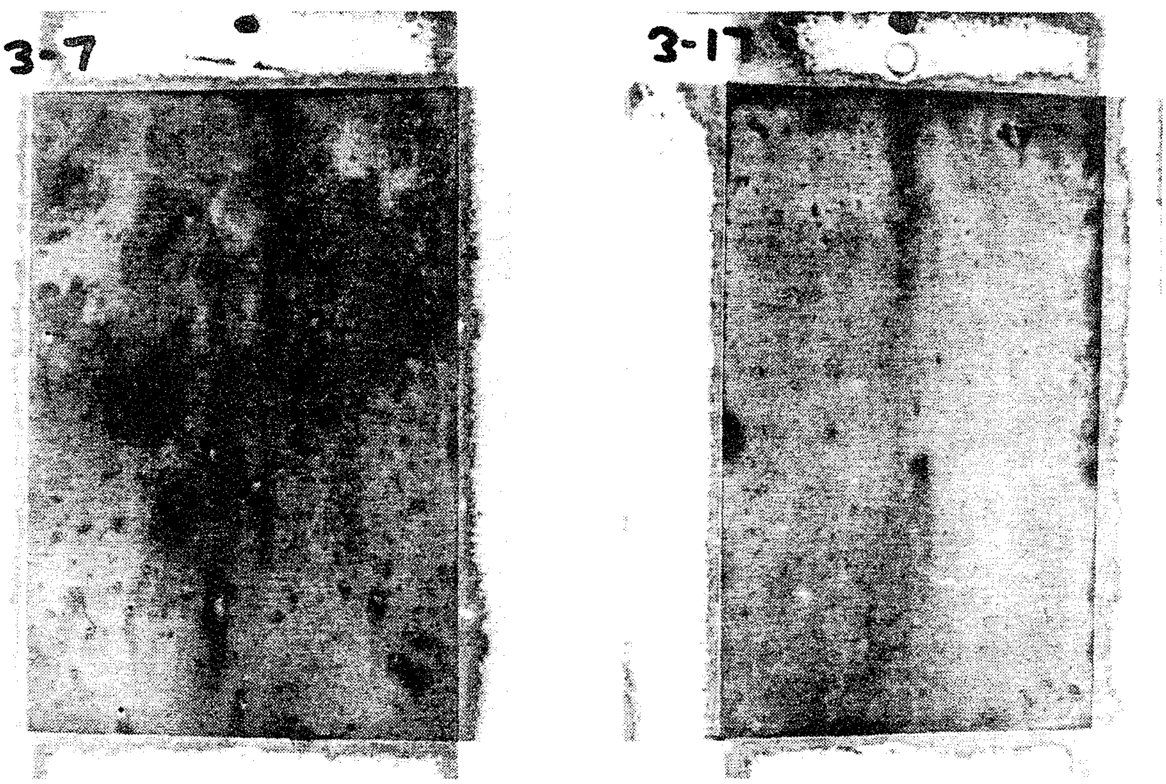

Figure 48. Sealing Steps II & III of the Sanchem-CC Process (After 8064 Hours of 5% Neutral Salt Fog Testing)

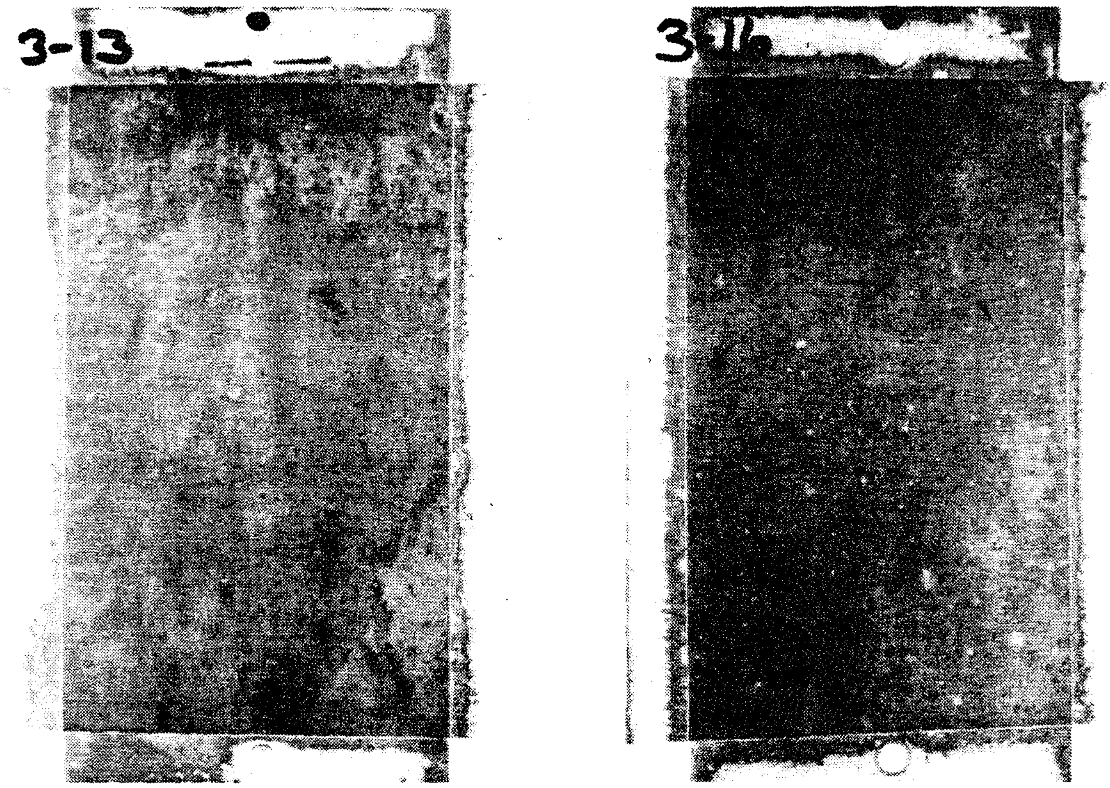

Figure 49. Sealing Steps II & III of the Sanchem-CC Process (After 8064 Hours of 5% Neutral Salt Fog Testing)

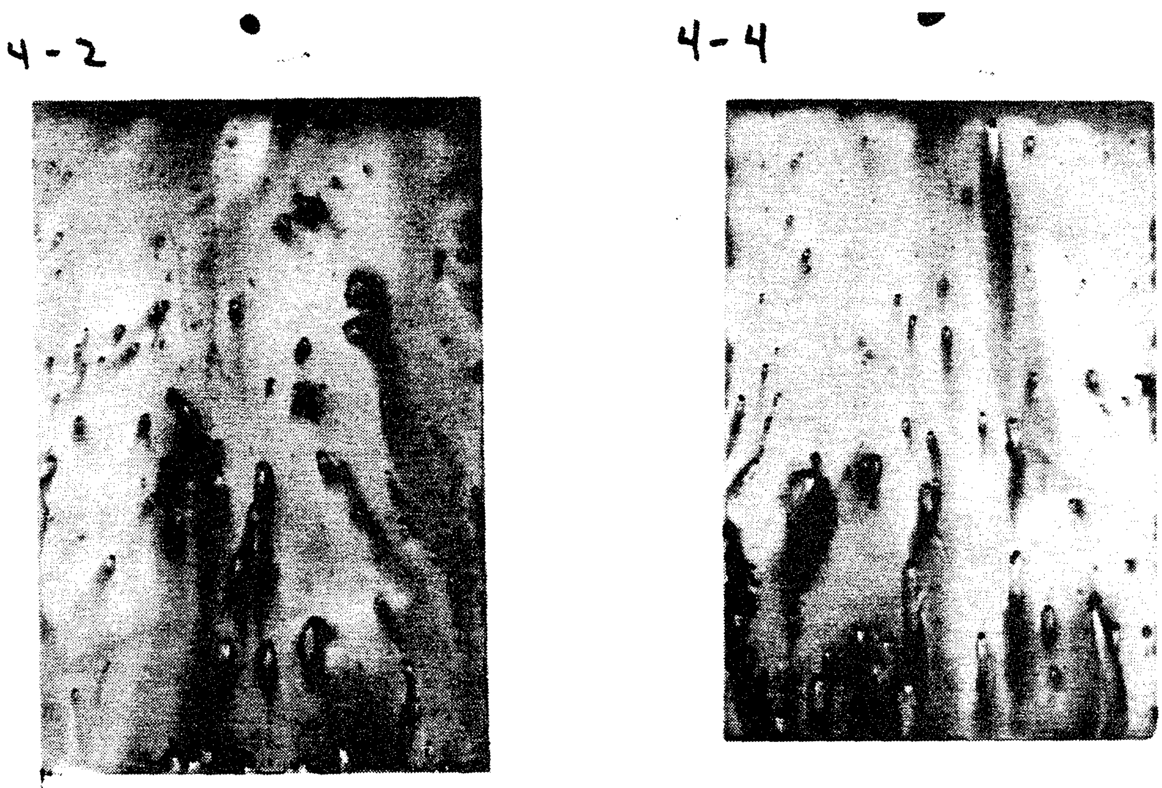

Figure 50. Permatreat 1001 (After 8064 Hours of 5% Neutral Salt Fog Testing)

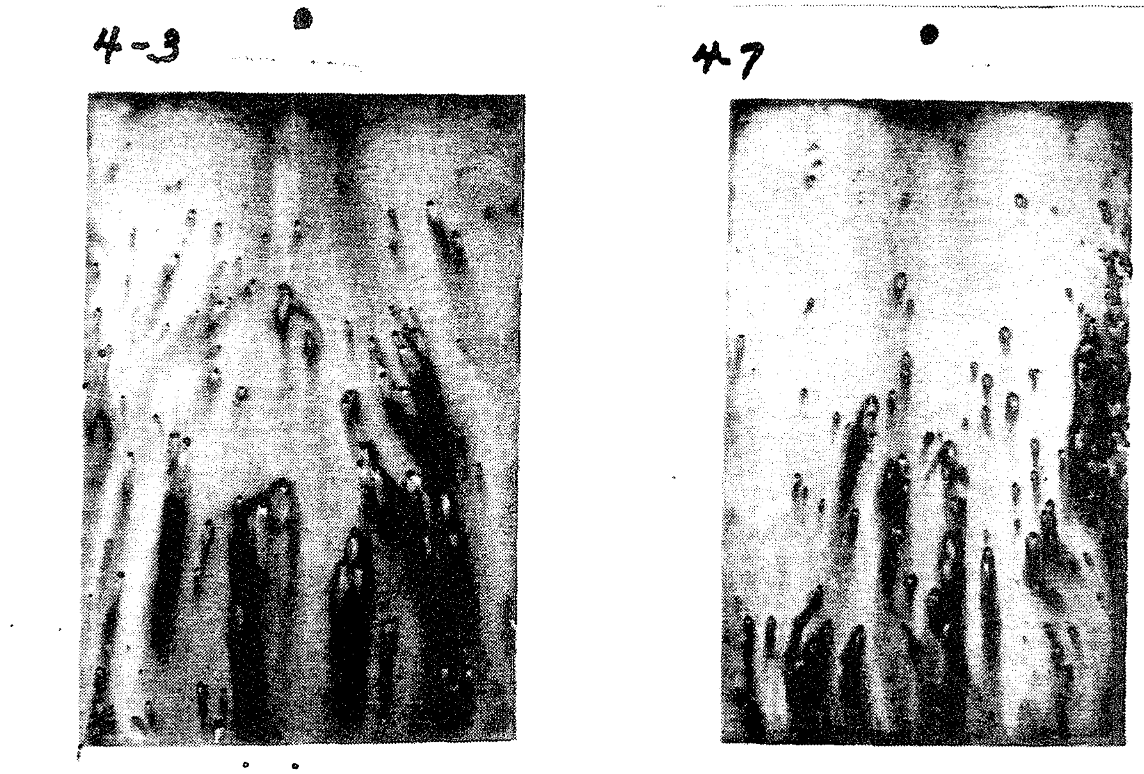

Figure 51. Permatreat 1001 (After 8064 Hours of 5% Neutral Salt Fog Testing)

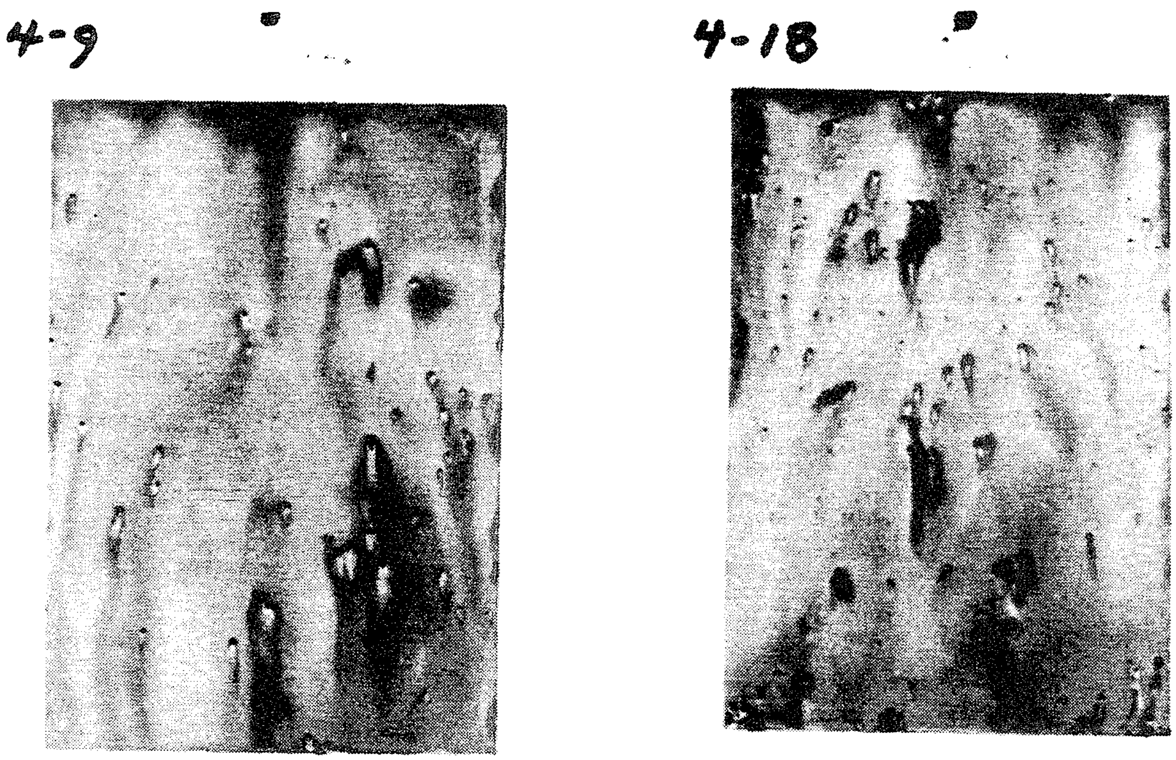

Figure 52. Permatreat 1001 (After 8064 Hours of 5% Neutral Salt Fog Testing)

4-14 4-15

Figure 53. Permatreat 1001 (After 8064 Hours of 5% Neutral Salt Fog Testing)

4-12 4-13

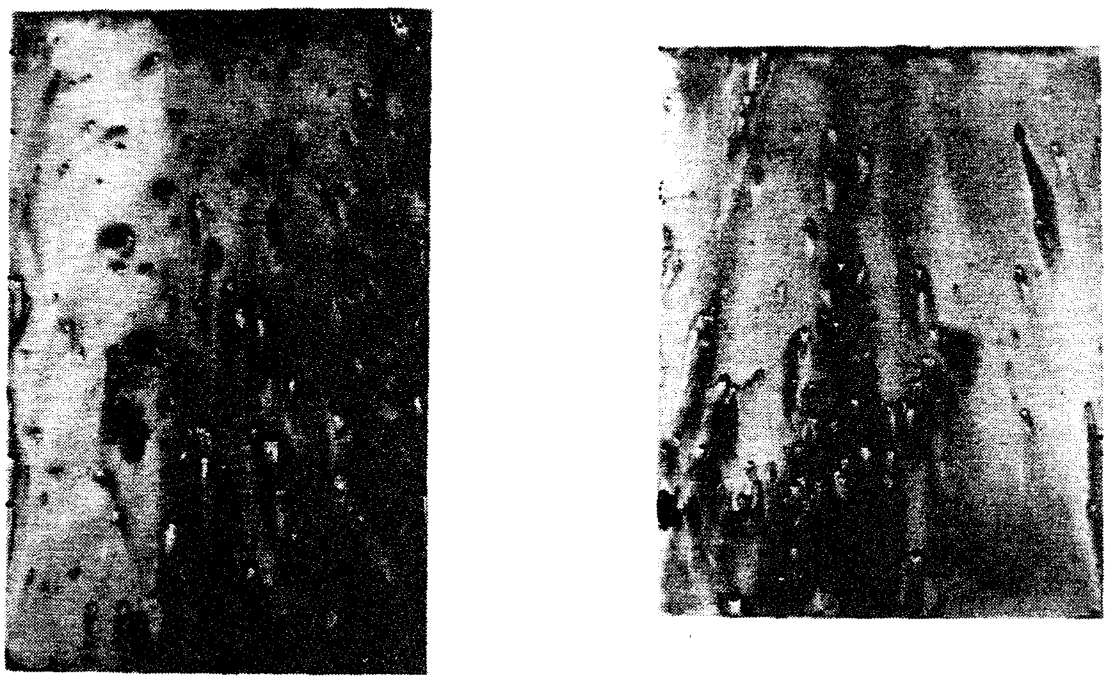

Figure 54. Permatreat 1001 (After 8064 Hours of 5% Neutral Salt Fog Testing)

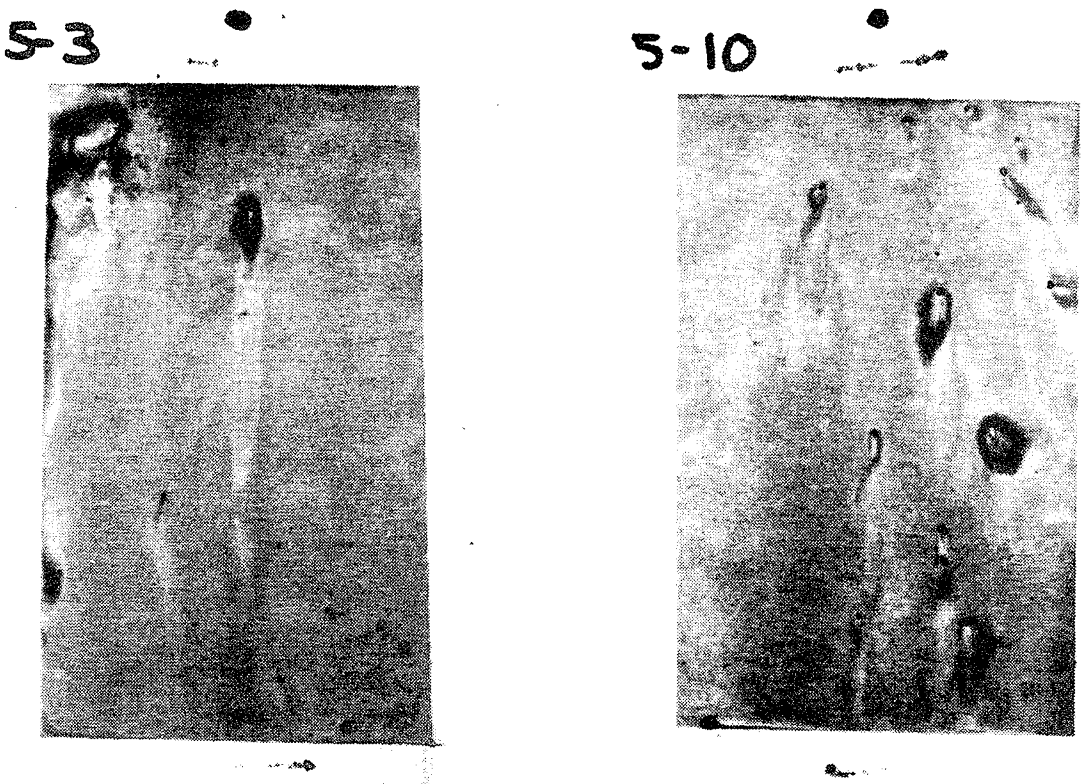

Figure 55. Alodine 2000 (After 8064 Hours of 5% Neutral Salt Fog Testing)

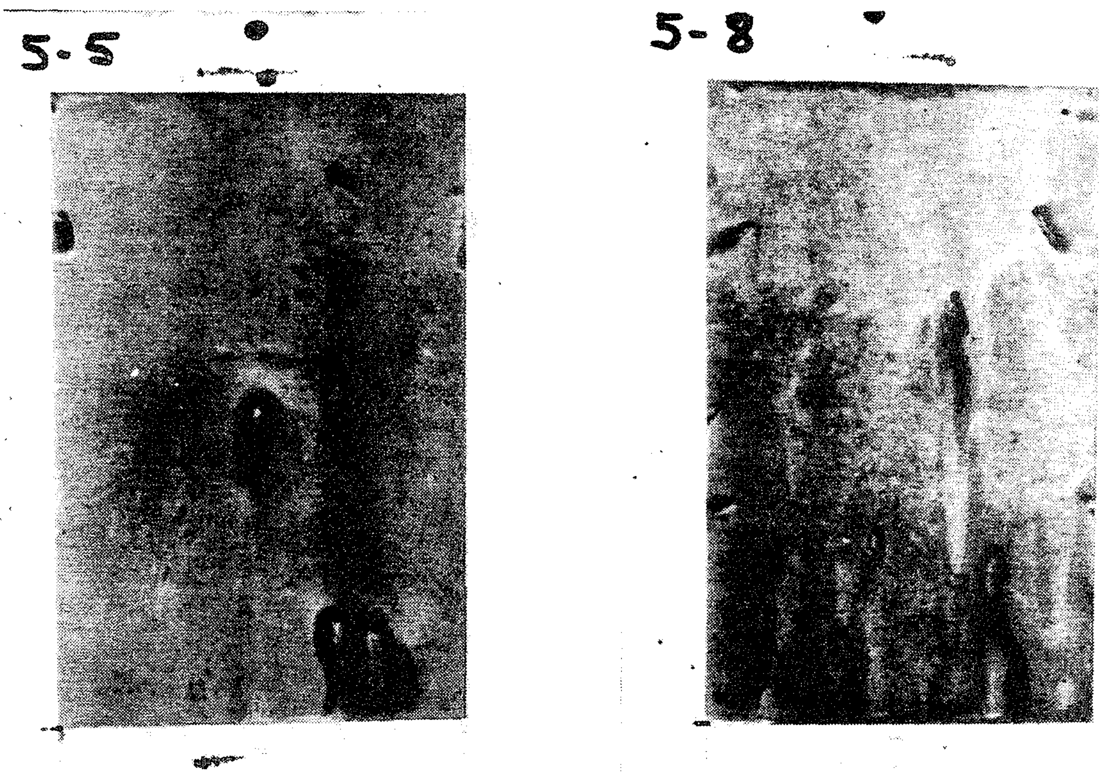

Figure 56. Alodine 2000 (After 8064 Hours of 5% Neutral Salt Fog Testing)

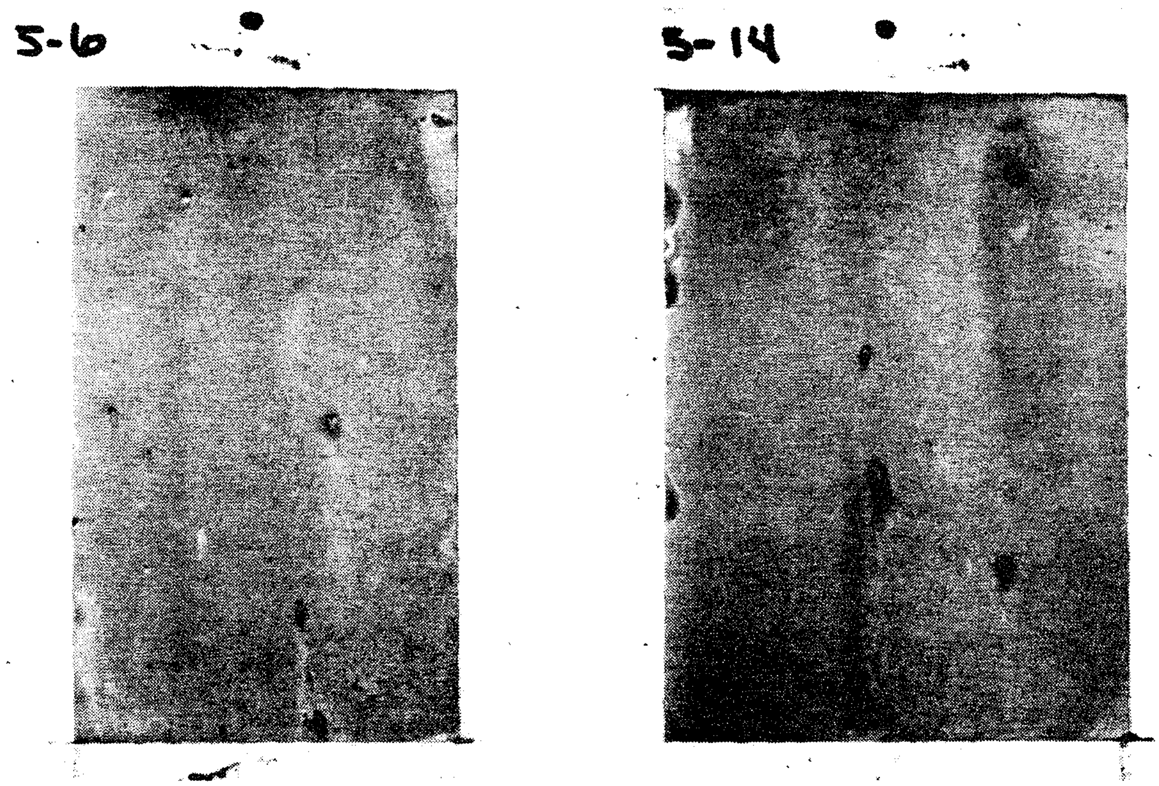

Figure 57. Alodine 2000 (After 8064 Hours of 5% Neutral Salt Fog Testing)

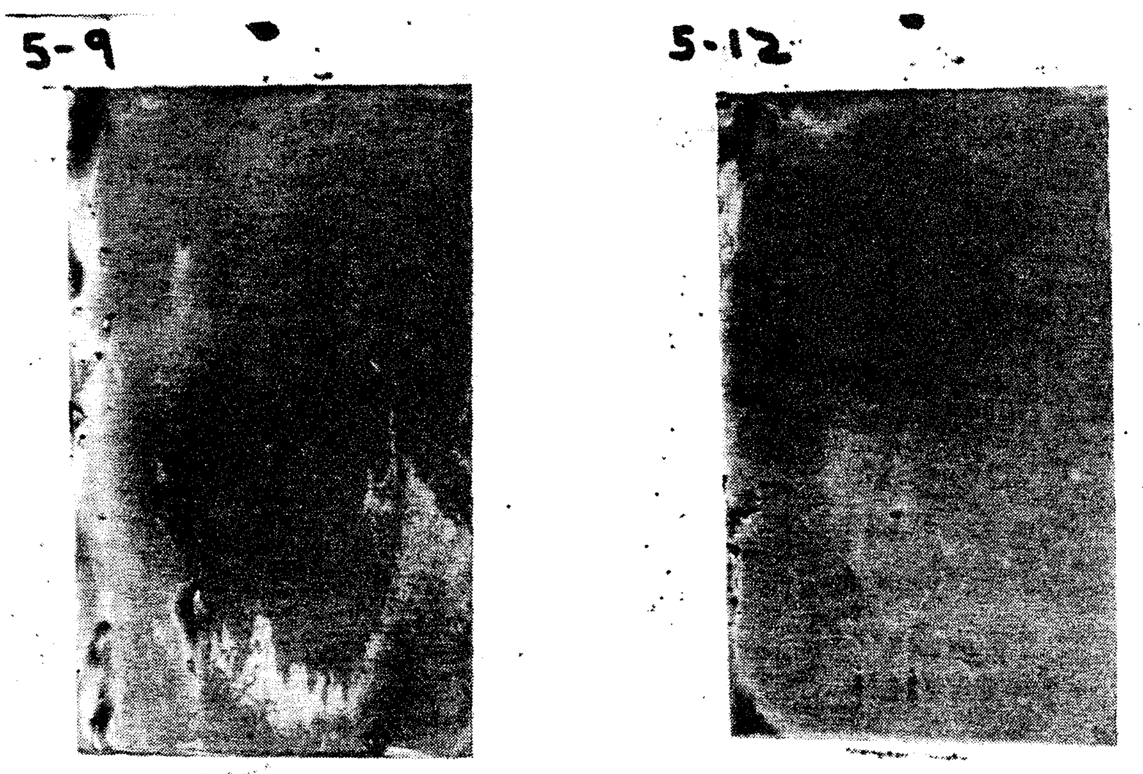

Figure 58. Alodine 2000 (After 8064 Hours of 5% Neutral Salt Fog Testing)

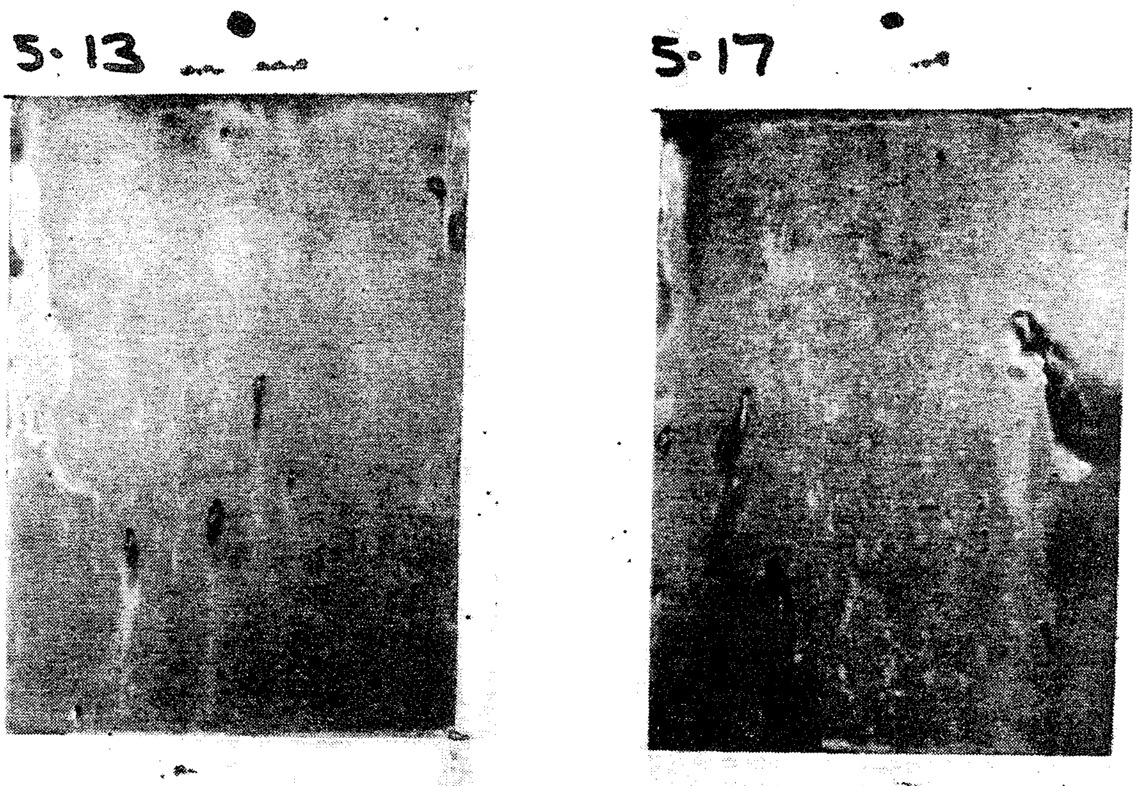

Figure 59. Alodine 2000 (After 8064 Hours of 5% Neutral Salt Fog Testing)

Photographs of the panels at the conclusion of testing show that the size and number of pits on the panels varies not only from treatment-to-treatment, in some instances, but also between panels for any given treatment. The important point, however, is that all of the panels for all of the treatments have met the 672 hour salt spray requirement of MIL-C-83488. Furthermore, based on the above, it can be concluded that all of the candidate nonchromated conversion coatings have provided comparable performance and performance comparable to the chromate conversion-coated controls.

4. Sulfur Dioxide (SO_2) Salt Fog Testing for 500 Hours

The 5% neutral salt fog environment required by MIL-C-83488 for corrosion resistance testing has long been the industry standard. However, the actual in-service environment is not always best simulated by testing in neutral salt fog. At many commercial and military industrial sites, there are emissions of SO_2 from smokestacks. The SO_2 emissions, in the presence of water, accelerates corrosion rates due to the formation of acids.

MDA uses an SO_2 salt fog test developed by the Naval Air Development Center (now Naval Air Warfare Center). It was originally conceived to simulate a ship board environment when virtually all Navy ships burned a high sulfur content fuel. Although this is not the case today, the test is still used by the Navy and considered a valuable accelerated test method.

It should be emphasized that the Navy SO_2 salt fog test represents a very severe, acidified environment. More specifically, synthetic sea salt conforming to ASTM D1141 is used which contains many corrosive compounds (e.g., 25.6% magnesium chloride and 2.8% calcium chloride) in addition to sodium chloride. Also, during every 6-hour period of exposure, 1500 cc of sulfur dioxide gas is introduced in the chamber which results in the formation of acids. It is introduced over a 60 minute period with a minimum of 6 hours between addition periods. It should also be noted that the Navy SO_2 salt fog test is typically specified for a complete system or component. Such a system or component would include a primer or a primer and topcoat. Based on the above facts, the ultimate purpose of SO_2 salt fog testing under this development program was to determine the relative performance of the various conversion coatings in a harsh or accelerated environment. Finally, there is no specification requirement applicable to IVD aluminum-coated substrates in regard to SO_2 salt fog testing.

Ten panels were subjected to SO_2 salt fog testing for each of the four candidate nonchromated conversion coatings and for the chromate conversion coating. The original plan was to expose the panels until failure. Due to the condition of the panels as testing progressed, however, it was considered more prudent to stop the test after 504 hours of exposure when meaningful comparisons could still be made. Figures 60 – 69 show representative panels for each of the treatments at the conclusion of testing.

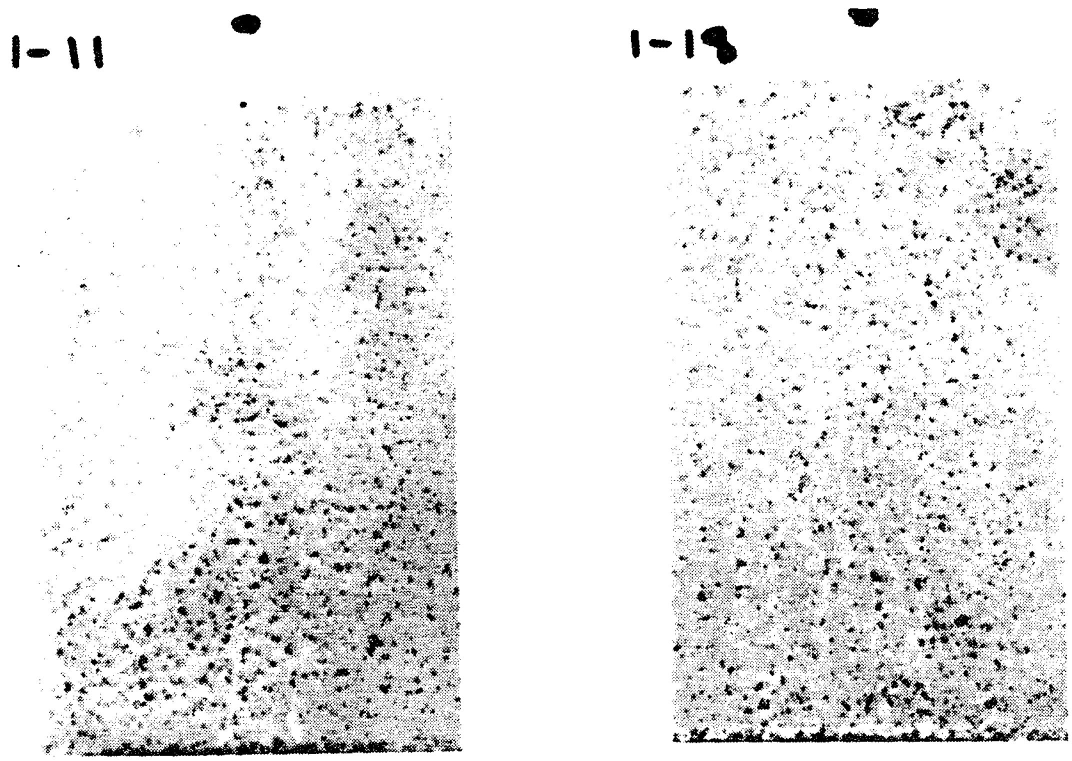

Figure 60. Chromate Conversion Coating (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

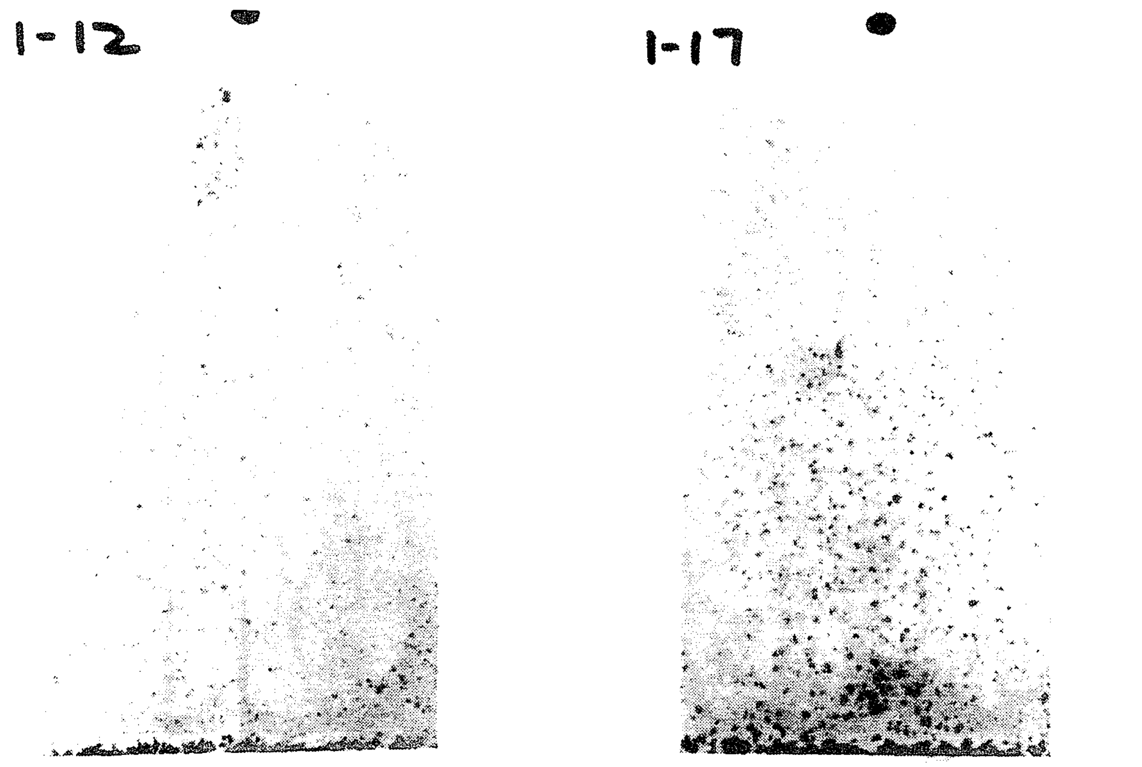

Figure 61. Chromate Conversion Coating (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

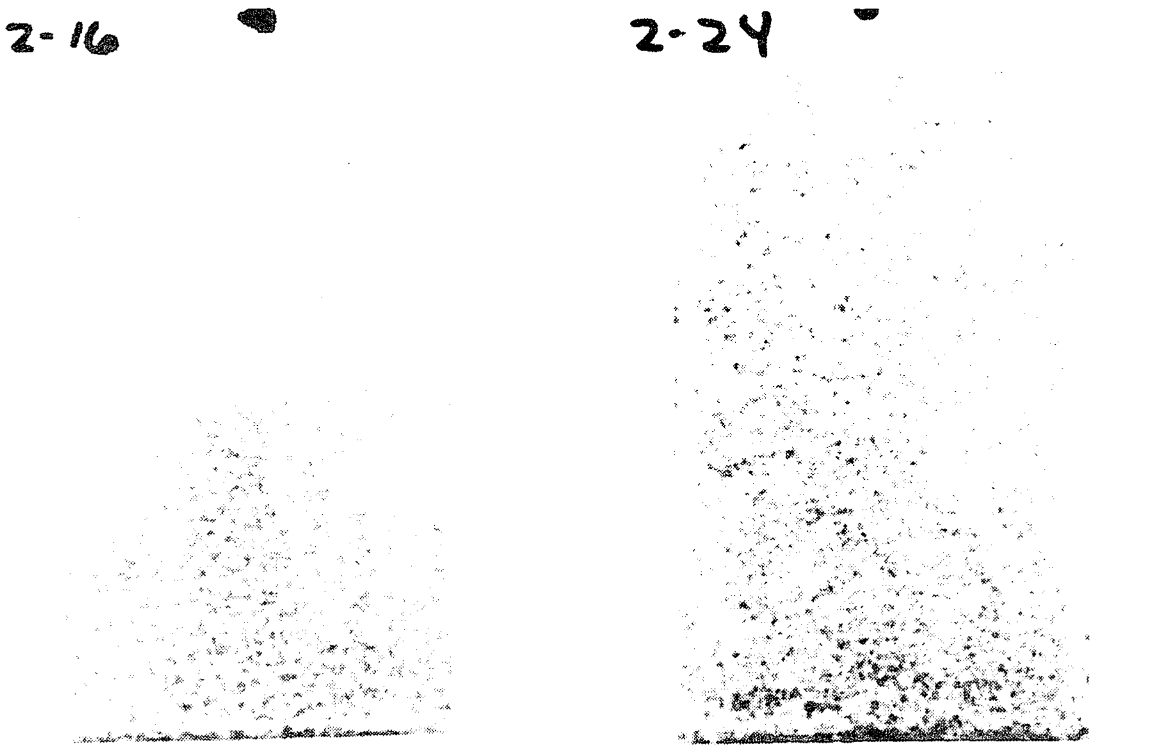

Figure 62. Sealing Step II of the Sanchem-CC Process (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

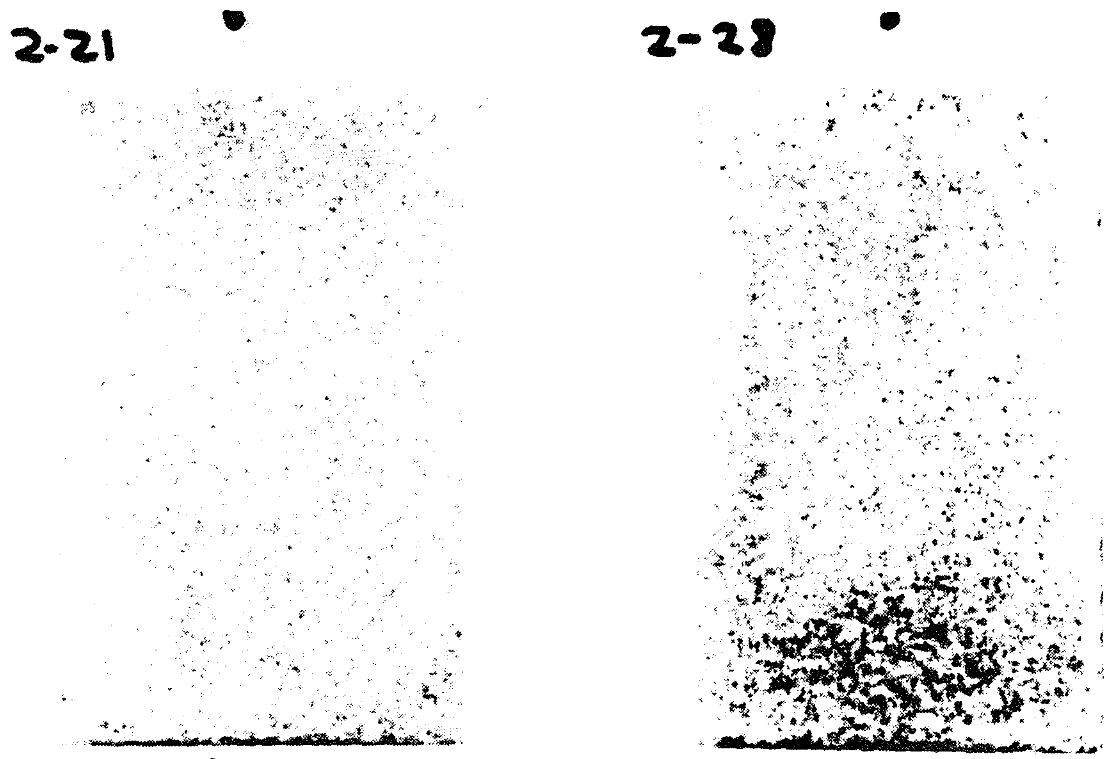

Figure 63. Sealing Step II of the Sanchem-CC Process (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

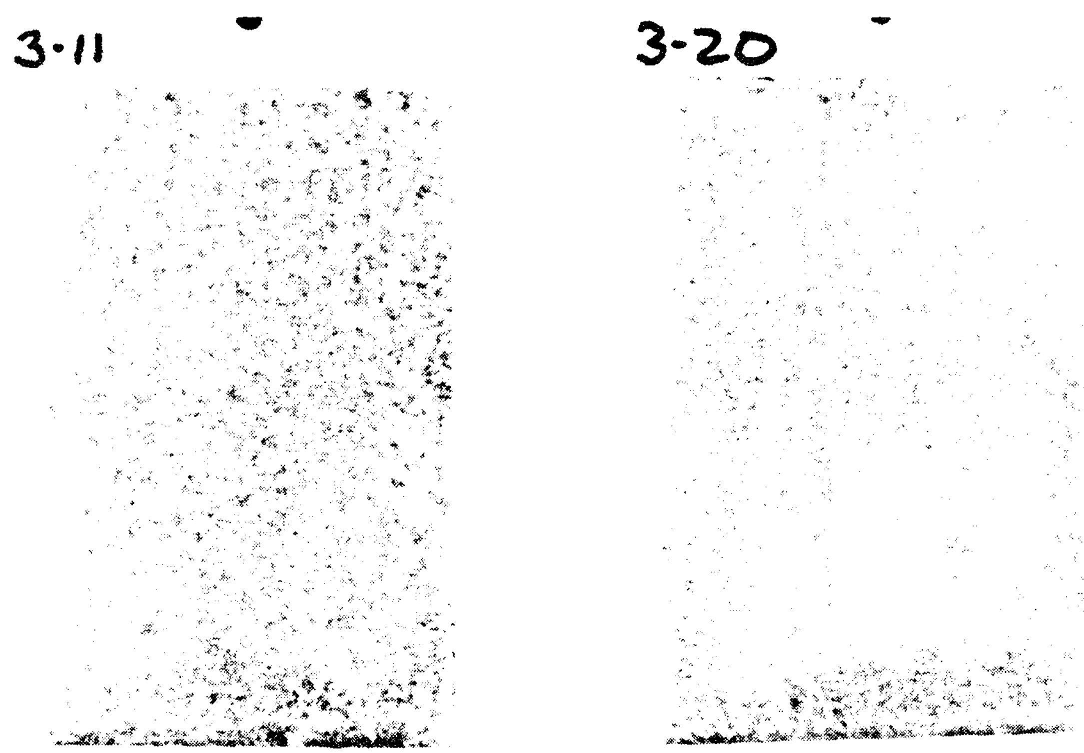

Figure 64. Sealing Steps II & III of the Sanchem-CC Process (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

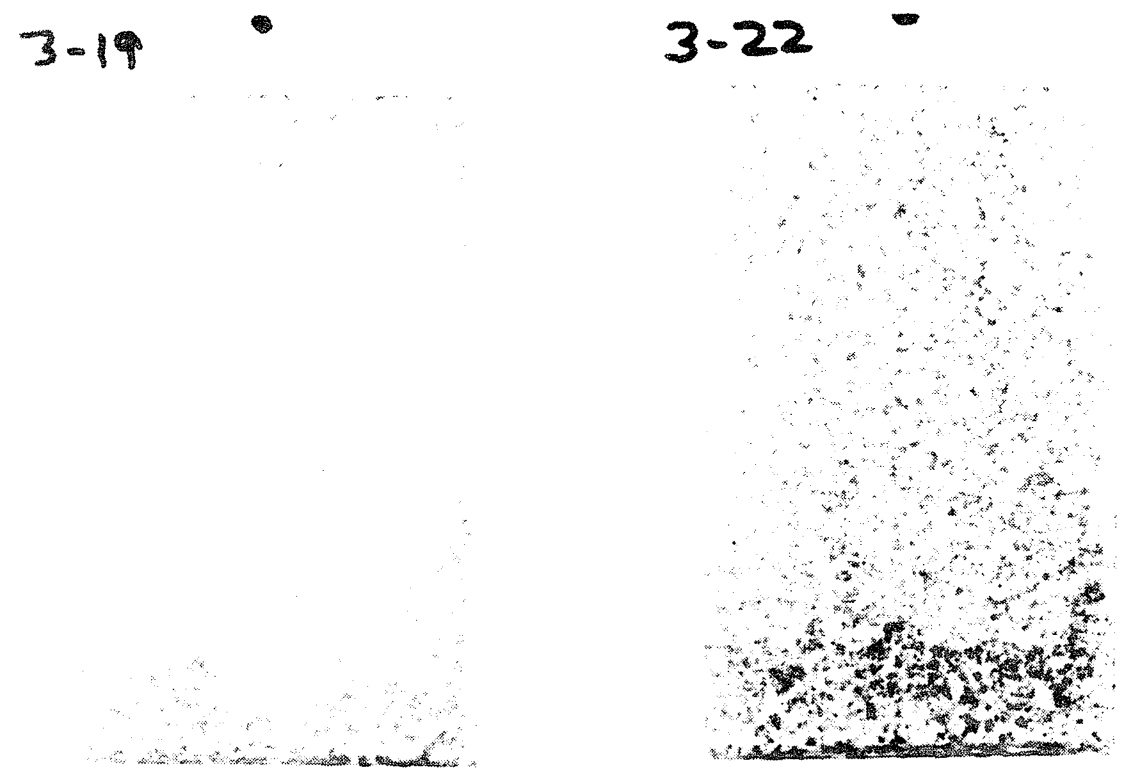

Figure 65. Sealing Steps II & III of the Sanchem-CC Process (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

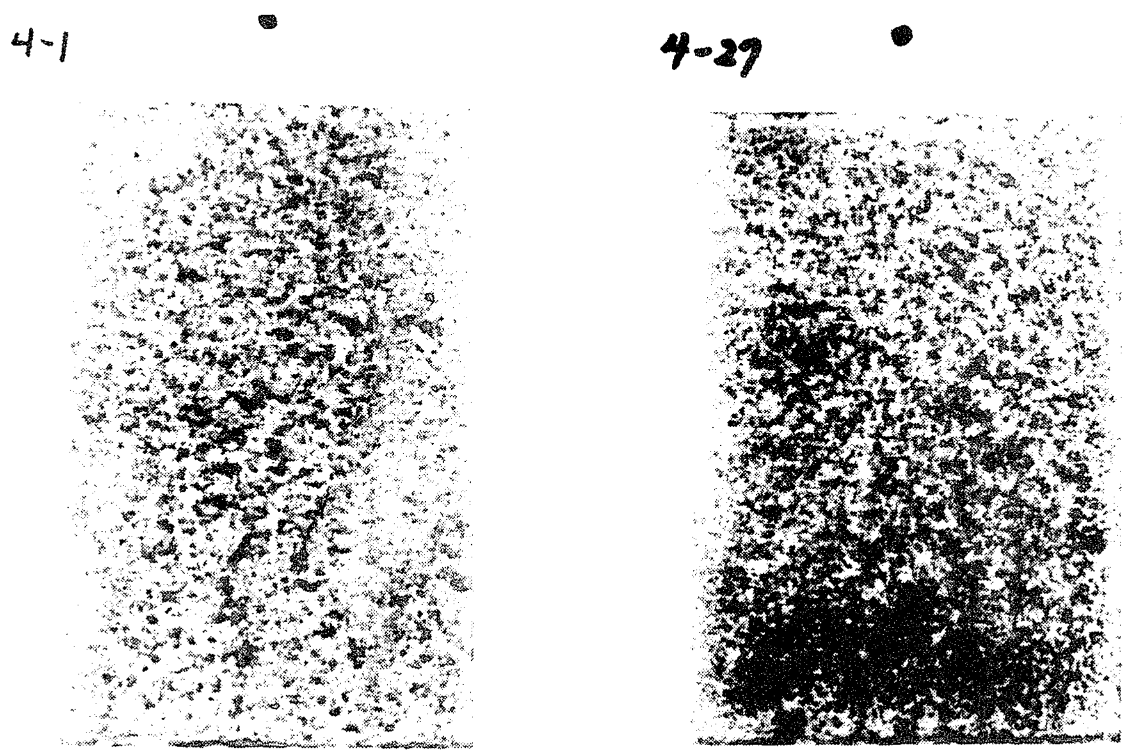

Figure 66. Permatreat 1001 (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

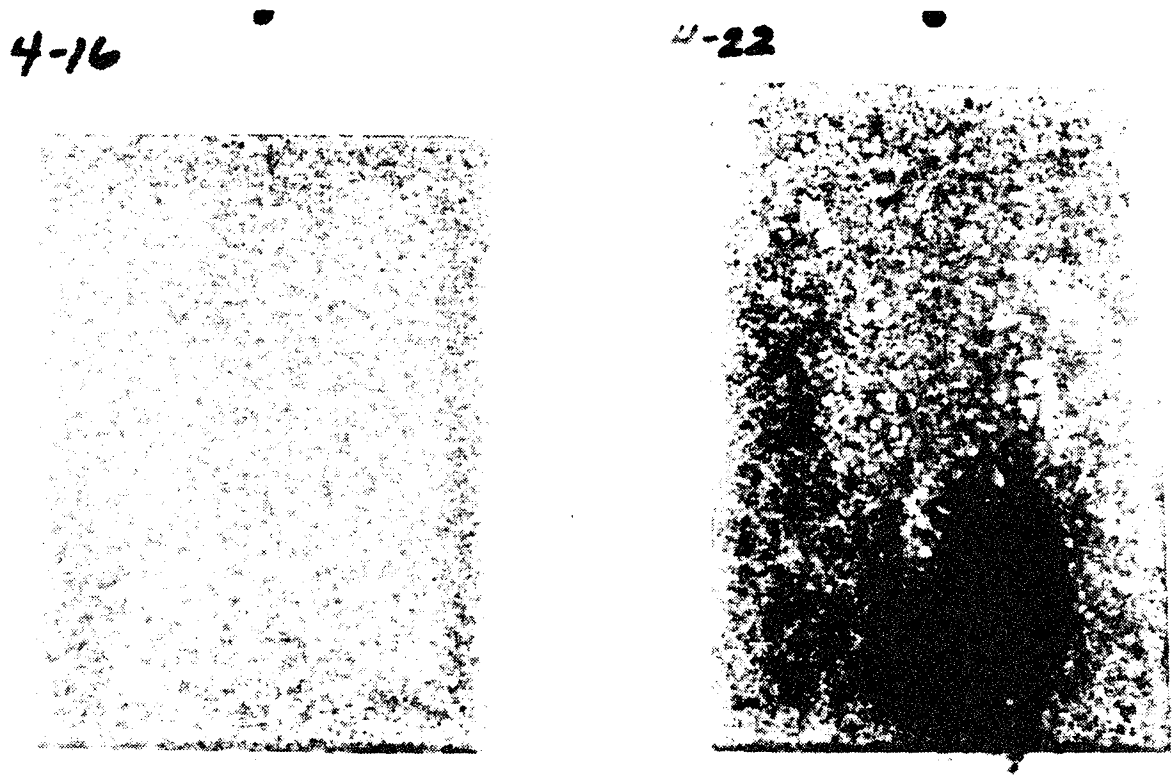

Figure 67. Permatreat 1001 (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

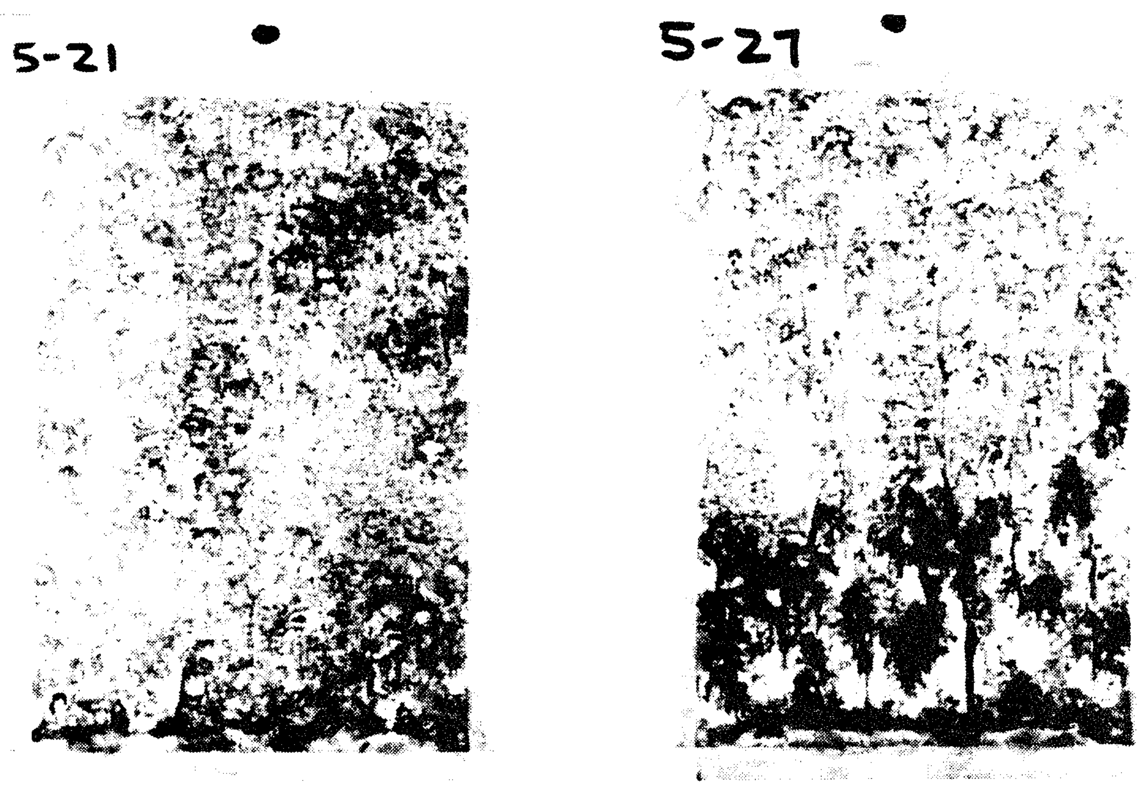

Figure 68. Alodine 2000 (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

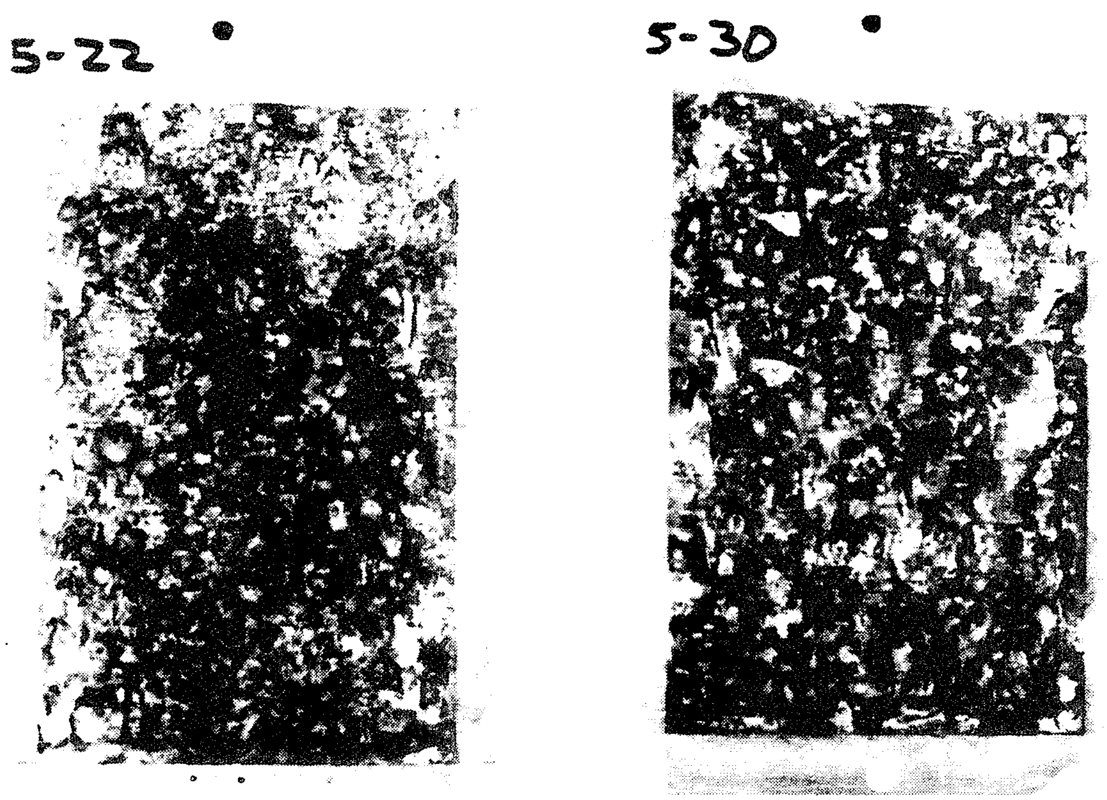

Figure 69. Alodine 2000 (After 504 Hours of Sulfur Dioxide Salt Fog Testing)

Out of all the panels tested, only one of the Permatreat 1001 panels showed evidence of failure (i.e., the appearance of red rust) at the conclusion of testing. Failure occurred right near the end of the exposure period.

The various candidate nonchromated conversion coatings and the chromate conversion coating controls were given a final performance ranking at the conclusion of SO_2 salt fog testing based on visual appearance and smoothness to the touch. This ranking, from best to worst, is noted below. Of interest, the Alodine 2000 panels displayed a significantly greater amount of corrosion products compared to the other treatments.

- Chromate Conversion Coating
- Sealing Step II of the Sanchem-CC process, except at increased temperature, and Sealing Steps II and III of the Sanchem-CC process
- PERMATREAT 1001
- Improved Alodine 2000

5. St. Louis Outdoor Exposure

Ten panels were subjected to outdoor exposure testing in St. Louis, MO for each of the four candidate nonchromated conversion coatings and for the chromate conversion coating. The plan was to expose the panels until failure. As expected, none of the panels have failed at the conclusion of this development program which corresponds to approximately 58 weeks of testing. In fact, none of them show any evidence of pitting or corrosion products. The panels will continue to be exposed and monitored until failure after the program ends.

Of some interest, a few distinct marks appeared on some of the panels for all of the treatments, except for the chromate conversion-coated controls, during the course of testing. Also, during testing, a water-spot type staining phenomenon occurred on the panels treated with Alodine 2000, Sealing Step II of the Sanchem-CC process, and Sealing Steps II and III of the Sanchem-CC process. None of the above anomalies are considered significant at the conclusion of this development program.

Figure 70 shows the panels on the exposure rack used for outdoor exposure testing soon after testing began. The panels are inclined at an angle of 30° from the vertical and face southward. The ten panels for each treatment are located across the rack in separate rows. Figures 71 – 75 show representative panels for each treatment after 55 weeks of exposure.

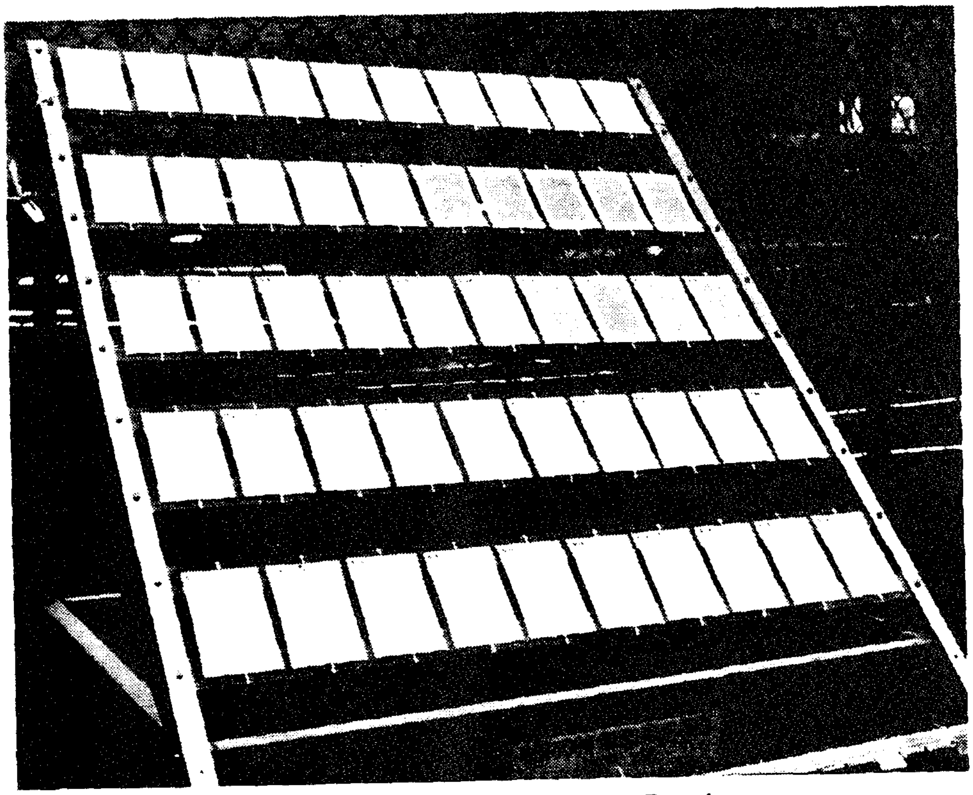

Figure 70. Outdoor Exposure Panels

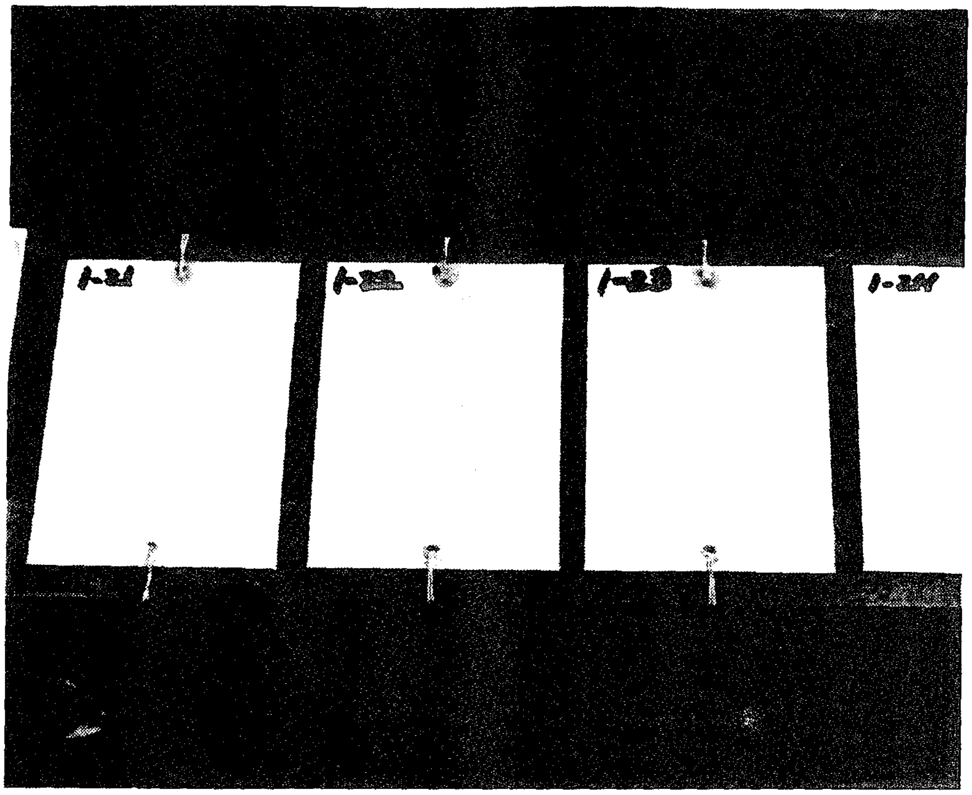

Figure 71. Chromate Conversion-Coated Panels After 55 Weeks of Outdoor Exposure

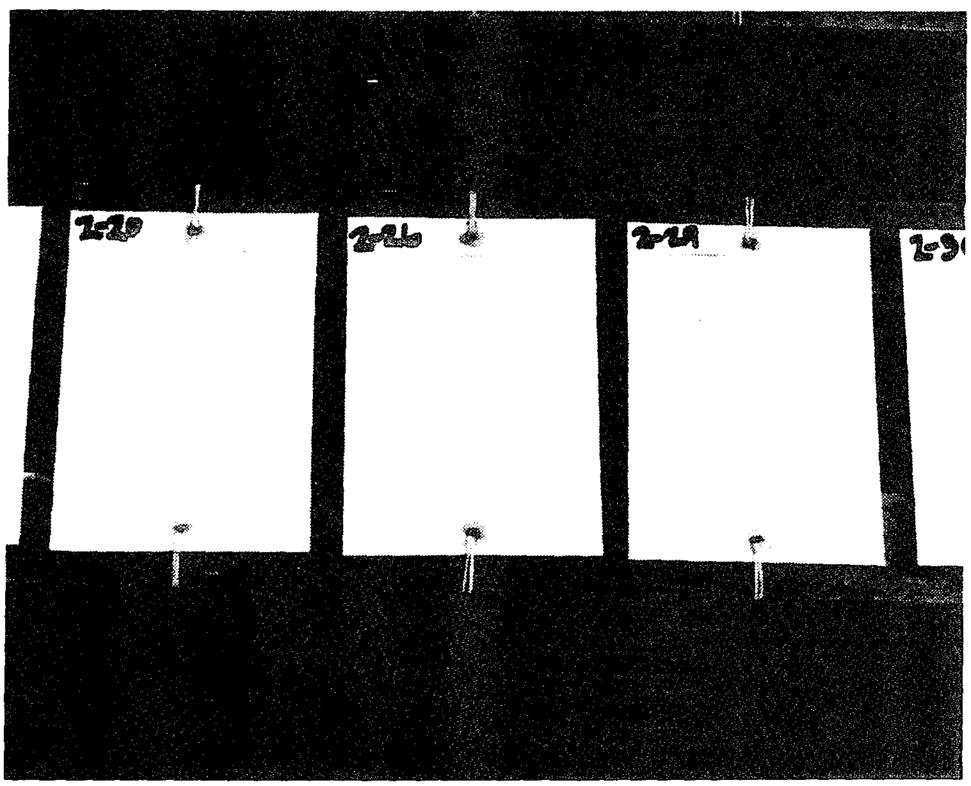

Figure 72. Sealing Step II of the Sanchem-CC Process Panels After 55 Weeks of Outdoor Exposure

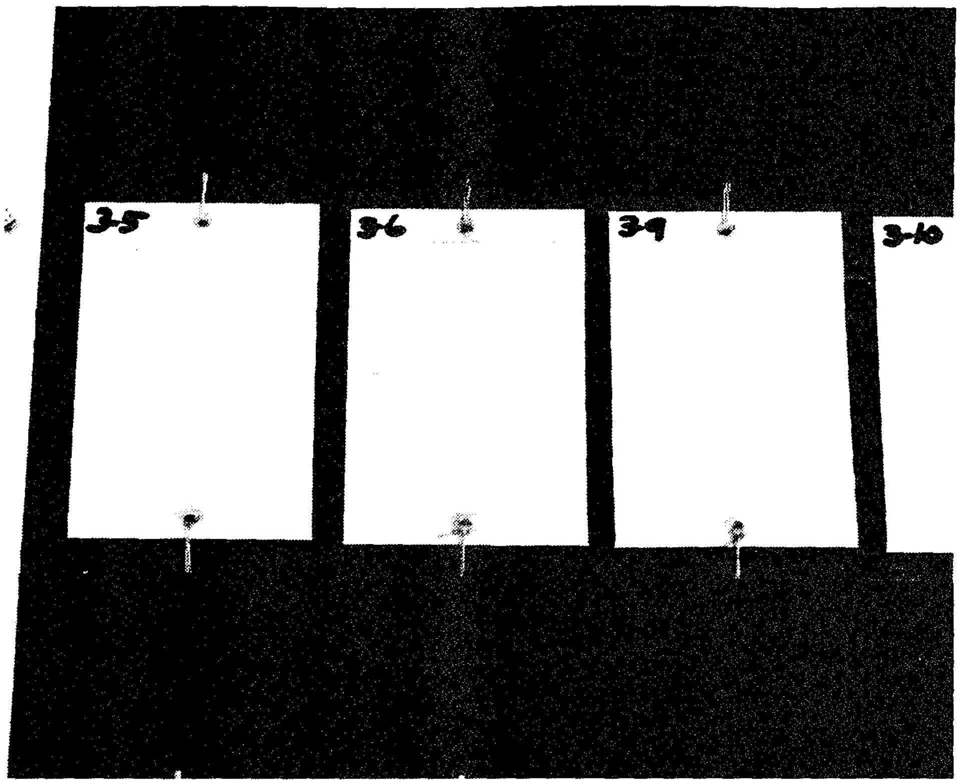

Figure 73. Sealing Steps II & III of the Sanchem-CC Process Panels After 55 Weeks of Outdoor Exposure

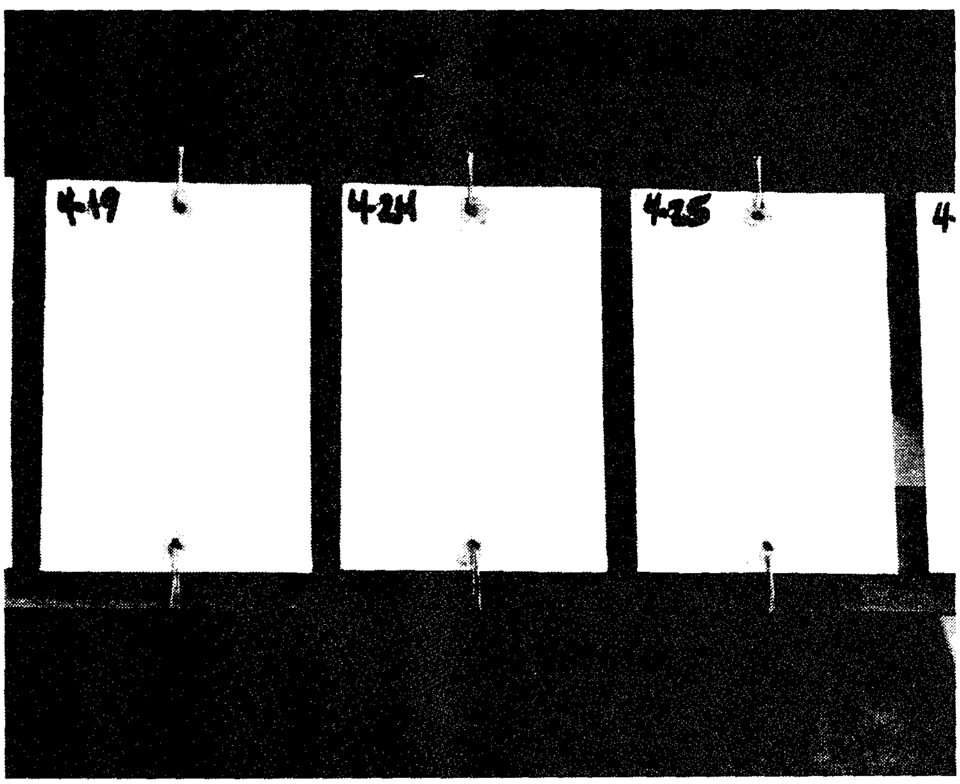

Figure 74. Permatreat 1001 Panels After 55 Weeks of Outdoor Exposure

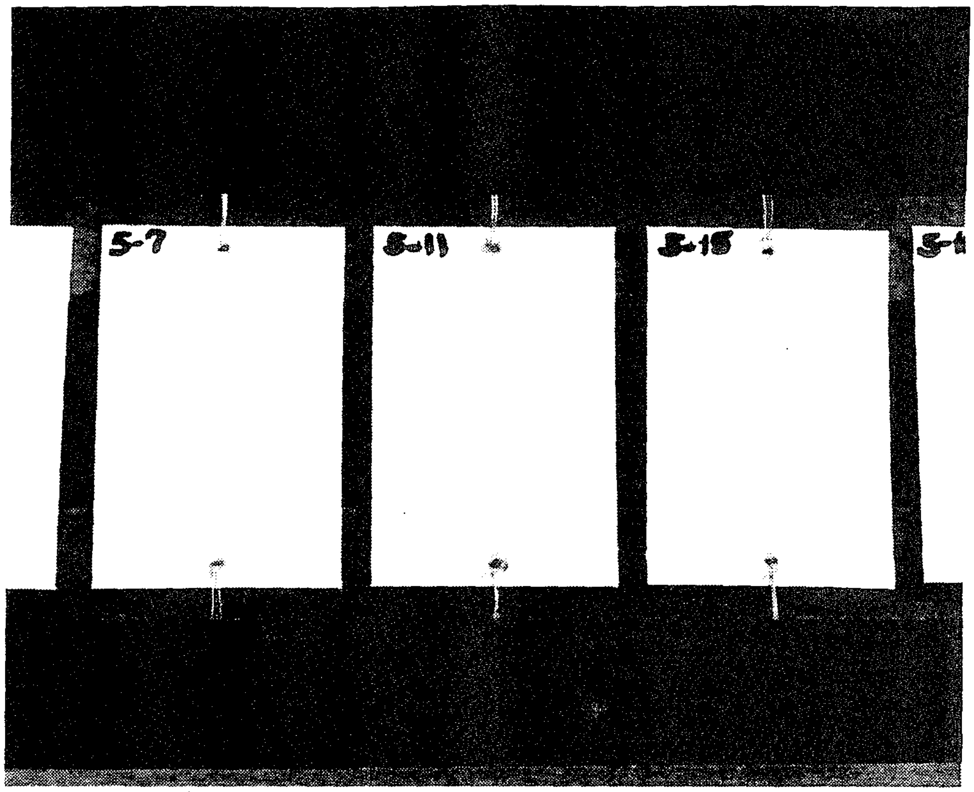

Figure 75. Alodine 2000 Panels After 55 Weeks of Outdoor Exposure

6. Supplementary Test 4

As salt spray testing progressed under Task 4, it was felt that the treatment consisting of Sealing Steps II and III of the Sanchem-CC process offered considerable promise. This being the case, but before committing this process to any type of scale-up evaluation, it was decided to verify required flexibility and further verify primer adhesion. Performance in this regard was of particular concern since Sealing Step III of the Sanchem-CC process seals the IVD aluminum coating using a potassium silicate solution. Silicates are typically inherently brittle.

IVD aluminum-coated 2024-T3 aluminum panels, 3 x 6 x 0.020 inch thick, were used for the flexibility and adhesion investigation. Initially, one set of the IVD aluminum-coated panels was treated using Sealing Steps II and III of the Sanchem-CC process and another set using chromate conversion coating. Then, panels from each set were spray primed with Courtauld's 513X408/910X831 water-borne epoxy primer and Courtauld's 519X303/910X357 solvent-based epoxy primer. Panels from each set were also primed by electrodeposition using BASF's G28AD012 epoxy primer.

The treated and primed panels described above were subjected to cross hatch, reverse impact, and mandrel bend testing. All panels passed all of the testing performed. The treatment consisting of Sealing Steps II and III of the Sanchem-CC process, therefore, was determined to be equivalent to the current chromate conversion coating process in regard to the tests which were performed. This performance, then, provided further confidence in the suitability of Sealing Steps II and III of the Sanchem-CC process as a viable production process.

A more complete description of the flexibility and primer adhesion testing performed under Supplementary Test 4 can be found in Appendix D.

SECTION VI
TASK 5 – EFFECT ON ALUMINUM ALLOYS

A. OBJECTIVE

The objective of Task 5 was to evaluate the same four candidate nonchromated conversion coatings downselected for Task 4 testing for their ability to protect aluminum alloys. More specifically, IVD aluminum is often used in place of anodize for fatigue critical aluminum alloy parts. Also, it is used on alloy steel and titanium fasteners to provide galvanic compatibility when such fasteners are installed in aluminum alloy structure. Any nonchromated conversion coating used as a replacement for chromate conversion coating in these applications, then, must provide the same level of corrosion resistance as chromate conversion coating.

B. OVERVIEW

IVD aluminum-coated 2024-T3 and 7075-T6 aluminum panels were treated with the four candidate nonchromated conversion coatings downselected under Task 4, and then subjected to 5% neutral salt fog testing per ASTM B117 for 3024 hours and sulfur dioxide (SO_2) salt fog testing for 168 hours. Five percent neutral salt fog test results were not as good as expected for one of the candidate treatments and the chromate conversion-coated controls, and this testing was repeated for the treatments in question. IVD aluminum-coated alloy steel and titanium fasteners were also treated with the same four candidate nonchromated conversion coatings and then installed in aluminum alloy bars with bare countersinks. These fastener bar specimens were then subjected to 5% neutral salt fog testing per ASTM B117 for up to six weeks, and SO_2 salt fog testing for one week. The performance of all the fastener bar specimens was less than expected based on historical testing at MDA. Reasons for this poorer than expected salt spray performance were identified. Improved fastener bar specimens were subsequently fabricated for two of the candidate nonchromated conversion coatings and the chromate conversion-coated controls, and they were subjected to 5% neutral salt fog testing per ASTM B117 for six weeks. The salt spray performance of these specimens was as expected, and much better than the initial specimens. At the conclusion of all Task 5 testing, the desired objective of determining a performance ranking for the four candidate nonchromated conversion coatings in regard to protecting aluminum alloys was accomplished.

C. DISCUSSION

1. Panel Material and Preparation

Two aluminum alloy panel materials, bare 2024-T3 per QQ-A-250/4 and bare 7075-T6 per QQ-A-250/12, were used for Task 5 testing. Panel size was 4 x 6 x 0.050 inch thick.

Initially, all of the aluminum alloy panels were cleaned in accordance with standard practice per MDA process specification P.S. 13143. The basic steps of this cleaning process include aqueous degreasing, alkaline cleaning, pickling, and deoxidizing.

After cleaning, all of the aluminum panels were IVD aluminum-coated with a coating conforming to Class 1 (1.0-mil thick minimum) of MIL-C-83488. The aluminum panels were coated in groups of 20 along with five steel panels. The steel panels were used to verify the thickness of the IVD aluminum coating on the aluminum panels since there is no non-destructive method available to measure the thickness of IVD aluminum on an aluminum substrate. Five thickness measurements were made on each steel panel. The average IVD aluminum coating thickness for all 50 of the steel panels coated (i.e., five panels x 20 individual coater runs) was 1.4 mils.

In accordance with standard practice, the IVD aluminum coating on all of the test panels was glass bead peened prior to application of the various conversion coatings. The conversion coatings were applied to the panels within 24 hours after the peening operation.

Figure 76 shows a representative 2024-T3 panel and a representative 7075-T6 panel treated with improved Alodine 2000 ready for salt fog testing. They are typical of the panels prepared for all of the conversion coatings tested.

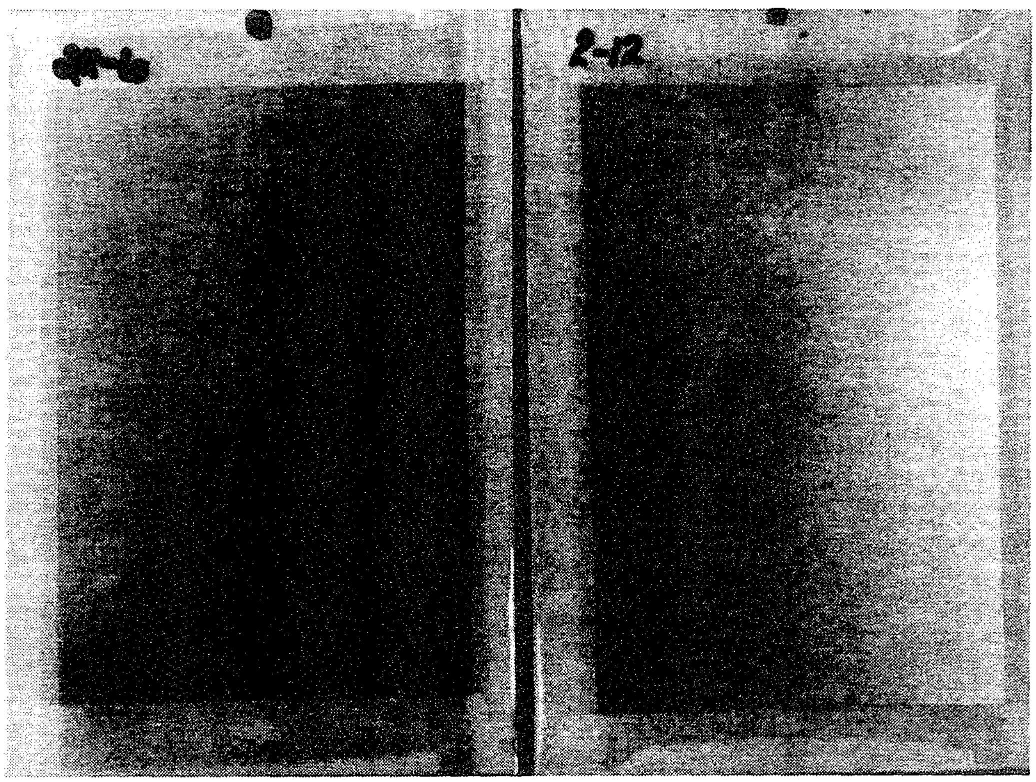

Figure 76. Alodine 2000 Panels Prior to Test (IVD Aluminum-Coated 2024-T3 Panel Left & IVD Aluminum-Coated 7075-T6 Panel Right)

2. Fastener Bar Material and Preparation

Alloy steel and titanium fasteners were used for Task 5 testing. They were installed in aluminum alloy bars or blocks which contained nine fastener holes.

The steel fasteners were 4130 alloy and conformed to NAS584-9T. These fasteners, as procured to the standard, were cadmium plated and chromate-conversion-coated. The cadmium was chemically stripped and then they were IVD aluminum-coated. The IVD aluminum coating was applied to conform to Class 3 (0.3-mil thick minimum) per MIL-C-83488. The actual applied thickness was reported to be in the range of 0.3-0.5 mil.

The titanium fasteners conformed to ST3M732-4L8. These fasteners, as procured to the standard, were IVD aluminum-coated and chromate conversion-coated. Since they were chromate conversion-coated, the IVD aluminum coating was chemically stripped from the fasteners and then they were recoated with IVD aluminum. The IVD aluminum coating was applied to conform to Class 3 (0.3-mil thick minimum) per MIL-C-83488. The actual applied thickness was reported to be in the range of 0.3-0.5 mil.

In accordance with standard practice, the IVD aluminum coating on all of the fasteners was glass-bead-peened prior to application of the various conversion coatings. The conversion coatings were applied to the fasteners within 24 hours after the peening operation.

The aluminum alloy bars or blocks were machined from 7075-T651 plate, 0.5 inch thick, conforming to QQ-A-250/12. The final size of each of the machined bars was 1 inch wide x 9 inches long x 0.5 inch thick. After machining, the bars were sulfuric-acid-anodized and dichromate-sealed per MDA process specification P.S. 13201, Type II, Class 1. After anodizing and sealing, nine 1/4-inch diameter holes on one inch centers were drilled in each bar. Finally, the required countersinks were drilled. Figure 77 shows a completed fastener bar and a representative conversion-coated fastener ready for installation.

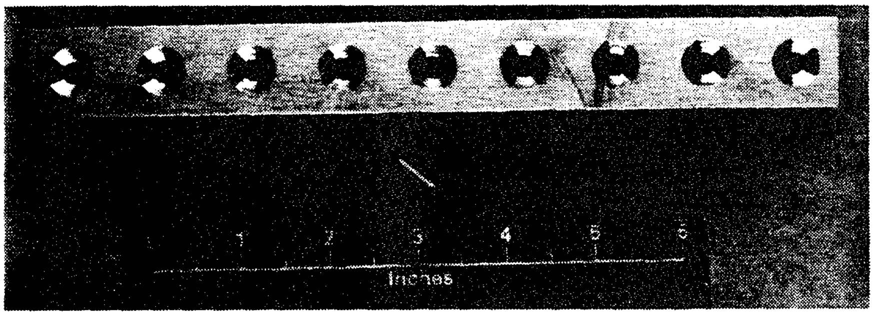

Figure 77. Aluminum-Alloy Bar Containing Countersunk Fastener Holes With Representative IVD Aluminum-Coated/Conversion-Coated Fastener Ready for Installation

The fasteners were torqued to approximately 50 in.-lbs. and the washers and nuts on the back side of the fastener bars were potted with MIL-S-83430, Class B polysulfide sealant. Figure 78 shows a completed fastener bar specimen ready for salt spray testing.

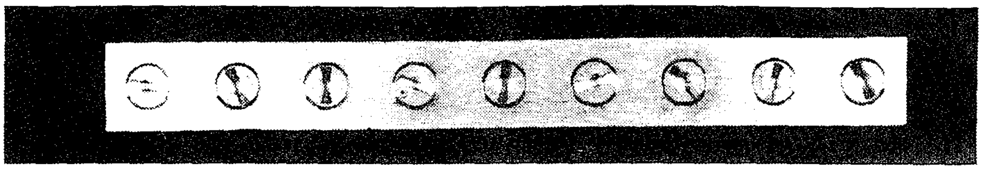

Figure 78. Completed Fastener Bar Specimen Ready For Salt Spray Testing

3. Summary of Task 5 Salt Spray Testing

A summary of the salt spray testing performed for the panels and fastener bar specimens under Task 5 is provided by Table 6. The sections which follow this paragraph discuss the results of each test in detail.

For information purposes, it should be noted that the IVD aluminum coating military specification (i.e., MIL-C-83488) does not require salt spray testing using aluminum as a panel substrate material. It was included in this development program, however, since IVD aluminum is used in production at MDA on a variety of aircraft, aluminum alloy parts.

There is no specification requirement to conduct salt spray testing for IVD aluminum-coated fasteners which have been conversion coated and then installed in aluminum bars with bare countersunk holes. The intent of fastener bar testing was to determine relative performance of the conversion coatings and compare this performance with historical data for chromate conversion coatings.

TABLE 6. SUMMARY OF TASK 5 SALT SPRAY TESTING

SPECIMENS	TREATMENTS	TEST	DURATION
Aluminum Panels (10, 2024-T3 & 10, 7075-T6)	Four Candidates & Chromate conversion-coated Controls	5% Neutral Salt Fog	3024 Hrs. (18 Wks.)
Aluminum Panels (10, 2024-T3 & 10, 7075-T6)	Four Candidates & Chromate conversion-coated Controls	SO_2 Salt Fog	168 Hrs. (1 Wk.)
Aluminum Panels (2024-T3 & 7075-T6)1/	Sealing Steps II & III of Sanchem-CC Process & Chromate conversion-coated Controls	5% Neutral Salt Fog (REPEAT TESTING)	3000 Hrs. (18 Wks.)
Fastener Bars (1 bar with steel fasteners & 1 bar with titanium fasteners)	Four Candidates & Chromate conversion-coated Controls	5% Neutral Salt Fog	672 Hrs. (4 Wks.) (steel fasteners); 1008 Hrs. (6 Wks.) (titanium fasteners)
Fastener Bars (1 bar with steel fasteners & 1 bar with titanium fasteners)	Four Candidates & Chromate conversion-coated Controls	SO_2 Salt Fog	168 Hrs. (1 Wk.) (steel & titanium fasteners)
Fastener Bars (1 bar with steel fasteners)	Sealing Step II & Sealing Steps II & III of the Sanchem-CC Process & Chromate conversion-coated Controls	5% Neutral Salt Fog (REPEAT TESTING)	1008 Hrs. (6 Wks.) (steel fasteners)

NOTE: 1/ Ten panels of each alloy for Sealing Steps II & III of the Sanchem-CC process, and five panels of each alloy for chromate conversion-coated controls.

4. Five Percent Neutral Salt Fog Testing of IVD Aluminum-Coated Aluminum Panels (3024 Hours)

The plan was to expose the panels to 5% neutral salt fog for 3000 hours. The panels were exposed for a total of 3024 hours.

In general, most of the candidate nonchromated conversion coatings performed exceptionally well on the IVD aluminum-coated aluminum panels. Most noteworthy in this respect were the panels treated with Sealing Step II of the Sanchem-CC process, improved Alodine 2000, and PERMATREAT 1001. Additional details related to the performance of each conversion coating are presented in the paragraphs which follow.

Both the IVD aluminum-coated 2024-T3 and 7075-T6 panels treated with Sealing Step II of the Sanchem-CC process showed outstanding performance at the conclusion of salt spray testing. A few significant pits occurred on a couple of the panels but, of significance, there was no evidence of corrosion products associated with these pits. Also, several of the panels had a number of pits in the IVD aluminum coating, but most importantly, all were very small and there was no evidence of IVD aluminum depletion or corrosion products associated with any of the pits. Figure 79 shows two representative 2024-T3 panels at the conclusion of salt spray testing, and Figure 80 shows two representative 7075-T6 panels at the conclusion of salt spray testing.

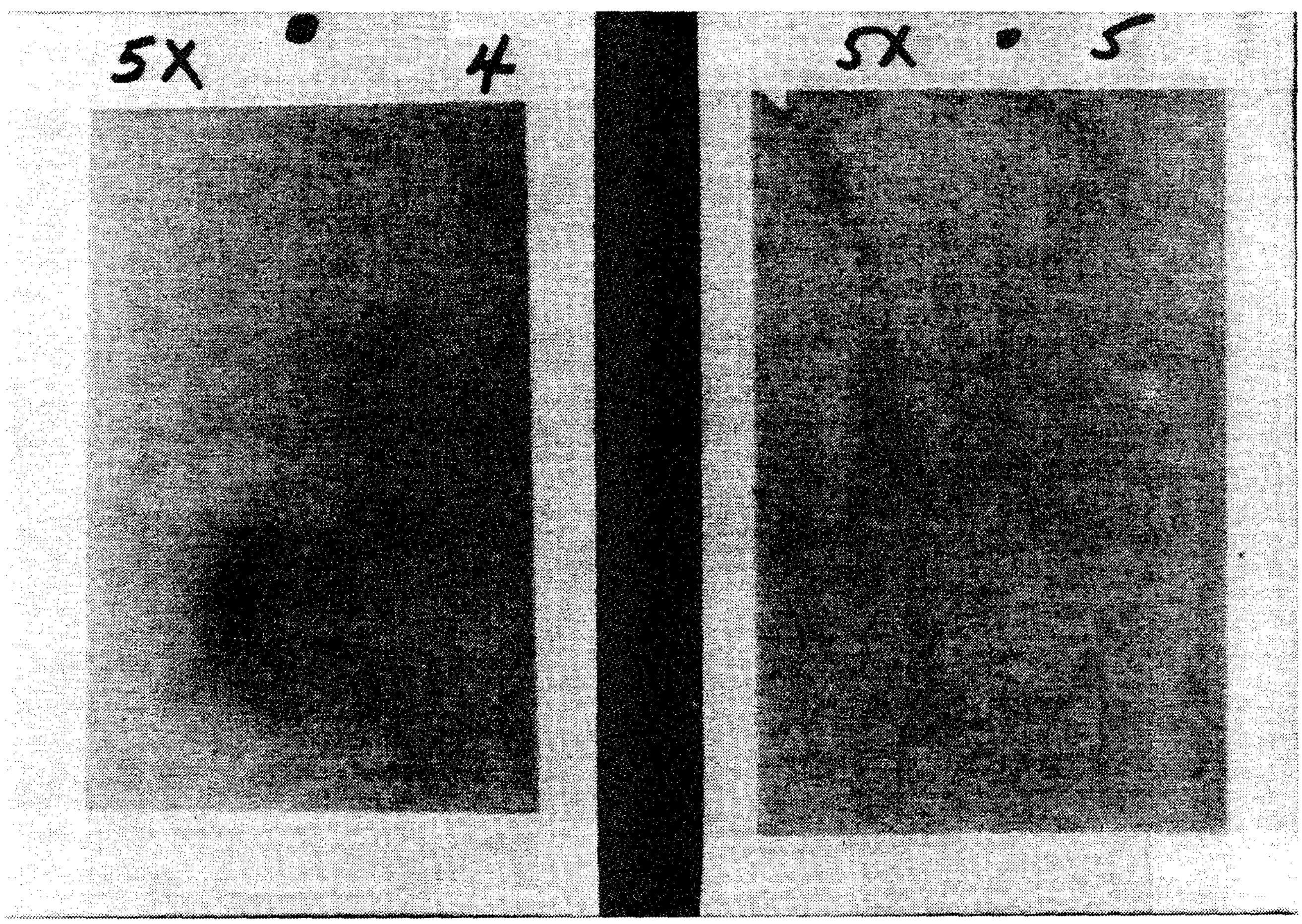

Figure 79. Sealing Step II of the Sanchem-CC Process/IVD Aluminum-Coated 2024-T3 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

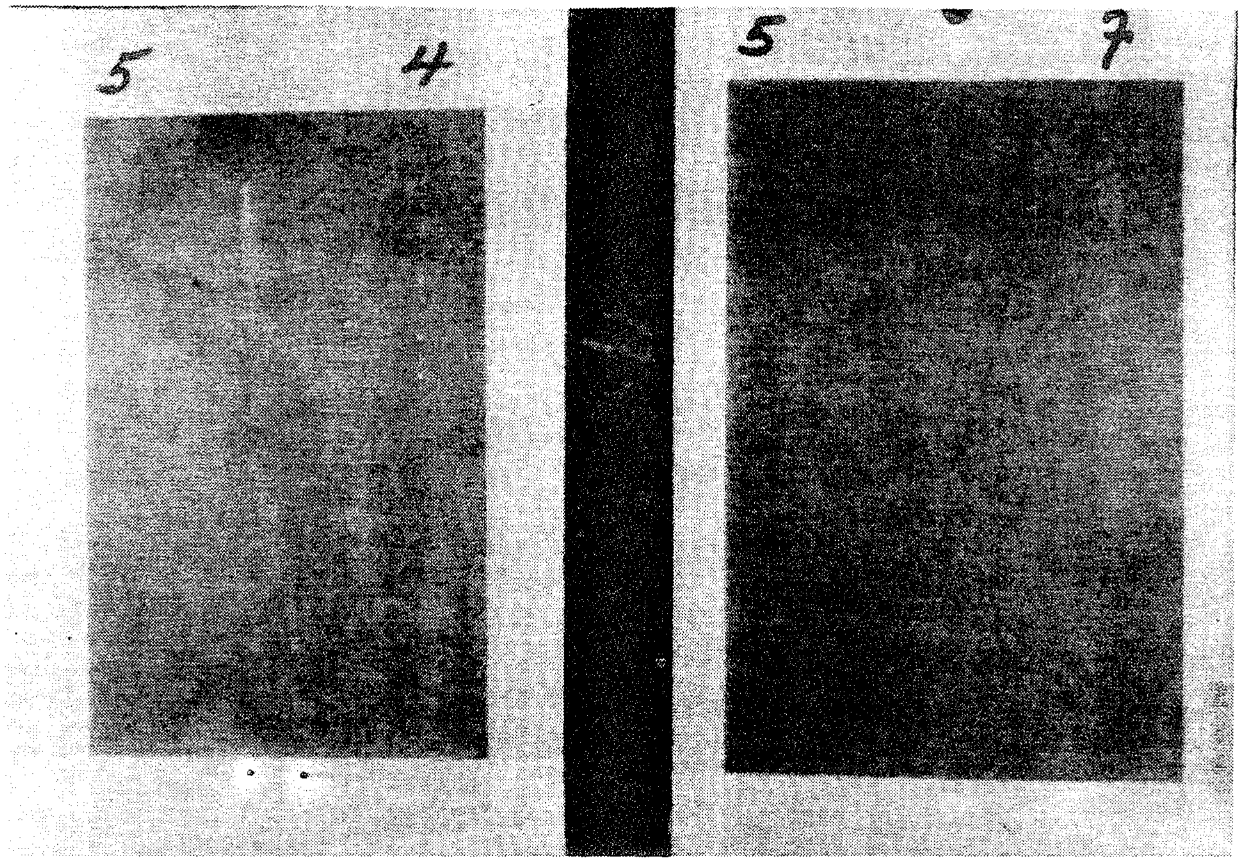

Figure 80. Sealing Step II of the Sanchem-CC Process/IVD Aluminum-Coated 7075-T6 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

Like the panels treated with Sealing Step II of the Sanchem-CC process, the panels treated with improved Alodine 2000 also showed outstanding performance at the conclusion of salt spray testing. Most of the panels had a number of pits in the IVD aluminum coating, but most importantly, all were very small and there was no evidence of IVD aluminum depletion or corrosion products associated with any of the pits. Figure 81 shows two representative 2024-T3 panels at the conclusion of salt spray testing, and Figure 82 shows two representative 7075-T6 panels at the conclusion of salt spray testing.

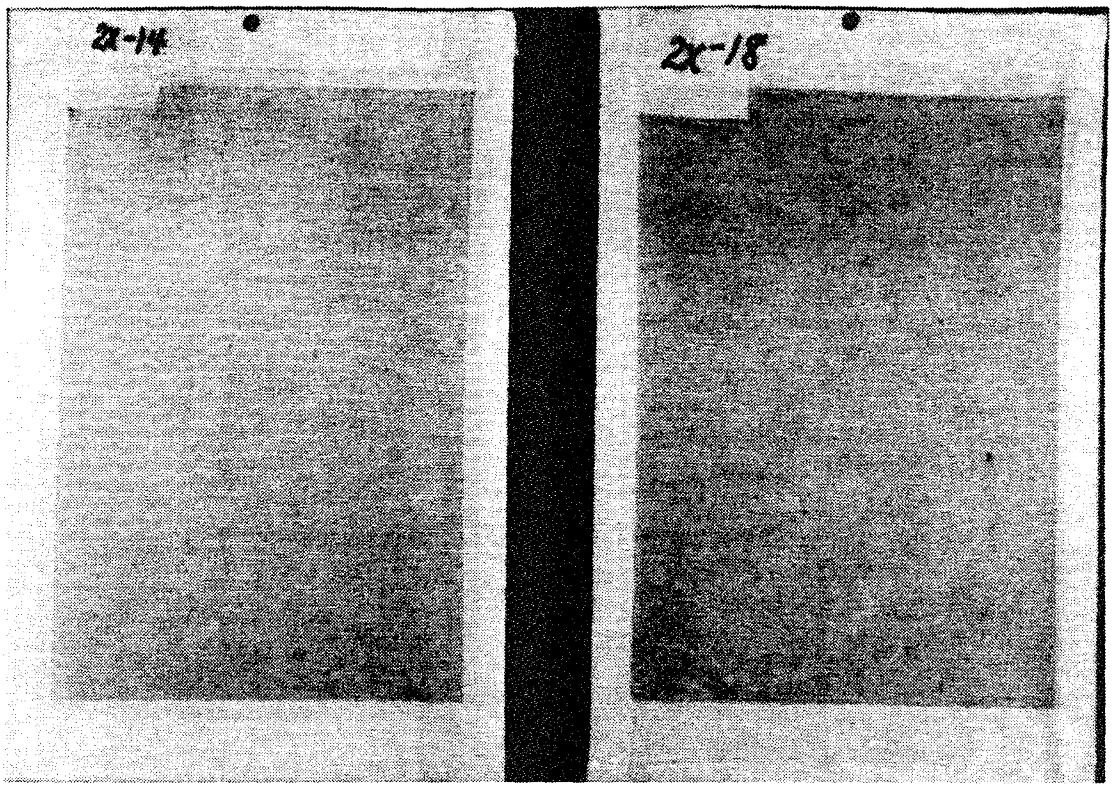

Figure 81. Alodine 2000/IVD Aluminum-Coated 2024-T3 Panels
(After 3024 Hours of 5% Neutral Salt Fog Testing)

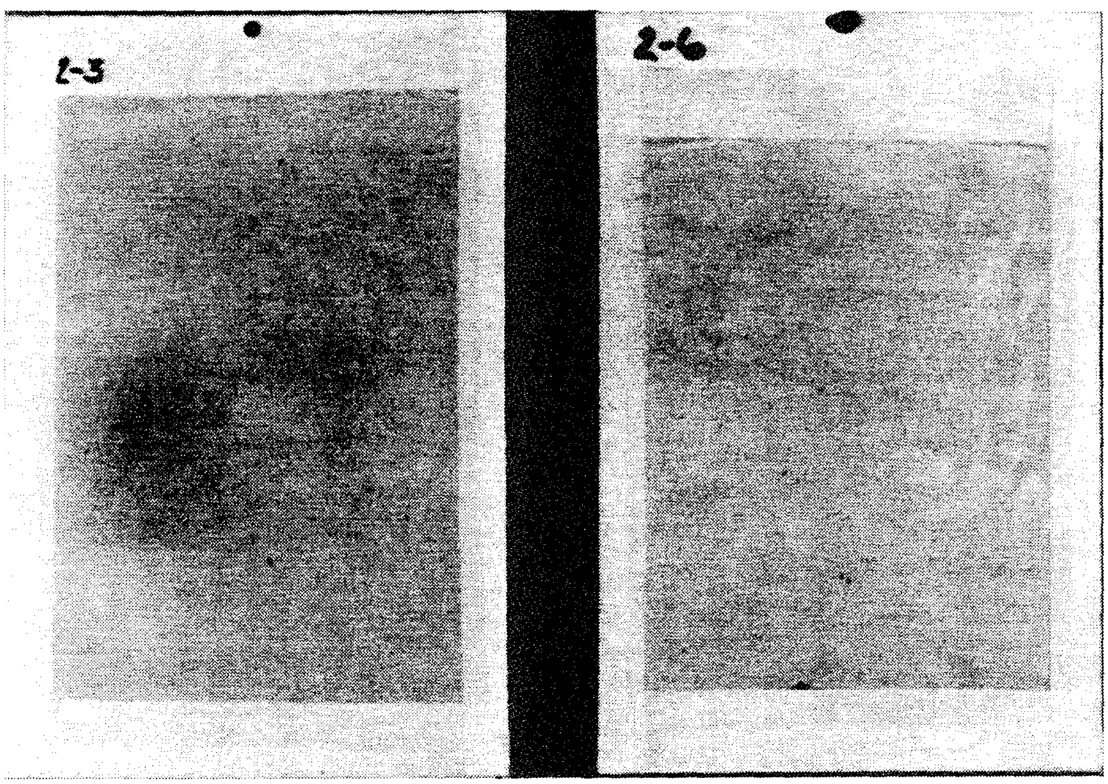

Figure 82. Alodine 2000/IVD Aluminum-Coated 7075-T6 Panels
(After 3024 Hours of 5% Neutral Salt Fog Testing)

Like the Sealing Step II and improved Alodine 2000 panels, the IVD aluminum-coated 2024-T3 and 7075-T6 panels treated with PERMATREAT 1001 also showed outstanding performance at the conclusion of salt spray testing. Most of the panels had a number of pits in the IVD aluminum coating but, most important, all were very small and there was no evidence of IVD aluminum depletion or corrosion products associated with any of the pits. There was one exception, however, where one of the panels had several pits with corrosion products. Figure 83 shows two representative 2024-T3 panels at the conclusion of salt spray testing, and Figure 84 shows two representative 7075-T6 panels at the conclusion of salt spray testing.

In general, the 7075-T6 panels treated with Sealing Steps II and III of the Sanchem-CC process showed good performance at the conclusion of salt spray testing. This performance was in spite of the fact that five of the original ten panels exhibited a wrinkled condition of the IVD aluminum coating very early in testing. One of the five wrinkled panels was removed after 336 hours of salt spray testing and then cross-sectioned. As expected, photomicrographs revealed a loss of adhesion between layers of the IVD aluminum coating. One of the panels was removed from test after 2592 hours of exposure due to reaching the arbitrary removal criteria of three pits with significant corrosion products. Two of the remaining eight 7075-T6 panels which completed the full 3024 hours of salt spray testing are shown in Figure 85.

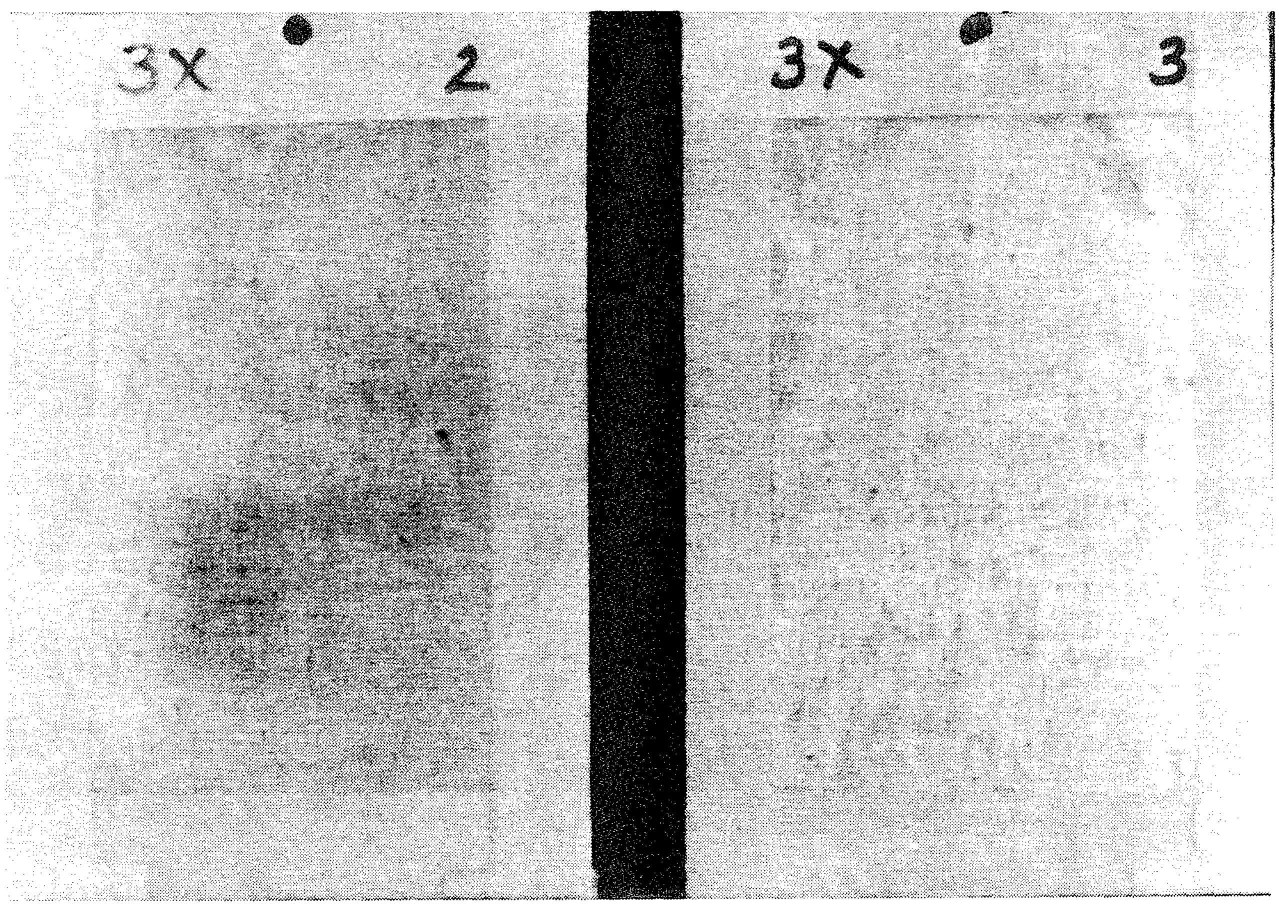

Figure 83. Permatreat 1001/IVD Aluminum-Coated 2024-T3 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

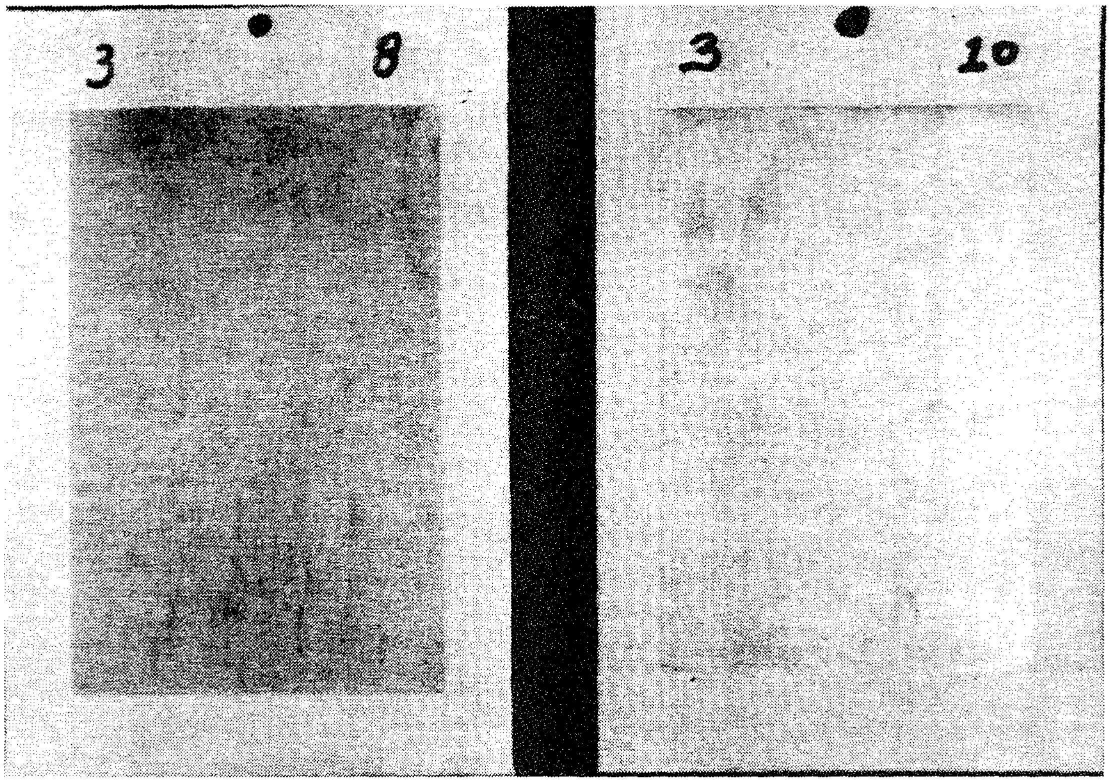

Figure 84. Permatreat 1001/IVD Aluminum-Coated 7075-T6 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

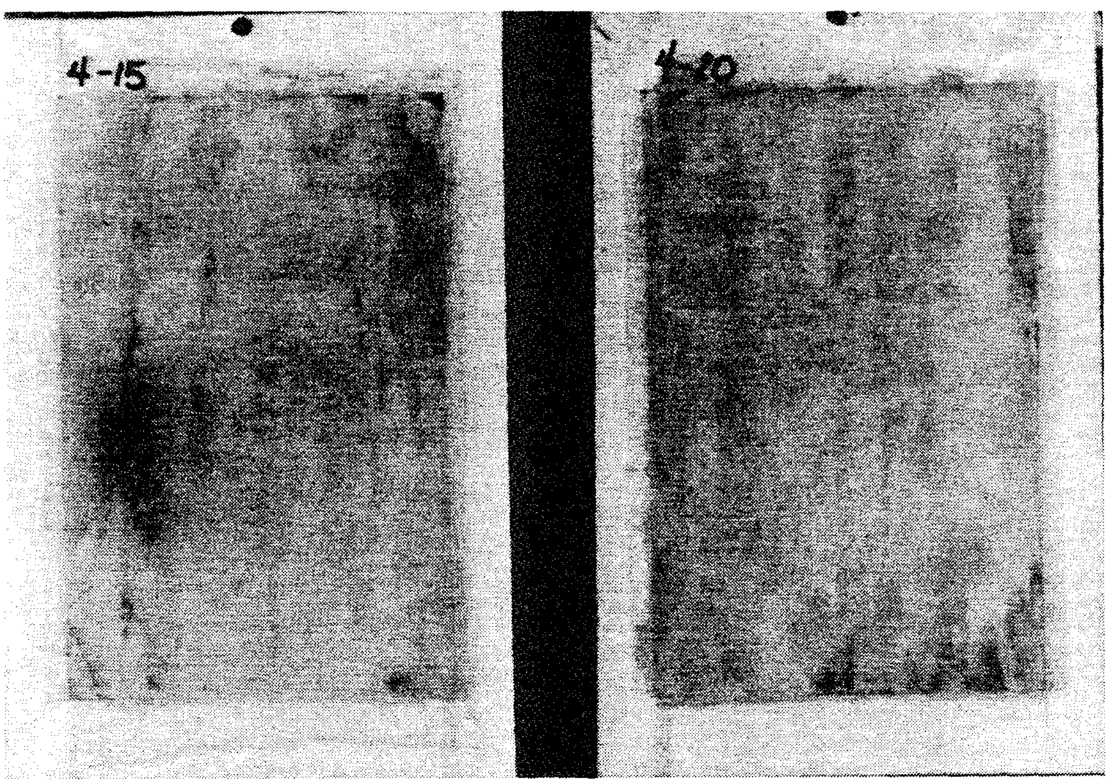

Figure 85. Sealing Steps II & III of the Sanchem-CC Process/IVD Aluminum-Coated 7075-T6 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

The 2024-T3 panels treated with Sealing Steps II and III of the Sanchem-CC process, on the other hand, did not perform as well as the 7075-T6 panels. In addition to the wrinkling, a number of the panels developed pits with corrosion products. Along this line, four of the ten test panels were removed after 2592 hours of salt spray exposure due to reaching the arbitrary removal criteria of three pits with significant corrosion products. Two of the remaining six 2024-T3 panels which completed the full 3024 hours of salt spray testing are shown in Figure 86.

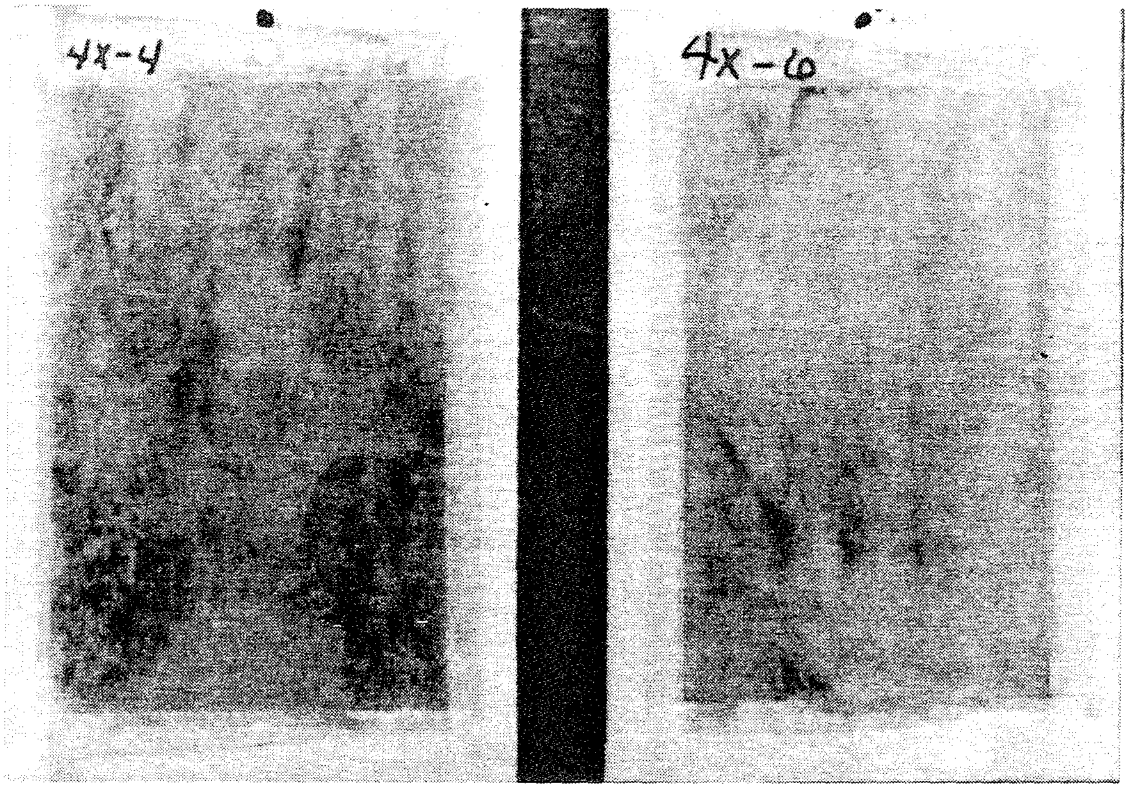

Figure 86. Sealing Steps II & III of the Sanchem-CC Process/IVD Aluminum-Coated 2024-T3 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

The chromate conversion-coated, 2024-T3 controls showed outstanding performance at the conclusion of salt spray testing. Like the Sealing Step II, improved Alodine 2000, and PERMATREAT 1001 treated panels, pits did occur in the IVD aluminum coating on some of the panels. However, also like these other treatments, the pits were very small and there was no evidence of IVD aluminum depletion or corrosion products associated with any of the pits. Figure 87 shows two representative panels at the conclusion of salt spray testing.

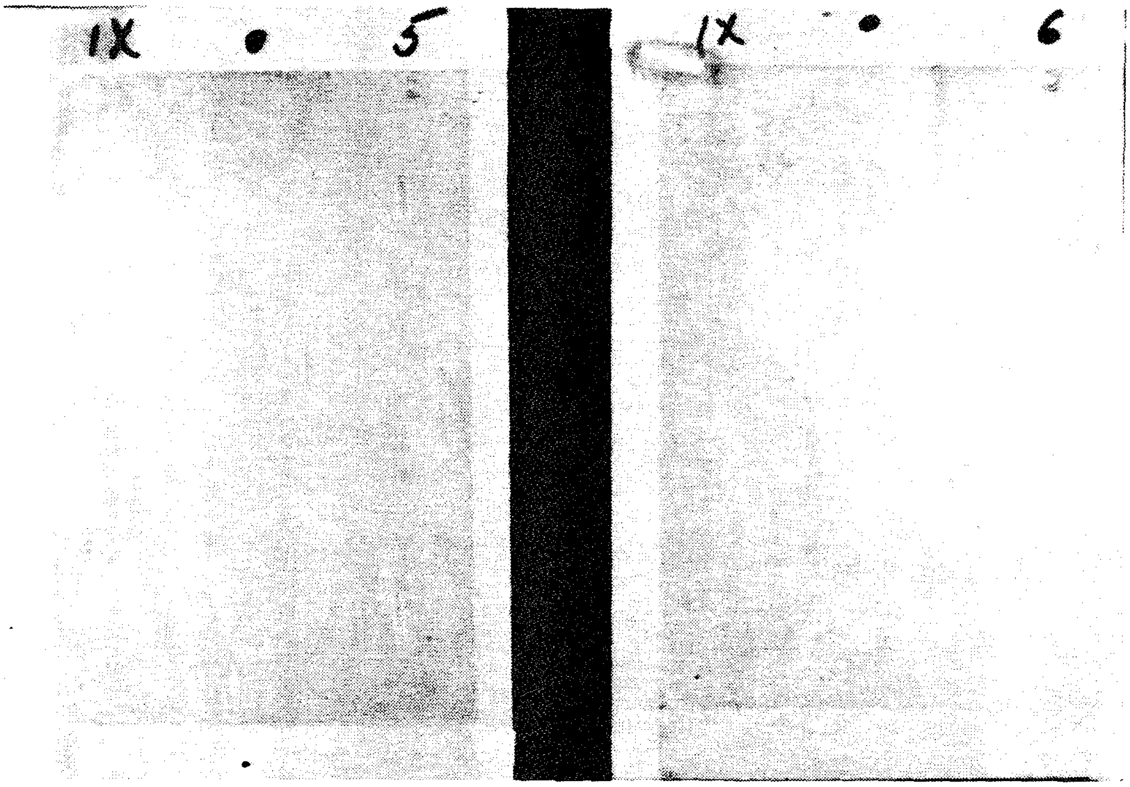

Figure 87. Chromate Conversion-Coated/IVD Aluminum-Coated 2024-T3 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

The chromate conversion-coated, 7075-T6 controls, on the other hand, did not perform as well as expected. A number of the panels developed pits with corrosion products. Along this line, one of the panels was removed after 336 hours of salt spray exposure for cross-sectioning due to pitting. Photomicrographs revealed that the pits extended into the base metal. Five other panels were removed after 2592 hours of salt spray exposure due to reaching the arbitrary removal criteria of three pits with significant corrosion products. Two of the remaining four panels which completed the full 3024 hours of salt spray testing are shown in Figure 88.

As previously mentioned, basically all of the panels treated with Sealing Step II of the Sanchem-CC process, improved Alodine 2000, and PERMATREAT 1001 showed outstanding performance at the conclusion of testing. It was further reported that there was some evidence of minor pitting in the IVD aluminum coating in regard to all three of these treatments. Of importance, this same type of minor pitting was observed for the chromate conversion-coated, 2024-T3 controls. Furthermore, all three of the aforementioned treatments performed much better than the chromate conversion-coated, 7075-T6 controls. Based on the above, then, it was concluded that Sealing Step II of the Sanchem-CC process, improved Alodine 2000, and PERMATREAT 1001 performed at least equal to the chromate conversion-coated controls.

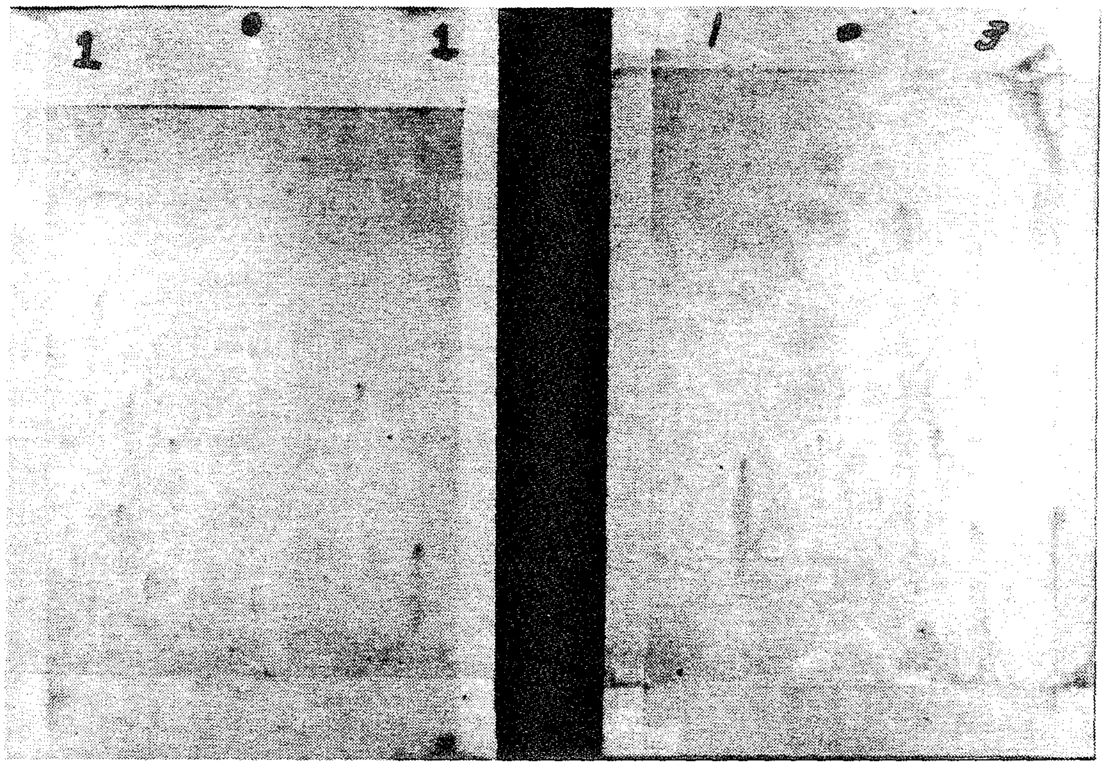

Figure 88. Chromate Conversion Coated/IVD Aluminum-Coated 7075-T6 Panels (After 3024 Hours of 5% Neutral Salt Fog Testing)

At the conclusion of 5% neutral salt fog testing, the performance of the conversion coatings on the 2024-T3 panels was ranked as follows:

1. Chromate Conversion Coating
   Sealing Step II of the Sanchem-CC Process
   Improved Alodine 2000
   PERMATREAT 1001

2. Sealing Steps II and III of the Sanchem-CC Process

At the conclusion of 5% neutral salt fog testing, the performance of the conversion coatings on the 7075-T6 panels was ranked as follows:

1. Sealing Step II of the Sanchem-CC Process
   Improved Alodine 2000

2. PERMATREAT 1001

3. Sealing Steps II and III of the Sanchem-CC Process

4. Chromate Conversion Coating

5. Five Percent Neutral Salt Fog Testing of IVD Aluminum-Coated Aluminum Panels (Repeat Testing for 3000 Hours)

A decision was made to repeat 5% neutral salt fog testing for IVD aluminum-coated 2024-T3 and 7075-T6 panels which had been treated with chromate conversion coating and Sealing Steps II and III of the Sanchem-CC process. This decision was based on the wrinkling associated with both the 2024-T3 and 7075-T6 panels treated with Sealing Steps II and III of the Sanchem-CC process. It was also based on significant pitting and corrosion products for the 2024-T3 panels treated with Sealing Steps II and III of the Sanchem-CC process and the 7075-T6 panels treated with chromate conversion coating.

Five 2024-T3 panels and five 7075-T6 panels were prepared for chromate conversion coating, and ten panels for each aluminum alloy were prepared for Sealing Steps II and III of the Sanchem-CC process. Preparation of the panels was identical to that previously described for the original Task 5 salt spray test panels. The average thickness of the IVD aluminum coating for these panels was 1.9 mils. This thickness was based on measurements made on steel panels which were IVD aluminum-coated at the same time as the aluminum panels. The treated panels were subjected to 5% neutral salt fog testing for 3000 hours.

As expected, none of the panels treated with Sealing Steps II and III of the Sanchem-CC process showed any evidence of the wrinkling encountered previously. However, all of these panels did show pits in the IVD aluminum coating, and these pits appeared sooner compared to the equivalent panels tested initially under Task 5. Of significance, it should be noted that the pits on most of the panels were relatively small, and there was no evidence of obvious corrosion products associated with any of the pits. Figure 89 shows two representative, 2024-T3 panels at the conclusion of salt fog testing, while Figure 90 shows two representative, 7075-T6 panels at the conclusion of salt fog testing.

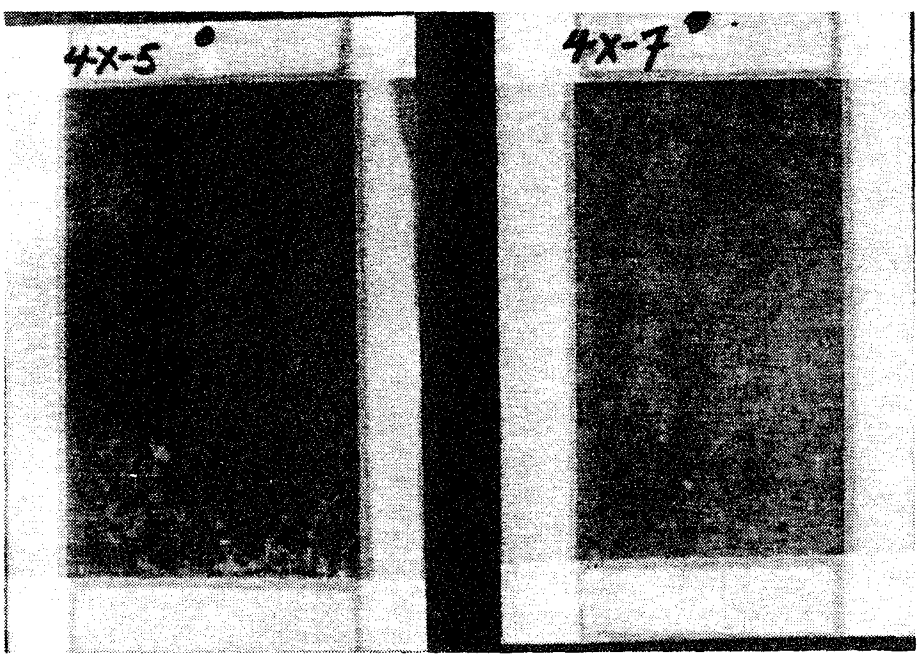

Figure 89. Sealing Steps II & III of the Sanchem-CC Process/IVD Aluminum-Coated 2024-T3 Panels (Task 5 Repeat Testing – After 3000 Hours of 5% Neutral Salt Fog Testing)

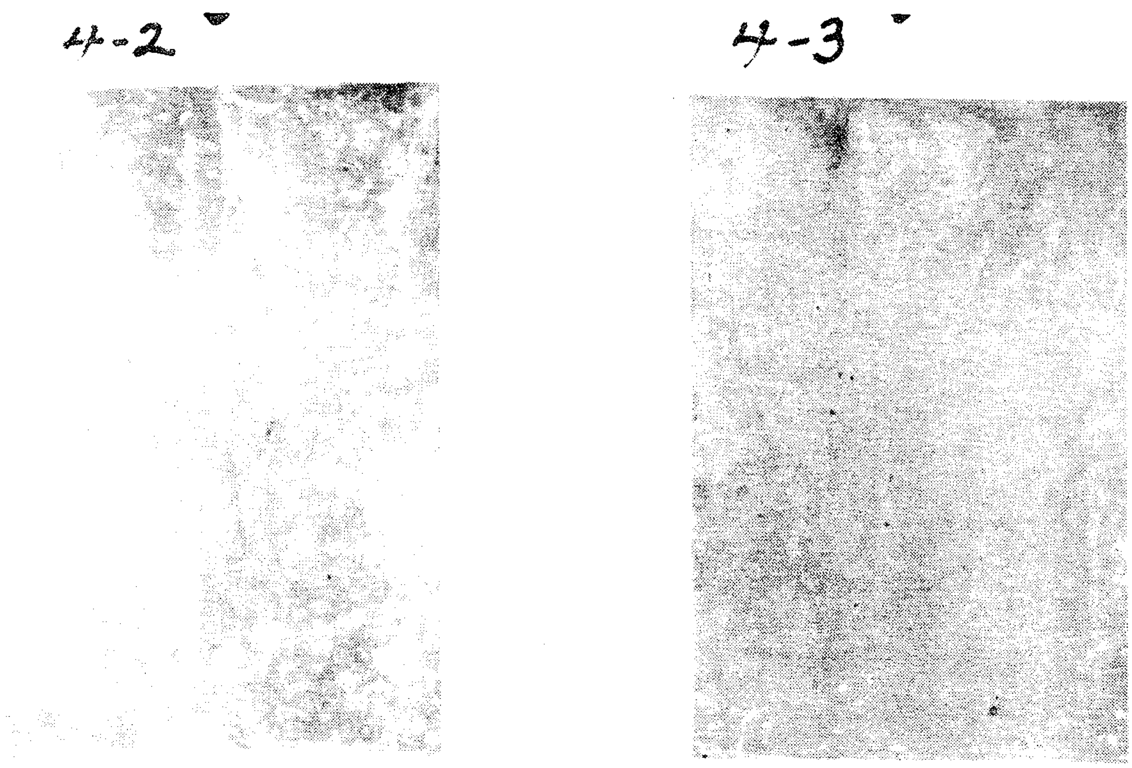

Figure 90. Sealing Steps II & III of the Sanchem-CC Process/IVD Aluminum-Coated 7075-T6 Panels (Task 5 Repeat Testing – After 3000 Hours of 5% Neutral Salt Fog Testing)

Additional 5% neutral salt fog testing was conducted for IVD aluminum-coated 2024-T3 and 7075-T6 panels which had been treated with Sealing Steps II and III of the Sanchem-CC process. It was conducted under Task 6 of this program as part of the process optimization study. Four 2024-T3 and four 7575-T6 panels were exposed to 5% neutral salt fog for 672 hours (four weeks). As in previous testing of equivalent panels under this Task 5, small pits occurred in the IVD aluminum coating early in testing in regard to these panels.

Four of the five chromate conversion-coated, 7075-T6 panels showed evidence of very minor pits in the IVD aluminum coating. However, there was no evidence of obvious corrosion products and all were smooth to the touch. The chromate conversion-coated, 7075-T6 panels are considered to have shown outstanding performance at the conclusion of testing, which was not the case for the equivalent panels tested initially under Task 5. Figure 91 shows two representative panels at the conclusion of salt fog testing.

The chromate conversion-coated, 2024-T3 panels did not show the same outstanding performance as the equivalent panels tested initially. Two of the five panels showed evidence of small pits in the IVD aluminum coating, and another panel had significant pits with corrosion products. In spite of the above, however, the chromate conversion-coated, 2024-T3 panels were still considered to have shown good performance based on 3000 hours of 5% neutral salt fog testing.

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Figure 91. Chromate Conversion-Coated/IVD Aluminum-Coated 7075-T6 Panels (Task 5 Repeat Testing – After 3000 Hours of 5% Neutral Salt Fog Testing)

6. Sulfur Dioxide (SO_2) Salt Fog Testing of IVD Aluminum-Coated Aluminum Panels (168 Hours)

The target exposure period for sulfur dioxide (SO_2) salt fog testing of the IVD aluminum-coated aluminum panels was 500 hours. This time period was more of a potential upper limit rather than a definite goal due to the severity of the environment (Reference the discussion under Task 4 SO_2 salt fog testing in regard to the severity of the environment).

Sulfur dioxide salt fog testing was stopped after 168 hours due to the condition of the panels. It was considered more prudent to stop at this point when meaningful comparisons could still be made. Representative panels, at the end of the exposure period, are shown in Figures 92 – 101. The 2024-T3 panels are shown in Figures 92 – 96, and the 7075-T6 panels are shown in Figures 97 – 101.

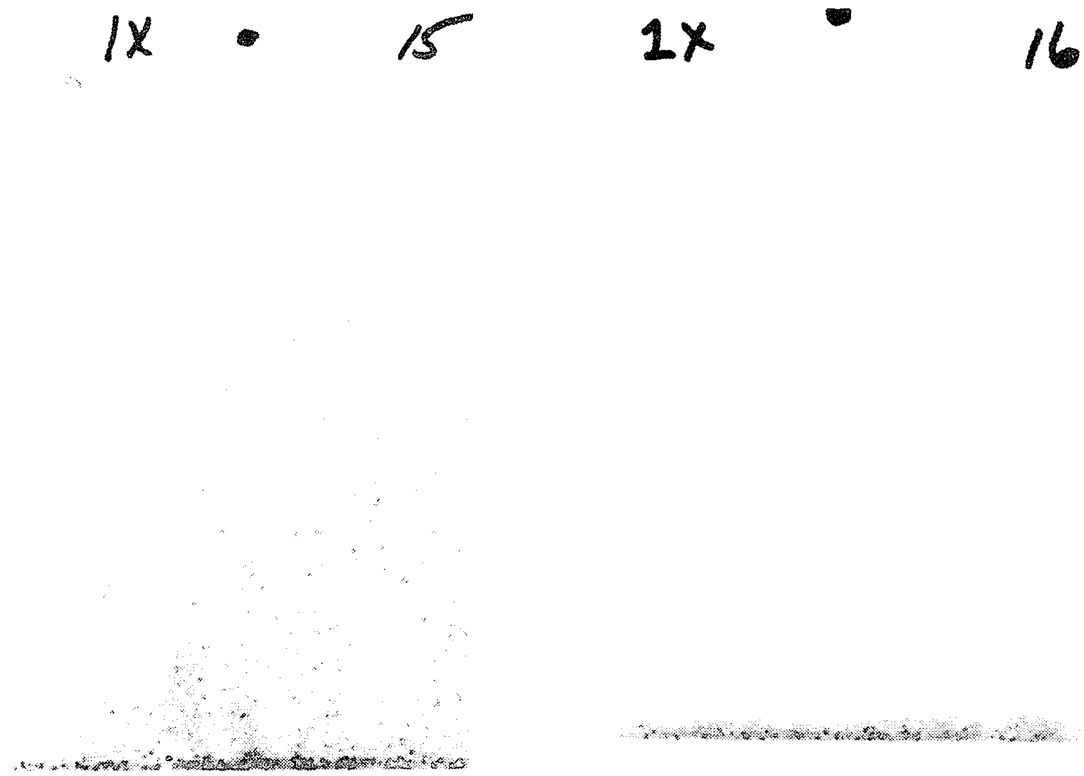

Figure 92. Chromate Conversion-Coated/IVD Aluminum-Coated 2024-T3 Panels (After 168 Hours of SO_2 Salt Fog Testing)

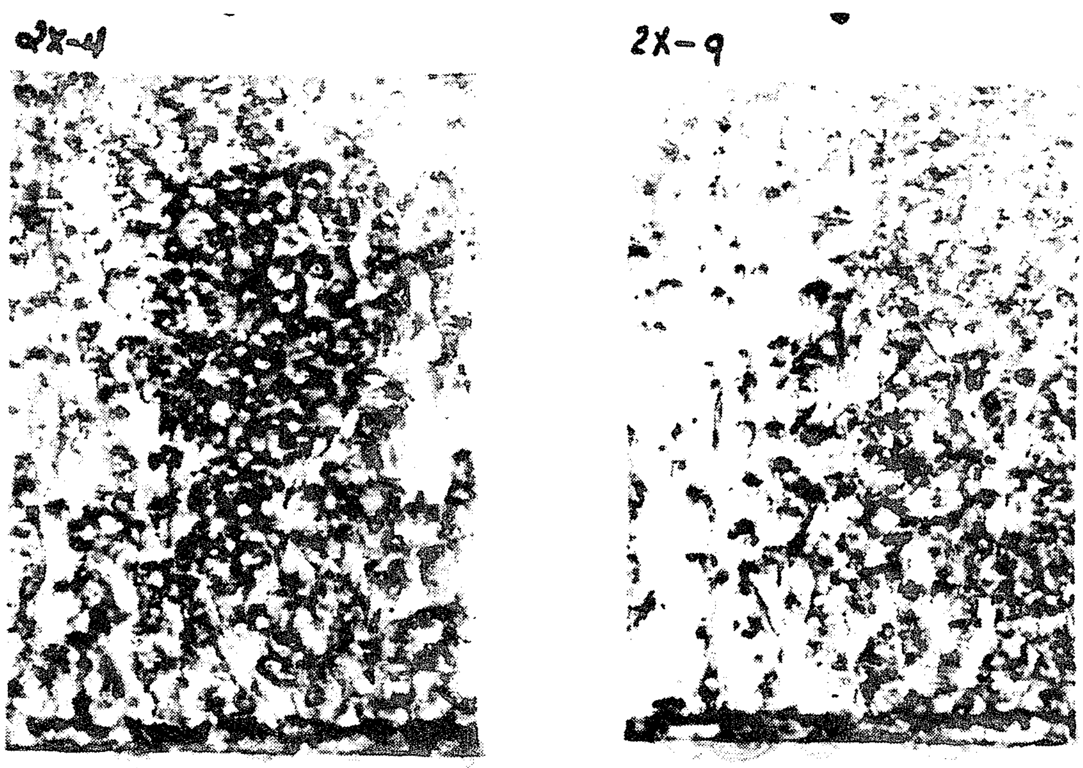

Figure 93. Alodine 2000/IVD Aluminum-Coated 2024-T3 Panels (After 168 Hours of SO_2 Salt Fog Testing)

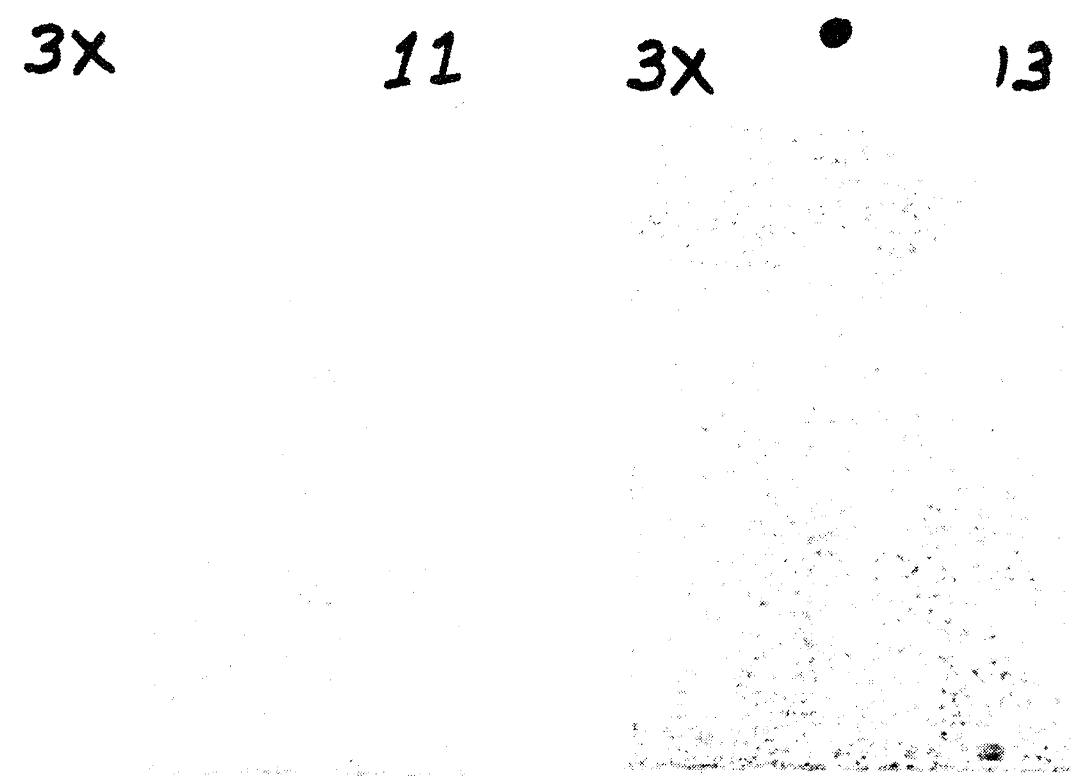

Figure 94. Permatreat 1001/IVD Aluminum-Coated 2024-T3 Panels (After 168 Hours of SO_2 Salt Fog Testing)

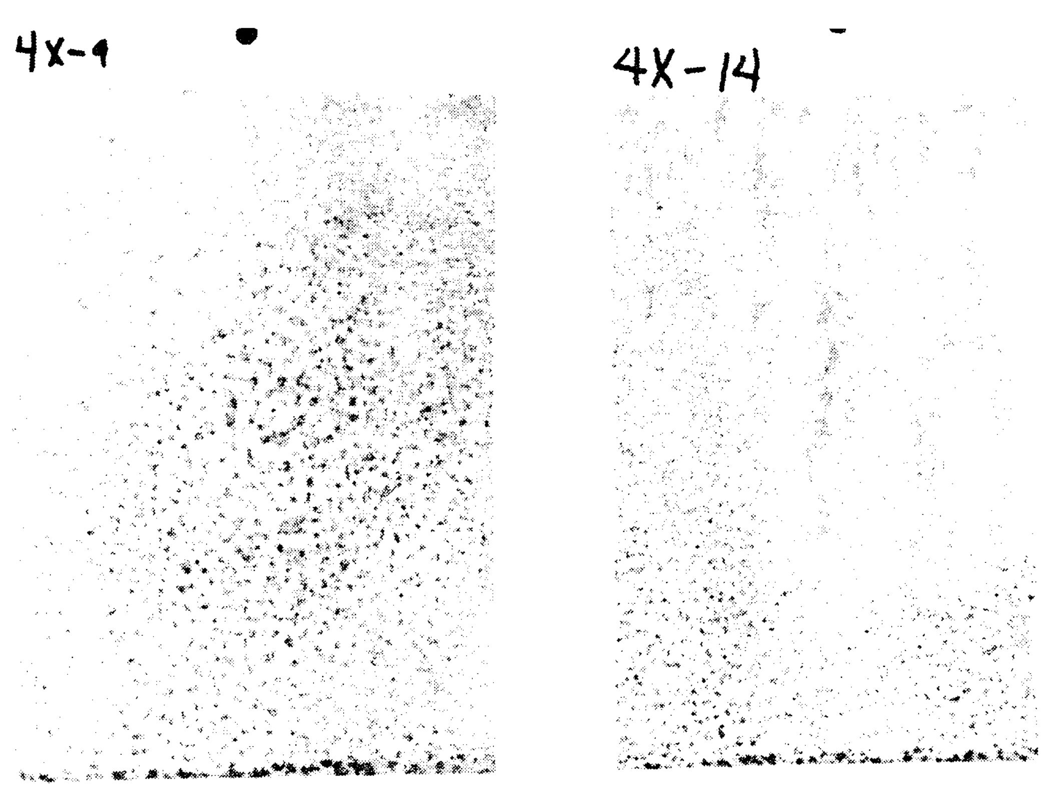

Figure 95. Sealing Steps II & III of the Sanchem-CC Process/IVD Aluminum-Coated 2024-T3 Panels (After 168 Hours of SO_2 Salt Fog Testing)

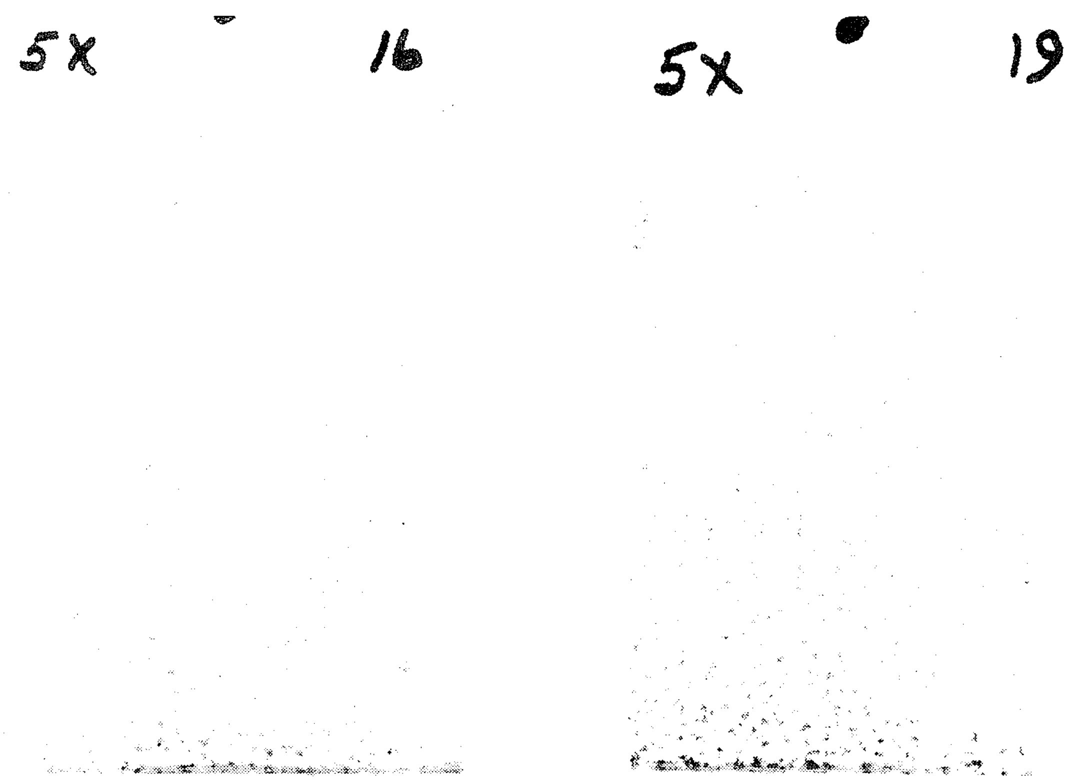

Figure 96. Sealing Step II of the Sanchem-CC Process/IVD Aluminum-Coated 2024-T3 Panels (After 168 Hours of SO_2 Salt Fog Testing)

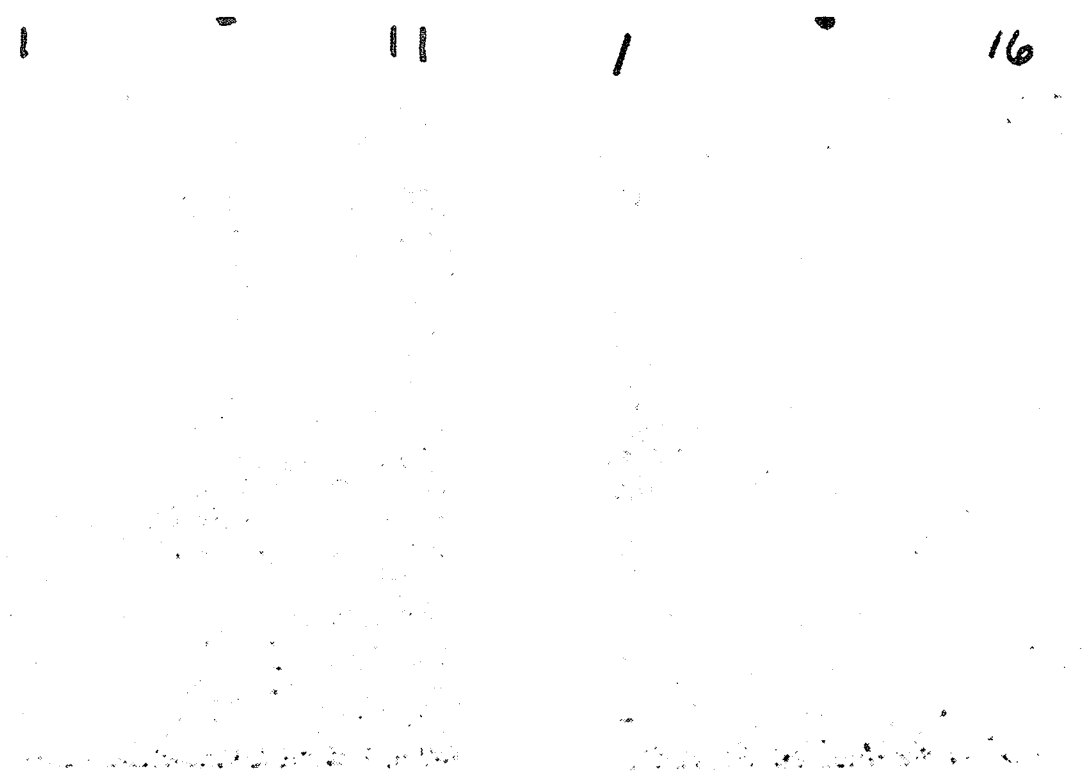

Figure 97. Chromate Conversion-Coated/IVD Aluminum-Coated 7075-T6 Panels (After 168 Hours of SO_2 Salt Fog Testing)

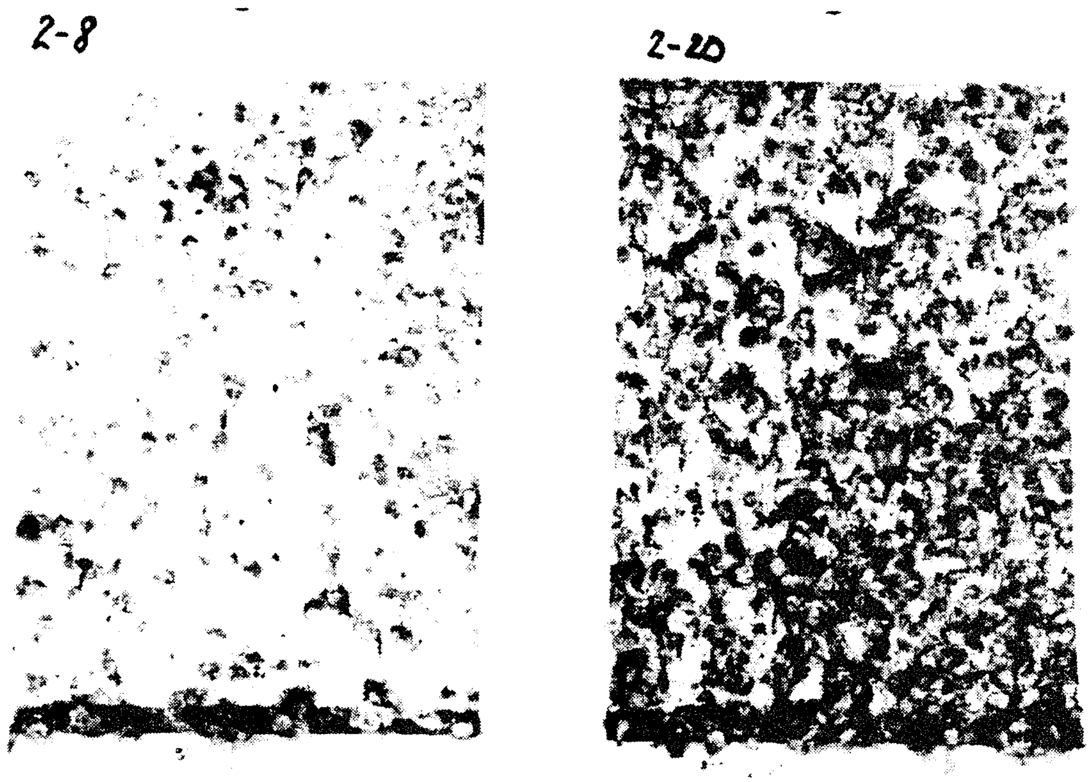

Figure 98. Alodine 2000/IVD Aluminum-Coated 7075-T6 Panels
(After 168 Hours of SO_2 Salt Fog Testing)

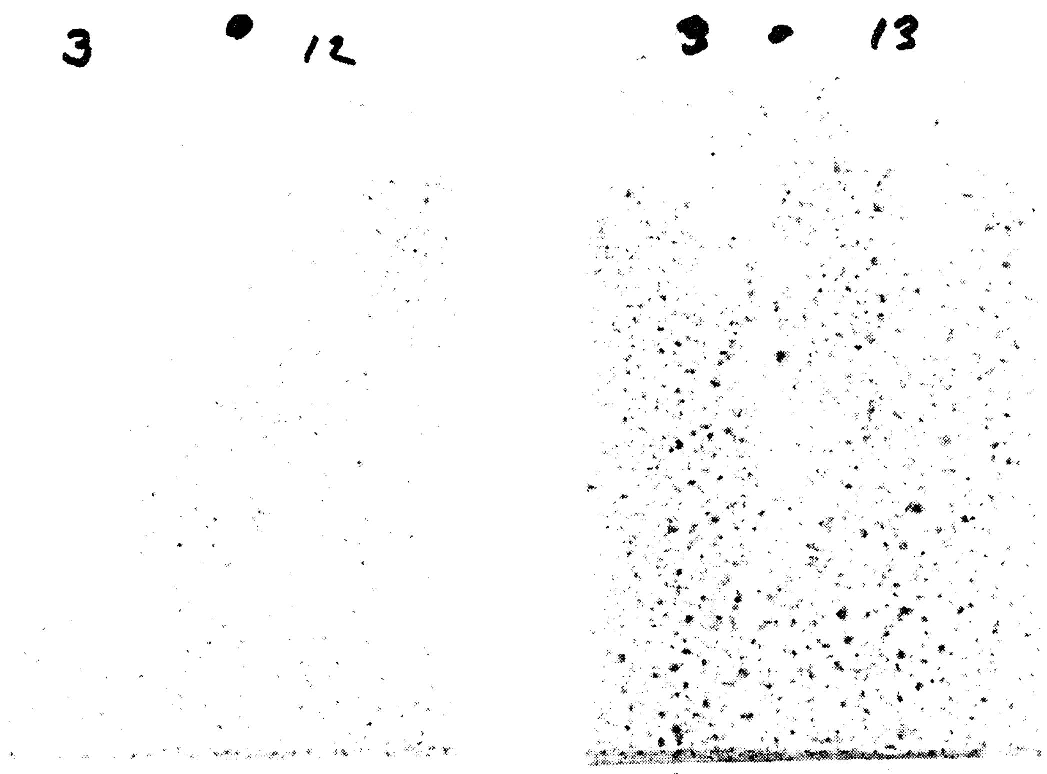

Figure 99. Permatreat 1001/IVD Aluminum-Coated 7075-T6 Panels
(After 168 Hours of SO_2 Salt Fog Testing)

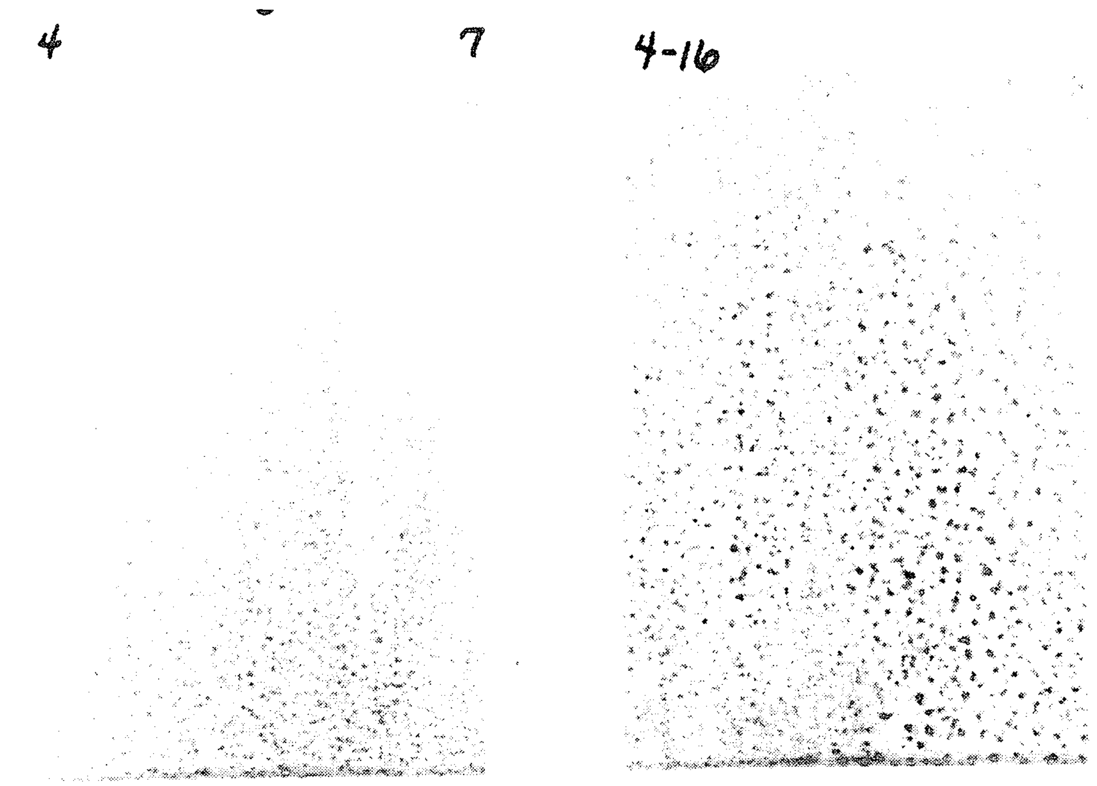

Figure 100. Sealing Steps II & III of the Sanchem-CC Process/IVD Aluminum-Coated 7075-T6 Panels (After 168 Hours of SO_2 Salt Fog Testing)

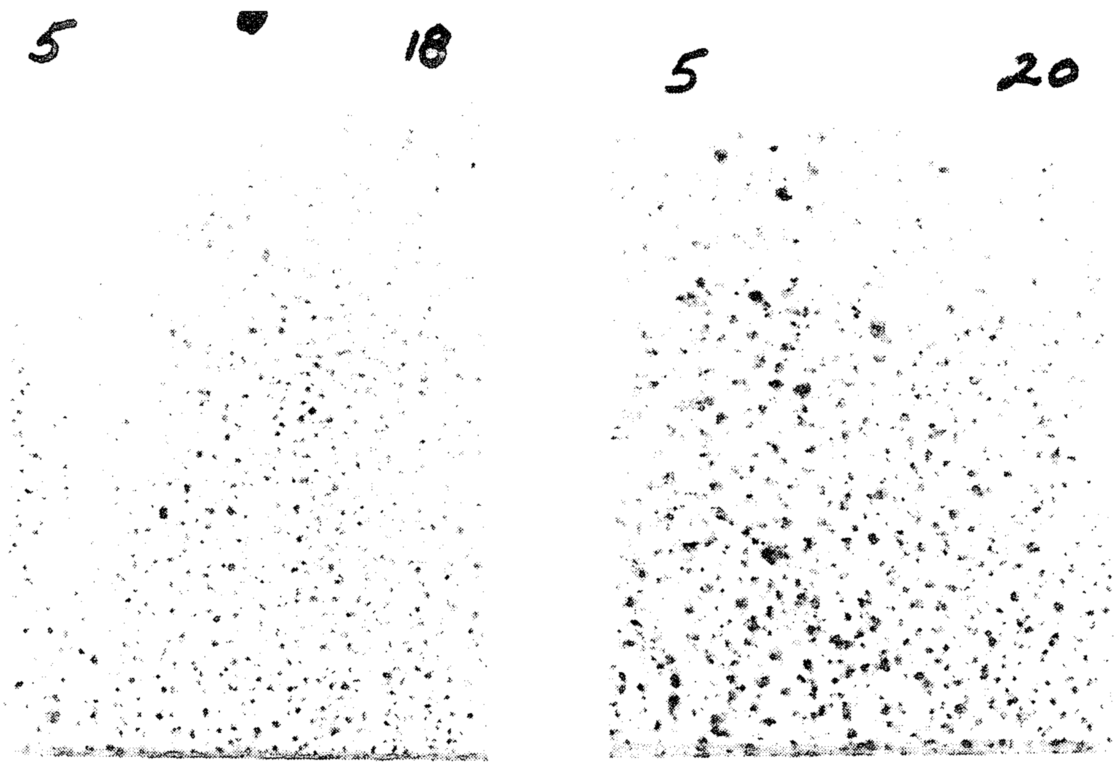

Figure 101. Sealing Step II of the Sanchem-CC Process/IVD Aluminum-Coated 7075-T6 Panels (After 168 Hours of SO_2 Salt Fog Testing)

Following below is the ranking of the 2024-T3 panels at the conclusion (168 hours) of SO_2 salt fog testing.

1. Chromate Conversion Coating

2. Sealing Steps II and III of the Sanchem-CC Process

3. Sealing Step II of the Sanchem-CC Process
   PERMATREAT 1001

4. Improved Alodine 2000

Following below is the ranking of the 7075-T6 panels at the conclusion (168 hours) of SO_2 salt fog testing.

1. Chromate Conversion Coating

2. Sealing Steps II and III of the Sanchem-CC Process

3. Sealing Step II of the Sanchem-CC Process
   PERMATREAT 1001

4. Improved Alodine 2000

In regard to the above completed testing, on the whole, the 2024-T3 panels performed slightly better than the 7075-T6 panels.

7. Five Percent Neutral Salt Fog Testing of Fastener Bar Specimens

The plan, as stated in the technical proposal, was to expose the fastener bar specimens containing both the alloy steel and titanium fasteners to 1008 hours (6 weeks) of 5% neutral salt fog.

Testing was stopped for the fastener bar specimens containing the IVD aluminum-coated steel fasteners after 672 hours (four weeks) of exposure. It was terminated at this time, rather than after the intended 1008 hours of exposure, due to greater than expected visual corrosion. Probable reasons for this less than expected performance in 5% neutral salt fog were identified and are discussed in a subsequent paragraph. As a result of this performance, a decision was made to repeat testing of the steel fasteners in 5% neutral salt fog.

The fastener bar specimens containing the IVD aluminum-coated titanium fasteners completed the planned 1008 hours of exposure to 5% neutral salt fog. These specimens, with the fasteners still installed, are shown in Figure 102 at the conclusion of testing. Following is a cross reference of the fastener bar numbers shown in Figure 102 with the corresponding conversion coating used to treat the IVD aluminum-coated titanium fasteners in the bars.

1T - Chromate Conversion Coating

2T - Improved Alodine 2000

3T - PERMATREAT 1001

4T - Sealing Steps II and III of the Sanchem-CC Process

5T - Sealing Step II of the Sanchem-CC Process

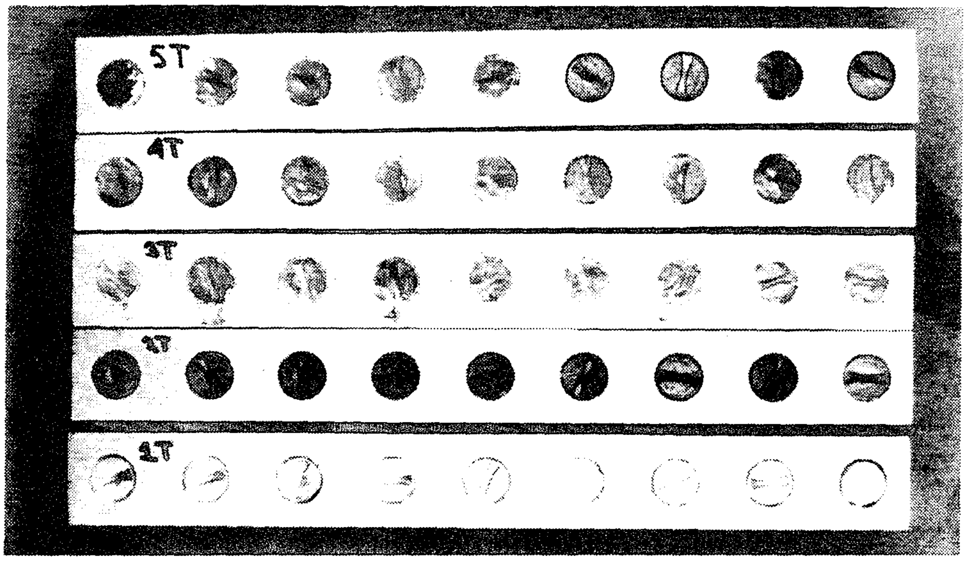

Figure 102. IVD Aluminum-Coated & Conversion-Coated Titanium Fasteners (After 1008 Hours of 5% Neutral Salt Fog Testing)

8. Sulfur Dioxide (SO_2) Salt Fog Testing of Fastener Bar Specimens

The plan, as stated in the technical proposal, was to expose the fastener bar specimens containing both the alloy steel and titanium fasteners to 336 hours (2 weeks) of sulfur dioxide (SO_2) salt fog. This time period was more of a potential upper limit rather than a definite goal due to the severity of the environment (Reference the discussion under Task 4 SO_2 salt fog testing in regard to the severity of the environment).

Testing of the fastener bar specimens was stopped after 168 hours of exposure to SO_2 salt fog. It was stopped at this time, rather than after the intended 336 hours of exposure, due to a level of visual corrosion beyond which it was felt that meaningful results might not be realized upon cross-sectioning of the countersinks.

The performance of the fastener bars containing the IVD aluminum-coated steel fasteners was considered suspect for the same reasons a decision was made to repeat 5% neutral salt fog testing for these fasteners. Therefore, the results of this testing are not presented.

Figure 103 shows the fastener bars with the installed titanium fasteners for each of the four candidate nonchromated conversion coatings and the chromate conversion-coated controls at the conclusion (168 hours) of

SO_2 salt fog testing. Following is a cross reference of the fastener bar numbers shown in Figure 103 with the corresponding conversion coating used to treat the IVD aluminum-coated fastener in the bars.

1T - Chromate Conversion Coating

2T - Improved Alodine 2000

3T - PERMATREAT 1001

4T - Sealing Steps II and III of the Sanchem-CC Process

5T - Sealing Step II of the Sanchem-CC Process

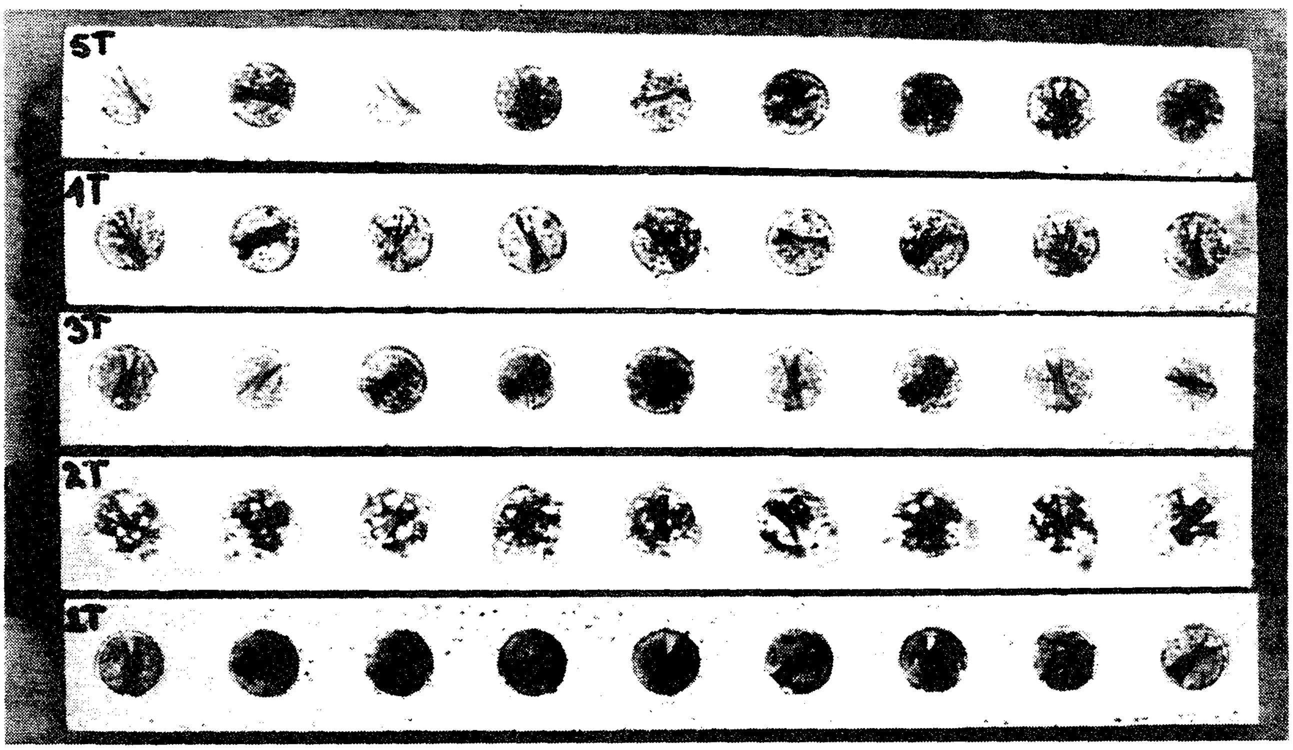

Figure 103. IVD Aluminum-Coated & Conversion-Coated Titanium Fasteners (After 168 Hours of SO_2 Salt Fog Testing)

9. Summary of Results of 5% Neutral Salt Fog and SO_2 Salt Fog Testing for Fastener Bar Specimens Containing IVD Aluminum-Coated Titanium Fasteners

At the conclusion of fastener bar salt spray testing, the fastener bar specimens were visually ranked with the titanium fasteners still installed in the bars. Table 7 summarizes this visual ranking.

TABLE 7. RANKING OF CONVERSION COATINGS BASED ON VISUAL INSPECTION OF FASTENER BAR SPECIMENS AFTER SALT SPRAY TESTING

FASTENER CATEGORY	SALT SPRAY TEST/DURATION	VISUAL RANKING WITH FASTENERS INSTALLED					VISUAL RANKING WITH FASTENERS REMOVED									
							FASTENER CONTACT AREA					"EYEBROW AREA"				
		A[a/]	B[b/]	C[c/]	D[d/]	E[e/]	A	B	C	D	E	A	B	C	D	E
IVD aluminum-coated TITANIUM	5%/6 WKS	1	1	3	2	2	1	2	2	2	2	2	3	3	1	1
	SO_2/1WK	1	3	2	2	2	1	2	2	1	1	2	5	3	3	3

NOTES
- a/ A - Chromate Conversion Coating
- b/ B - Improved Alodine 2000
- c/ C - Permatreat 1001
- d/ D - Sealing Steps II and III of the Sanchem-CC Process
- e/ E - Sealing Step II of the Sanchem-CC Process
- f. Lowest number for ranking indicates best salt spray performance

After the initial visual ranking with the titanium fasteners installed, the fasteners were removed from the fastener bars and the conversion coatings were ranked for each fastener category based on visual appearance of the countersinks. This visual ranking of the countersinks was divided into two categories. One category was for the upper portion of the countersink referred to as the "eyebrow" (i.e., area of bare aluminum between the edge of the fastener and edge of the countersink all around the countersink). The other category was for the remaining area of the bare aluminum countersink where the fastener was in direct contact with the countersink. Figures 104 and 105 show the fastener bar specimens with the titanium fasteners removed. The results of the visual ranking of the bare countersinks for the various conversion coatings are also summarized in Table 7.

After visual examination of the countersinks, two of the nine fastener holes in each of the fastener bar specimens were cross-sectioned. These cross sections were subsequently mounted and polished, and then photomicrographs were taken of the cross sections to determine the degree of corrosion in the countersinks. The two countersinks selected for cross-sectioning in each fastener bar were intended to provide representation for all the countersinks in that particular bar. Figure 106 shows representative photomicrographs of countersinks for the fastener bars subjected to 5% neutral salt fog testing for 6 weeks (1008 hours), and Figure 107 shows representative photomicrographs of countersinks for the fastener bars subjected to SO_2 salt fog testing for 1 week (168 hours).

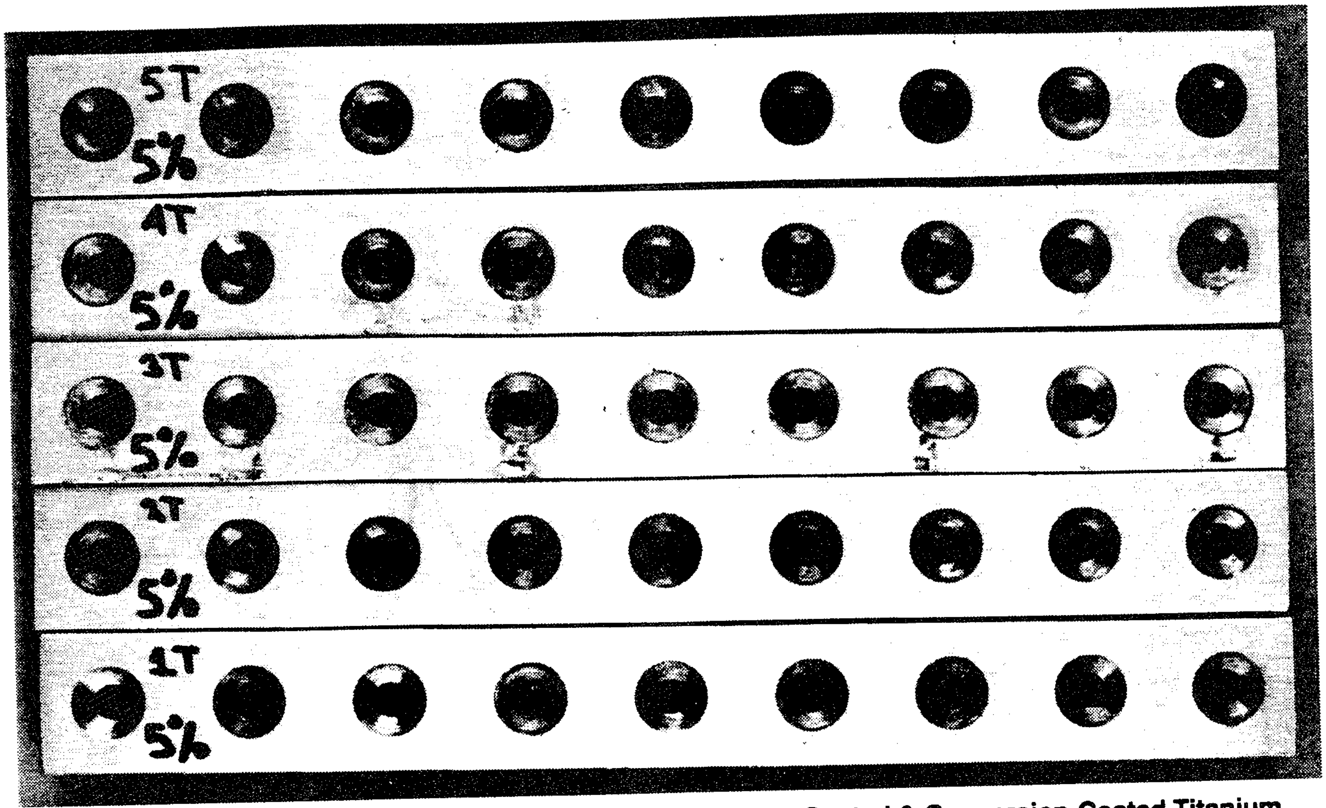

Figure 104. Fastener Bars Which Contained IVD Aluminum-Coated & Conversion-Coated Titanium Fasteners at the Conclusion (1008 Hours) of 5% Neutral Salt Fog Testing

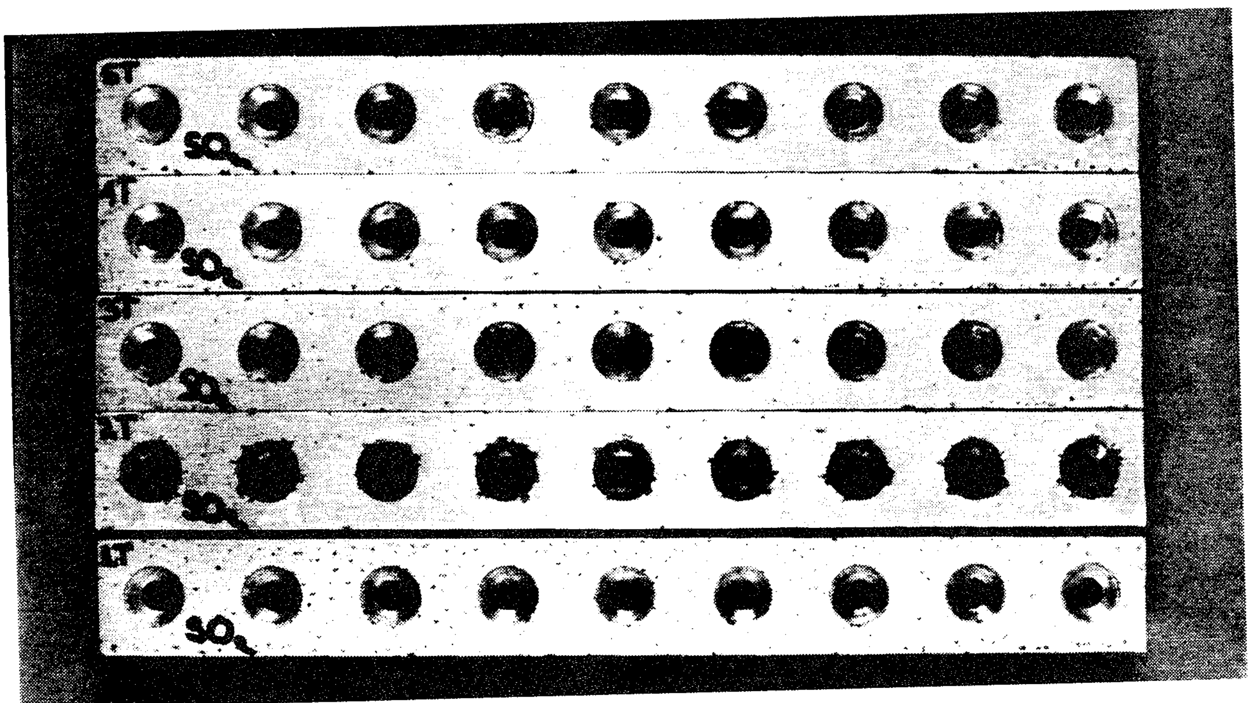

Figure 105. Fastener Bars Which Contained IVD Aluminum-Coated & Conversion-Coated Titanium Fasteners at the Conclusion (168 Hours) of SO_2 Salt Fog Testing

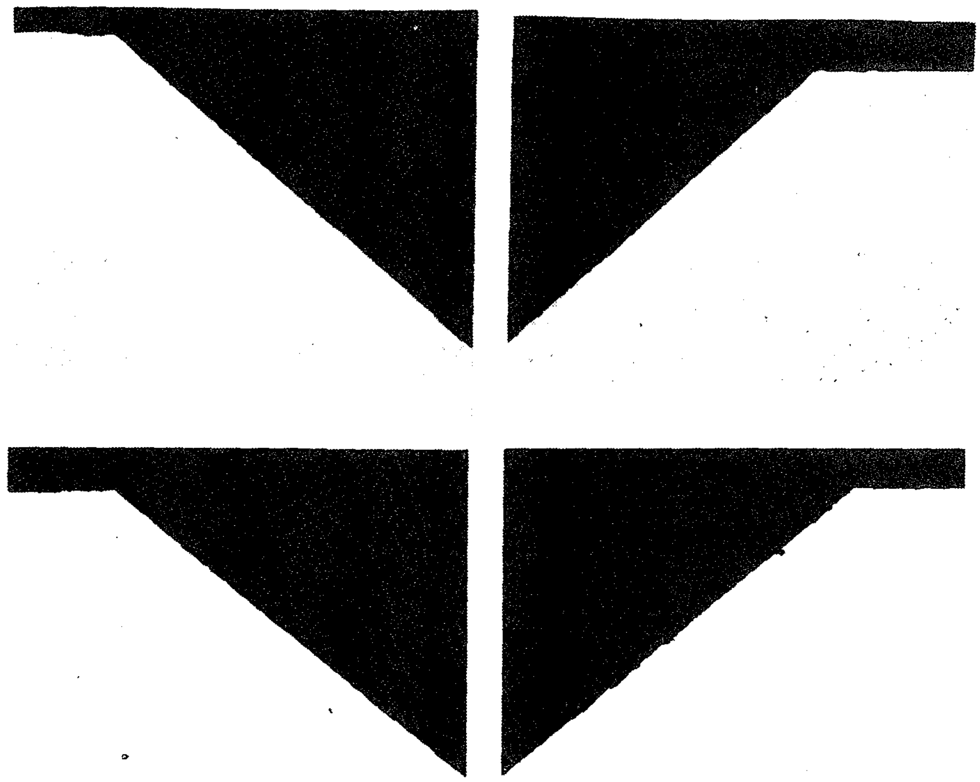

Figure 106. Representative Countersinks From Fastener Bars With Titanium Fasteners After 1008 Hours Exposure to 5% Neutral Salt Fog

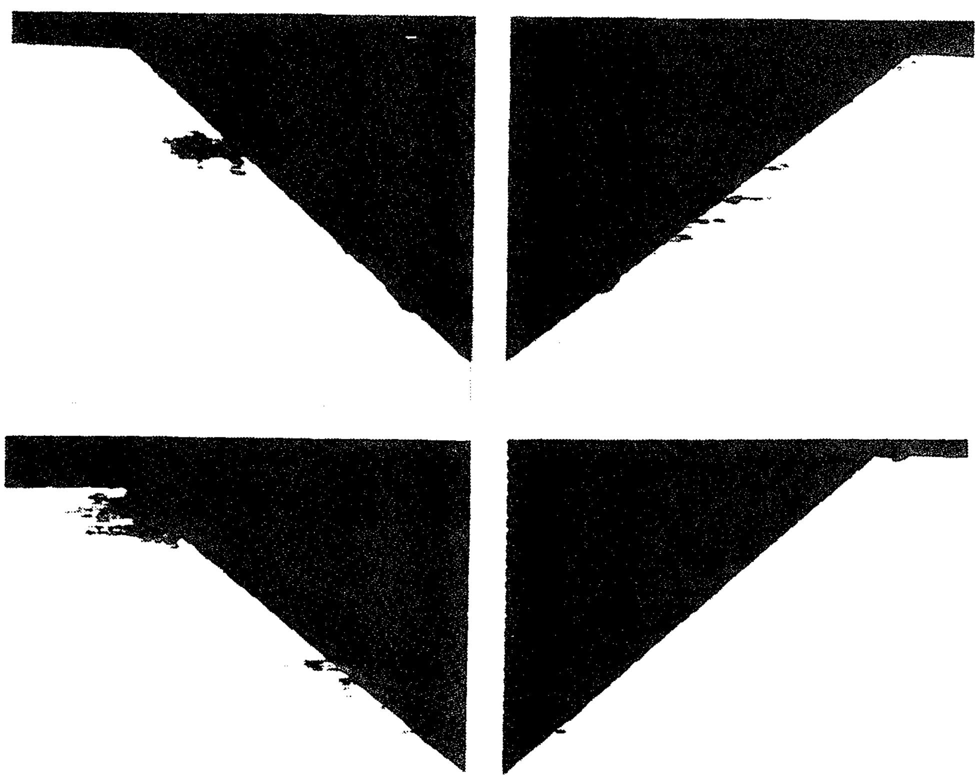

Figure 107. Representative Countersinks From Fastener Bars With Titanium Fasteners After 168 Hours Exposure to SO_2 Salt Fog

In regard to the fastener bars containing the IVD aluminum-coated titanium fasteners which were subjected to 5% neutral salt fog testing for 6 weeks, all of the conversion coatings, including the chromate conversion-coated controls, were generally comparable. Furthermore, all did an acceptable job of protecting the bare countersinks.

In regard to the fastener bars containing the IVD aluminum-coated titanium fasteners which were subjected to SO_2 salt fog testing for one week, the chromate conversion-coated controls appeared to perform slightly better than the candidate nonchromated conversion coatings.

Table 8 provides the final ranking of the various conversion coatings for each of the fastener categories. This final ranking is based on both visual appearance of the countersinks and all the photomicrographs taken.

TABLE 8. FINAL RANKING OF CONVERSION COATINGS AFTER SALT SPRAY TESTING OF FASTENER BAR SPECIMENS

IVD ALUMINUM-COATED TITANIUM FASTENERS (5% NEUTRAL SALT FOG FOR 6 WEEKS)	
1.	Chromate Conversion Coating
2.	Sealing Step II of the Sanchem-CC Process
	Sealing Steps II and III of the Sanchem-CC Process
	Improved Alodine 2000
3.	PERMATREAT 1001
IVD ALUMINUM-COATED TITANIUM FASTENERS (SO_2 SALT FOG FOR 1 WEEK)	
1.	Chromate Conversion Coating
2.	Sealing Step II of the Sanchem-CC Process
	Sealing Steps II and III of the Sanchem-CC Process
	PERMATREAT 1001
3.	Improved Alodine 2000

10. Probable Reasons for Less Than Expected Performance of Fastener Bar Specimens Containing IVD Aluminum-Coated Steel Fasteners

As previously discussed, none of the conversion coatings, including the chromated controls, performed as well as expected in salt spray in regard to the fastener bars containing the IVD aluminum-coated steel fasteners. This reduced performance compared to historical testing at MDA was probably the result of several factors. First, the "eyebrow" of bare aluminum around the countersinks was larger than normal. This "eyebrow" condition between the edge of the fastener and the edge of the countersink, all around the fastener, is visible in Figure 78. Another factor which probably adversely affected salt spray performance concerns the fact that the IVD aluminum coating on the fasteners, in most instances, was below the allowable specification limit of 0.3 mil. This was first determined "after-the-fact" by measuring the IVD aluminum thickness on several fasteners using a Magna Gage. These were extra fasteners which had been prepared for testing, but were not used. The less than required thickness was further confirmed by cross-sectioning and taking photomicrographs of these same fasteners. A final factor which probably affected salt spray performance concerns the fact that the fasteners, inadvertently, were not removed in a timely manner at the conclusion of salt spray testing. As required, polysulfide sealant had been applied over the washers and nuts on the back side of the fastener bar specimens to prevent a water entry path from the back side of the specimen. However, the quantity applied was excessive and it proved to be very difficult and time consuming to remove. Corrosion probably continued to occur in the countersinks until the fasteners were removed. It also is important to remove visible corrosion products in the countersinks immediately after fastener removal. This is best accomplished using water and a nylon bristle brush and, if not performed properly, corrosion can continue. The countersinks were cleaned after fastener removal, but not in this optimum manner.

11. Five Percent Neutral Salt Fog Testing of Fastener Bars Containing IVD Aluminum-Coated Steel Fasteners (Repeat Testing)

As a result of the less than expected salt spray performance of the fastener bar specimens containing the IVD aluminum-coated steel fasteners, 5% neutral salt fog testing was repeated for IVD aluminum-coated steel fasteners which were treated with Sealing Step II of the Sanchem-CC process, Sealing Steps II and III of the Sanchem-CC process, and chromate conversion coating. The two variations of the Sanchem-CC process were selected for retest since they had shown the most promise up to this point based on all testing conducted under this development program. Proper steps were taken to eliminate the discrepancies encountered in fabrication of the initial fastener bar specimens. Figure 108 shows the completed specimens ready for test.

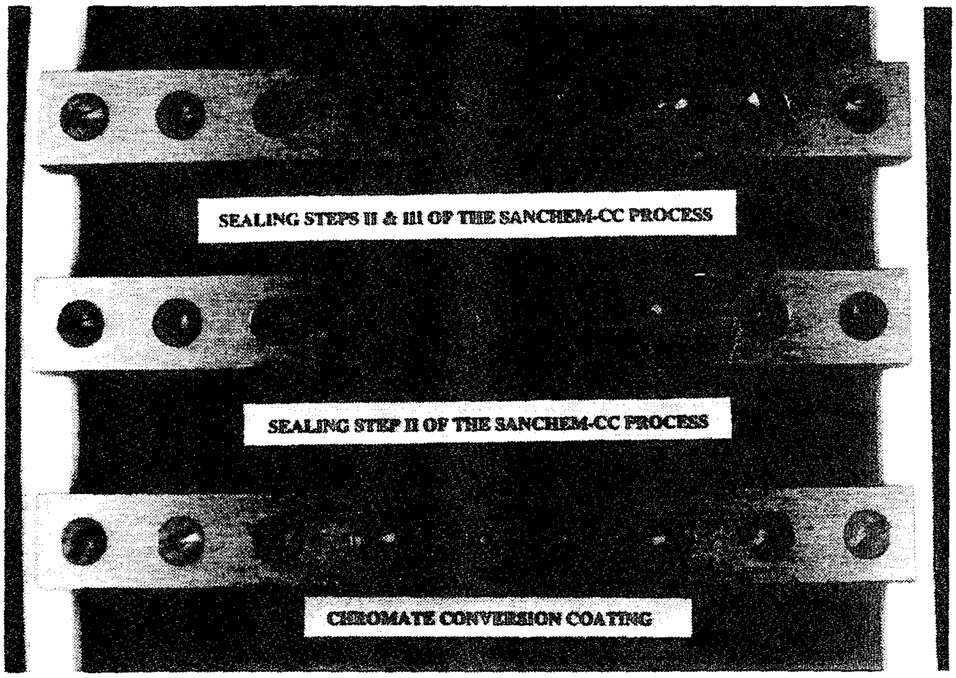

Figure 108. Aluminum-Alloy Bars With IVD Aluminum-Coated Steel Fasteners Installed Ready for Repeat 5% Neutral Salt Fog Testing

As had been the plan for the initial fastener bar specimens containing the steel fasteners, the repeat specimens did complete 1008 hours (6 weeks) of exposure to 5% neutral salt fog. Figure 109 shows these specimens at the conclusion of testing with the fasteners still installed. As expected, these repeat specimens performed much better than the initial specimens tested. The repeat fastener bar specimens are shown in Figure 110 after removal of the fasteners. Based on visual examination of the countersinks, it appeared that both variations of the Sanchem process provided performance at lease comparable to the chromate conversion-coated controls.

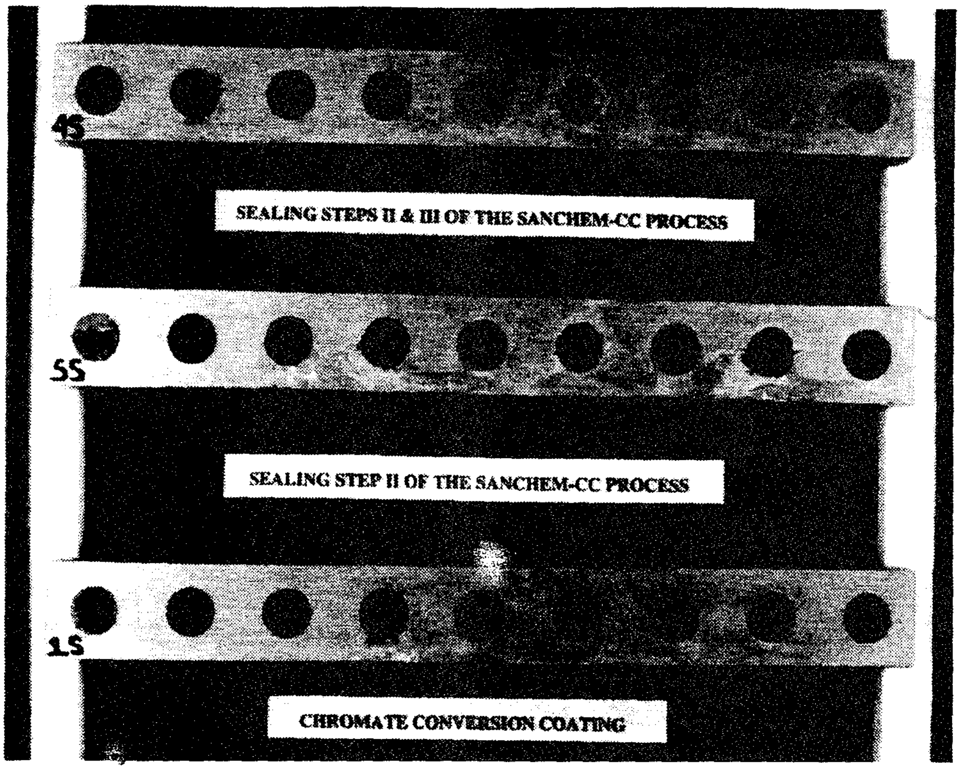

Figure 109. Aluminum-Alloy Bars With IVD Aluminum-Coated Steel Fasteners Installed After 1008 Hours of 5% Neutral Salt Fog Testing (Repeat Testing)

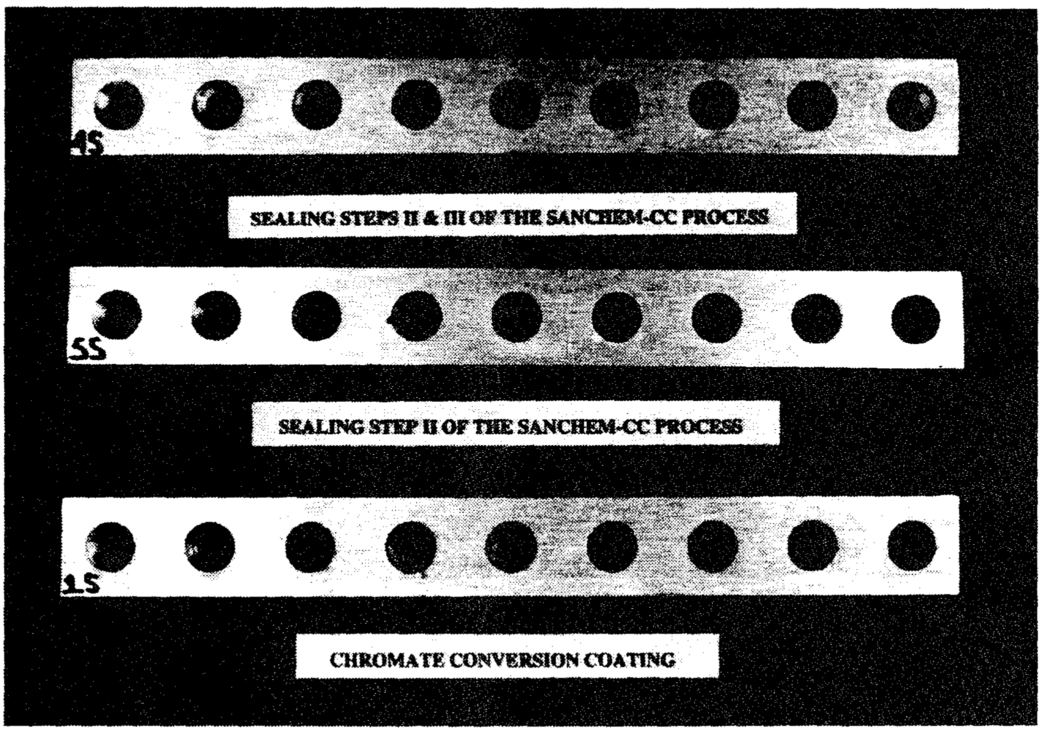

Figure 110. Aluminum-Alloy Bars After 1008 Hours of 5% Neutral Salt Fog Testing and Removal of IVD Aluminum-Coated Steel Fasteners (Repeat Testing)

SECTION VII
TASK 6 – IMPLEMENT PILOT PRODUCTION

A. OBJECTIVE

The objective of Task 6 was to set-up a pilot production processing line at MDA-St. Louis using the most promising nonchromated conversion coating based on testing through Task 5. Then, after initial runs to establish/verify procedural steps, and testing to assure compliance to the minimum requirements of MIL-C-83488, the plan was to obtain Customer approval to begin processing of IVD aluminum-coated production parts.

B. OVERVIEW

Testing through Task 5 indicated that many of the candidate nonchromated conversion coatings were not quite as effective as once thought based on in-house testing prior to contract award and/or demonstrated inconsistencies that are typical with developmental programs. Therefore, it was considered prudent to conduct a more limited scale-up of the final treatment selected. Under this scenario, processing parameters could be more closely monitored each day and additional optimization could be conducted if necessary. Accordingly, a 20-gallon processing line was set-up for a scale-up evaluation.

Task 6 was divided into two parts. The first part involved further optimization studies in regard to Sealing Step II of the Sanchem-CC process and Sealing Steps II and III of the Sanchem-CC process. Variables investigated in regard to this optimization study included immersion time, solution temperature, and solution concentration. Testing performed to determine the effect of these variables included 5% neutral salt fog per ASTM B117 for four weeks, primer adhesion, and contact electrical resistance. Based on the results of the process optimization study, a final optimized process utilizing Sealing Steps II and III of the Sanchem-CC process was selected. This final selected process was then verified under the second part of Task 6. Verification involved processing of 20 IVD aluminum-coated steel panels each working day over a four week period. Panels processed every other day were subjected to 5% neutral salt fog testing per ASTM B117 for four weeks. Also, panels processed on the first, eleventh and twentieth working day were subjected to primer adhesion and contact electrical-resistance testing. The chemistry of the two solutions involved in processing was monitored daily.

C. DISCUSSION

1. Downselection of Candidate Treatments

As testing progressed under Task 5 and all testing under the program was taken into account, it became apparent that both Sealing Step II of the Sanchem-CC process and Sealing Steps II and III of the Sanchem-CC process were the most promising treatments. It was also recognized that it would be beneficial to the overall success of the development program to continue optimization studies in regard to these treatments. As a result, the two Sanchem treatments were selected for evaluation under Task 6, and Task 6 was divided into two parts. The first part focused on continued optimization of both treatments which, in-turn, led to selection of a final optimized treatment. The second part of Task 6, then, involved verification of the final treatment selected by processing panels over an extended period of time.

Improved Alodine 2000 also showed considerable promise based on testing through Task 5. It might have been selected as the final treatment, except for one limitation. More specifically, when IVD aluminum-coated panels treated with improved Alodine 2000 were exposed to SO_2 salt fog, a significant amount of corrosion products were formed in a relatively short period of time. Although SO_2 salt fog testing is not an Air Force required test, it still raises the question concerning the long term performance of Alodine 2000 treated parts in an acidic or acid rain environment. Such environments are common in industrial areas with sulfur emissions.

PERMATREAT 1001 was dropped from further consideration under this development program at the conclusion of Task 5 testing. Although this treatment demonstrated very good salt spray performance on IVD aluminum-coated aluminum panels, it generally was the least desirable candidate treatment in all other tests. It also has another disadvantage. It is most effective when squirted on a part and then dispersed over the surface of the part by spinning the part or blowing air on the part. Conventional tank processing by itself can result in thickness build-ups which are detrimental to paint adhesion.

2. Twenty-Gallon Scale-Up Line

A 20-gallon scale-up line was set-up in a laboratory at MDA to facilitate Task 6 testing. An overall view of this processing line is shown in Figure 111, while a close-up of the primary tanks in the line is shown in Figure 112. The tank in the middle contains the heated permanganate solution (i.e., Sealing Step II of the Sanchem-CC process), and the tank on the left contains the heated potassium silicate solution (i.e., Sealing Step III of the Sanchem-CC process). The tank on the right is a deionized water rinse tank which was used to rinse panels after treatment in the permanganate solution. A second deionized water rinse tank was used for rinsing panels after treatment in the potassium silicate solution. It is located in an adjacent module and is not shown.

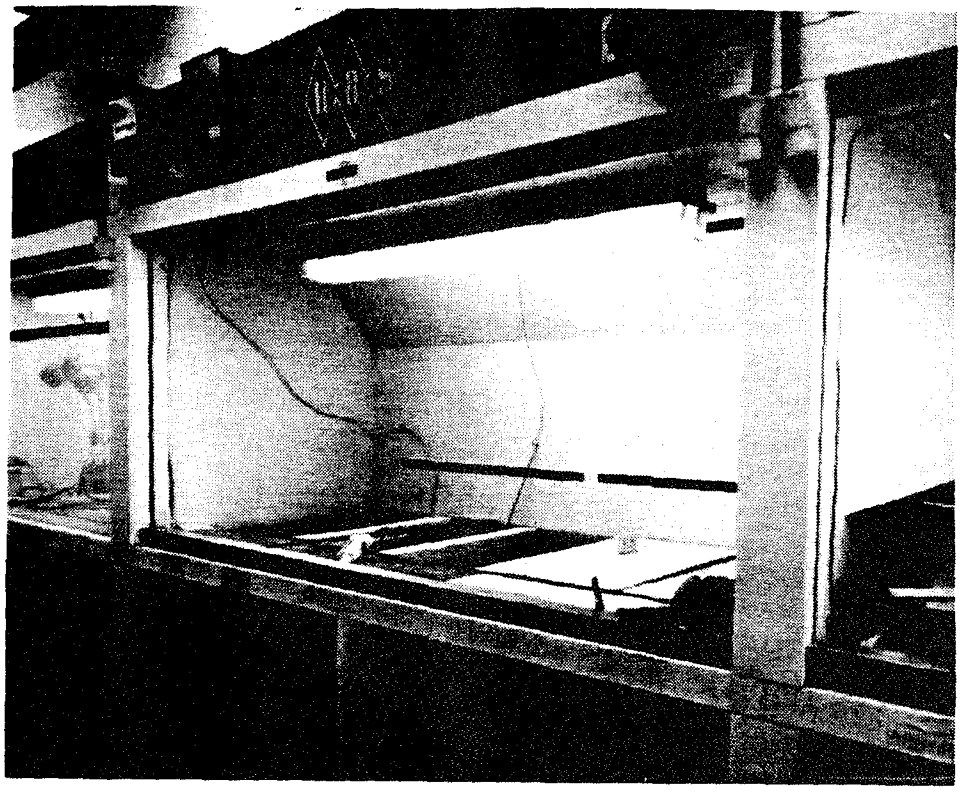

Figure 111. Overall View of 20-Gallon Processing Line For Task 6 Testing

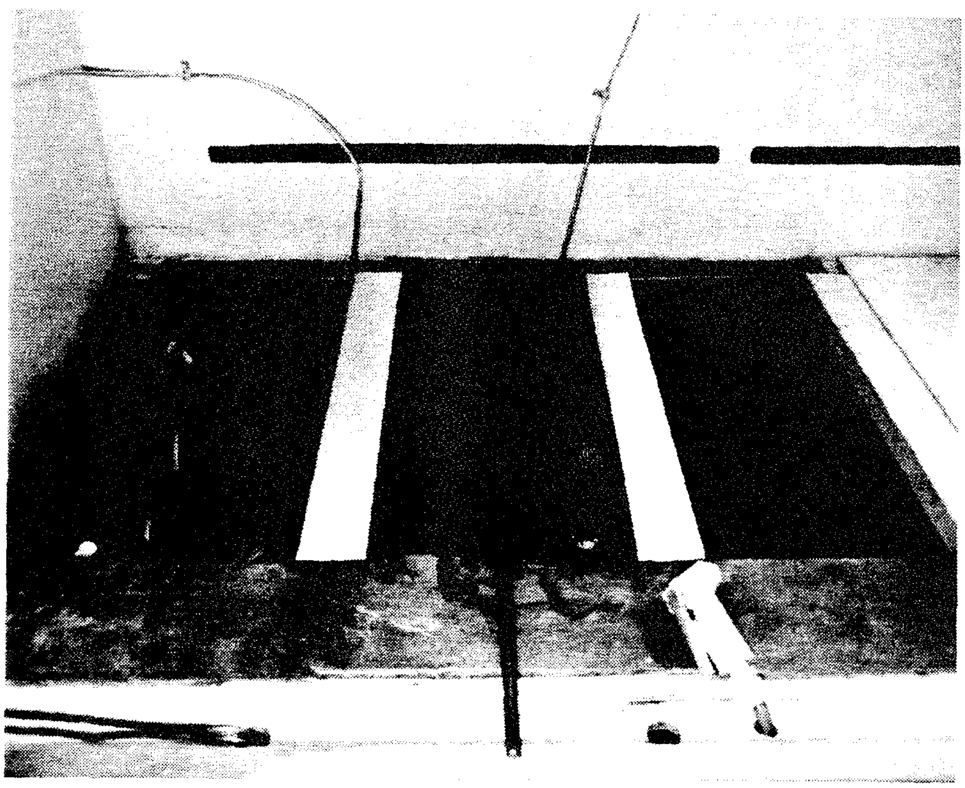

Figure 112. Close Up View of Tanks in 20-Gallon Processing Line

3. Process Optimization Study

a. Panel Material and Preparation

Both IVD aluminum-coated steel panels and IVD aluminum-coated aluminum panels were used for the process optimization study. The steel panels were AISI 4130, 4 x 6 x 0.040 inch thick, conforming to MIL-S-18729. The aluminum panels were 2024-T3 and 7075-T6, 4 x 6 x 0.050 inch thick. The steel panels were degreased and then grit-blasted prior to IVD aluminum coating. The aluminum panels were chemically cleaned prior to IVD aluminum coating which involved aqueous degreasing, alkaline cleaning, pickling, and deoxidizing. The IVD aluminum coating was applied to conform to MIL-C-83488, Class 1 (1.0-mil thick minimum). The average thickness of the IVD aluminum coating for all the panels was 1.5 mils.

Table 9 summarizes all of the panels prepared and the tests performed (i.e., 5% neutral salt fog, primer adhesion, and contact electrical resistance) under the process optimization study. In regard to panel processing, Figure 113 shows a group of panels prior to immersion in the permanganate solution. Figure 114 shows all of the salt spray panels in their racks ready for testing.

b. Five Percent Neutral Salt Fog Testing

As planned, the salt spray test panels were exposed to 5% neutral salt fog for 672 hours (4 weeks). As was the case in most of the equivalent testing performed earlier, most of the test panels developed pits in the IVD aluminum coating early in testing. Of importance, however, none of the steel panels showed any evidence of failure (i.e., no appearance of red rust) for any of the process variations tested. In other words, all the treated IVD aluminum coated steel panels met the salt spray requirement of MIL-C-83488 for a Class 1 IVD aluminum coating (i.e., 1.0-mil thick minimum coating).

Conclusions from the Task 6 process optimization study are summarized below in regard to Sealing Step II of the Sanchem-CC process, and Sealing Steps II and III of the Sanchem-CC process.

(1) Sealing Step II of the Sanchem-CC Process

- Increasing the immersion time from three minutes to six or ten minutes for any of the 12 process variations investigated did not appear to improve salt spray performance from the standpoint of initial pitting. In fact, in some instances, performance seemed to be adversely affected with increased immersion time. The above conclusion is applicable to both the steel and aluminum panels tested.

- In regard to the steel panels, double concentration at 140°F was superior to standard concentration at either 140°F or 170°F in regard to preventing pitting in the IVD aluminum coating. Also, in regard to the steel panels, double concentration at 170°F did not appear to improve performance over double concentration at 140°F.

TABLE 9. TASK 6 PROCESS OPTIMIZATION STUDY

TREATMENT	NO. OF SALT SPRAY PANELS			NO. OF 4130 STEEL PANELS FOR CONTACT ELECTRICAL RESISTANCE TESTING	NO. OF 4130 STEEL PANELS FOR PRIMER ADHESION TESTING
	4130 STEEL	2024-T3 ALUM.	7075-T6 ALUM.		
Sealing Step II of the Sanchem - CC Process					
3 min at 170°F (Baseline)	4	4	4	1	1
6 min at 170°F	4	4	4	1	1
10 min at 170°F	4	4	4	1	1
3 min at 140°F	4	4	4	1	1
6 min at 140°F	4	4	4	1	1
10 min at 140°F	4	4	4	1	1
Sealing Steps II & III of the Sanchem - CC Process					
3 min at 140°F & 1 min at 200°F (Baseline)	4	4	4	1	1
3 min at 140°F & 3 min at 200°F	4	4	4	1	1
3 min at 170°F & 1 min at 200°F	4	4	4	1	1
3 min at 170°F & 3 min at 200°F	4	4	4	1	1
10 min at 170°F & 1 min at 200°F	4	4	4	1	1
10 min at 170°F & 3 min at 200°F	4	4	4	1	1
Sealing Step II of the Sanchem - CC Process (Double Concentration)					
3 min at 170°F	4	3	4	1	1
6 min at 170°F	4	4	4	1	1
10 min at 170°F	4		2	1	1
3 min at 140°F	4	4	4	1	1
6 min at 140°F	4		4	1	1
10 min at 140°F	4		4	1	1

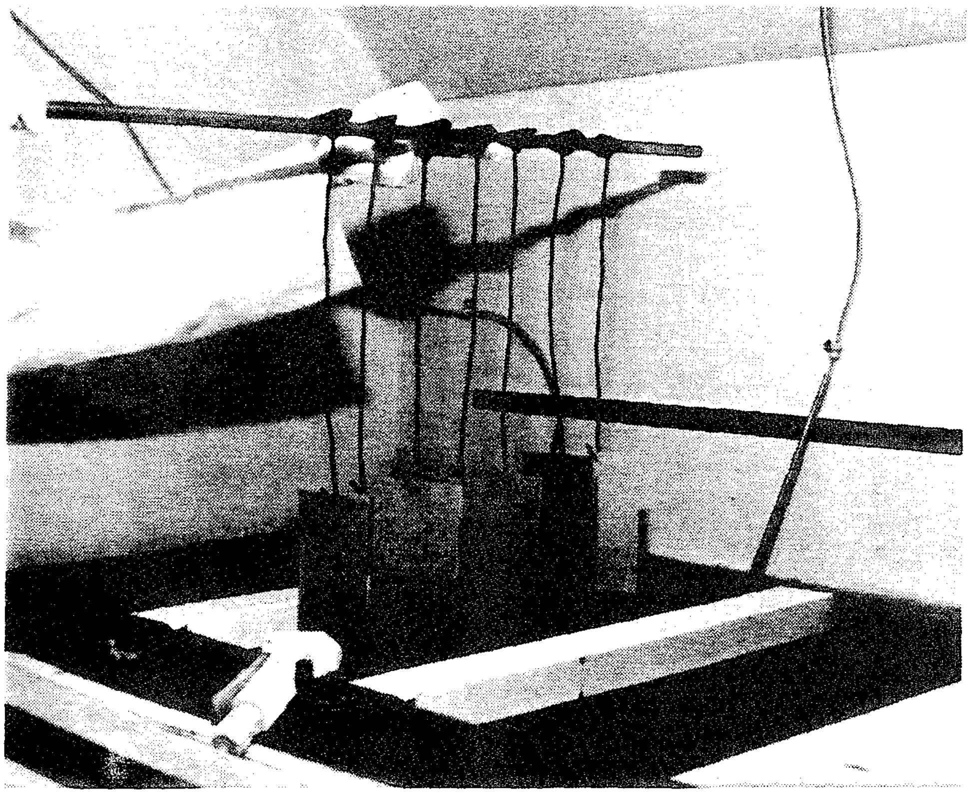

Figure 113. IVD Aluminum-Coated Panels Prior to Immersion in Permanganate Solution

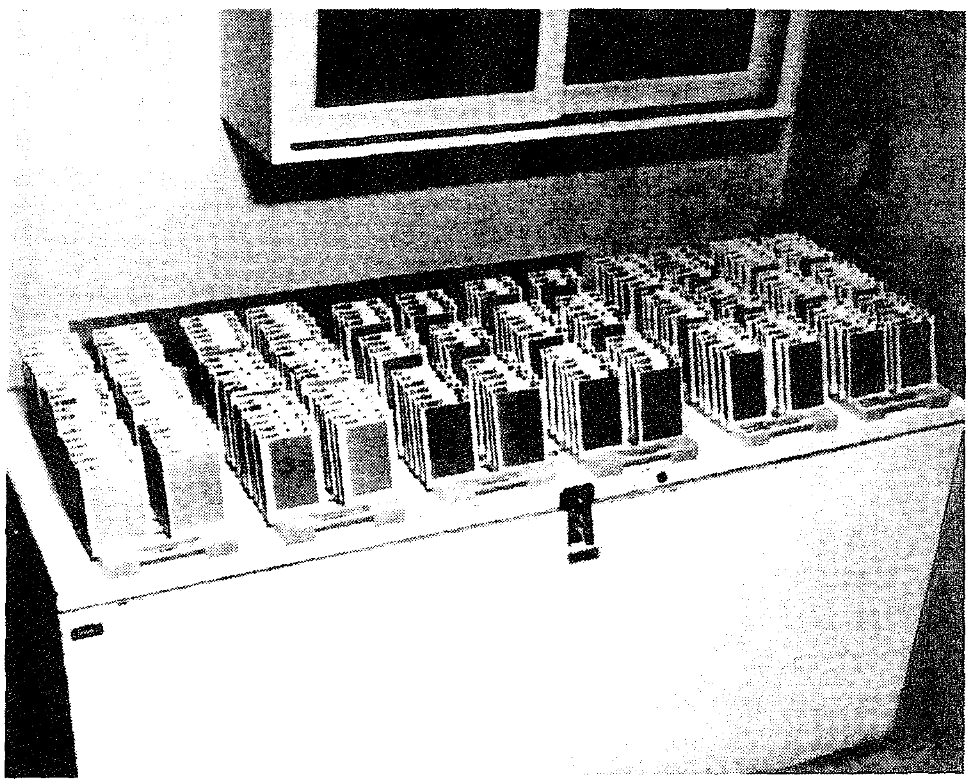

Figure 114. Test Panels Ready for 5% Neutral Salt Fog Testing Under Task 6 Process Optimization Study

- In regard to the aluminum panels, standard concentration at 170°F appeared to perform as well as double concentration at 140°F or 170°F, and better than standard concentration at 140°F.

(2) Sealing Steps II and III of the Sanchem-CC Process

- Panels treated for 10 minutes at 170°F in Sealing Step II followed by either one minute or three minutes at 200°F in Sealing Step III did not perform as well as the other four variations evaluated. The above conclusion is applicable to both the steel and aluminum panels.

- The other four processing variations noted in the previous paragraph (i.e., three minutes at 140°F and one minute at 200°F, three minutes at 140°F and three minutes at 200°F, three minutes at 170°F and one minute at 200°F, three minutes at 170°F and three minutes at 200°F) were, in general, comparable in regard to number of pits and condition of the pits at the conclusion of testing. From these results, it would appear that at least one repeat test, using optimum quality IVD aluminum-coated panels, would be required to distinguish differences between these four variations.

c. Contact Electrical Resistance Testing

As noted in Table 9, one panel for each of the processing variations or treatments was subjected to contact electrical resistance testing before and after seven days of exposure to 5% neutral salt fog. This testing was identical to that conducted previously under Task 2 which is described in detail in Section III of this report.

The results of contact electrical resistance testing are summarized in Table 10. All of the variations were below the 5-milliohm maximum requirement of MIL-C-81706 prior to salt spray exposure, but not all met the 10-milliohm maximum requirement of the military specification after salt spray exposure. Most of the panels which failed had both high and low resistance readings, depending on the location on the panel. Selected panels were retested on subsequent, successive days as noted in Table 10. Surprisingly, the results did not always correspond to the original results. Also, in some instances, panels tested twice on the same day did not always provide the same results. The reason for these inconsistencies in contact electrical resistance testing is not known. The testing apparatus itself is not thought to be the primary culprit because, in many instances, when the panel passed initially after salt spray testing, it also passed in regard to subsequent determinations.

As also shown in Table 10, six of the eight panels retested on 10 April 1995 were again tested that same day, after scrubbing with water and a nylon bristle brush. The intention was to determine if residual products on the surface of the panels from salt spray testing may have been contributing to the inconsistencies in contact electrical resistance testing test results. The results of this investigation were inconclusive as two of the panels which failed initially on 10 April 1995 passed after scrubbing, while three other panels which failed initially still failed after the scrubbing operation. Finally, as noted in Table 10, two of the panels were retested on 20 April 1995 both before and after lightly abrading the surface using a silicon carbide coated nylon abrasive pad (i.e., Scotchbrite) wetted with methyl ethyl ketone. In both cases, the panels failed contact electrical resistance testing prior to abrading, but passed after abrading.

TABLE 10. RESULTS OF CONTACT ELECTRICAL RESISTANCE TESTING

TREATMENT	AVG. CONTACT ELECTRICAL RESISTANCE (MILLIOHMS) 1/						
		AFTER 7 DAYS EXPOSURE TO 5% NEUTRAL SALT FOG					
	Prior to Exposure (3/22/95)	Initial Test (4/5/95)	Retest (4/7/95)	Retest (4/10/95) Before Scrubbing	Retest (4/10/95) After Scrubbing	Retest (4/20/95) Before Abrading	Retest (4/20/95) After Abrading
Sealing Step II of the Sanchem - CC Process							
3 min at 170° F (Baseline)	0.86	34.3	37.8, 13.3	12.9	29.7	13.3	1.3
6 min at 170° F	0.51	21.9	3.3				
10 min at 170° F	0.79	23.8	11.8, 37.3	38.1	16.7		
3 min at 140° F	0.15	2.4					
6 min at 140° F	0.22	0.9					
10 min at 140° F	0.27	1.2		3.7			
Sealing Steps II & III of the Sanchem - CC Process							
3 min at 140° F & 1 min at 200° F (Baseline)	0.41	0.6		0.9			
3 min at 140° F & 3 min at 200° F	0.37	4.7					
3 min at 170° F & 1 min at 200° F	0.75	18.8	11.3, 5.7	25.0, 13.9	8.5		
3 min at 170° F & 3 min at 200° F	0.27	3.8	4.9				
10 min at 170° F & 1 min at 200° F	0.47	28.0	11.3, 7.1				
10 min at 170° F & 3 min at 200° F	0.32	2.4	4.1				
Sealing Step II of the Sanchem - CC Process (Double Concentration)							
3 min at 170° F (Baseline)	0.63	28.7		9.1	4.0		
6 min at 170° F	1.24	108.0		122.8	92.0	46.1	4.4
10 min at 170° F	0.86	5.3		31.1	5.2		
3 min at 140° F	0.52	23.6					
6 min at 140° F	0.75	11.0					
10 min at 140° F	0.69	4.5					

NOTES: 1/ Typically, nine individual measurements made on each panel and then average calculated. MIL-C-81706 requires a maximum resistance of 5-milliohms as treated and a maximum of 10-milliohms after 7 days exposure to 5% neutral salt fog.

Contact electrical resistance testing was repeated for five of the processing variations. Unlike the initial testing, two panels rather than one were evaluated for each of the processing variations. The results of this testing are summarized in Table 11. As before, all of the treatments met the 5-milliohm maximum requirement of MIL-C-81706 prior to salt spray testing. Also, as before, not all of the panels failed after salt spray testing. Again, most of the panels which failed had both high and low readings depending on location on the panel.

At first glance, one could assume that the results of contact electrical resistance testing might be of some concern. This is not the case, however, for a couple of reasons. First, in spite of the inconsistencies and failure to meet MIL-C-81706 requirements after salt spray exposure in some cases, all of the resistance values are still relatively low. Second, low electrical resistance is required for electrical bonding and grounding and, of significance, electrical bonds are not often required for IVD aluminum-coated steel parts. Along this line, even if they are required, standard practice is to first abrade the area and then touch it up with conversion coating immediately before making the electrical bond. As previously noted, abrading the surface after salt spray exposure resulted in acceptable resistance readings. In a final note, it should be mentioned that the reason for the inconsistencies in contact electrical resistance test results would have been investigated further if the budget under this program would have permitted it.

TABLE 11. RESULTS OF REPEAT CONTACT ELECTRICAL RESISTANCE TESTING

TREATMENT	AVERAGE CONTACT ELECTRICAL RESISTANCE (MILLIOHMS) 1/	
	PRIOR TO EXPOSURE	AFTER 7 DAYS EXPOSURE TO 5% NUETRAL SALT FOG
Sealing Step II of the Sanchem - CC Process		
3 min at 170° F		
Panel No.1	1.7	69.2
Panel No. 2	1.2	10.3
3 min at 140° F		
Panel No.1	2.8	40.7
Panel No. 2	1.9	262.2
Sealing Steps II & III of the Sanchem - CC Process		
3 min at 140° F & 1 min at 200° F		
Panel No. 1	1.5	2.2
Panel No. 2	1.2	5.6
3 min at 170° F & 1 min at 200° F		
Panel No. 1	1.4	250.3
Panel No. 2	1.6	16.6
Sealing Step II of the Sanchem - CC Process (Double Concentration)		
3 min at 140° F		
Panel No. 1	1.1	61.9
Panel No. 2	0.6	54.8

NOTES: 1/ Typically, nine individual measurements made on each panel and then average calculated. MIL-C-81706 requires a maximum resistance of 5-milliohms as treated and a maximum of 10-milliohms after 7 days exposure to 5% neutral salt fog.

In spite of the inconsistencies in regard to contact electrical resistance testing, several conclusions could still be made. These are summarized below in regard to Sealing Step II of the Sanchem-CC process, and Sealing Steps II and III of the Sanchem-CC process.

(1) Sealing Step II of the Sanchem-CC Process

- With one exception (Ref. Table 11 repeat testing), all of the panels treated in standard concentration at 140°F met contact electrical resistance requirements after salt spray exposure. Other than the one exception noted, the test results are consistent with contact electrical resistance testing performed under Task 2 in regard to Sealing Step II of the Sanchem-CC process.

(2) Sealing Steps II and III of the Sanchem-CC Process

- Sealing Steps II and III consisting of 3 minutes at 140°F and 1 minute at 200°F met contact electrical resistance requirements for all testing performed.

d. Primer Adhesion Testing

As noted in Table 9, one panel for each of the processing variations or treatments was subjected to primer adhesion testing. Two coats of Deft 44-GN-36 low density, epoxy, water-borne primer were spray applied to one side of each of the panels to provide a final dry film thickness of 0.0008 to 0.0012 inch. The primed panels were air dried for approximately 30 minutes at room temperature and then oven dried for one hour at 150°F. They were then air dried for an additional 14 days at room temperature prior to tape adhesion testing. Initially, a dry, tape adhesion test was performed on each panel. Then, a scribed, wet, tape adhesion test was performed on each panel per Method 6301 of Federal Test Method Standard 141. Testing per the Federal Standard involved immersion of the test panels in distilled water at room temperature for 24 hours followed by scribing and tape testing.

All of the panels passed the dry, tape adhesion test.

Table 12 summarizes the results of scribed, wet, tape adhesion testing for all of the processing variations of Table 9. It also provides the results for some limited repeat testing also conducted. As can be seen from this table, all of the panels treated with Sealing Step II of the Sanchem-CC process, with one exception, failed the scribed, wet, tape adhesion test. All of the panels treated with Sealing Steps II and III of the Sanchem-CC process, on the other hand, passed this test. Figure 115 shows a typical panel treated with Sealing Steps II and III of the Sanchem-CC process after successfully passing the test.

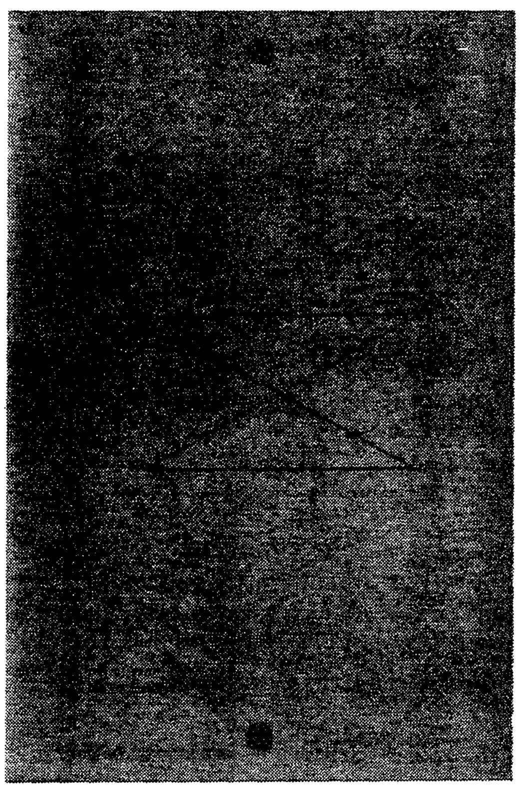

Figure 115. Primed Panel Which Passed Scribed, Wet, Tape Adhesion Testing (Sealing Steps II & III of the Sanchem-CC Process)

TABLE 12. RESULTS OF SCRIBED, WET, TAPE ADHESION TESTING UNDER TASK 6 OPTIMIZATION STUDY

	PRIMER ADHESION (P=PASS; F=FAIL)	
TREATMENT	INITIAL TESTING	REPEAT TESTING
SEALING STEP II OF THE SANCHEM-CC PROCESS		
3 MIN AT 170°F (BASELINE)	P	F
6 MIN AT 170°F	F	
10 MIN AT 170°F	F	
3 MIN AT 140°F	F	F
6 MIN AT 140°F	F	
10 MIN AT 140°F	F	
SEALING STEP II & III OF THE SANCHEM-CC PROCESS		
3 MIN AT 140°F & 1 MIN AT 200°F (BASELINE)	P	P
3 MIN AT 140°F & 3 MIN AT 200°F	P	
3 MIN AT 170°F & 1 MIN AT 200°F	P	P
3 MIN AT 170°F & 3 MIN AT 200°F	P	
10 MIN AT 170°F & 1 MIN AT 200°F	P	
10 MIN AT 170°F & 3 MIN AT 200°F	P	
SEALING STEP II OF THE SANCHEM-CC PROCESS (DOUBLE CONCENTRATION)		
3 MIN AT 170°F	F	
6 MIN AT 170°F	F	
10 MIN AT 170°F	F	
3 MIN AT 140°F	F	F
6 MIN AT 140°F	F	
10 MIN AT 140°F	F	

In general, primer adhesion tests results under this Task 6 process optimization study were consistent with previous testing. More specifically, Task 2 IVD aluminum-coated steel panels treated with the full Sanchem-CC process, which includes Sealing Step III, and then primed with a different water-borne primer (i.e., Courtauld's 513X408/910X831 per MIL-C-85582, Type I and MMS-423) also passed scribed, wet, tape adhesion testing. Panels treated with only Sealing Step II of the Sanchem-CC process, on the other hand, have always failed this test when water-borne primers are used. Table 13 summarizes scribed, wet, tape adhesion testing conducted under this program for IVD aluminum-coated steel panels treated with Sealing Step II of the Sanchem-CC process.

TABLE 13. SUMMARY OF SCRIBED, WET, TAPE ADHESION TESTING FOR SEALING STEP OF THE SANCHEM-CC PROCESS

PRIMER	PRIMER TYPE		TEST NO. 1 (TASK 2)	TEST NO. 2 (TASK 2 REPEAT)	TEST NO. 3 (TASK 6)	TEST NO. 4 (TASK 6 REPEAT)
	WATER BASED	SOLVENT BASED				
COURTAULDS 513X408/910X831 PER MIL-C-85582 TYPE I & MMS-423	X		Failed ("Pin-point Type" primer to substrate failure)	Failed ("Pin-point Type" primer to substrate failure)		
COURTAULDS 519X303/910X357 PER MMS-425		X		PASSED		
DEFT 44-GN-36	X				Failed (Primer to substrate failure)	Failed (Primer to substrate failure)

The Courtauld's 513X408/910X831 water-borne primer conforming to MIL-C-85582, Type I and MMS-423 is a first-generation water-borne primer. Furthermore, although it is currently used in some production applications, it is recognized that improved water-borne primers are available. Along this line, effort is under way to qualify improved products for production use. Based on the above, then, the fact that the Courtauld's 513X408/910X831 primer failed the scribed, wet, tape-adhesion test in regard to Sealing Step II of the Sanchem-CC process was originally thought to be related to this specific primer. This theory was further supported when the currently used solvent-based primer (i.e., Courtauld's 519X303/910X357 per MMS-425) passed this same adhesion test in regard to Sealing Step II of the Sanchem-CC process. Later, when the newer generation DEFT 44-GN-36 water-borne primer also failed the scribed, wet, tape-adhesion test in regard to Sealing Step II of the Sanchem-CC process, it became apparent that perhaps water-borne primers, in general, were not totally compatible with Sealing Step II of the Sanchem-CC process.

e. Selection of Final Treatment for Process Verification Testing

Based on the results of the process optimization study, the Sanchem-CC process consisting of 3 minutes at 140°F in Sealing Step II followed by 1 minute at 200°F in Sealing Step III was selected as the best treatment and the treatment to be used for process verification under Task 6. This decision was based on the following factors:

- Performance comparable to chromate conversion coating in regard to IVD aluminum-coated steel panels exposed to 5% neutral salt fog for 8000 hours.
- Five percent neutral salt fog performance comparable to chromate conversion coating in regard to IVD aluminum-coated steel fasteners installed in 7075-T6 aluminum bars with bare countersinks.
- Adequate 5% neutral salt fog performance in regard to IVD aluminum-coated titanium fasteners installed in 7075-T76 aluminum bars.
- Adequate performance on IVD aluminum-coated aluminum panels in regard to salt spray testing.

- Required primer adhesion.
- Required contact electrical resistance before and after 7 days of exposure to 5% neutral salt fog.

4. Process Verification of Final Treatment Selected

a. Panel Material and Preparation

IVD aluminum-coated steel panels were used for Task 6 process verification testing. The panels were AISI 4130 steel, 4 x 6 x 0.040 inch thick, conforming to MIL-S-18729. The panels were degreased and grit-blasted prior to IVD aluminum coating. The IVD aluminum coating on the side of the panels to be tested was applied to conform to MIL-C-83488, Class 1 (1.0-mil thick minimum). The average thickness of the IVD aluminum coating for all the panels was 1.6 mils.

In accordance with standard practice, the IVD aluminum coating on all of the panels was glass-bead-peened prior to conversion coating. The conversion coating was applied to the panels within 24 hours after the peening operation.

As planned, 20 panels were processed each day over a 4-week or 20-working-day period. Processing involved immersion in a permanganate solution at 140°F for 3 minutes (i.e., Sealing Step II) followed by immersion in a potassium silicate solution at 200°F for 1 minute (i.e., Sealing Step III). SAFEGARD 3000 is the Sanchem product designation for the Sealing Step II solution and SAFEGARD 4000 is the Sanchem product designation for the Sealing Step III solution.

b. Five Percent Neutral Salt Fog Testing

Five panels processed on the 1st, 3rd, 5th, 7th, 9th, 11th, 13th, 15th, 17th, 19th and 20th working days were exposed to 5% neutral salt fog for 672 hours (4 weeks). Most of the panels up through the 11th day of processing developed small pits in the IVD aluminum coating. In general, the overall performance of these panels was not quite as good as equivalent panels tested earlier in the program. After the 11th day and continuing until the end of processing, however, the amount of pitting on the panels decreased and their overall appearance improved. The reason for this change is not known. Figure 116 shows two representative panels processed prior to the 11th day, while Figure 117 shows two representative panels processed after the 11th day. The dark streaks on the panels shown in Figure 117 are a result of the thin IVD aluminum coating associated with the holes and hangar marks. In a final note, it is important to mention that there was no evidence of red rust on any of the panels tested. Therefore, all met the 672-hour requirement of MIL-C-83488 for a Class 1 IVD aluminum coating (i.e., 1.0-mil thick minimum coating).

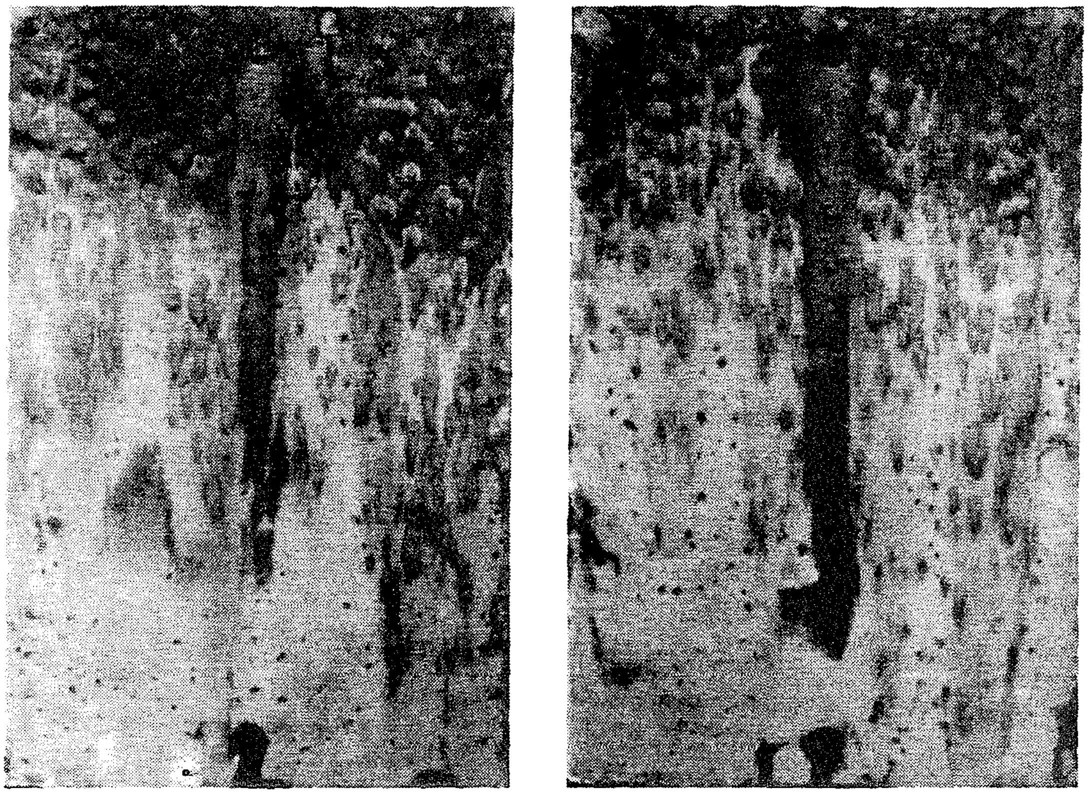

Figure 116. Representative Task 6 Process Verification Panels After 672 Hours Exposure to 5% Neutral Salt Fog (Prior to 11th Day of Processing)

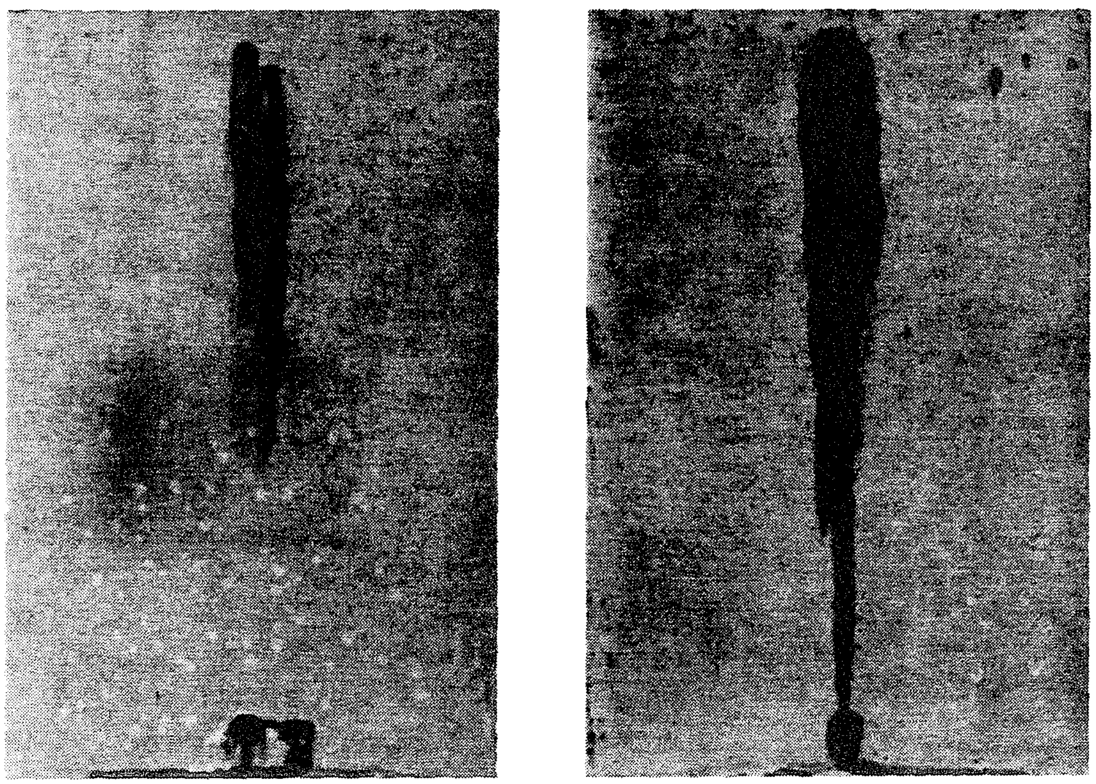

Figure 117. Representative Task 6 Process Verification Panels After 672 Hours Exposure to 5% Neutral Salt Fog (After 11th Day of Processing)

c. Contact Electrical Resistance Testing

Two panels processed on the 1st, 11th and 20th working days were subjected to contact electrical resistance testing both before and after seven days exposure to 5% neutral salt fog. This testing was identical to that conducted previously under Task 2 which is described in detail in Section III of this report.

The results of contact electrical resistance testing are summarized in Table 14. There were some inconsistencies in the test results which was not totally unexpected based on previous testing. The important fact, however, is that all but one of the six panels met the 5-milliohm maximum requirement of MIL-C-81706 prior to salt spray exposure, and the one that did not, still had a relatively low value. Also, after salt spray exposure, all values were relatively low for the panels treated on the first and twentieth or final day of processing. Of greatest significance in regard to the above test results, electrical bonds are not often required for IVD aluminum-coated steel parts and, even if they are, standard practice is to first abrade the area, then touch it up with conversion coating immediately before making the electrical bond.

TABLE 14. CONTACT ELECTRICAL RESISTANCE TEST RESULTS FOR TASK 6 PROCESS VERIFICATION TESTING

DAY OF PROCESSING	PANEL NO.	AVERAGE CONTACT ELECTRICAL RESISTANCE (MILLIOHMS) 1/	
		PRIOR TO EXPOSURE	AFTER 7 DAYS EXPOSURE TO 5% NEUTRAL SALT FOG
FIRST	24	0.8	6.4
	25	2.3	10.4
ELEVENTH	99	12.2	123.2
	100	3.2	1387.0
TWENTIETH	178	2.1	34.3
	182	1.3	11.7

NOTES: 1/ Nine individual measurements made on each panel and then average calculated. MIL-C-81706 requires a maximum of 5-milliohms as treated and a maximum of 10-milliohms after 7 days exposure to 5% neutral salt fog.

d. Primer Adhesion Testing

Two panels processed on the 1st, 11th and 20th working days were tested for primer adhesion. The primer used and all testing was identical to that described for primer adhesion testing under the process optimization study of this Task 6. All panels passed both the dry, tape-adhesion test, and the scribed, wet, tape-adhesion test.

e. Solution Chemistry

Chemical treatments, such as conversion coating, developed and qualified based on solutions prepared and applied in the laboratory can change once the process is scaled-up. Changes may be related to unique requirements for maintaining solution chemistry and operating parameters once the tanks are increased in size.

Also, once solutions are used to process a large number of parts, changes not experienced or envisioned in the laboratory may occur. Hence, there may be the need for special controls or solution analysis techniques.

Based on the above, the chemistry of the two solutions involved in conversion coating during process verification was monitored daily. More specifically, each day of processing, the pH was checked for the SAFEGARD 3000 (i.e., Sealing Step II permanganate solution) and the SAFEGARD 4000 (i.e., Sealing Step III potassium silicate solution). Sanchem specified a pH range of 6.0-7.0 for the SAFEGARD 3000 solution and a pH range of 11.5-12.0 for the SAFEGARD 4000 solution. Also, each day of processing, a titration was performed for the SAFEGARD 4000 to verify the required level of permanganate ion. Sanchem specified a range of 2.8-4.8 mL in regard to the amount of solution required for the titration. The results of the solution analysis testing described above are shown in Tables 15 and 16 for the SAFEGARD 3000 and SAFEGARD 4000 solutions, respectively.

During processing of panels over the 20-working-day period, the pH of the SAFEGARD 3000 solution tended to increase. This, then, required periodic additions of nitric acid. Of significance, however, the quantity required was very small. Also, during the processing period, the pH of the SAFEGARD 4000 solution tended to decrease requiring additions of 50 percent potassium hydroxide. Of significance, the additions were somewhat large. However, once several additions had been made up through the halfway point in processing, no more were required. This behavior indicates that the solution eventually became stable. Water additions were required each day for both solutions. The amount added for the SAFEGARD 4000 was greater than that for the SAFEGARD 3000 due to its higher operating temperature. A summary of all the additions made to both solutions throughout the 20-working-day period is also provided by Tables 15 and 16.

TABLE 15. SOLUTION MAINTENANCE FOR SAFEGARD 3000 UNDER TASK 6 PROCESS VERIFICATION

DATE OF PROCESSING	pH[3/]	ADDITION OF NITRIC ACID TO LOWER pH (ML)	TITRATION FOR PER-MANGANATE ION (ML OF TITER[4/])	WATER ADDITION (INCH)
4/26	6.9		4.7	
4/27	7.5		4.5	0.5
4/28	7.0		4.5	0.5
5/1	7.7		4.7	0.5
5/2	7.9		4.6	0.5
5/3	8.0	2.0	4.5	0.5
5/4	7.1		4.5	0.5
5/5	7.9		4.4	0.5
5/8	7.7		4.1	1.0
5/9	7.9	1.5	4.0	1.0
5/10[1/]	7.0		4.0	1.0
5/11	7.3		4.0	1.0
5/12	7.6		3.9	1.0
5/15	7.8	1.0	3.8	1.0
5/16	6.8		3.9	1.0
5/17	7.8		3.9	0.5
5/18	7.6		3.8	0.5
5/19	7.7		3.8	0.5
5/22	7.8		3.9	1.0
5/23	7.9	1.0	3.9	1.0
5/24	7.2		3.9	1.0
5/25[2/]	7.3		3.7	1.0

NOTES:
1/ Did not process any panels on this day.
2/ Day after end of test. Did not process panels on this day.
3/ Sanchem data sheet specified a range of 5.0 to 9.0. MDA elected to use a range of 6.0-8.0. Later, during above process verification, Sanchem recommended a range of 6.0-7.0.
4/ Allowable range is 2.8 to 4.8 mL. A freshly prepared solution of SAFEGARD 3000 requires 4.4 to 4.8 mL.

TABLE 16. SOLUTION MAINTENANCE FOR SAFEGARD 4000 UNDER TASK 6 PROCESS VERIFICATION

DATE OF PROCESSING	pH3/	ADDITION OF 50% POTASSIUM HYDROXIDE TO RAISE pH (GRAMS)	EVIDENCE OF PRECIPITATE	WATER ADDITION (INCH)
4/26	11.6		None	
4/27	11.5		None	2.0
4/28	11.6		None	3.0
5/1	11.6		None	3.5
5/2	11.6		None	3.0
5/3	11.5	200	None	3.0
5/4	11.6		None	3.5
5/5	11.5	300	None	3.5
5/8	11.7		Solution Cloudy	4.0
5/9	11.8	300	4/	4.0
5/10 1/	11.7		5/	3.5
5/11	11.8		5/	4.0
5/12	11.8		5/	4.0
5/15	11.8		5/	4.5
5/16	11.8		5/	4.0
5/17	11.8		5/	4.0
5/18	11.7		5/	4.0
5/19	11.8		5/	4.0
5/22	11.7		5/	4.5
5/23	11.7		5/	4.0
5/24	11.7		5/	4.0
5/25 2/	11.6		5/	4.0

NOTES:
1/ Did not process any panels on this day.
2/ Day after end of test. Did not process panels on this day.
3/ Required range is 11.5 – 12.0.
4/ Appears something floats to surface directly above panels as they are being processed in the solution.
5/ Same as 4/ above plus precipitate visible in bottom of tank prior to agitation of the solution. Precipitate was analyzed by Electron Dispersive Spectroscopy (EDS) and elements detected indicated it was likely potassium aluminosilicate (i.e., $KAlSi_3O_8$) which, according to Sanchem, Incorporated, would be expected and is not a problem.

f. Process Specification

Appendix E provides a process specification applicable to treatment of IVD aluminum-coated metal parts using Sealing Steps II and III of the Sanchem-CC process. This process specification is based primarily on the verification testing performed under Task 6. For information purposes, the Sanchem designation for the Sealing Step II solution is SAFEGARD 3000 and the Sanchem designation for the Sealing Step III solution is SAFEGARD 4000.

SECTION VIII
CONCLUSIONS

The conclusions of this program are broken down into two categories. More specifically, those directly applicable to IVD aluminum-coated alloy steel parts, and those applicable to IVD aluminum-coated aluminum parts.

A. IVD ALUMINUM-COATED ALLOY STEEL PARTS INCLUDING FASTENERS

1. The process consisting of Sealing Steps II and III of the Sanchem-CC process is a satisfactory alternative to chromate conversion coating. This conclusion is based on comparison testing of the two treatments on IVD aluminum-coated steel panels in regard to 5% neutral salt fog for 8000 hours, sulfur dioxide salt fog for 500 hours, and outdoor exposure in St. Louis, MO for 58 weeks. It is also based on comparable performance of the two treatments in regard to treated IVD aluminum-coated steel fasteners which were installed in aluminum alloy bars with bare countersinks and then subjected to 5% neutral salt fog testing. Finally, it is based on required performance in regard to primer adhesion and contact electrical resistance. Sealing Steps II and III of the Sanchem-CC process, like chromate conversion coating, meet all the requirements of the IVD aluminum coating military specification, MIL-C-83488.

2. Sealing Steps II and III of the Sanchem-CC process appears to be a viable production process based on the process verification work performed under Task 6.

3. Alodine 2000 is also a satisfactory alternative to chromate conversion coating, except for its performance in sulfur dioxide salt fog. With this one exception, it is acceptable for all the same reasons noted in Paragraph 1 for Sealing Steps II and III of the Sanchem-CC process.

4. Sealing Step II of the Sanchem-CC process by itself provides comparable salt spray performance to chromate conversion coating in regard to 5% neutral salt fog, and acceptable performance in regard to sulfur dioxide salt fog testing. However, it is much more prone to initial pitting compared to chromate conversion coating and the other treatments investigated. Also, on the negative side, water-based primers do not adhere as required when treated panels are primed, immersed in distilled water for 24 hours, and then subjected to a scribed, tape adhesion test. Finally, on the negative side, treated panels, after 7 days exposure to 5% neutral salt fog, often exceed the contact electrical resistance requirement of 10-milliohms required by MIL-C-81706 for a chromate conversion coating.

5. Permatreat 1001 is the least effective of all the candidate nonchromated conversion coatings evaluated in regard to salt spray performance. It does, however, provide required performance in regard to primer adhesion and contact electrical resistance.

B. IVD ALUMINUM-COATED ALUMINUM PARTS INCLUDING IVD ALUMINUM COATED FASTENERS (ALLOY STEEL AND TITANIUM) INSTALLED IN ALUMINUM ALLOY WITH BARE COUNTERSINKS

1. Alodine 2000 is a satisfactory alternative to chromate conversion coating, except for its performance in sulfur dioxide salt fog. With the exception noted, this conclusion is based on comparable performance of the two treatments in regard to IVD aluminum-coated aluminum panels subjected to 5% neutral salt fog testing for 3000 hours. It is also based on required performance in regard to primer adhesion and contact electrical resistance.

2. Sealing Steps II and III of the Sanchem-CC process is a satisfactory process, but it is not as effective as chromate conversion coating. This conclusion is based primarily on comparison testing of the two treatments on IVD aluminum-coated aluminum panels in regard to 5% neutral salt fog for 3000 hours. More specifically, taking all of the initial and repeat tests into account for both treatments, the Sanchem treated panels tended to pit more, and earlier in testing compared to the chromate conversion-coated panels. On the positive side, repeat fastener bar testing using IVD aluminum-coated steel fasteners demonstrated comparable performance for the two treatments after exposure to 5% neutral salt fog for 1008 hours. Also, on the positive side, Sealing Steps II and III of the Sanchem-CC process provide required performance in regard to primer adhesion and contact electrical resistance.

3. PERMATREAT 1001 is possibly a satisfactory alternative to chromate conversion coating. On the positive side, its performance on IVD aluminum-coated aluminum panels subjected to 5% neutral salt fog testing for 3000 hours was comparable to chromate conversion coating. Also, on the positive side, it provided required performance in regard to primer adhesion and contact electrical resistance. On the negative side, its performance was the least acceptable of all the candidate treatments in regard to fastener bar testing of IVD aluminum-coated titanium fasteners where the fasteners were installed in aluminum alloy bars with bare countersinks and then subjected to 5% neutral salt fog testing. Also, on the negative side, PERMATREAT 1001 is most effective when squirted on a part and then dispersed over the surface of the part by spinning the part or blowing air on the part. Conventional tank processing by itself for this non-rinse treatment can result in thickness build-ups which are detrimental to paint adhesion.

4. Sealing Step II of the Sanchem-CC process by itself is not a satisfactory alternative to chromate conversion coating. This is basically due to primer adhesion. More specifically, water-based primers do not adhere as required when primed panels are immersed in distilled water for 24 hours and then subjected to a scribed, tape-adhesion test. It should also be noted that Sealing Step II treated panels, after seven days exposure to 5% neutral salt fog, often exceed the contact electrical resistance requirement of 10-milliohms required by MIL-C-81706 for a chromate conversion coating. On the positive side, the performance of Sealing Step II on IVD aluminum-coated aluminum panels subjected to 5% neutral salt fog for 3000 hours was comparable to chromate conversion coating. Also, on the positive side, repeat fastener bar testing, using IVD aluminum-coated steel fasteners, demonstrated comparable performance for the two treatments after exposure to 5% neutral salt fog for 1008 hours.

SECTION IX
RECOMMENDATIONS

A. PRIMARY RECOMMENDATION

1. Proceed with set-up of a pilot production line for alloy steel parts using the final process selected and verified under this program. This process consists of Sealing Steps II (immersion in a permanganate solution at 140°F for 3 minutes) and III (immersion in a potassium silicate solution at 200°F for 1 minute) of the Sanchem-CC process. Prior to processing any production parts, verify required salt spray performance and primer adhesion. Also, prior to processing any production parts, define/establish contact electrical resistance requirements and verify required performance. Use IVD aluminum-coated alloy steel panels for the above investigations.

B. SUGGESTIONS/RECOMMENDATIONS RELATED TO THE PILOT PRODUCTION LINE (BASED ON THE UNDERSTANDING THAT A PILOT PRODUCTION PROCESSING LINE WILL BE SET UP AT ROBINS AIR FORCE BASE UNDER A PHASE II FOLLOW-ON EFFORT TO THIS JUST COMPLETED PROGRAM)

1. Use a tank approximately 4 feet long x 4 feet wide x 4 feet deep which corresponds to approximately 480 gallons of solution. Two such tanks will be required; one for the SAFEGARD 3000 solution and one for the SAFEGARD 4000 solution. The tank for the SAFEGARD 3000 solution should be constructed from 304 stainless steel, mild steel lined with polypropylene or Koroseal, polypropylene, or equivalent. The tank for the SAFEGARD 4000 solution should be constructed from stainless steel, mild steel, polypropylene, or equivalent. If two tanks are not available, an alternative might be to place two tanks of the recommended size into a larger available tank. The recommended tank size of 4 feet x 4 feet x 4 feet is based on the largest IVD aluminum-coated part currently chromate conversion coated at Robins Air Force Base (C-130 propeller hub), provisions for heating, and adequate working space. Refer to the Appendix E process specification concerning the requirements for heating and agitation of the tanks.

C. SUGGESTIONS/RECOMMENDATIONS ADDRESSING FURTHER ENHANCEMENT OF THE PERFORMANCE OF SEALING STEPS II AND III OF THE SANCHEM-CC PROCESS

1. Investigate the salt spray performance of Sealing Steps II and III of the Sanchem-CC process when the normal concentration of the Sealing Step II is doubled. Five percent neutral salt fog testing under the process optimization study of Task 6 indicated that Sealing Step II by itself at double concentration was effective in preventing initial pitting compared to the standard recommended concentration. Five IVD aluminum-coated alloy steel panels have been prepared using this recommended treatment and are performing very well in 5% neutral salt fog testing in regard to initial pitting. These panels were prepared in the laboratory based on the results of the process optimization study and have not been

previously reported. In conjunction with the above testing, prepare and test controls using the standard iteration of Sealing Steps II and III of the Sanchem-CC process.

2. Investigate the salt spray performance of Sealing Steps II and III of the Sanchem-CC process when the temperature of the permanganate or Sealing Step II solution is raised to 170°F rather than the normal 140°F. Salt spray testing under Supplementary Test No. 2 of Task 2 indicated that this temperature change was effective in reducing initial pitting for Sealing Step II by itself. Subsequent salt spray testing under the process optimization study of Task 6 at both temperatures, combined with Sealing Step III, did not show a difference in performance. This, then, is considered an unresolved issue worthy of further investigation. In conjunction with the above testing, prepare and test controls using the standard iteration of Sealing Steps II and III of the Sanchem-CC process.

3. If inconsistencies are encountered in regard to contact electrical resistance test results for either the pilot production line investigation or any of the above processing variations, conduct an appropriate investigation to determine the reason(s).

D. SUGGESTIONS/RECOMMENDATIONS RELATED TO OTHER CONVERSION COATING SYSTEMS

1. Evaluate the sulfur dioxide salt fog performance of Alodine 2000 using Parker+Amchem's latest generation seal which they identify as TD-3095-Y and describe as being inorganic. The TD-3095-J seal used for testing under this development program was described by Parker+Amchem as being a combination organic/inorganic material.

2. Investigate the use of other seals in conjunction with Sealing Step II of the Sanchem-CC process.

APPENDIX A

SUPPLEMENTARY TEST 1

FURTHER INVESTIGATION OF SANCHEM-CC PROCESS CONSISTING OF OXIDE FILM FORMATION STEP FOLLOWED BY SEALING STEPS I AND II

Supplementary Test 1 was conducted to resolve the discrepancy in salt spray test results between the initial test performed under in-house funding and the Task 2 testing. More specifically, 16 IVD aluminum-coated 4130 steel panels were prepared using steel sheet material totally free of any rust or surface imperfections. The IVD aluminum coating was applied to conform to MIL-C-83488, Class 2 (0.5-mil thick minimum). The actual average thicknesses on the panels ranged from 0.7 mil to 1.0 mil.

Eight of the above panels were sent to Sanchem, Incorporated for treatment using the Sanchem-CC process consisting of the Oxide Film Formation Step followed by Sealing Steps I and II. They were sent in increments of two panels over a two week period, and treatment was performed accordingly at four different times. The other eight panels were chromate conversion coated in-house. Glass bead peening and chromate conversion coating of these panels was performed at exactly the same time intervals as the panels sent to Sanchem, Incorporated.

Before sending the panels to Sanchem, Incorporated for treatment, each was examined under a microscope at 30X and any flaws in the IVD aluminum coating were noted. Similarly, after chromate conversion coating of the control panels in-house, they were examined under a microscope at 30X. The above examinations revealed small flaws in the IVD aluminum coating in the form of voids or pits. The number of pits varied from panel-to-panel. Some panels had one or two pits while others had up to seven or eight pits.

The Sanchem treated panels and the MDA treated chromate conversion-coated control panels were subjected to 5% neutral salt fog testing. One set of the panels (i.e., four Sanchem panels and four chromate conversion coated panels treated at the same intervals over a 1-week period) was exposed for a total of 1872 hours while the other set was exposed for a total of 1704 hours.

After one set of the panels had been subjected to 336 hours of salt spray testing and the other 168 hours of testing, several items were considered noteworthy. These items are noted below.

- A significant number of additional pits (i.e., over and above those found under the microscope) appeared in all but one of the Sanchem treated panels after only one to two days of salt spray testing. Also, in general, the number of pits continued to increase over seven days of salt spray exposure. In regard to the chromate conversion-coated panels, significantly fewer pits appeared over seven days of testing compared to the Sanchem treated panels.
- Once pits appeared in the Sanchem treated panels, they tended to grow slowly with increased exposure time. However, with only a couple of exceptions, there was no evidence of IVD aluminum depletion products associated with this growth in pit size over 7 days of salt spray exposure. With a few minor exceptions, pits in the chromate conversion-coated panels did not tend to grow in size with increased exposure time.

- Small areas on the test side of the panels corresponding to the location of hangars during IVD aluminum coating had little or no IVD aluminum coating present. Depletion of the IVD aluminum coating was significant around these areas after 336 hours of salt spray exposure for the Sanchem treated panels. The chromate conversion-coated panels, on the other hand, did not show any evidence of aluminum depletion at the hangar marks after 336 hours of salt spray exposure.

After one set of the panels had been subjected to 1056 hours of salt spray testing and the other set 888 hours of testing, it was evident, as shown in Figure A-1, that the Sanchem-treated panels were performing significantly better than the equivalent panels tested initially under Task 2. One phenomenon still evident and significant, however, concerned the rate of IVD aluminum depletion. More specifically, IVD aluminum depletion occurred sooner and then continued more rapidly for the Sanchem-CC process (i.e., Oxide Film Formation Step followed by Sealing Steps I and II) compared to the chromate conversion coating process. This fact is evident in Figures A-2 and A-3 at the panel hangar locations near the holes. It should be noted that the tape was applied to the top and bottom of the Sanchem treated panel shown in Figure A-2 after 744 hours of salt spray testing, and to the Sanchem treated panels shown in Figure A-1 after 576 hours of salt spray testing. This was done when it was decided that these panels would be subjected to long term salt spray testing rather than the originally planned three weeks of testing. Standard practice for any long term salt spray testing is to tape the back side and all exposed edges of the panel.

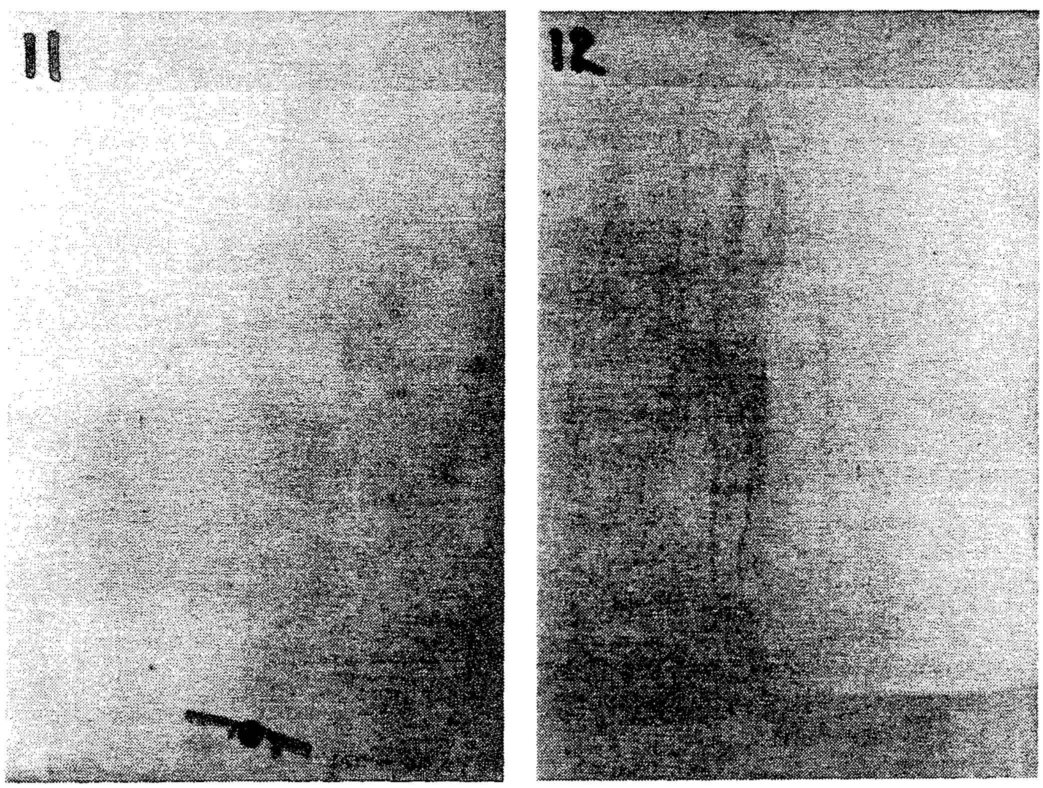

Figure A-1. Sanchem-CC Process (Oxide Film Formation Step Followed By Sealing Steps I and II) After 888 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 11 & 12)

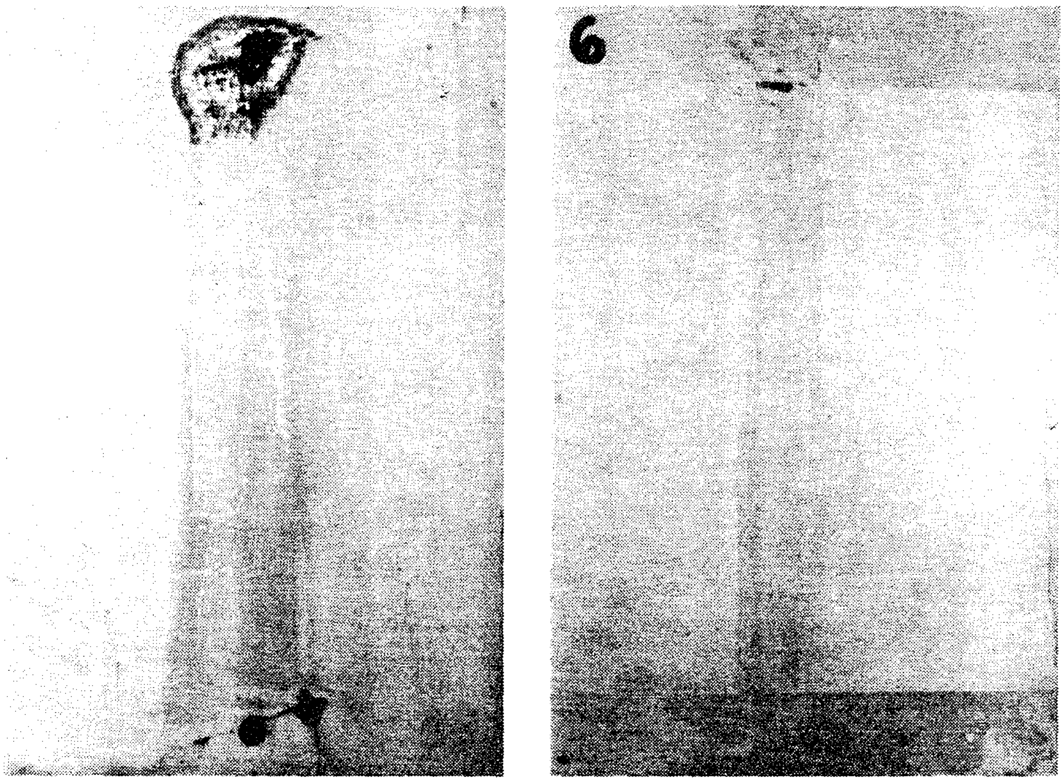

Figure A-2. Sanchem-CC Process (Oxide Film Formation Step Followed By Sealing Steps I and II) After 1056 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 5 & 6)

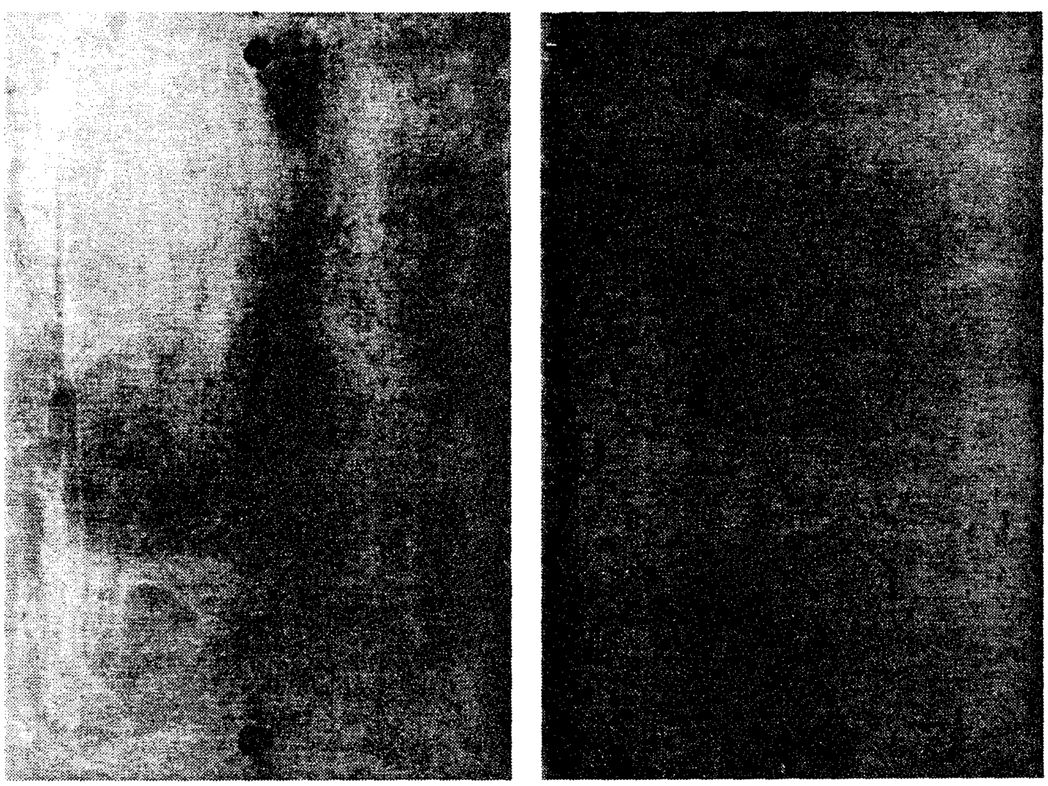

Figure A-3. Chromate Conversion Coating Process After 1056 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 7 & 8)

Salt spray testing was concluded under Supplementary Test 1 after one set of the panels had been subjected to 1872 hours of salt spray testing and the other set 1704 hours of testing. As was the case after 1056 and 888 hours of testing, the Sanchem treated panels, as shown in Figure A-4, performed significantly better than the equivalent panels tested initially under Task 2. Also, as noted before, the rate of depletion of the IVD aluminum coating was more rapid for the Sanchem treated panels compared to the chromate conversion-coated panels. This fact is evident in Figures A-5 and A-6, at the panel hangar locations near the holes.

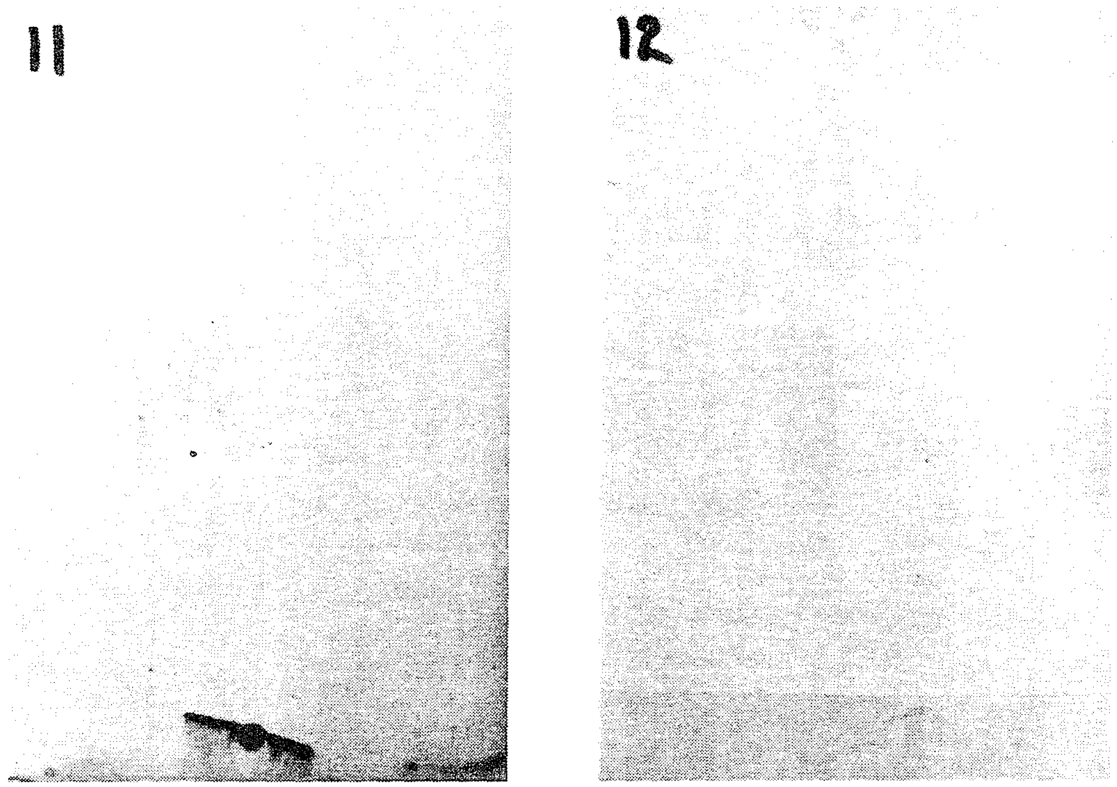

Figure A-4. Sanchem-CC Process (Oxide Film Formation Step Followed By Sealing Steps I and II) After 1704 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 11 & 12)

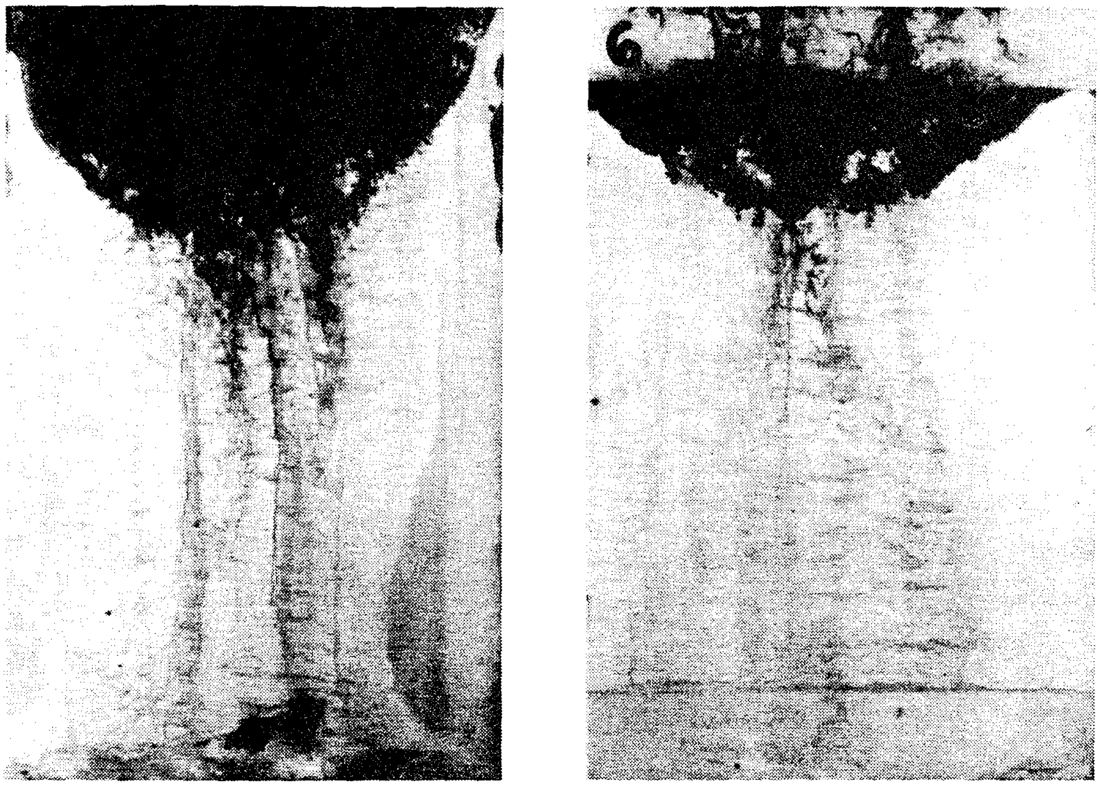

Figure A-5. Sanchem-CC Process (Oxide Film Formation Step Followe By Sealing Steps I and II) After 1872 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 5 & 6)

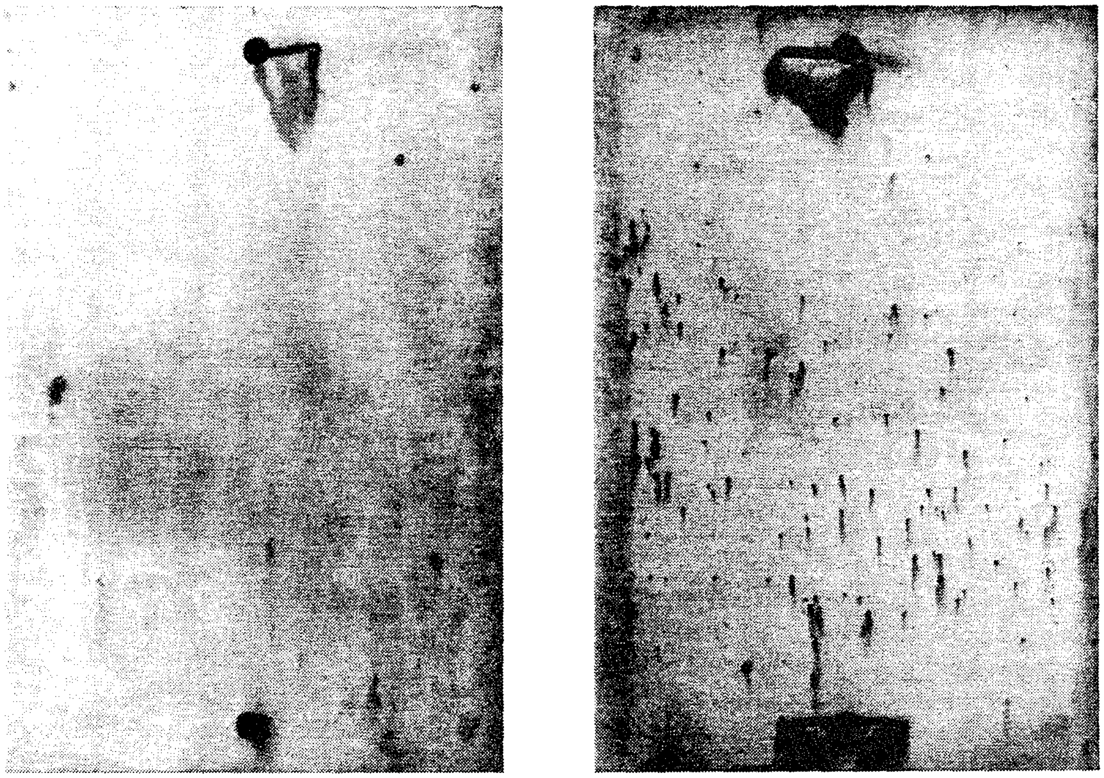

Figure A-6. Chromate Conversion Coating Process After 1872 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 7 & 8)

As a result of the testing performed under Supplementary Test 1, several conclusions can be made in regard to the performance of chromate conversion coating versus the Sanchem-CC process consisting of the Oxide Film Formation Step followed by Sealing Steps I and II. These are summarized below.

- A chromated conversion coating is more effective in preventing pits from occurring initially compared to the Sanchem, nonchromated conversion coating.
- A chromated conversion coating is more effective in preventing pits from growing compared to the Sanchem, nonchromated conversion coating.
- When a flaw or pit is present in the IVD aluminum coating, a chromated conversion coating will significantly delay the onset of aluminum depletion compared to the Sanchem, nonchromated conversion coating.
- Once depletion of the IVD aluminum coating begins, a chromate conversion coating is significantly more effective in reducing the rate of depletion compared to the Sanchem, nonchromated conversion coating.

APPENDIX B

SUPPLEMENTARY TEST 2

INVESTIGATION OF VARIATIONS OF SEALING STEP II OF THE SANCHEM-CC PROCESS

Under Task 2 salt spray testing, the IVD aluminum-coated steel panels treated with only Sealing Step II of the Sanchem-CC process (i.e., immersion for 1 minute in a permanganate solution at 140°F) were the only panels out of all six candidate nonchromated conversion coatings which passed 3000 hours of salt spray testing. This performance was somewhat surprising because numerous dark spots or pits appeared in the coating on these panels after only one day of salt spray testing. Some of these initial appearing small dark spots or pits displayed short dark tails which would tend to indicate a leaching type process was taking place. As time passed, the dark spots or pits became larger, and there was more evidence of staining or leaching associated with the dark spots or pits. Also, at the conclusion of testing, two of the three Sealing Step II panels of Task 2 showed evidence of IVD aluminum depletion, but it was minor and confined to a few localized areas. It is evident, then, that the permanganate solution treatment by itself did not prevent initial pitting, but was very effective in preventing IVD aluminum depletion and subsequent corrosion of the steel substrate.

As a result of the salt spray performance of the panels treated with only Sealing Step II of the Sanchem-CC process, an investigation was initiated which used this step as the focal point for possibly reducing initial pitting. More specifically, IVD aluminum-coated steel panels were prepared using steel sheet material totally free of any rust or surface imperfections. The average thickness of the IVD aluminum coating on the various panels ranged from 0.8 to 1.0 mil. Table B-1 provides a summary of the Sealing Step II variations used to treat the panels. Two panels were prepared for each treatment. Treatments 9 and 12 involved first alkaline cleaning and then deoxidizing prior to the Sealing Step II, permanganate solution treatment. These variations were suggested by John Bibber at Sanchem. The alkaline cleaning was intended to remove any traces of silica or any other contaminates which might have been present from previous glass bead peening of the IVD aluminum coating. This, in turn, might further open up the pores in the IVD aluminum coating. The nitric acid deoxidation step was intended to passivate the surface and provide a thin, uniform layer of aluminum oxide. The overall theory was that better performance of the permanganate treatment could be achieved if the IVD aluminum surface was clean, passivated, and exhibited a thin, uniform layer of aluminum oxide.

Figures B-1 – B-8 show the various panels treated at Sanchem, Incorporated after 336 hours of 5% neutral salt fog testing, and Figure B-9 shows the chromate conversion-coated control panels. Of significance, dark spots or pits appeared on the panels subjected to Treatments 1 and 6 after only 24 hours of salt spray testing. These dark spots grew in size with increasing exposure time, and after seven days of salt spray testing, they began to show distinct dark tails. The performance of these panels was similar to those tested under Task 2 which were treated with only Sealing Step II of the Sanchem-CC process, except there were far fewer pits after 24 hours of salt spray testing. For information purposes, it should be noted that the permanganate treatment used for Treatments 1 and 6 under Supplementary Test 2 is the same treatment as Sealing Step II of the Sanchem-CC process used for the Task 2 panels, except the immersion times were three and ten minutes respectively instead of one minute. In regard to the panels subjected to Treatments 7 and 8, pits also appeared after only 24 hours of salt spray testing. Compared to Treatments 1 and 6, however, these pits were smaller and less distinct. In general, they were only observable under slight magnification. After seven days of salt spray exposure, some of the dark spots or pits on the Treatment 8 panels had become prominent and exhibited dark tails. After seven days of salt spray exposure, there were a few more dark spots or pits on the Treatment 7 panels, but all were small and exhibited no dark tails. In regard to the panels subjected to Treatments 9, 11 and 12, there was anywhere from 2 to 15 very small pits after 24 hours of salt spray testing, and, in general, these were only observable under slight magnification. The number and size of these pits did not change appreciably up to seven days of salt spray testing. In a final note, the panels subjected to Treatment 3 appeared to show evidence of IVD aluminum depletion after only 24 hours of salt spray testing. This condition became more pronounced after 7 days of salt spray testing. It is possible that the salt spray may have diffused through the polyurethane coating and became trapped between the IVD aluminum coating and the polyurethane coating. The salt spray performance described above for the various treatments evaluated under this Supplementary Test 2 generally held true through the 336 hours (2 weeks) of salt spray testing. Of significance, all were effective in significantly reducing initial pitting compared to the initial Task 2 panels treated with Sealing Step II of the Sanchem-CC process.

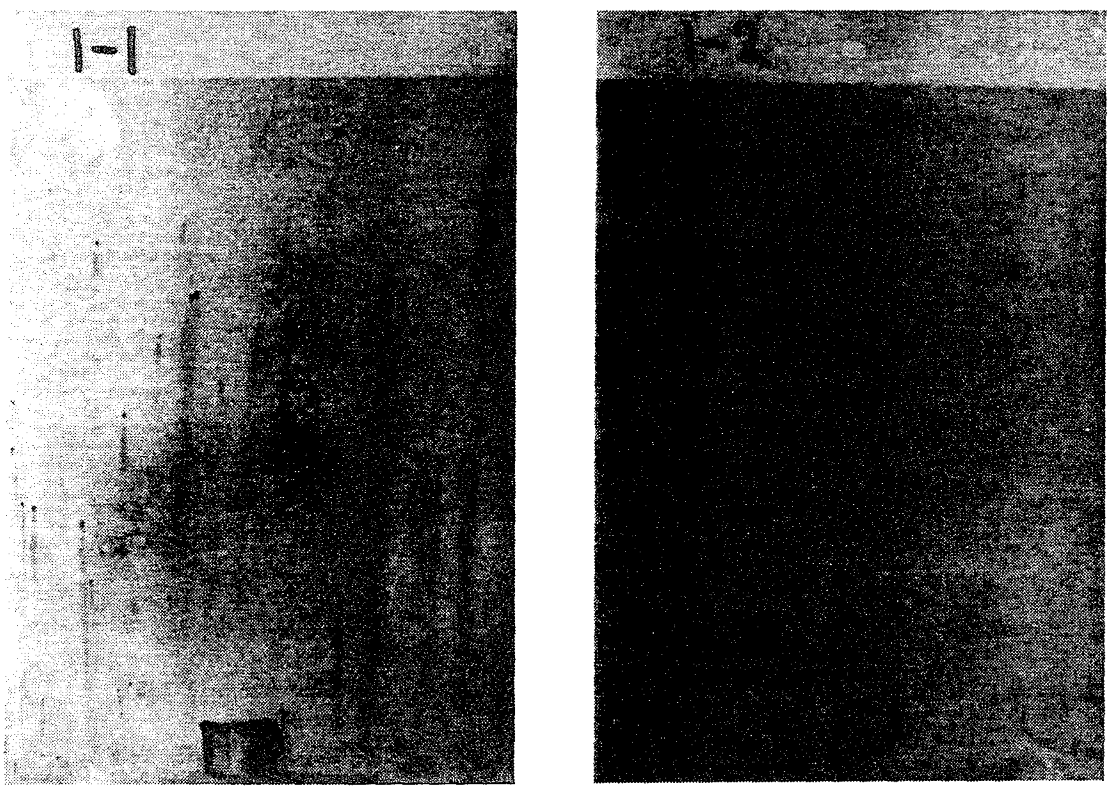

Figure B-1. Standard Permanganate Treatment (140°F for 3 Minutes) After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 1-1 & 1-2)

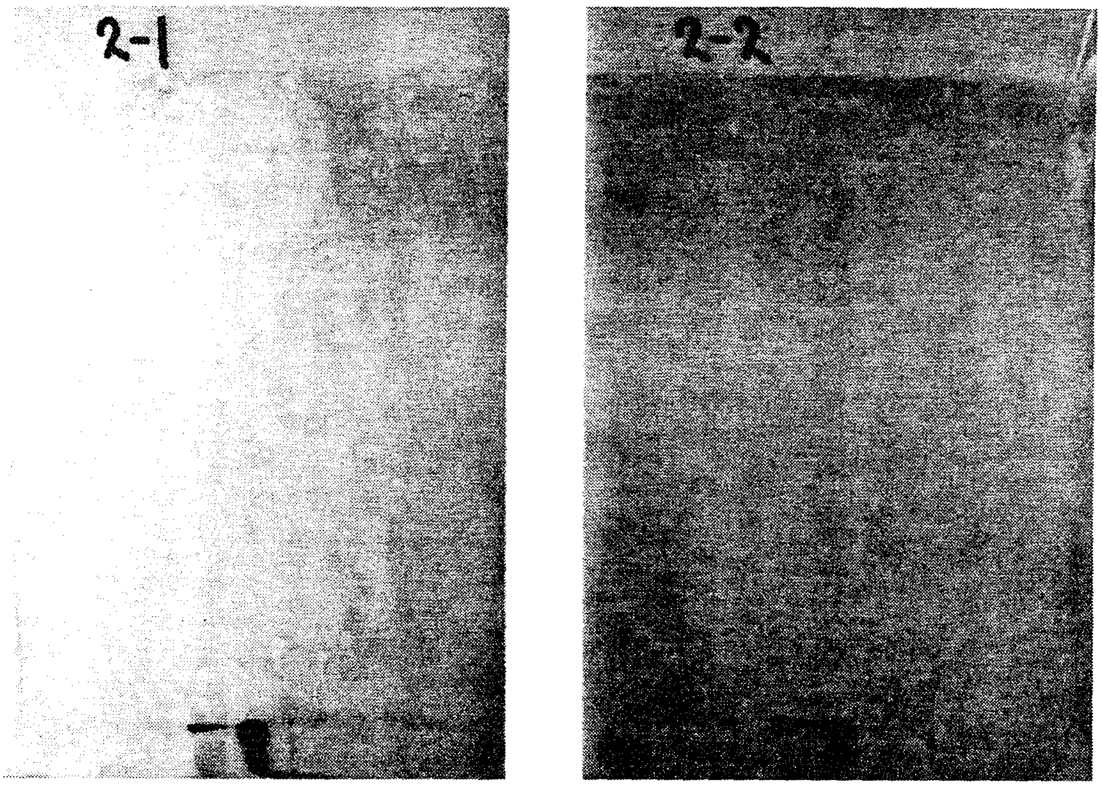

Figure B-2. Standard Permanganate Treatment with Potassium Silicate Seal After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 2-1 & 2-2)

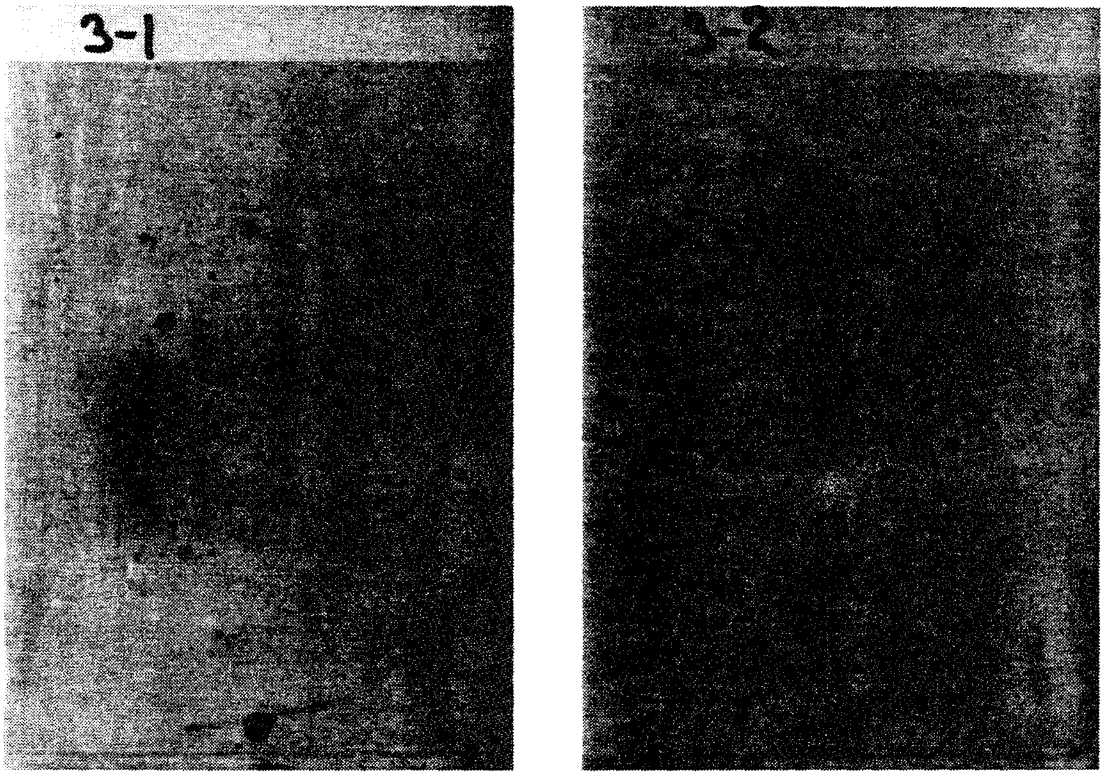

Figure B-3. Standard Permanganate Treatment with Polyurethane Seal After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 3-1 & 3-2)

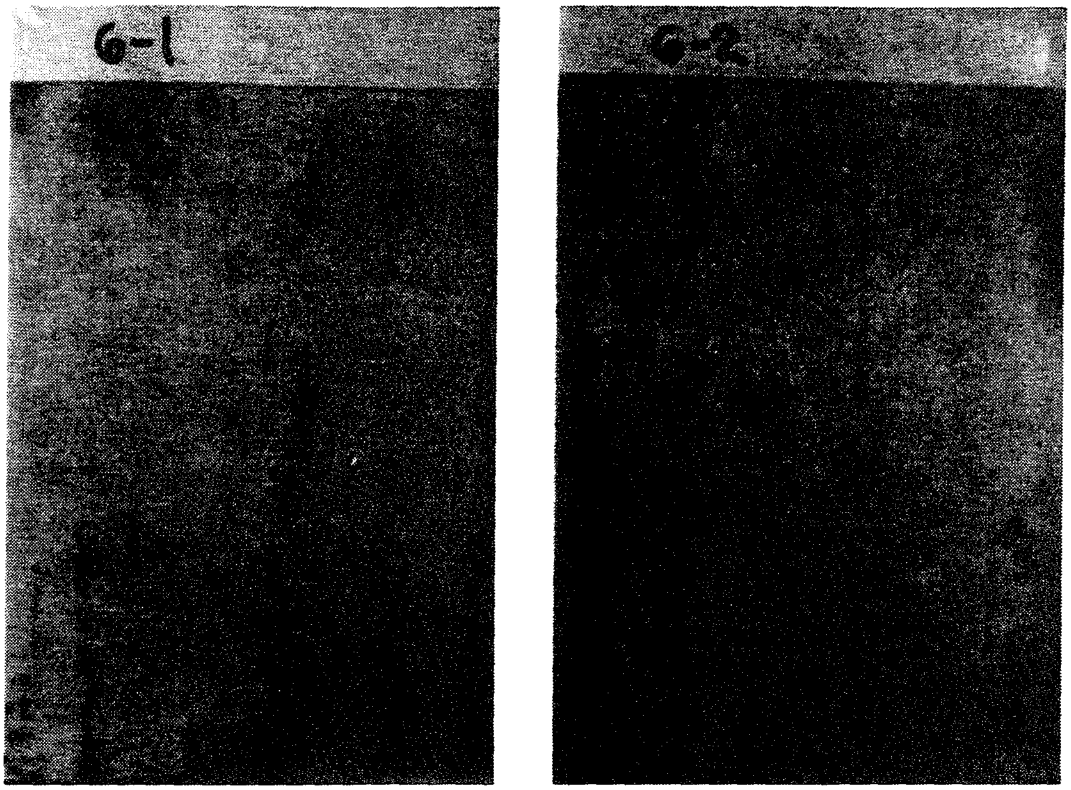

Figure B-4. Standard Permanganate Treatment Except Increased Time (10 Minutes) After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 6-1 & 6-2)

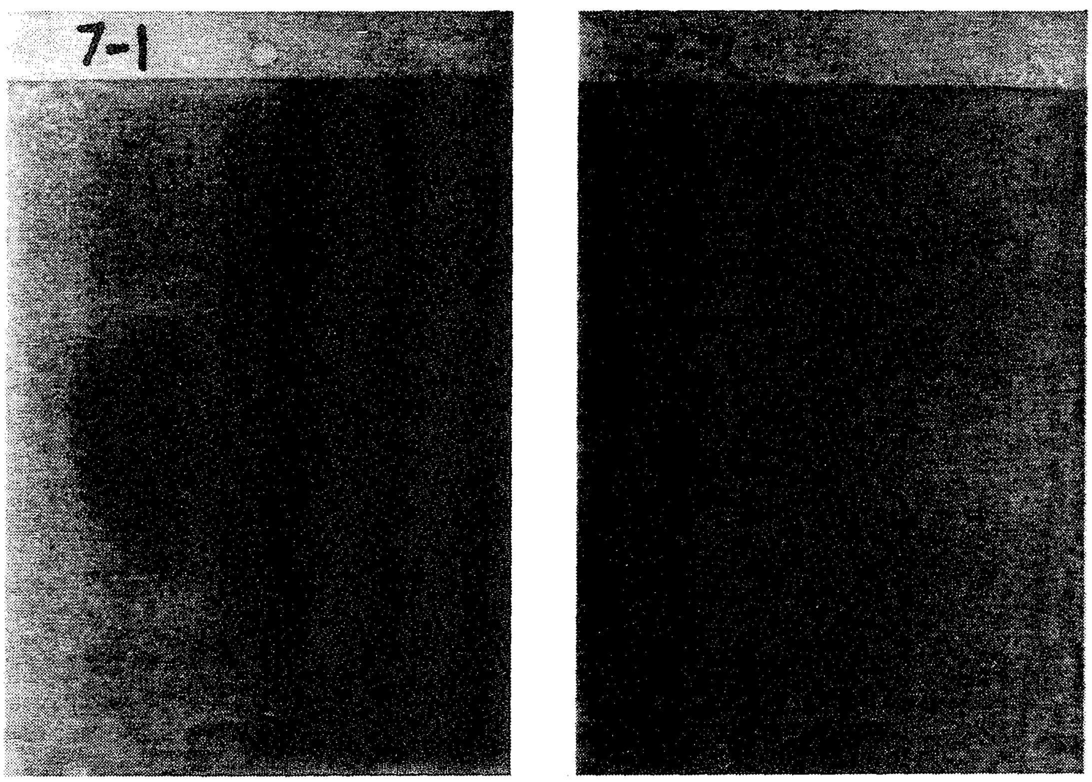

Figure B-5. Standard Permanganate Treatment Except Increased Temperature (170°F) After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 7-1 & 7-2)

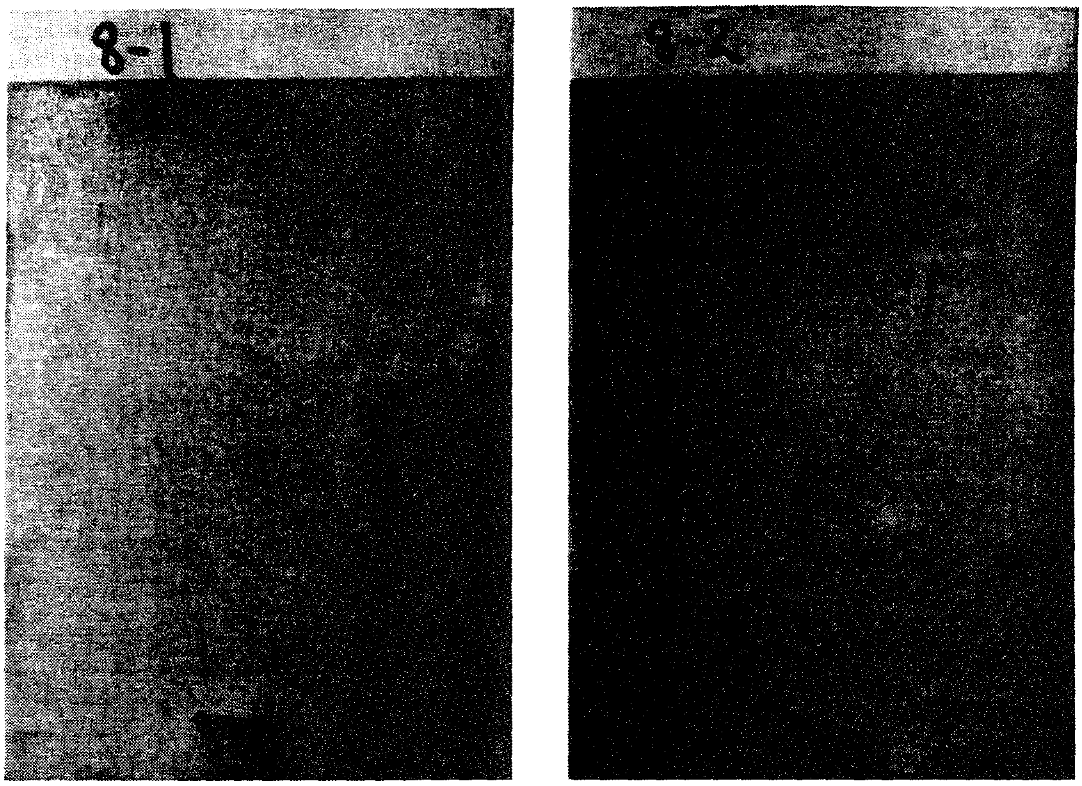

Figure B-6. Standard Permanganate Treatment Except Increased Time and Temperature (170°F for 10 Minutes) After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 8-1 & 8-2)

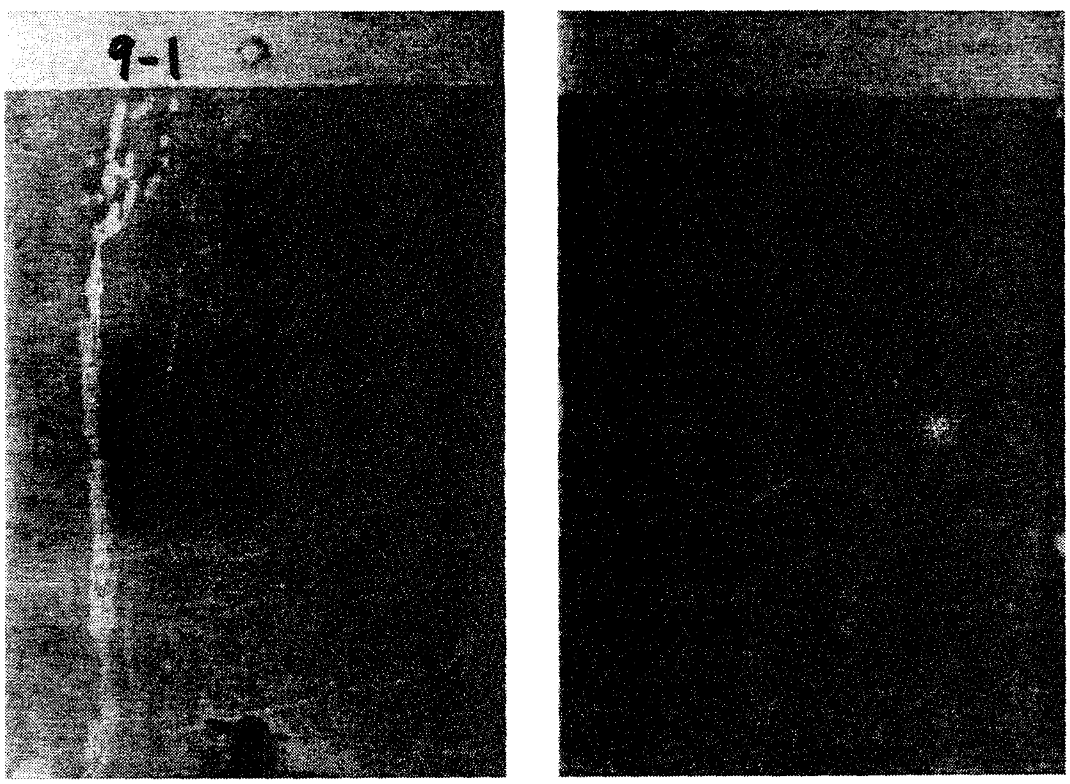

Figure B-7. Alkaline Clean, Nitric Acid Deoxidize (Deox 1000), and Standard Permanganate Treatment After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 9-1 & 9-2)

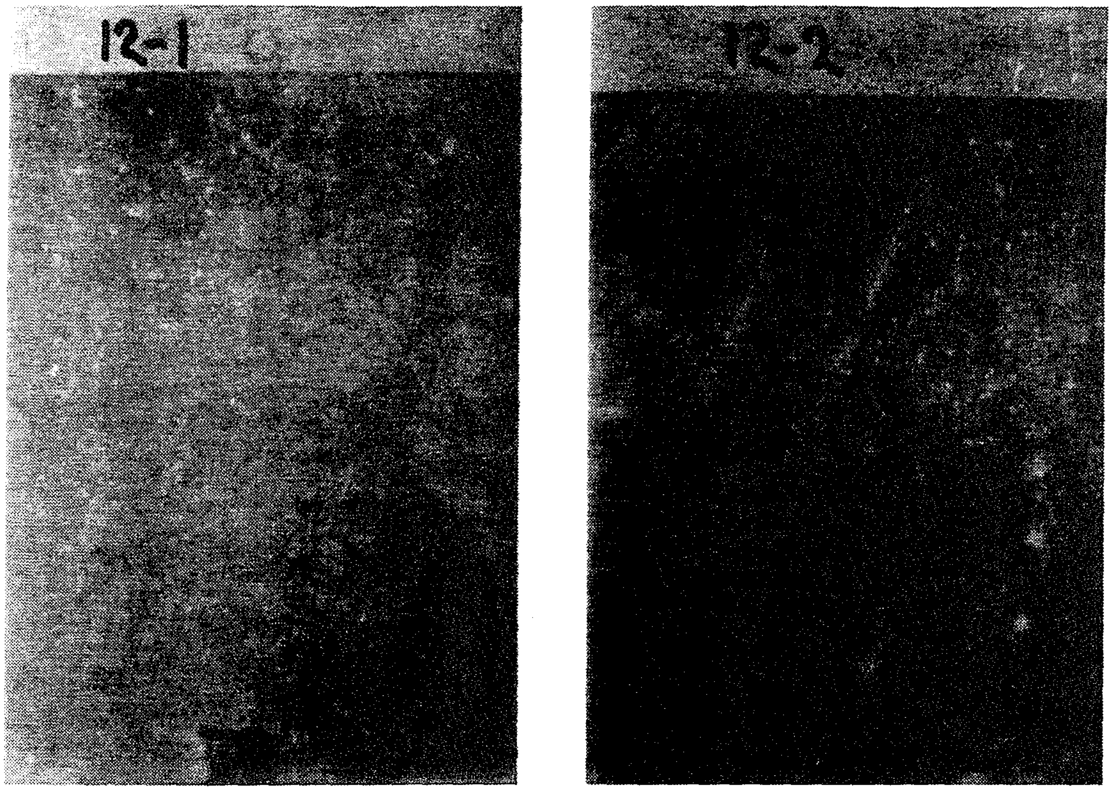

Figure B-8. Alkaline Clean, Nitric Acid Deoxidize (10% Nitric Acid), and Standard Permanganate Treatment After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 12-1 & 12-2)

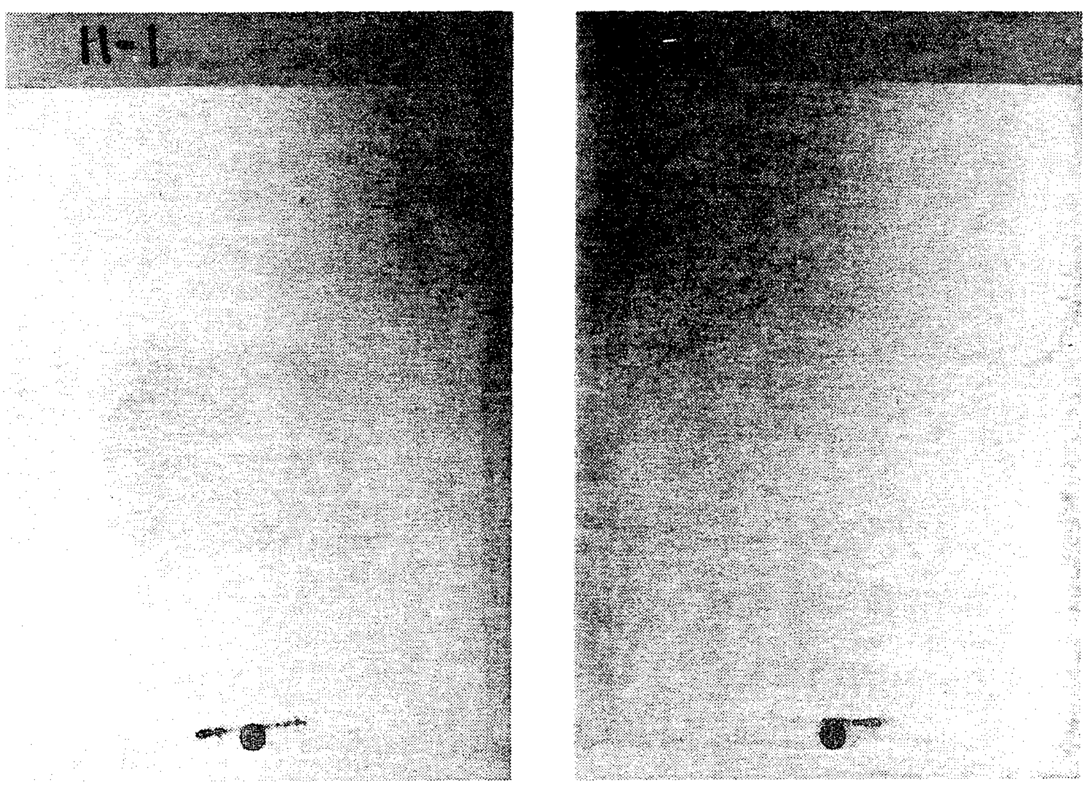

Figure B-9. Chromate Conversion Coating Process After 336 Hours Exposure to 5% Neutral Salt Fog (Panel Numbers 11-1 & 11-2)

The panels defined by Table B-1 representing variations of Sealing Step II of the Sanchem-CC process were subjected to a total of 1152 hours of 5% neutral salt fog testing. As was the case after 336 hours of salt spray testing, it still could be concluded after 1152 hours of exposure that most of the variations significantly reduced initial pitting compared to the initial Task 2 panels treated with Sealing Step II of the Sanchem-CC process.

At the end of the 1152 hours of salt spray exposure, the Sealing Step II variations of Table B-1 were ranked in regard to performance. This ranking is noted below:

7 and 11	Most Desirable
2 and 12	↓
1, 6 and 8	↓
3	↓
9	Least Desirable

There are small areas at the one end of each panel which have little or no IVD aluminum coating. These areas correspond to the location of the hangars which were used to suspend the panels during IVD aluminum coating. These areas were monitored in regard to IVD aluminum depletion and/or corrosion during the 1152 hours of salt spray testing. The various treatments were then ranked as noted below in regard to preventing IVD aluminum depletion and/or corrosion after 1152 hours of salt spray exposure.

3, 8 and 11	Most Desirable
6 and 12	↓
7	↓
1	↓
2	↓
9	Least Desirable

Each of the Sealing Step variations of Table B-1 was also ranked in regard to preventing IVD aluminum depletion and/or corrosion on the back or non-test side of the panel. It should be noted that the back side of the panel is characterized by an IVD aluminum coating which is both thinner and varies in thickness (i.e., coating is thicker along the periphery of the back side of the panel). This condition is due to the fact that the test side of the panels faced the "boats" used to vaporize the aluminum in the chamber and the coating on the back of the panel is deposited via a wrap around effect. As with the ranking associated with the hangar marks, the intention or ranking the back side of the panels was to make a judgement of the treatment variations in regard to their ability to prevent IVD aluminum depletion and/or corrosion of the steel substrate. The panels were ranked as follows:

11 and 8	Most Desirable
6	↓
7	↓
1, 2 and 3	↓
12	↓
9	Least Desirable

Based on all of the above rankings made (i.e., test side, hangar locations on test side, and back side), one could conclude that Treatment 8 was the most desirable. It could further be surmised that Treatment 8, followed by a potassium silicate seal, would be desirable. Although the above conclusions have been made, it should be emphasized that they must be considered preliminary since they are based on such a small number of specimens.

TABLE B-1. VARIATIONS OF SEALING STEP II (PERMANGANATE TREATMENT) INVESTIGATED

TREATMENT NO.	TREATMENT
1	Standard permanganate treatment (140°F for 3 minutes).
2	Standard permanganate treatment with potassium silicate seal for 1 minute.
3	Standard permanganate treatment with polyurethane seal (CYDROTHANE HP-1035 – polyurethane dispersion in water).
6	Standard permanganate treatment, except increased time (10 minutes).
7	Standard permanganate treatment, except increased temperature (170°F).
8	Standard permanganate treatment, except increased time and temperature (170°F for 10 minutes).
9	Alkaline clean (Alkaline Cleaner 500 – 150°F for 30 seconds), nitric acid deoxidize (DEOX 1000 – nitric acid and sodium bromate – 100°F for 30 seconds) and standard permanganate treatment.
12	Alkaline clean (Alkaline Cleaner 500 – 150°F for 3 minutes), nitric acid deoxidize (10% nitric acid – R.T. for 1 minute) and standard permanganate treatment.
11	Chromate conversion coat (Controls).

NOTE: Two panels prepared for each treatment.

APPENDIX C

SUPPLEMENTARY TEST 3

INVESTIGATION OF IMPROVED ALODINE 2000

Near the end of Task 2 salt spray testing for 3000 hours, MDA learned that Parker+Amchem had made changes to their Alodine 2000 non-chromated conversion coating. These changes were the result of problems encountered in an 80 gallon scale-up evaluation performed at Boeing in Renton, WA. One change involved addition of an ingredient to the conversion coating solution to improve bath life stability. The other change, which is of greater significance, concerned the use of an organic/inorganic seal rather than the previous completely organic seal. This latter change was probably made due to the salt spray and paint adhesion failures encountered during the scale-up evaluation at Boeing. The Parker+Amchem product designation for this improved seal was TD-3095-J.

The original version of Alodine 2000 was evaluated under Task 2 of this program in regard to salt spray performance, contact electrical resistance, and primer adhesion. Since this testing showed it was a promising candidate, eight IVD aluminum-coated steel panels were sent to Parker+Amchem for treatment with the improved Alodine 2000. The steel sheet material used for these panels was totally free of any rust or surface imperfections. The IVD aluminum coating applied to the eight panels conformed to MIL-C-83488, Class 2 (0.5 mil thick minimum). The average thickness of the IVD aluminum coating on the various panels ranged from 0.84 to 0.98 mil.

Three of the eight improved Alodine 2000 panels were exposed to 5% neutral salt fog testing for a total of 3864 hours (161 days). Of interest, early in testing, these panels performed somewhat similar to the Task 2 panels treated with Sealing Step II of the Sanchem-CC process. More specifically, a significant number of pits appeared in the coating after only a few days of salt spray exposure. Also, like the Task 2 panels, the pits on these panels became somewhat larger and darker, and exhibited dark tails as the exposure time increased. The Alodine 2000 panels are shown in Figure C-1 at the conclusion of salt spray testing. Of significance, there is no evidence of IVD aluminum depletion let alone failure. The performance of these panels was far superior to the original Alodine 2000 panels tested under Task 2.

Another three of the eight panels were subjected to contact electrical resistance testing identical to that conducted previously under Task 2. The improved Alodine 2000 met the contact electrical resistance requirements of MIL-C-81706 both before and after salt spray testing. The results of this testing are presented in Table C-1.

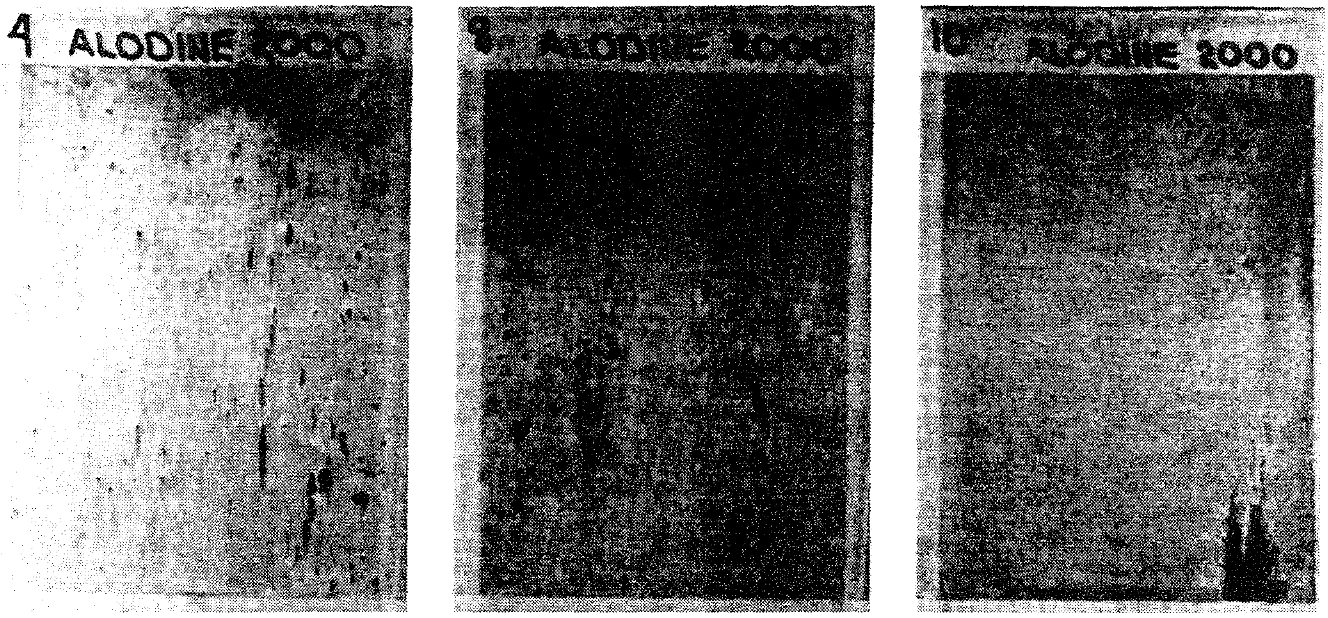

Figure C-1. Improved Alodine 2000 Panels After 3864 Hours of 5% Neutral Salt Fog Testing

TABLE C-1. RESULTS OF CONTACT ELECTRICAL RESISTANCE TESTING

CONVERSION COATING	DATE OF CONVERSION COATING	PANEL NO.	AVG. CONTACT ELECTRICAL RESISTANCE (MILLIOHMS) 1/		
			4/1/94 (NO EXPOSURE)	4/18/94 (NO EXPOSURE)	4/27/94 (AFTER 7 DAYS EXPOSURE TO 5% NEUTRAL SALT FOG)
IMPROVED ALODINE 2000	3/29/94	7 13	1.04 1.30	1.17 1.65	5.90 7.18

NOTES: 1/ Average values noted are based on five measurements taken over the surface of the panel. The maximum allowable values per MIL-C-81706 are 5.00-milliohms as treated, and 10.00-milliohms after salt fog exposure.

The remaining two panels treated with the improved Alodine 2000 were primed with a compliant, water-borne, epoxy primer. They were then subjected to a dry, tape adhesion test followed by a scribed, wet, tape adhesion test. The water-borne primer used and all parameters related to tape testing were identical to those described earlier for Task 2 testing. There were no primer adhesion failures associated with the improved Alodine 2000 treatment.

In summary, the improved Alodine 2000 treatment met contact electrical resistance and primer adhesion screening requirements. Also, and of significance, it demonstrated vastly improved salt fog performance compared to the original version of Alodine 2000 tested under Task 2.

APPENDIX D

SUPPLEMENTARY TEST 4

FLEXIBILITY AND ADHESION EVALUATION OF SEALING STEPS II AND III OF THE SANCHEM-CC PROCESS

As salt spray testing progressed under Task 4, it was felt that the treatment consisting of Sealing Steps II and III of the Sanchem-CC process offered considerable promise. This being the case, but prior to committing this process to any type of scale-up evaluation, it was felt necessary to verify required flexibility and further verify primer adhesion. Performance in this regard was of particular concern since Sealing Step III of the Sanchem-CC process seals the IVD aluminum coating using a potassium silicate solution. Silicates are typically inherently brittle.

The panel material used for all flexibility and adhesion testing was bare 2024-T3 aluminum per QQ-A-205/4. All 72 panels involved in the test program were 3 inches x 6 inches x 0.020 inch thick. Initially, the panels were chemically cleaned per McDonnell Process Specification P.S. 13143 which involved aqueous degreasing, alkaline cleaning, pickling and deoxidizing. Next, the panels were IVD aluminum-coated. Then, after the normal glass bead peening operation, 36 of the panels were chromate conversion-coated and the remaining 36 panels were treated with Sealing Steps II and III of the Sanchem-CC process. After conversion coating, 12 panels for each of the conversion coatings were primed with two coats of Courtauld's 513X408/910X831 water-borne primer. This primer conforms to MIL-P-85582, Type I and McDonnell Material Specification MMS-423. A second group of 12 panels for each conversion coating was primed with two coats of Courtauld's 519X303/910X357 solvent-based primer which conforms to MMS-425. Both of the above Courtauld's primers are currently in production use at MDA in St. Louis. The final group of 12 panels for each conversion coating was primed by electro-deposition using BASF's G28AD012 epoxy primer.

The treated and primed panels described above were subjected to cross hatch, reverse impact, and mandrel bend testing. Figure D-1 shows a summary of the testing performed, while Figure D-2 shows a representative specimen for each of the tests performed. Following is a description of the exact test procedure used for each of the three tests.

CROSS HATCH TEST: Scribe through the primer eleven, 2-inch long parallel lines, 1/16 inch apart. Then, scribe a similar set of lines at 45° across the field of the first set of lines to form 100 small diamond-shaped patches. Wipe the area free of loose primer. Perform a dry tape test on the scribed cross-hatch area per McDonnell Process Specification P.S. 21313. Express adhesion in numerical terms by counting the number of patches left intact after removal of the tape. Report the percent retention of the primer within the diamond patches formed by the cross-hatch scribed lines.

CONVERSION COATING

- **MMS-423**
  - Cross Hatch (3)
  - Reverse Impact (3)
  - Mandrel Bend
    - R.T. (3)
    - -70°F (3)
- **MMS-425**
  - Cross Hatch (3)
  - Reverse Impact (3)
  - Mandrel Bend
    - R.T. (3)
    - -70°F (3)
- **E COAT**
  - Cross Hatch (3)
  - Reverse Impact (3)
  - Mandrel Bend
    - R.T. (3)
    - -70°F (3)

NOTES
1. Above testing conducted for both conversion coatings (i.e., chromate conversion coating and Sealing Steps II & III of the Sanchem-CC Process).
2. Number in parenthesis for each test denotes number of specimens tested.

Figure D-1. Summary of Testing Performed for Both Conversion Coatings

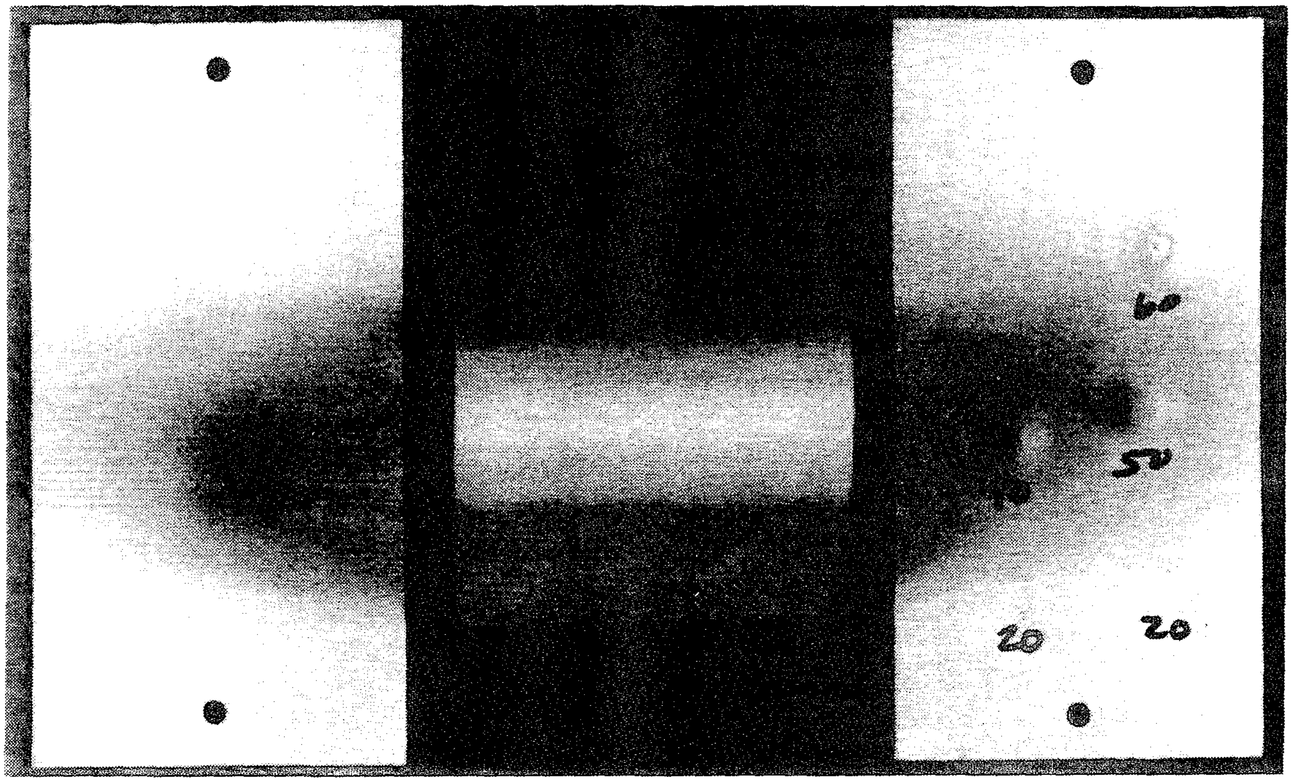

Figure D-2. Cross Hatch, Mandrel Bend and Reverse Impact Test Specimens (All Primed with MMS-423 Primer)

REVERSE IMPACT: Test the panel on the Gardner Impact Tester using the 5/8 inch diameter ball and the two pound weight. Verify that there is no loss of adhesion or cracking of the primer after subjecting the panel to this 20 inch-pounds of reverse impact. If this is the case, perform a dry tape adhesion test on the impacted area per MDA process specification P.S. 21313. Examine the panel for evidence of primer removal. Verify that there is no loss of primer adhesion.

MANDREL BEND (R.T.): Bend the primed panel through 180° over a mandrel in accordance with Method 6221 of Federal Test Method Standard 141. The diameter of the mandrel shall be in accordance with the following:

- MMS-423 Primer – 1" diameter mandrel
- MMS-425 Primer – 1/4" diameter mandrel
- Electro-Prime – 1/2" diameter mandrel.

Verify that the primer shows satisfactory adhesion with no flaking or cracking. Cracks occurring at either end and extending no more than 1/4 inch shall be disregarded.

MANDREL BEND (-70°F): Place the panel in a cold box maintained at -70° ± 5°F for 5 hours. Immediately (within seconds) after exposure, bend the panel rapidly over a 1-inch diameter mandrel which has been conditioned at the same temperature. Verify that the primer shows no loss of adhesion or other evidence of film failure.

All panels passed all of the testing performed. The process consisting of Sealing Steps II and III of the Sanchem-CC process, therefore, was determined to be equivalent to the current chromate conversion coating process in regard to the tests which were performed. This performance, then, provides further confidence in the suitability of Sealing Steps II and III of the Sanchem-CC process as a viable production process.

APPENDIX E

PROCESS SPECIFICATION

TREATMENT OF IVD ALUMINUM-COATED METAL PARTS USING A NON-CHROMATED CONVERSION COATING

1.0 APPLICATION

1.1 This Specification defines the requirements and procedures for applying a non-chromated conversion coating to IVD aluminum-coated metal parts using an immersion process.

1.2 IVD aluminum-coated alloy steel parts treated per this Specification will meet the corrosion resistance requirements of MIL-C-83488, Classes 1, 2, and 3, Type II.

2.0 APPLICABLE DOCUMENTS

2.1 MILITARY SPECIFICATIONS

2.1.1 MIL-S-18729 – Steel Plate, Sheet, and Strip, Alloy 4130, Aircraft Quality

2.1.2 MIL-C-81706 – Chemical Conversion Materials for Coating Aluminum and Aluminum alloys

2.1.3 MIL-C-83488 – Coating, Aluminum, High Purity

2.2 FEDERAL STANDARDS

2.2.1 FED-STD-141 – Paint, Varnish, Lacquer and Related Materials; Methods of Inspection, Sampling and Testing

2.3 ASTM SPECIFICATIONS

2.3.1 ASTM B117 – Standard Method of Salt Spray (Fog) Testing

3.0 MATERIALS AND/OR SOLUTIONS

3.1 PROCESSING SOLUTIONS

3.1.1 SAFEGARD 3000, Sanchem, Inc., Chicago, IL (potassium permanganate solution also referred to as Sealing Step II of the Sanchem-CC process)

3.1.2 SAFEGARD 4000, Sanchem, Inc., Chicago, IL (potassium silicate solution also referred to as Sealing Step III of the Sanchem-CC process)

3.2 CHEMICALS FOR SOLUTION MAINTENANCE

3.2.1 Fifty Percent Sodium Hydroxide

3.2.2 Nitric Acid, 42° Baume per O-N-350

3.2.3 Sulfuric Acid, Reagent Grade

3.2.4 Iron (II) Sulfate Heptahydrate

4.0 EQUIPMENT

4.1 SAFEGARD 3000

4.1.1 304 Stainless Steel Tank, Mild Steel Tank Lined with Polypropylene or Koroseal, or Polypropylene Tank

4.1.2 The tank shall have heating provisions for maintaining the solution at 140°-150°F.

4.1.3 The tank shall have provisions for mild agitation of the solution. Pump type agitation as opposed to air sparging is adequate.

4.2 SAFEGARD 4000

4.2.1 Stainless Steel, Mild Steel, or Polypropylene Tank

4.2.2 The tank shall have heating provisions for maintaining the solution at 200°-212°F. Heating by a steam coil is preferred.

4.2.3 The tank shall have provisions for air sparge type or equivalent agitation.

4.2.4 The tank shall have an appropriate cover which can be placed over the tank when it is not in use.

4.3 RINSE TANKS

4.3.1 Room Temperature Deionized Water Immersion Rinse Tanks

NOTE: Two tanks will be required. One for rinsing after processing in the SAFEGARD 3000 solution and one for rinsing after processing in the SAFEGARD 4000 solution.

4.3.2 Both tanks shall have provisions for continuous overflow and air sparge type agitation.

4.4 DRYING OVEN

4.4.1 An open top type oven operated at 140°F maximum may be used as an aid in drying parts. Use of such an oven is not mandatory.

4.5 RACKS OR BASKETS

4.5.1 Stainless Steel or Polypropylene Racks or Baskets

5.0 REQUIREMENTS

5.1 IVD ALUMINUM COATING ON PARTS TO BE TREATED

5.1.1 The IVD aluminum coating on parts to be treated per this specification shall conform to MIL-C-83488, Classes 1, 2, or 3.

5.2 SAFEGARD 3000

5.2.1 The SAFEGARD 3000 solution shall be maintained at 140°-150°F and a pH of 6.0-7.0 during processing.

NOTE: The temperature of the solution may be reduced to 100°F over weekends and holidays.

5.2.2 The permanganate ion concentration shall be maintained at a level equivalent to 2.8-4.8 mL of titer.

5.2.3 The SAFEGARD 3000 solution is furnished by Sanchem, Inc. as a concentrate. For solution make-up, 1 gallon of concentrate shall be added to each 9 gallons of deionized water.

NOTE: After make-up of the solution, check the pH. If it is not within the required range, add nitric acid or fifty percent sodium hydroxide as required.

5.3 SAFEGARD 4000

5.3.1 The SAFEGARD 4000 solution shall be maintained at 200°-212°F and a pH of 11.5-12.0.

NOTE: The temperature of the solution may be reduced to 140°F over weekends and holidays.

5.3.2 The SAFEGARD 4000 is furnished by Sanchem, Inc. as a concentrate. For solution make-up, 0.88 gallon of concentrate shall be added to each 10 gallons of deionized water.

NOTE: After make-up of the solution, check the pH. If it is below the required range, add fifty percent sodium hydroxide, as required, to bring it within range.

5.4 SET-UP OF NEW PROCESSING LINE

5.4.1 The pH and permanganate ion concentration of the SAFEGARD 3000 solution shall be checked daily. After one month of processing, the pH and permanganate ion concentration of the solution shall be checked as determine appropriate, but at least once a week. Check the permanganate ion concentration per Paragraph 7.1.3.

5.4.2 The pH of the SAFEGARD 4000 solution shall be checked daily. After one month of processing, the pH of the solution shall be checked as determined appropriate, but at least once a week.

5.5 PROCESSING OF PARTS

5.5.1 Metal parts shall be arranged in racks or baskets with minimum contact between the parts.

5.6 SALT SPRAY PERFORMANCE

5.6.1 The test panel material shall be AISI 4130 steel, 4" X 6" X 0.040-0.060" thick, conforming to MIL-S-18729. The test panels shall be IVD aluminum coated per MIL-C-83488, Class 2.

5.6.2 Initially, when a new processing line is set-up, salt spray testing shall be performed on five test panels processed every other day for the first month of processing. Salt spray testing shall be in accordance with ASTM B117 and shall meet the 504 hour corrosion resistance requirement of MIL-C-83488.

5.6.3 After the required testing of 5.6.2, salt spray test panels shall be prepared and tested at the frequency determined appropriate.

5.7 PRIMER ADHESION

5.7.1 Test panels shall conform to the requirements of 5.6.1.

5.7.2 Initially, when a new processing line is set up, primer adhesion testing shall be performed on three test panels processed every other day for the first month of processing. The primer used shall be one which is currently in production use.

5.7.3 Initially, a dry, tape adhesion test shall be performed on each panel. Next, a scribed, tape adhesion test shall be performed on each panel per Method 6301 of Federal Test Method Standard 141. Testing per the Federal Standard will involve immersion of the test panels in distilled water for 24 hours followed by scribing and then tape testing. There shall be no intercoat separation between the primer and conversion coating.

5.7.4 After the required testing of 5.7.2, primer adhesion test panels shall be prepared and tested at the frequency determined appropriate.

5.8 CONTACT ELECTRICAL RESISTANCE

5.8.1 Test panels shall conform to the requirements of 5.6.1.

5.8.2 Initially, when a new processing line is set-up, contact electrical resistance testing shall be performed on three test panels processed every other day for the first month of processing.

5.8.3 The test method and equipment for contact electrical resistance testing shall be in accordance with MIL-C-81706.

5.8.4 After the required testing of 5.8.2, contact electrical resistance test panels shall be prepared and tested at the frequency determined appropriate.

6.0 PROCEDURES

6.1 Immerse the parts in the SAFEGARD 3000 solution at 140°-150°F for 3 minutes.

6.2 Immediately after removal from the SAFEGARD 3000 solution, immersion rinse the parts in room temperature deionized water for 3 minutes.

6.3 Immediately after deionized water rinsing, immerse the parts in the SAFEGARD 4000 solution at 200°-212°F for 1 minute.

6.4 Immediately after removal from the SAFEGARD 4000 solution, immersion rinse the parts in room temperature deionized water for 3 minutes.

6.5 Air dry the parts, or oven dry the parts in an open-top dryer.

7.0 QUALITY ASSURANCE PROVISIONS

7.1 SAFEGARD 3000

7.1.1 Add deionized water daily as required to maintain the tank operating level.

7.1.2 Check the pH of the solution per the frequency requirements of 5.4.1. Add nitric acid or 50 percent sodium hydroxide as required to maintain the required range of 6.0-7.0.

7.1.3 Check the concentration of the permanganate ion per the frequency requirements of 5.4.1. Perform the determination as follows: Add one gram of the used SAFEGARD 3000 solution to 100 ml of deionized water containing 1.0 gram of sulfuric acid and titrate immediately with a solution of 5.0 g/L iron (II) sulfate heptahydrate, containing 0.5 gram sulfuric acid, to a clear solution. A fresh solution will require 4.6±0.2 mL while a spent solution will require 3.0±0.2 mL. To recharge the spent SAFEGARD 3000 solution, add one gallon of concentrate to each 29 gallons of spent solution. After recharging, add nitric acid or fifty percent sodium hydroxide, as required, to maintain the desired pH range of 6.0-7.0.

7.2 SAFEGARD 4000

7.2.1 Add deionized water daily as required to maintain the tank operating level.

7.2.2 Check the pH of the solution per the requirements of 5.4.2. Add fifty percent potassium hydroxide as required to maintain the required pH range of 11.5-12.0.

7.3 PROCESS CONTROL SPECIMENS

7.3.1 Verify conformance to all requirements of 5.6, 5.7 and 5.8 for process control specimens.

8.0 DISPOSAL OF SPENT SOLUTIONS

8.1 SAFEGARD 3000

8.1.1 SAFEGARD 3000 contains no toxic chemicals and may be discharged at its normal pH operating range. If color is a consideration, however, the solution may be decolorized as follows: First, lower the pH to 2.0 or lower. Next, add sodium sulfite solution to discharge the color. Finally, after decolorization, raise the pH to a level acceptable for discharge.

NOTE: An iron (II) sulfate solution may also be used to discharge the color using the above procedure. Use of this solution, however, will result in a precipitate of iron and manganese oxides.

8.2 SAFEGARD 4000

8.2.1 SAFEGARD 4000 contains no toxic chemicals. It is only necessary to lower the pH of the spent solution to 9.0 or lower before discharge.