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# Duct Protection Coating Concept Development and Test

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This report has been reviewed and approved.



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FOR THE COMMANDER



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**DUCT PROTECTION COATING CONCEPT  
DEVELOPMENT AND TEST**

**CONTRACT: F40600-89-C-0002**

**FINAL TECHNICAL REPORT (CDRL A002)**

15 February 1992

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## PREFACE

This Final Technical Report (CDRL Item A002) is submitted to the Arnold Engineering Development Center (AEDC) as the final requirement for Contract F40600-89-C-0002. This report documents the technical activities and results performed and obtained by Mission Research Corporation (MRC) during this Small Business Innovation Research Phase II program. Contact Mr. C. David Newlander for any additional information.

This report is submitted in three parts. This part contains the body of the technical report and documents the activities performed and results obtained. The second part contains the test data sheets which document the history of each article fabricated, environmentally conditioned, and tested during the program. The third part is a set of photographs which provides a visual documentation of the program activities. These are referenced from the technical text as required.

The following persons are responsible for the technical success achieved under this contract:

Mission Research Corporation: Albert H. Koivu  
Mark N. West

Consultants: Dr. Kenneth G. Mayhan (Applied Research Labs)  
Dr. Ralph Kafesjian (Applied Research Labs)  
Dr. Adrian MacNab (Failure Analysis Associates)

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# SECTION I

## EXECUTIVE SUMMARY

### 1.1 Program Objectives and Scope

The overall objective of the program was to develop and test a surface cleaning/preparation and corrosion resistant protective polymer coating for the interior of the AEDC Environmental Test Facility (ETF) ferrous metal air supply ducts. The coating was required to withstand operating temperatures ranging from -120°F to +600°F, high air mass flow (up to 1100 ft/sec), high internal pressures (up to 150 psi), and high humidity (up to 100%). In addition, besides the survival against this hostile environment, a number of other desired characteristics and constraints were specified. In particular, the types of cleaning techniques acceptable to the AEDC were basically limited to chemical approaches. Typically, sand or abrasive blasting is used to produce corrosion-free, clean surfaces for coating steel substrates. These particular techniques are strongly discouraged and basically "outlawed" at the AEDC facilities. Water blasting is an acceptable technique, but requires a high pressure water stream, expensive equipment, and considerable clean up. Other mechanical techniques such as wire brushing are inefficient and would not produce the required degree of surface cleanliness. Additional desired coating characteristics included:

- o A well understood failure mode based on a gradual wear of the coating rather than a catastrophic failure which would release large masses of material into the air flow.
- o Ease of application with low tolerances to parameters such as thickness, temperature, and humidity (i.e., a system which required laboratory-type conditions during application would not be acceptable).
- o A short-time, room temperature cure cycle, or at a minimum, a cure which could be affected by utilizing the duct heating system.
- o Ease of maintenance, repair, and ultimately removal if necessary.
- o Safe and environmentally acceptable.

This report documents the second phase of the Small Business Innovation Research (SBIR) contract. The results of the Phase I contract are documented in Reference 1. During the Phase I contract, MRC evaluated a number of concepts and techniques for providing the required protection system. A number of these went beyond the development of a polymer coating or paint system. The Phase II contract was constrained to utilize polymer coatings. However, a number of these other ideas are summarized in Section 2.11 for completeness.

The overall SBIR program is designed to provide "seed" money to small businesses for the development of technologies which can be transferred to commercial activities and products. Two products were developed, optimized, and tested under this contract which appear to have significant commercial application, and MRC has prepared a preliminary plan to commercialize and market these products. This plan is summarized in Section 3.0. These activities will be performed in the Phase III Program and will be accomplished without government funding. This program is an excellent example of how the SBIR program and the Air Force can support small research and development-oriented businesses. The overall plan for the three phase SBIR program is presented in Figure 1.

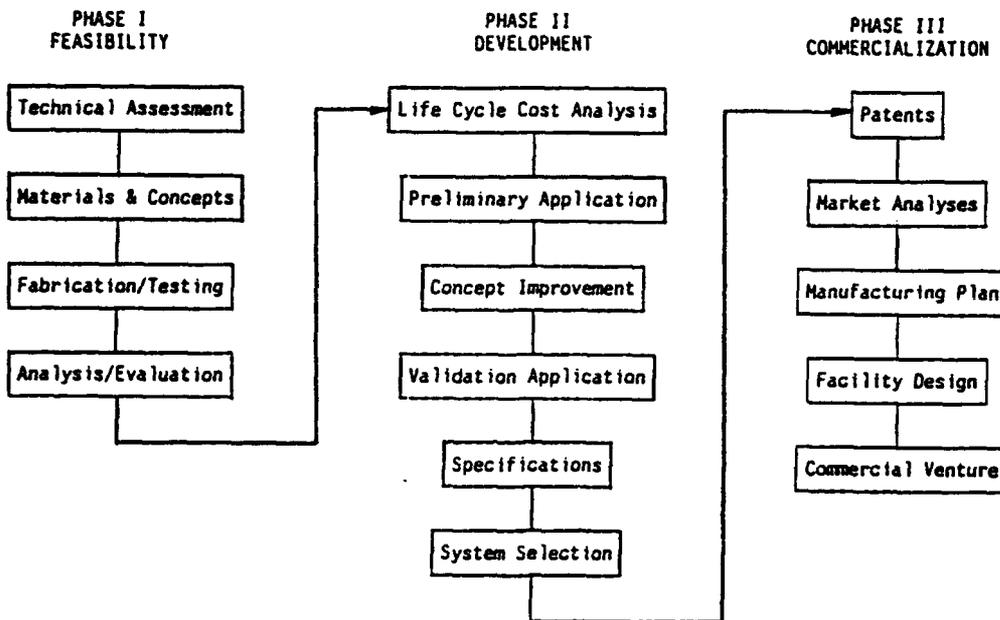


Figure 1. Schematic of Three Phase Program

## 1.2 Program Approach

In order to achieve the overall program objectives, a number of specific goals were established. The following list briefly describes those goals and milestones and is a summary of the overall program technical approach.

- o Develop methods for producing corroded steel panels in the laboratory which simulate the conditions found in the ducts.
- o Define and develop optimized methods of steel duct cleaning and surface preparation which are acceptable to the AEDC and provide the required degree of cleanliness and anchorage.
- o Develop an appropriate coating system which protects the steel from corrosion, adheres to the prepared surface, and resists erosion and high/low temperature cycling while maintaining the required flexibility.
- o Define and use laboratory techniques which adequately test the coating system for adhesion, aging, erosion/ablation, temperature/moisture resistance, and corrosion protection.
- o Procure accelerated aging equipment for conducting tests which simulate the AEDC duct temperatures, humidities, and static stresses and cycling of these variables.

- o Perform preliminary and validation testing of the developed concepts in the AEDC ducts.
- o Develop coating solutions for specific problem areas such as expansion joints, edges, bends, and interfaces.
- o Determine acceptable techniques and methods for applying the developed coating systems on the duct surfaces.
- o Determine maintenance procedures for the coating concepts.
- o Develop repair kits and procedures for the developed coating concepts.
- o Develop detailed specifications for the coating systems including surface cleaning and preparation, coating materials, application, processing, maintenance, and repair.

### 1.3 Results and Conclusions

The following summarizes the activities performed by MRC, the results generated by those activities, and the overall conclusions drawn from the results:

- o Laboratory procedures for producing corroded steel specimens were developed using the steel alloys present in the AEDC ducts. It was intended that the corrosion generated on these panels would simulate and duplicate the duct wall corrosion conditions, and could therefore be used to directly evaluate surface preparation and cleaning techniques. However, cleaning agents which successfully performed on these panels were not capable of efficiently removing the corrosion on the walls of the B-Header. Based upon these tests in the ducts, we conclude that the B-Header corrosion is significantly different than that generated in our laboratory. It is not clear that AEDC duct-like corrosion can be produced in any laboratory in a timely manner. The conditions of the cycled high temperatures and humidities over many years may have produced a near unique corrosion layer in the duct system. If so, then attempts to develop a cleaning technique cannot succeed without either continual access to the duct interior for direct testing or access to materials exposed in the ducts for long periods of time (i.e., removal of a section of the ducts for laboratory test and evaluation).

**We conclude that duct-like corrosion may be very difficult to efficiently generate in the laboratory for cleaner evaluation.**

- o Laboratory procedures were developed for producing conditioning tests which simulated the temperature and humidity environments in the ducts. These procedures included subjecting the test specimens to a heating/cooling cycle with temperatures ranging from 600°F to -100°F with rates ranging from 6°F/minute to 17°F/minute. These rates were evaluated by AEDC personnel as being representative of duct conditions. After temperature cycling for a day, the specimens were subjected to high humidity (80% RH) and temperatures (120°F) for up to twelve hours. This conditioning was repeated until the coating on the panels failed or the testing was completed. In some cases, this cycling continued over periods of three to six months.

o A wide variety of surface preparation techniques were evaluated including mechanical (wire brush, glass bead blast, and abrasive/water blasting) and chemical (solvent wipe, hydrochloric acid, commercial cleaners, and specially prepared cleaners). This evaluation was performed on the laboratory-generated corroded steel panels. The results of this evaluation indicated that wire brushing, solvent wipe, and commercial cleaners do not provide a surface which is clean enough for application of a polymer coating. Glass bead and abrasive blasting techniques are not acceptable to the AEDC. Water blasting can provide an acceptable surface, but is too slow and expensive for actual use in the duct system. Hydrochloric acid can provide an acceptable surface but generates toxic waste and is probably environmentally unacceptable. The cleaner developed by MRC is ineffective in removing the B-Header corrosion.

**We conclude that there is no one acceptable method for cleaning the duct surfaces for application of a polymer coating or paint.**

o A "low toxicity" chemical cleaning process utilizing mild organic acids known as (MORC) was developed which in the laboratory provided an acceptably clean steel surface for coating. This cleaning material was modified into a gel appropriate for application to vertical and "over-the-head" surfaces. The cleaner has proved to be very effective in all of the laboratory studies and therefore has promise as a commercial product. The performance of the cleaner was shown to be sensitive to temperature. The cleaner was ineffective in removing the B-Header corrosion in tests conducted by both MRC and the AEDC and therefore cannot be used by itself to solve the AEDC problem. A combined mechanical/cleaner approach might be successful for cleaning the very tough areas. This combination might include water blasting or mechanically sanding the surfaces followed by several applications of the cleaning compound.

**We conclude that the MRC cleaner has commercial potential**

o A wide range of available high temperature paints and coating materials were evaluated. These included both permanent as well as "replenishable" coatings including both commercial products and MRC-blended materials. Based upon numerous accelerated aging tests, the MRC-developed aluminum flake-filled silicone resin coating (known as MARB) proved to be capable of withstanding the simulated harsh duct environmental conditions without significant degradation. Laboratory testing indicated that this coating is superior to other commercially available paints. This is probably due to the high solids content of the coating, the use of aluminum flake rather than aluminum paste, and the expressed intent of blending a material for the wide temperature extremes seen in the duct system. The adhesion of the coating to the ferrous surfaces does not appear to be sensitive to the cleaning technique used as long as all of the corrosion is removed. Tests conducted on coated panels cleaned with hydrochloric acid, organic acids, water blasting, and glass bead blasting did not show significant differences in performance.

**We conclude that this coating would work in the AEDC duct system if adequate cleaning could be performed, and that this coating material has commercial potential as a high quality, high performance paint.**

- o Four test patch applications have been attempted in the B-Header. The first used a preliminary formulation of the organic acid cleaner. Insufficient time was available for a true evaluation of the cleaner. The poorly-prepared test area was coated with a brushed application of the developed aluminum-silicone paint. At this date, the coating is failing as expected due to the growth of corrosion under the coating. This is attributed to both the poor initial surface condition and the thin coating areas due to the brush stroke texture. A second attempt at using the organic acid cleaner was performed using a more concentrated formulation and a gelling ingredient to reduce moisture loss. This was also unsuccessful due to limited application time and potentially by an underlying layer of previously applied paint. The third application was an attempt to validate the coating performance. An area of the duct wall surface was cleaned using a belt sander and an application of organic acid cleaner. This was then sprayed with the MRC aluminum-silicone paint. Recent evaluation of this test patch by AEDC personnel indicates that the coating has an unusual surface texture, but there is no sign of corrosion growth under the surface. However, the duct system has not been used in a severe environment mode and the coating capability has not been severely tested. The surface texture is probably the result of aluminum flake separation and surfacing, and the lack of a complete cure of the coating (which requires temperatures on the order of 490°F for complete cure). The results of the final test patch assessment are encouraging and the patch should continue to be evaluated at periodic intervals. A final cleaner evaluation was performed by the AEDC which assessed if long application periods (8 to 16 hours) would increase its performance to an acceptable level. The cleaner was ineffective in removing the B-Header corrosion over those long periods of application.

#### 1.4 Overall Program Summary and Recommendations

- o A coating, superior to those commercially available, has been developed which can withstand temperature extremes and high humidity. This coating has commercial potential.
- o A rust cleaner with strong commercial applications has been developed. Although this cleaner has been successful in removing all corrosion tested in the laboratory, it was unsuccessful in removing the B-Header corrosion.
- o The B-Header corrosion may be a unique form of corrosion formed by the unusual environmental conditions within the duct system over a substantial period of time.
- o The development of a technique for cleaning the B-Header cannot be developed without continual access to the duct interior or access to materials exposed in the ducts for long periods of time.
- o Water blasting does not appear to be a viable cleaning technique for the ducts.
- o A polymer coating material or paint will only be effective if undesirable cleaning techniques (i.e., hydrochloric acid or abrasive blasting) are utilized.
- o Other protection techniques such as cladding with aluminum or stainless steel are probably more achievable than the use of a coating or paint, but will still require the cleaning of some portions of the duct wall surfaces..

## SECTION II

### DETAILED TECHNICAL RESULTS

#### 2.1 Documentation

The technical results of this program have been well documented in Technical Progress/Status Reports (References 2 through 8), a Year 1 Interim Technical Report (Reference 9), and four Program Reviews (References 10 through 13). These documents contains the details of the program activities and provide a chronological history of the program. The following paragraphs provide a summary of the activities, the results, and any conclusions.

#### 2.2 Description of Ducts, Materials, and Environments

The environment test facility (ETF) at the Arnold Engineering Development Center (AEDC) contains a series of ducts that are used to channel temperature-conditioned air to jet engine test cells so that the engines can be characterized to a wide range of operating conditions. The complete ducting system is about three miles long and was constructed around 1951 using three low-carbon steel alloys, namely A516-70, A285, and A203. A516-70 and A285 are low carbon steels that have little restriction on the chemistry of the alloys. The A203 is a nickel steel which has good low temperature properties and which corrodes less than non-nickel alloys. The A203 has proven to be very difficult to clean and its mill scale is very adhering. Drawings of the duct work were obtained from the AEDC which provided diameters and peak operating temperatures and pressures. AEDC personnel also indicated on the drawings, what sections of the ducts required coating. From these, it was determined that approximately 38,000 square feet of surface area would require coating. Table 1 contains a description of the various duct regions and the operating temperature ranges. From this table, the ducts were subdivided into regions of similar environment as shown in Table 2. The intent of this subdivision was to evaluate the potential for using different coatings in regions of similar environmental extremes if an overall solution could not be developed. It is now believed that a single coating system could be used on all of the required duct locations if proper cleaning and curing could be performed.

Table 1. Surface Area Estimate

Section	Location	Temp Range (°F)	Diameter (ft)	Length (ft)	Area (ft <sup>2</sup> )
1	Extension, left	-10 to 100	3	208	1960
2	Extension, left	-10 to 300	5	150	2356
3	Connector, duct	-20 to 400	5	167	2623
4	Extension, duct	-50 to 400	5	20	314
5	Extension, duct	-50 to 400	7	20	440
6	Crossover, duct	-100 to 475	4	110	1382
7	Header, "A"	-120 to 575	6	248	4675
8	Spheres, 4	-120 to 575	20		5027
9	Extensions	-120 to 575	4	208	2614
10	Header, "B"	-20 to 650	6	256	4825
11	Spheres, 6	-20 to 650	20		7540
12	Crossover	-20 to 650	3.5	117	1286
13	Concentric, duct	-50 to 800	7	100	2199
14	Concentric, duct	-65 to 800	4	48	603
				Total	37,844

Table 2. Subdivision Versus Environment Versus Surface Area

Sections*	Environment (°F)	Surface Area (ft <sup>2</sup> )
1, 2, 3, 4, 5	-50 to +400	7,693
6, 7, 8, 9	-120 to +575	13,701
10, 11, 12	-20 to +650	13,651
13, 14	-60 to +800	2,803

\*As defined in Table 1

### 2.3 Selection/Description of Laboratory Testing Techniques

The two most important characteristics required for the duct coating system are substrate adhesion and abrasion resistance. The coatings were tested for adhesion using a 1000 psi Elcometer tester (See Photograph 1) in accordance with ASTM D4541-85. In many cases, coating/substrate adhesion and coating cohesion exceeded the 1000 psi maximum which could be tested using this equipment. AEDC personnel indicated that they believed that an adhesion strength of 200 psi was sufficient. Scrape adhesion/scratch hardness was measured using a balance beam scraper (See Photograph 2) in accordance with ASTM D2197-86.

A testing methodology was developed which consisted of the following:

1. Completely clean a steel panel with hydrochloric acid (HCl).
2. Allow the panel to corrode by exposing to high humidity, heat, and, in some cases, salt spray.
3. Clean and prepare the corroded surface and document the technique.
4. Apply inhibitors, primers, and coatings and cure as desired.
5. Subject the coated panels to high (>600°F) and low (-100°F) temperatures for a day.
6. Subject the coated panels to high (80% RH) humidities and temperatures (120°F) overnight.
7. Bond on adhesion test dollies and cure overnight.
8. Perform adhesion tests and record data and failure mode.
9. Repeat steps 5 through 9 as required.

The high and low temperatures were achieved using an environmental chamber procured from Applied Test Systems (see Photograph 3). A used Blue M humidity chamber (See Photograph 4) was used to provide corroding environments. The heating/cooling cycle used is shown in Figure 2. This simulates the rise times actually achieved at the ETF ducts. A special form was

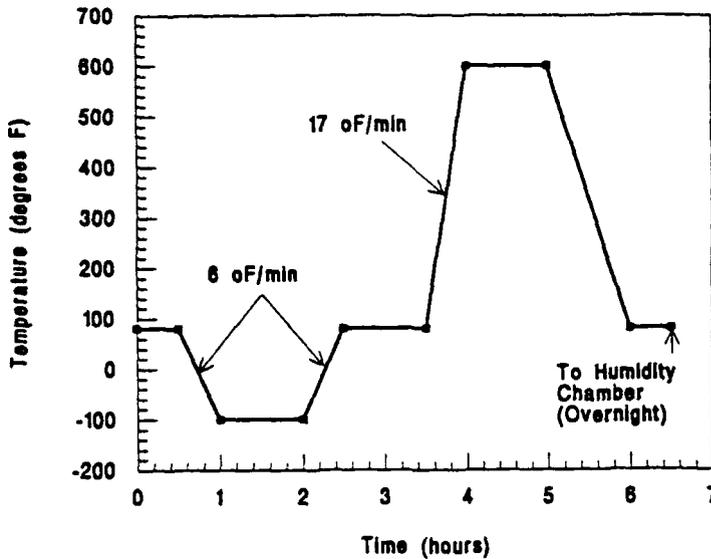


Figure 2. Heating/Cooling Cycle Used in Test Chamber

designed which provides detailed documentation of how each test specimen was fabricated (including preparation, material formulations, curing), conditioned (temperatures, time at temperature, humidity conditions, and equipment used), and tested (when, at what locations, and measured results/ failure modes). An example of such a form is shown in Figure 3.

**NRC Fabrication/Conditioning/Test Form 90-1**

Set: 18      Sample: 9001      Comments:

Materials: 50% HCl Acid, Sodium Bicarbonate, MEK, NRC MARB (5.0 SR240, 5.0 Toluene, 10.0 DC6-2230, 10.0 Xylene, 7.4 MD 7100 Al, 0.15 ZnO)

Equip Definitions: O - Oven; C - Heat/Cooling Chamber; H - Humidity Chamber; A - Adhesion Tester

Date	Action	Equip	Results/Comments
06/08/90	Acid Clean		Remove Mill Scale
06/08/90	Dip in NaHCO <sub>3</sub>		Neutralize, Dry
06/12/90	Wipe with MEK		
06/12/90	Coat with MARB	Brush	
06/12/90	1 hr @ 487	O	Cure
06/13/90	3.5 hr @ 140	H	Humid
06/14/90	Test (Location 1)	A	800 psi (97% Epoxy, 3% Adhesion to Rusty Substrate)
06/14/90	4 hr @ 600	C	
06/15/90	30 min @ 150	H	Humid
06/15/90	30 min @ 15	H	
06/15/90	30 min @ 150	H	Humid
06/18/90	Test (Location 2)	A	600 psi (85% Adhesion, 15% Epoxy)
06/18/90	3 hr @ 600	O	

Figure 3. Example of Completed Specimen Documentation Form

## 2.4 Hardware Selection and Procurement

A number of environmental test chambers were examined and evaluated, and bids from selected suppliers were obtained and reviewed. An Applied Test Systems (ATS) Series 3720 heating and cooling chamber was selected and procured. The chamber has a heavy-gauge stainless steel exterior and interior and is a split box type which could interface with our Instron tensile tester if required. The equipment has a wide temperature range of -340 to 800°F. The cooling is provided by a bottle of liquid nitrogen. The Series 2010 heat/cool controller allows for 8 ramp or 8 soak intervals. The chamber can reach maximum temperature in about 30 minutes which is consistent with the AEDC conditions. This temperature chamber is not available with a controlled humidity capability. It was decided to purchase a separate controlled humidity chamber. Specimens would be temperature conditioned in the heating/cooling chamber and then moved into the humidity chamber. This would simulate the actual duct conditions, where the ducts are first exposed to high or low temperature air flow, allowed to return to ambient temperature, and exposed to the natural Tennessee humidity. A used Blue M, closed-system, humidity chamber was procured from Harold Johns, Inc. It has a six cubic foot capacity with a temperature range of 0 to 200°F with 5 to 98% relative humidity. It has a manual set point control for wet and dry bulbs. The auxiliary de-mineralized water system was also procured and installed.

## 2.5 Steel Substrate Analyses and Procurement

The ducts are constructed of three different steel alloys: A516-86 grade 70, A285 grade C, and A203 grade E. These are all low carbon steels. The A203 is a nickel steel and contains between 3 and 4% nickel. Table 3 presents the elemental composition and mechanical properties of these alloys. The A516 and A285 are typical steel alloys used for welded pressure vessels and boilers. The A203 has better low temperature properties and has higher notch toughness. There is little difference in the corrosion behavior exhibited by these materials although the A203 should have a lower corrosion rate than the other two materials. The presence of copper, nickel, chromium, and phosphorus should improve atmospheric corrosion resistance. Forty square feet of the A516 alloy was purchased from Tell Steel and sheared into 6" by 6" squares (see Photograph 5). The thickness was 0.275". Twenty square feet each of the A285 and A203 alloys were also

Table 3. Characteristics of Procured Steel Plates

Composition (%)	A203 grade E	A285 grade C	A516 grade 70
Iron	95.0	97.0	98.0
Carbon	0.20	0.28	0.27
Nickel	3.18 - 3.82	0.0	0.0
Copper	0.0	0.18 - 0.37	0.0
Manganese	0.78	0.98	0.79 - 1.30
Phosphorous	0.035	0.035	0.035
Sulfur	0.040	0.040	0.040
Silicon	0.13 - 0.45	0.0	0.13 - 0.45
<b>Properties</b>			
Tensile Strength (ksi)	70 - 90	55 - 75	70 - 90
Yield (ksi)	40	30	38
Elongation (%)	21	27	21

purchased from Tell Steel and sheared into 6" by 6" plates. The A285 was 0.255" thick and the A203 was 0.265" thick. In addition to these steel plates, steel "Q-panels" were purchased for use as substrates for the coating development activities. These panels provided a clean ferrous surface for application of preliminary coating concepts. These types of substrates are commonly used for coating evaluation studies. The description of these "Q-panels" is presented in Table 4.

Table 4. Q-Panels Procured by MRC

Designation	Thickness (in)	Size	Finish	Temper
QD-39	0.020	3" x 9"	D609 type III	A366 (1/4 Hard)
S-39	0.030	3" x 9"	D609 type II	A366 (1/4 Hard)
RS-14	0.062	1" x 4"	Ground	A366 (1/2 Hard)

Note: ASTM D609: type I - grit blast rolled  
           : type II - flat polished on one side  
           : type III - smooth rolled

## 2.6 Surface Preparation Studies and Results

Coating adhesion is maximized when the coating is applied to a clean surface. Both mechanical and chemical cleaning techniques were evaluated including:

- o Detergent wash
- o Solvent wipe
- o Wire Brush
- o Acid clean (Hydrochloric and Phosphoric)
- o Blasting (Water, glass bead, ice, and abrasive)
- o Commercial cleaners
- o Conversion coatings
- o Non-commercial cleaners

The initial cleaning technique evaluations used the A516-70 panels as received from the steel distributor. The panels were covered with a wide range of corrosion - from a light layer of rust to heavy mill scale, and even to other contamination such as paint. The methodology was to "clean" the panels using the available techniques and then to coat the panels with candidate paints. The panels were then exposed to cyclic high and low temperatures and high humidity. Adhesion tests were then conducted and the coating/substrate strength evaluated. There was considerable scatter in the results, and an improved procedure was adopted in order to more closely simulate the corrosion conditions in the ducts. All tests panels numbered 8000 or above were prepared using the following procedure:

- o Completely clean the panel using hydrochloric acid. This procedure removes all mill scale and other contaminants.
- o Wash and neutralize the panels with sodium bicarbonate.

- o Subject the panels to high humidity and heating to generate a rusty surface.
- o In some cases, a salt water spray was used to generate a very heavy rust layer and to induce pitting.

These rusted panels were then subjected to controlled cleaning procedures. A description of the techniques and the advantages/disadvantages is given below:

- o Detergent wash: The panels were washed with commercial detergents until no red color was present. The detergent removes grease, oils, and loose debris, but does not provide a satisfactory surface for coating.
- o Solvent wipe: The panels were wiped with MEK or Xylene-loaded paper towels until no red color was present on the paper towels. The solvent removes grease, oils, and loose debris, but does not provide a satisfactory surface for coating.
- o Wire Brush: The panels were wire brushed using an electric grinding wheel motor with a wire wheel. The surfaces were then further cleaned using a solvent wipe to remove any loose debris. Wire brushing removes a high percentage of the corrosion, but does not adequately remove any rust inside the pitting and texture. Wire brushing does not generate an anchor pattern and can possibly enhance the corrosion problem due to dissimilar materials. Wire brushing is also very labor intensive.
- o Acid clean: A 50% solution of hydrochloric acid can be used to thoroughly clean the steel surfaces of rust and mill scale. It does leave behind a residue which must be removed before coating. The residue left behind by phosphoric acid was very difficult to remove and hydrochloric acid appears to be a better selection. Acid does not remove oils or grease very well, and therefore, a solvent or detergent cleaning was used before the acid cleaning. After using the acid, the surface needs to be sprayed with a mild base, such as a sodium bicarbonate/water solution. Although, acid could be used to perform the cleaning, it has several disadvantages - the workers would need to wear protective, acid proof clothing, adequate ventilation would need to be provided, and neutralization/clean-up/disposal of the used acid would be necessary.
- o Blasting: The AEDC has effectively "outlawed" blasting with abrasives which eliminates glass bead and particulate blasting from consideration. Water blasting was a strong contender as a cleaning technique. Unfortunately, water blasting requires a 20,000+ psi stream to obtain a white metal surface. These blasters cost over \$200,000 and would produce a considerable water disposal problem. Water blasting can be used to provide a clean surface, but will not produce an anchor pattern unless very high pressures are used. Additional discussion of water blasting techniques are contained in Section 2.8. Other innovative blasting techniques were investigated. Blasting with ice or other materials which are abrasive at room temperature, but which melt or evaporate at higher temperatures appeared to be a novel solution. However, in experiments, ice did not appear to be abrasive enough and materials with the correct mix of properties could not be located.
- o Commercial cleaners: Several commercially available cleaners (Naval Jelly, Kano X-Rust Liquid, and Cortec VSI-421) were evaluated. The cleaned panels were washed in a sodium bicarbonate solution. In general, these cleaners removed a significant fraction of the corrosion on the panels. However, they

were slow to perform, messy, and left a difficult-to-remove residue. The residue suggests that these cleaners contain phosphoric acid as their active ingredient.

- o **Conversion coats:** Several commercially available "conversion coat" materials such as Nybco Bar-B-Que Black and Neutra Rust were evaluated. The technique investigated involved a solvent wipe or wire brushing which was followed by an application of the conversion coat. After curing, the conversion coat (and converted corrosion) was then removed by wire brushing. This approach did not produce a suitably clean surface.
- o **Non-commercial cleaners:** Specially formulated, non-commercial cleaners were evaluated. These cleaners had initially been developed for cleaning corroded, antique firearms. Standard hydrochloric or phosphoric acid-based cleaners were too strong for antiques and attacked the base metals as they dissolved the corrosion. These cleaners are unique in that they were designed to dissolve the FeO layer present between the stronger forms of corrosion ( $\text{Fe}_2\text{O}_3$ , and  $\text{Fe}_3\text{O}_4$ ) and the base steel. This allows the stronger forms of corrosion to fall off or be easily washed away. The main ingredients of the cleaners are a series of organic acids. Formula modifications have been evaluated which investigated the effects of small quantities of hydrochloric or phosphoric acids, gelling agents, and skin-forming additives. Several series of evaluations were performed to optimize the cleaners as a function of degree of corrosion. The best cleaners could easily clean most of the corroded surface within an hour of application. Some difficulties have been encountered in cleaning the pits, since the cleaner works best when a clear path to the FeO exists. This path is minimal on very heavily corroded surfaces or on surfaces with deep pitting. Good performance was obtained in the laboratory by applying a coat of the cleaner, mechanically "swirling" the cleaner around every ten minutes with a brush, finally scraping the cleaner off after an hour using a squeegee, and reapplying until a clean surface was obtained. As will be shown in the next section, the silicone resin-based paints developed under this program, work extremely well in conjunction with surfaces prepared using these cleaners. Although this MORC cleaner was mostly ineffective in cleaning the B-Header corrosion, a final system has been optimized for commercial use. The features of this cleaning system will include:
  - o Water soluble
  - o Low toxicity - no hydrochloric or phosphoric acids
    - minimal protective clothing required
    - no breathing apparatus necessary
  - o Easy to apply - brush or spray
  - o Thick - gelling agents used to enhance attachment to vertical/overhead surfaces
  - o Continual action - skin maintains moisture content within the gel

## **2.6.1 Summary of the Development of Non-Commercial Cleaners.**

### **2.6.1.1 Initial Applications/Results:**

MRC consultants, Dr. Ken Mayhan and Dr. Ralph Kafesjian, have been developing ferrous corrosion cleaners for a number of years. This organic acid cleaner technology was investigated for possible use in the AEDC duct environment. An initial laboratory demonstration of a cleaner provided a clean surface on a laboratory-rusted panel after a five to ten minute application. A set of six panels were then cleaned and prepared for coating using the MARB and other advanced

coatings, and were then subjected to high and low temperatures and high humidity. The results were very impressive. Adhesion tests indicated strengths above 1000 psi after numerous environmental cycling. These results were presented at the 2nd Program Review Meeting and suggested that further investigation was warranted.

The results were so promising that a very preliminary duct patch application was performed which took advantage of an opening of the duct system for maintenance on 6 July 1990 which was one week after the 2nd Review Meeting held at the AEDC. During this duct opening, the preliminary cleaner was used to clean a patch in the "B"-Header. Due to a lack of time, only a very preliminary cleaner application could be applied. This failed to completely clean the surface. However, the laboratory results were so promising, that further investigations were undertaken in an attempt to strengthen and optimize the cleaner formulation.

#### 2.6.1.2 Cleaner Formulations/Evaluations #1:

Seventeen different cleaner variations were evaluated in terms of cleaning efficiency and residue formation. These formulations were applied to heavily corroded and pitted steel panels and observations made over time. Among the variables to be evaluated were phosphoric acid content, improved film forming characteristics, incorporation of shear thinning properties, and retardation of water evaporation. The results are presented in Table 5. After review of the results it appeared that the film formers used may be associating with the organic acid and interfering with its penetration into the rust film. It also appeared that formulations IV-01, V-01, VIII-01, and IX-01 had merit for further development.

Table 5. Evaluation of Cleaners #1

Designation	Characteristics	Rating	Comments
I-01	Organic + Phosphoric, Klucel	OK	Difficult to Remove
I-02	Organic, Klucel	OK	Too Thin
II-01	Organic + Phosphoric, PVP	No	Evaporates
II-02	Organic, PVP	No	Poor Cleaning
III-01	Organic + Phosphoric, PVA	No	Poor Cleaning
III-02	Organic, PVA	No	Poor Cleaning
IV-01	Organic + Phosphoric, PA	OK	Good Potential
IV-02	Organic, PA	Fair	Good Potential
V-01	Organic + Phosphoric, Aerosil	Best	Clean Tough Rust
V-02	Organic, Aerosil	No	Separated on Panel
VI-01	Organic + Phosphoric, PA, Klucel	Poor	Requires Shaking
VI-02	Organic + Phosphoric, PA, Klucel	Fair	Too Thin
VII-01	Organic + Phosphoric, Klucel, Cyanomer	No	Incompatible Formulation
VIII-01	Organic + Phosphoric, Klucel, Cab-O- Sil (Aerosil)	Good	Good Potential
IX-01	Organic + Phosphoric, Klucel, Hydrophobic Cab-O-Sil	Good	Good Potential, But Thin
X-01	Organic + Phosphoric, Klucel, Cab-O- Sil, Cyclobutane, IPA	OK	Too Thin
X-02	Organic, Klucel, Cab-O-Sil, Cyclobutane, IPA	OK	Did not Clean Pits

### 2.6.1.3 Cleaner Formulations/Evaluations #2:

A second set of cleaners was formulated based upon the results of the initial formulation testing. These cleaners contained two organic acids and Aerosil with and without small amounts of phosphoric acid. These cleaners removed most of the rust in two thirty minute treatments. The best cleaner was XII-04 which contained both organic acids, phosphoric acid, DMF, and Aerosil. A series of screening studies were then performed to evaluate:

- o Thixotropic agents compared to the baseline Aerosil 200.
- o Different organic acid ratios. An optimum ratio was established.
- o Non-ionic surfactants.
- o Mixtures of phosphoric and hydrochloric acids using formulation V-01 as the baseline.

Three new cleaners were formulated and evaluated by both the AEDC and MRC. The results are presented in Table 6. From these results a base formulation was decided upon. The ingredients are:

Ingredients for Baseline Cleaner (10/08/90): Saturated Organic Acid  
 Unsaturated Organic Acid  
 DHDKCB  
 Surfactant C  
 Formamide  
 De-ionized Water  
 Aerosil 200

Table 6. Evaluation of Cleaners #2

Designation	Characteristics	Rating	Comments
XVII-01	Organics + Phosphoric, Aerosil	OK	Cleans in 2, 1 hour-applications.
XVIII-01	Organics + Phosphoric + Hydrochloric, Aerosil	Best	
XIX-01	Organics + Phosphoric, COK Silica	Poor	

### 2.6.1.4 Cleaner Formulations/Evaluations #3:

These formulations and tests were performed to evaluate the effects of the addition of auxiliary acids to the base formulation. Formulation parameters included the organic acids concentration ratios, addition of phosphoric and hydrochloric acids, and additions of sulphonic acid, DNSA, and EDTA. The results are shown in Table 7. Based upon these results, the baseline cleaner was selected for use during the second duct cleaning/coating application. This second test patch application was attempted in November 1990. A best laboratory cleaner was used, but it did not easily remove the corrosion. At this time it was learned that the B-Header had been painted in the mid-1970s with an aluminum based paint. It was suspected that the corrosion on the surface may have come through weak spots in that coating and spread across and over the paint. Therefore the cleaner could not be effective since it could not easily get down to the FeO layer

next to the virgin steel. At this time it was also learned that the "A" header has been previously painted. This raised several questions concerning the conditions of the remaining portions of the ducts which have a high priority for coating application. If these duct sections have also been previously painted, then a technique would be required to remove that paint. The developed cleaners could then be used to prepare the duct surfaces for coating. An effort was undertaken to investigate and find such a paint remover, specifically formulated for aluminum-filled silicone resin paints.

Table 7. Evaluation of Cleaners #3

Designation	Characteristics	Rating	Comments
XX-01	High Phosphoric Concentration	Poor	Slow
XX-02	Dilute Baseline	Excellent	#2 Selection
XX-03	Lower Phosphoric Concentration	OK	Slow
XX-04	Baseline	Best	#1 Selection
XX-05	Hydrochloric	Fair	Good for Light Rust
XX-06	Different Organic Ratios	Good	
XX-07	Sulphonic Acid	Good	#4 Selection
XX-08	DNSA	Very Good	#3 Selection, Leaves no film
XX-09	EDTA	Good	Not effective on worst rust

#### 2.6.1.5 Chemical Removal of Silicone/Aluminum Paint

MRC completed a thorough investigation into the removal of high temperature aluminum-silicone paints with a wide variety of solvents as summarized in Table 8. None of these materials were successful in adequately removing the paint. MRC contacted Dow Chemical about the existence of chemical strippers for high temperature silicone/aluminum paint. Two potential materials (Dowanol TPM and Dowanol EPH glycol-ether) were obtained and evaluated. Neither performed adequately.

Table 8. Solvents Screened as Chemical Strippers

Acetone	Chlorosulfonic Acid	Cellosolve	Ethyl Acetate
DMF	Methyl Isobutyl Ketone	Toluene	Dichlorobenzene
Formamide	Tetrahydroforan	Dioxane-1,4	2 Butoxy Ethanol
Acetonitrile	Chloroform	Xylene	Toluenesulfonic Acid
MEK	Methylene Chloride	50% H <sub>2</sub> SO <sub>4</sub>	Concentrated H <sub>2</sub> SO <sub>4</sub>
20% NaOH	Commercial Paint Remover	DMSO	Benzenesulfonic Acid
IPA	Concentrated HCl	Pyridine	50% HCl

#### 2.6.1.6 Cleaner Formulations/Evaluations #4:

The results of the November test patch application lead to investigation of formulations using a different class of organic acids. Six organic acid cleaners were formulated and tested. The recipes are shown in Table 9. Formulation #6 is basically that of the standard MORC.

Laboratory-rusted panels were immersed into these formulations and the amount of cleaning recorded as a function of time as shown in Table 10. The order of effectiveness was:

- Formulation #5
- Formulation #6
- Formulation #3
- Formulation #4
- Formulation #2
- Formulation #1

Although formulation #5 was ranked above the standard MORC formulation, no significant advantage is seen in this formulation.

Table 9. Formulation of Organic Acid Cleaners Used in Investigation #4

Component (gms)	#1	#2	#3	#4	#5	#6
Organic Acid 1	10.0	10.0	10.0	10.0	10.0	10.0
Organic Acid 2	40.0					10.0
Organic Acid 3		40.0				
Organic Acid 4			40.0			
Organic Acid 5				40.0		
Organic Acid 6					40.0	
Surfactant C	1.0	1.0	1.0	1.0	1.0	1.0
DHDKCB	0.05	0.05	0.05	0.05	0.05	0.05
Formamide	15.0	15.0	15.0	15.0	15.0	15.0
Water	100.0	100.0	100.0	100.0	100.0	100.0

Table 10. Test Results for Organic Acid Cleaners #4

Time (hr)	#1	#2	#3	#4	#5	#6
1	0-5*	0-5	10	0-10	40-50	30-40
2	20-30	20-30	20-30	30-40	70-80	60-70
3	50	50	70	80	100	90
4	80-90	90	100	90	100	100

\* Percentage of exposed area cleaned

### 2.6.1.7 MORC Gel Cleaning Performance - Temperature and Time Tests

A series of tests were performed to determine the cleaning effectiveness of the MORC gel on laboratory rusted steel panels as a function of temperature and time. The duct cleaning tests were normally performed in relatively short (several hour) time periods and the walls of the duct was generally cool to cold. These variables may have been a factor in the poor performance of the MORC gel at the ducts, while maintaining excellent performance in the lab.

Six 6" by 6" A203 panels (identified as Set 26R-9621 through 9626) were MEK wiped and thoroughly cleaned on both sides with Aqua Regia. The panels were then neutralized, rinsed and quickly dried. The panels then underwent one month of conditioning to produce a moderately heavy rust with some surface attack. The panels were often removed from the chambers under wet, cold, or hot conditions to try to achieve a thorough rusting in the shortest period of time. High temperatures in the dry oven were deliberate, and an attempt to generate a high temperature oxide effect. The panels were cut into 2" by 2" square test specimens and were identified by a stamped numeral.

The bottom of a stainless steel pan was coated with MORC gel. Nine of the small specimens were placed in the pan and covered with additional gel so that they were completely submerged in the MORC and coated on both sides (see Photograph 6). A test temperature was selected and the controlled environmental chamber was pre-conditioned to that temperature. The pan was instrumented with a thermocouple and placed into the chamber (see Photograph 7). At selected time intervals, the chamber was opened and a test specimen extracted. The pan was returned to the chamber and the temperature again controlled. The extracted specimen was rinsed with high pressure water and quickly forced air dried. The specimens were evaluated, the degree of cleanliness and the amount of rust removed was visually determined, and the results documented. The process was repeated until all specimens were evaluated. The cleanliness of the specimens was further evaluated using a stereoscope.

Tests were conducted at 50, 70, and 90°F. Additionally, a set of specimens were tested at lab ambient conditions as a control and for comparisons with previous tests. Photograph 8 shows the specimens tested at 50°F. At the two hour point, the temperature of the chamber drifted down to about 18°F for a short period. The recovery was quick and probably did not significantly affect the cleaning results. As can be seen, the light red rust was quickly removed at the 15 minute point. However, additional cleaning did not become evident until after four or five hours. The specimen became a bright silver color after overnight exposure to the gel. At that point, it was estimated that the surface was 98% clean.

Two sets of tests were conducted at 70°F. Photographs 9, and 10 show these test specimens.. Again the loose rust was quickly removed. However, significant cleaning began to occur at the one to two hour mark. When left overnight, the cleaner evaporated and dried out on the specimens. This left a grey discoloration, but the surface looked cleaned under the stereoscope. The results from the ambient conditions are shown in Photograph 11 and are similar to those obtained for the 70°F tests.

The specimens tested at 90°F are shown in Photograph 12. After several hours the gel began to exhibit cracking as it began to dry out. The photograph shows that the cleaning of the surface follows a typical "mud-cracking" structure which one would associate with the drying of the gel. The center of the crack platelets are the locations which showed the best cleaning.

Figures 4 and 5 summarize the results of the tests and plot percent cleanliness and percent rust removal as functions of cleaning time, respectively. The test results are shown as separate graphs for each test temperature condition. All of the tests showed evidence of loose rust

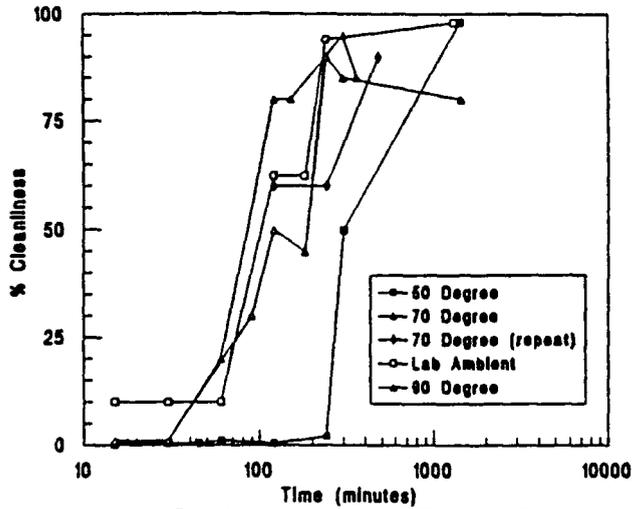


Figure 4. Cleaning Effectiveness Versus Time and Temperature

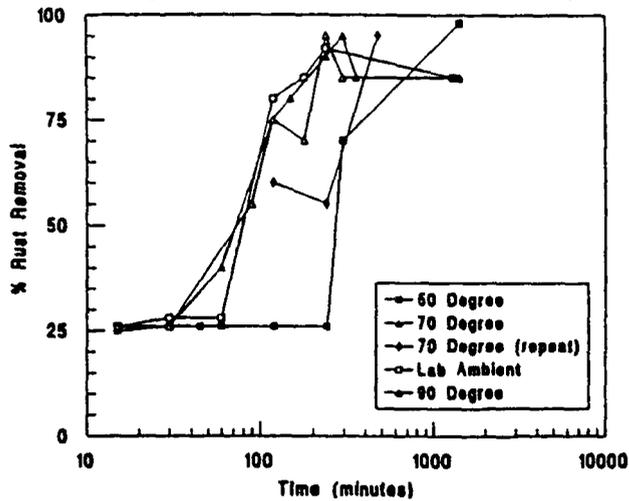


Figure 5. Rust Removal Versus Time and Temperature

removal after 15 minutes. All but the 50°F tests showed a significant increase in cleaning at the 1 to 2 hour mark as the gel presumably gets under the rust layer. This suggests that the rust softens, and a removal and reapplication of the gel at this point may optimize and increase the cleaner performance. Drying out and cracking of the gel occurred in as little as two hours for the 90°F tests. Drying out at ambient conditions did not occur until eight hours. An optimized procedure would remove and reapply the cleaner at the six to eight hour mark. The graphs suggest that the fastest cleaning occurred at 70°F after two hours. However all of the temperatures cleaned to 80% levels after 5 to 8 hours of application. All of the panels showed cleanliness after an overnight exposure.

These results indicate that the cleaner can provide an adequate surface if applied for a long enough period. The results were, however, obtained on laboratory-rusted test panels. Tests conducted by AEDC at the ducts applied the cleaner for long (overnight) periods of time. Although improvement in performance was evident, the cleaner was judged to be ineffective against the B-Header corrosion.

## 2.7 Analysis of Rust Specimens

Several rust scrapings were taken from the duct B-Header wall during the March 1991 test patch application. The rust samples obtained by MRC from the ducts were then subjected to powder X-ray analysis along with samples of salt spray rust generated in MRC's laboratory and a sample of known ferric oxide. This work was carried out at the Graduate Center for Materials Research, University of Missouri-Rolla by Drs. G. Mayhan and J. Li and Professor W. J. James. A copy of the report submitted to MRC is contained in Appendix B. The following samples were analyzed:

- (1) Rust from B-Header Chamber Opening (overhead)
- (2) Rust from B-Header, 30 ft. from Chamber (side)
- (3) Rust from B-Header, 40 ft. from Chamber (side)
- (4) Rust from B-Header, 50 ft. from Chamber (side)
- (5) Rust from B-Header, 50 ft. from Chamber (overhead)
- (6) Rust from B-Header, 20 ft. from Chamber, treated with MORC organic acid rust remover solution and washed with tap water
- (7) Rust from B-Header Extension, about 30 ft. from point of entry
- (8) Rust from panel which was salt spray rusted (MRC)
- (9) 'Pure' ferric oxide sample.

It is noted that no samples were obtained from the A-Header. Also, no samples were taken from the bottom of the ducts because this surface had been exposed to running water and did not represent corrosion products from the rest of the duct surfaces.

### 2.7.1 Sample Preparation

All rust samples were ground to fine powders using a porcelain mortar and pestle. Some samples were easier to powder than others and, therefore, the fineness of grind varied somewhat. In those cases where the rust presented problems, the grinding time was extended from three to five minutes. The difficult-to-powder samples appeared much more crystalline than the salt spray rust, being much more densely packed. The powders were then spread and packed onto a glass slide to which a thin layer of petroleum had been applied over an area measuring approximately 1.5 cm x 4.0 cm. The powder was further compressed by covering the powder with a clean glass plate and applying pressure. The top glass plate was removed and the glass plate containing the sample was positioned in a DIANO-XRD-8000 scanning x-ray spectrometer. In general, the samples were scanned from 30 to 80 degrees, in some instances specific ranges were rescanned to confirm the presence of specific peaks.

All final spectra were obtained using Co K-alpha radiation. It is noted that the customary Cu K-alpha radiation can cause fluorescence with the iron and produce interferences which make interpretation of any results difficult. Both the unadulterated and the powdered samples were microscopically examined in the presence of a strong magnetic field below the glass slides upon which the rust was placed. With the exception of the control  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>, all of the samples exhibited some magnetic character; the salt spray rust showing the least and the rust from the

chemically cleaned section showing by far the most activity. It was possible to roughly separate the magnetic from the non-magnetic particles in the powders; however, it was not possible to obtain enough separated particles to perform an independent analysis.

The basis for interpreting the x-ray powder patterns was the application of the Bragg relationship:

$$n\lambda = 2d \sin \Theta$$

where  $\lambda$  is the wavelength of radiation

$\Theta$  is Bragg reflection angle

d is the atomic spacing relating to this reflection angle

n is an integer (order of reflection)

The d values were then indexed and specific combinations identified and then confirmed through the voluminous X-ray File Numbers. The initial assignments were made based upon the three most intense peaks expected for a particular oxide and then the lesser intense peaks were correlated and through experience and the process of elimination, the final assignments of structure were completed.

### 2.7.2 X-ray Analysis Results and Discussion

The principal findings from the powder x-ray analysis can be summarized:

- (1) All rust samples from the ducts were found to contain  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> with varying amounts of Fe<sub>3</sub>O<sub>4</sub> (magnetite), with the exception of Sample (6) which had been treated with the MORC rust remover solution.
- (2) The rust sample No. 6 consisted almost entirely of Fe<sub>3</sub>O<sub>4</sub>. This sample was the most crystalline and the most difficult to powder. This rust represents a tightly packed crystalline film next to the base metal. The MORC rust remover would require multiple applications and very long exposure times in order to permeate and react with the thin layer of FeO which most probably exists beneath this layer.
- (3) The salt spray rust analyzed was predominantly  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> with a small amount of Fe<sub>3</sub>O<sub>4</sub>. This rust was easy to remove from the steel panel and was easy to powder using a mortar and pestle. This rust is not as tenaciously held to the base metal as that found in the ducts. In general, the salt spray products do not appear to simulate the actual duct corrosion products either from the standpoint of composition or crystalline morphology.

The rust obtained from the B-Header gave a somewhat unique x-ray pattern. This spectrum shows the probable presence of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> in addition to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>. In addition to the splitting of the peaks in the vicinity of 42°, several minor peaks were corollary with  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>. The major peak for  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> is about 39° which corresponds to a d-spacing of 2.69. Also,  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> has a peak corresponding to a d-spacing of 2.51 which is one-half to one-third of the intensity of the 2.69 peak. The samples from the ducts do not show this correspondence indicating the presence of a second major component. Since the major peaks of magnetite (Fe<sub>3</sub>O<sub>4</sub>) and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> occur at approximately the same d-spacing (2.49 - 2.53), the relative intensities of these peaks indicate

the mixture. It should be noted that there is much similarity in the structures of  $\gamma\text{-Fe}_2\text{O}_3$  and  $\text{Fe}_3\text{O}_4$ . Both have magnetic properties where the  $\alpha\text{-Fe}_2\text{O}_3$  is essentially non-magnetic.

It should be noted that the peak at  $39^\circ$  is missing. This peak is associated with  $\alpha\text{-Fe}_2\text{O}_3$  and it must be concluded that the  $\text{Fe}_2\text{O}_3$  that was originally a part of the rust has been undermined and removed by the cleaner. In order for this to occur, at least a thin layer of FeO had to exist between the layers. This is reasonable since  $\text{Fe}_3\text{O}_4$  is thought to really be a product of  $\text{FeO} + \text{Fe}_2\text{O}_3$ . A similar layer probably exists below the primary  $\text{Fe}_3\text{O}_4$  layer, but to reach it this layer must be permeated. The underlying layer of the ferrous oxide would be expected to be oxygen deficient, have a higher metal content and be held more tenaciously to the iron surface.

Under the environmental conditions found in the ducts (temperature range from freezing to  $350^\circ\text{C}$ ), it is expected that the corroded iron would form three layers of oxide:

Ferrous oxide (FeO)

Magnetite ( $\text{Fe}_3\text{O}_4$ )  $\rightarrow$  ( $\text{FeO} + \text{Fe}_2\text{O}_3$ )

Ferric oxide ( $\alpha\text{-Fe}_2\text{O}_3$ )

and obviously under some conditions some  $\gamma\text{-Fe}_2\text{O}_3$  is formed. The FeO,  $\text{Fe}_3\text{O}_4$  and  $\alpha\text{-Fe}_2\text{O}_3$  are defined by cubic structures while the  $\gamma\text{-Fe}_2\text{O}_3$  is rhombohedral. It is believed that the  $\gamma\text{-Fe}_2\text{O}_3$  is unstable at higher temperatures, but there is some controversy over this point. Under the duct conditions, the primary compound is expected to be  $\alpha\text{-Fe}_2\text{O}_3$ . The  $\gamma\text{-Fe}_2\text{O}_3$  could form at  $300\text{-}320^\circ\text{C}$ ., but would only be expected to do so in reduced oxygen atmosphere. It could therefore possibly form under the outer layers, since activity of oxygen is reduced there. The oxide films of  $\alpha\text{-Fe}_2\text{O}_3$  might be expected to form an overlayer of  $\gamma\text{-Fe}_2\text{O}_3$ , but below  $180^\circ\text{C}$  this would be expected to convert to  $\text{Fe}_3\text{O}_4$  at a very slow rate. The thickness of the FeO layer will depend upon the temperature and oxygen partial pressure at the time it is formed. The subsequent film of  $\text{Fe}_3\text{O}_4$  can be a stable tightly packed oxide layer. This will depend upon the ferrous state and how it converts to the hydrated forms of the higher oxides. When removing the scale by scraping, it is unlikely that any FeO present would be removed and detected (as such) by the X-ray analysis.

To be able to make more meaningful statements, it is necessary to know the precise composition of the steel used to fabricate the ducts (drawing available to MRC did not call out the steel alloy used in the B-Header) and a more accurate record of the environmental conditions to which the ducts were subjected and for what time periods. The basic compositions of the oxide layers and the extent to which they are formed depends upon the composition of the steel and the environment to which it is subjected. The nature of the adhesion and cohesion of the oxide layers obviously depend upon the same parameters.

Based upon the available results, there is strong evidence that the rust present in the ducts is substantially different than that formed in the laboratory using salt spray. The salt spray rust contains less magnetite and is more permeable to the rust removing solution than the rust layers in the ducts. At this time it, appears that the present MORC rust remover would require multiple applications and significantly longer exposure times to undermine the  $\text{Fe}_3\text{O}_4$  layer. This time could be reduced by mechanically breaking the external corrosion layers.

## 2.8 Water Blasting Evaluation

### 2.8.1 Test Panels

At the beginning of the project, water blasting appeared to be a feasible candidate for cleaning the corrosion from the duct walls. High pressure water can be used to cut steel, therefore, its use for cleaning corrosion appeared to a possible solution. Rust inhibitors could be added into the water to prevent flash rusting from forming before the coating could be applied. The addition of abrasives into the water was considered, but quickly disapproved by the AEDC personnel. MRC held several meetings with representatives from water blasting manufacturers. Panels were three times sent outside to water blast facilities for cleaning, in order to evaluate the technique.

MRC first sent three steel test specimens to Ingersoll Rand for water jet cleaning. Two of the specimens were coated with high temperature aluminum paint and the third had severe rusting. The paints used were MRC-MARB and Sheffield 326 aluminum-silicone paint. A video tape was made of the process and an engineering report was also prepared and received. The video tape showed that there was no problem in achieving a shiny metallic surface on any of the specimens that were sent. High pressure water at 45,000 psi containing no abrasive grit was used. The pressure nozzle was spun at 1250 rpm and traverse speed of the nozzle was varied between 20 and 200 inches per minute (ipm). Orifice diameter of the nozzle was .01 inches. Except for areas of difficult rust, this method might be capable of providing a paintable surface. We could not definitely determine this, since the plates had developed a light coat of rust by the time we had received them back.

In the second evaluation, eleven A203 panels were sent to Ingersoll-Rand for water blast cleaning. The conditions of the panels were:

- Panels 9701-9703 - MEK washed; mill scale not removed
- Panels 9711-9713 - MORC-cleaned 1 hour each side; mill scale not removed
- Panels 9721-9723 - Mill scale removed using aqua-regia
- Panels 9731&9732 - Mill scale removed using aqua-regia; coated with MARB

Once cleaned, the specimens were rusted in the laboratory using twelve thermal/humidity cycling events. The panels were then shipped to Ingersoll-Rand. They cleaned the panels as indicated in Table 11 and returned them to MRC for evaluation. Table 11 also contains MRC's observations as to the cleanliness produced by the blasting.

All of the blasting was accomplished using 0.01 diameter orifices with a rotation rate of 600 rpm. No abrasives were used and the jet cleaned a path about 1.0 inch wide. Although Ingersoll-Rand indicated that mill scale was completely removed, a residual dark grey oxide was evident which we were able to remove using MORC. Panels 9721 through 9723 were 100% cleaned of this oxide within two hours of MORC application. Photographs 13 through 16 show several of the after being returned from Ingersoll-Rand and after gel cleaning. We conclude that water jet blasting at 20-40 inches per minute at 47 ksi will remove aluminum-silicone paint and normal rusting. Only partial removal of mill-scale was accomplished at 35 ksi. Even at 47 ksi the mill-scale was not completely removed at speeds above 20 ipm. Speeds above 40 ipm leave rough grey colored marks. Water blasting at 40 ipm with 47 ksi pressure is a possible technique to clean off heavy rust (no mill-scale) as well as MARB coating removal. The blasting should be followed by a 30 to 60 minute application of MORC for 100% cleanliness.

The third evaluation was on specimens blasted by National Liquid Blasters (NLB) of Wixom, Michigan. Six panels were provided as described in Table 12. Photographs 17 and 18 show the panels as they were shipped to NLB. These were cleaned using pressures of 20 and 35 ksi and

speeds ranging from 4 to 12 ft/minute. Photograph 19 is an NLB picture taken after cleaning. The cleaning performance was mixed with the low velocities providing reasonable cleanliness. Applications of MORC cleaner after the blasting provided surfaces capable of coating. (See Photograph 20 to observe cleaning after one hour of MORC application).

Table 11. Summary of Ingersoll-Rand Water Blast Removal

Panel ID	Preparation	Speed (ipm)	Press (ksi)	MRC Observations
9701	MEK wash	40	35	Some mill scale flakes
		40	47	Mill scale removed
9702	MEK wash	60	47	Mill scale removed
		60	35	Mill scale specks evident
9703	MEK wash	40	47	Some mill scale removed
		40	35	Some mill scale removed
		20	47	Some mill scale removed
		40	47	Some mill scale removed
9721	Aqua Regia	60	47	Rough texture marks
		60	35	Rough texture marks
9722	Aqua Regia	40	35	Good removal
		40	47	Best rust removal
9723	Aqua Regia	100	35	Very rough surface
		100	47	Very rough surface
9731	MARB coat	40	47	Best removal of MARB coating
		40	35	

Table 12. Summary of NLB Panels

Panel	Steel	Side A	Side B	Mill Scale?	Cleaning Rating
1	A516	Light Rust	Light Rust/Coated	No	2
2	A203	Heavy Rust	Heavy Rust	No	4
3	A203	Rust on Scale	Clean	Yes	6
4	A203	Heavy Rust	Clean	Yes	5
5	A516	Medium Rust	MARB Coated	1	
6	A516	Medium Rust	MARB Coated	3	

## **2.8.2 Requirements and Feasibility for Water Blasting**

Discussions with NLB employees provided critical information for assessing the feasibility of using water blasting to remove the duct corrosion. NLB markets a portable system Appendix C contains a cost estimate for a system which could be used in cleaning the ducts. There are a number of critical issues associated with this cleaning technique. These include water requirements, manhours associated with cleaning the ducts, size and weight of the unit, and hardware costs. These are discussed in the following paragraphs.

### Water Requirements:

The water used with the blasting equipment needs to be of drinking quality and low in calcium and iron. A 20 psi system (the minimum pressure for removing any tough corrosion) requires between 3 and 9 gallons/minute during operation. A 40 inch/minute cleaning speed (again the minimum useful operating speed) which cleans a 1.5 inch wide strip, uses between 7 and 21 gallons of water per square foot. This implies that the B-Header (about 5000 square feet) would require about 100,000 gallons of drinking quality water to perform the cleaning. This does not account for waste or system start-up. In addition, this 100,000 gallons of water would be contaminated by the rust residue and require proper disposal.

### Cleaning Time Requirements:

A reasonable operating speed for removing the tough corrosion is about 40 inches/minute with a 1.5 inch wide strip. This is equal to 0.41 sq ft/minute. The B-Header (5000 sq ft) would require 12000 minutes of actual cleaning time (not including waste and set up). This is 200 hours which equals 25 shifts of 8 hours each. This suggests that the B-Header would be tied up for at least a month if not considerably more. The time required to clean the portion of the duct system of interest (38000) sq ft would take almost 200 shifts (or over six months) at a minimum.

### Size and Weight Constraints:

The portable unit of interest weighs about 4500 pounds and would require a crane for placement within the ducting. The volume of the system is about 120 cubic feet, suggesting that placement between the headers would be difficult at best.

### Hardware Costs:

The estimated hardware costs are contained in the Appendix. The hardware costs is the equivalent of one to two fully burdened manyears.

### Blasting Feasibility:

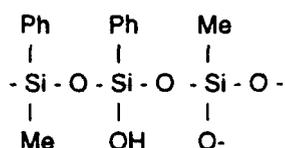
Based upon the water consumption and disposal requirements, the very slow cleaning times and the months required to clean any major portion of the duct system, and the size of the hardware, it does not appear to be feasible to use water blasting as a technique for cleaning the corrosion, and preparing the surface for coating application.

## 2.9 Coating Development Studies and Results

The coating development studies can be divided into three distinct groupings. In the first, various commercial products were evaluated as potential coatings and silicone resin blends were investigated and formulated. The second set evaluated both commercial and developed materials on "prepared" steel panels as a function of temperature and humidity conditioning. The third focused on long term and accelerated aging effects on the selected metallic powder-filled, silicone resin blended paints. Each of these are discussed in the next sections.

### 2.9.1. Initial Coating Developments and Evaluations

The only known polymeric coatings which have properties which can withstand the extreme duct environmental conditions are silicone resins. Silicones are made from organo-substituted chlorosilanes which, in turn, are prepared from elemental silicon derived from silica sand. Most of the resins used for coatings are of the type siloxane, which consists of alternating silicon and oxygen atoms. These in turn are linked directly to at least one monofunctional organic radical. An example of a poly(siloxane) is shown below:



Methyl (Me) and Phenyl (Ph) are the most common organic substituents in silicones because of their superior heat resistance. Each of these monomers produces certain physical properties to the silicone resin. The contributions of the two groups are generalized as follows:

Properties - High Methyl Content	Properties - High Phenyl Content
Flexibility	Heat Stability
Water Repellency	Flexibility Retention on Heat Aging
Low Weight Loss	Toughness
Low Temperature Flexibility	Air-Drying
Fast Cure Rate	
Heat-Shock Resistance	

The weight loss of the high phenyl content resins is greater than that of the similar high methyl resins because the phenyl resins cure more slowly and the lower polymers evaporate before they can cure with the matrix. After curing, however, the high phenyl resins change more slowly on thermal exposure since they possess higher oxidation resistance. The superior chemical resistance of the methyl resins is due to the faster and more complete cure. They may exhibit lower weight loss than the phenyl resins but are less resistant to crazing at elevated temperatures.

As can be seen above, the properties of the available silicone resins vary considerably and require blending to obtain the exact material formulation of interest. An infinite array of blends can be formulated. Commercially available paints have been blended to address several generic sets of environmental conditions. Most of these are directed towards very high temperature applications such as boilers or stacks. These paints are not blended for both the extremely high and low temperatures and high humidity conditions which exist at the duct system. Because of

the expense of silicone resins, most of these formulations contain only the minimal amount of silicone to satisfy their stated applicability. The major objective of the initial coating investigation was to mix phenyl and methyl rich resins to produce a hybrid which optimized the performance in the categories important to the duct environment. Table 13 lists the silicone resins studied and their characteristics.

Table 13. Silicone Resins Studied and Their Characteristics

Manufacturers	Resin	Characteristics
Dow Corning	805	Flexible, Good with pigments
Dow Corning	806A	Semi-hard, Good with aluminum
Dow Corning	808	Flexible, High temperature
Dow Corning	6-2230	Solid, Non-flexible, High temperature, must be blended
General Electric	SR-882M	Hard, Good blending, High solids
General Electric	SR-125	Tough, Flexible, Good thermal shock
General Electric	SR-240	Very high temperatures (>800°F), plasticizers

An important consideration in the formulation of silicone resins is the use of metallic salts as "dryers" which decrease the cure temperatures and times. Without these dryers, silicone resins can take weeks to fully cure at room temperatures or require high temperatures (around 480°F) to fully cure. Neither of these conditions is desirable for application in the duct system. The most popular dryer is zinc octoate.

A second consideration is the use of a metal filler in the silicone resin. The standard material used is aluminum flake. The flake provides a leafing condition and an attachment point for the silicone chain and is required for increased high temperature performance. Zinc powder can also be used and it provides cathodic protection against corrosion. Investigations were performed to evaluate both of these materials and their appropriate concentrations.

Ten series of test specimens were fabricated to investigate silicone resin formulations, evaluate specific materials, blends, and fillers, and to gain experience with these materials, fabrication processes, and test techniques. Of particular interest were commercially available inorganic zinc ethyl silicates. These materials have good high temperature properties, provide good cathodic protection, and have been used on selected duct exterior surfaces. All of the test specimens used Q-panel substrates so that good adhesion to a smooth surface would be obtained. Because the objectives were to study the coating materials themselves, adhesion and surface preparation were not of concern. The following paragraphs very briefly describe the objectives and results obtained from each specimen fabrication and test series.

Set 1: The basic objective was to obtain additional experience with silicone resins, to gain experience with the test and fabrication equipment, to compare the different unblended materials, and to investigate different cure cycles. The results indicated that the coatings were too thick, did not cure well, and that the metal dryer content was too low. (See Photograph 21).

- Set 2:** The objectives were to fabricate thin coatings using flexible, heat tolerant resins, and to investigate the effects of metal dryer concentration. The resins used were DC-808 and GE SR-125 and a 480°F cure temperature was used. The resulting specimens looked good, but the 1/2 mil was probably too thin to provide good properties. No apparent difference in the materials was observed. (See Photograph 22).
- Set 3:** The objectives were to evaluate ambient cure cycles and to decrease drying time by increasing metal dryer concentration. The coating thicknesses were also varied between 0.5 and 1.5 mils. The resins used were DC-808 and GE SR-125. Poor results were obtained; the specimens were gummy and poorly cured. Additional work was needed to develop a ambient curing material. (See Photograph 23).
- Set 4:** The objective was to use the knowledge gained from the previous specimens and to fabricate a high quality set of 1 mil specimens. This was achieved using DC-808 and GE SR-125 with an accelerated curing cycle and a moderate concentration of the dryer material. In all cases, the dryer used was Zinc Octoate (ZO) with a zinc concentration of 8%. High quality specimens were fabricated and a one mil thickness appears to be optimal. (See Photograph 24).
- Set 5:** The objective was to investigate application methods, namely, knife, spray, and brush. In addition, we evaluated a very hard, high temperature resin (GE SR-240). The results show a wide variation in thicknesses was obtained as a function of application technique. Additional work needed to be performed, but both spray and brush techniques should be applicable for the duct coating. The GE SR-240 looks like a material with high potential. (See Photograph 25).
- Set 6:** The objective was to investigate zinc powder (#201 dust) and aluminum flake (MD-7100) fillers in the SR-240 resin. Two filler concentrations were used (20 and 50%). The fabricated specimens exhibited high scratch hardness, but some problems were encountered with thickness control. Additional processing work would be required. (See Photograph 26).
- Set 7:** The objective was to evaluate silicone resin blends to produce a material with the proper high and low temperature, abrasion, and flexibility properties. The materials blended were DC 6-2230 flake (as a control), 50/50 DC-6-2230/GE SR-240, 25/75 and 50/50 DC-805/DC-806A, and 50/50 GE SR-125/GE SR-240. The resulting DC-6-2230 control was very brittle. The DC-6-2230/GE SR-240 exhibited a good ambient cure and had high scratch hardness. The other blends produced materials with moderate hardness. The blend using the silicone resins produced by the two different companies looked promising. (See Photograph 27).
- Set 8:** The objective was to gain experience in fabricating inorganic zinc ethyl silicates. Two commercial products (Sherwin-Williams Zinc Clad I and II) were used. Several of these were overcoated with a layer of GE SR-240. The specimens exhibited very poor properties, which was traced to a less than optimum fabrication process. The topcoated specimen showed high end joint adhesion. (See Photograph 28).

**Set 9:** The objective was to investigate several different resins (GE SR-112 and GE SR-882M) and to look at the effect of Q-panel thickness and surface condition upon the adhesion of the resins. The results showed that Q-panel thickness was a parameter and that the new resins produced hard, tough coatings. (See Photograph 29).

**Set 10:** The objective was to investigate graphite fillers in SR-240 resin. Several different graphites were evaluated. The specimens exhibited high hardness and good adhesion. Although graphite has several desirable properties, its poor electrochemical corrosion properties when associated with carbon steel, eliminated the use of this concept. (See Photograph 30).

Simple tests were conducted to screen the promising candidates from these test series for high temperature application. They were heated in the oven to 600°F and then scraped using a spatula. The specimen with the highest performance was an aluminum flake-filled GE SR-240 coating. The unfilled silicones showed poor performance by softening.

Based upon the results of these first preliminary experiments, it was concluded:

- o Silicone resins can be blended to produce coatings of potential use for the duct application. The 50/50 blend of DC-6-2230 and GE SR-240 produced a material of high quality which warranted further development.
- o Coating thickness is a critical parameter, with an optimal thickness being about 1 mil.
- o Filling the blends with aluminum flake and/or zinc powder increases the coating materials' performance in terms of hardness and resistance. High fill volumes (50%) are achievable.
- o A room temperature cure will be difficult to achieve.
- o Application techniques such as spray and brush can be used to apply the silicone resins. However, thickness control is an issue, especially with the brush which may lead to corrosion protection problems.

## **2.9.2. Coating Developments and Evaluations**

Under this portion of the development effort, over 100 specimens were evaluated and documented. The general objectives of these specimen series were to investigate the performance of commercial and MRC-produced coatings on ferrous surfaces with different surface preparations and degrees of cleanliness. The initial sets of specimens used the A516-70 steel panels as received from the distributor. These were covered with a wide range of corrosion and contamination and were therefore not optimally controlled. Later specimen sets used panels which were first cleaned with hydrochloric acid and allowed to grow a controlled layer of known corrosion.

The initial sets of specimens were subjected to several sets of temperature and humidity cycling. These test conditions were selected to exercise the coatings in a screening mode. For the later sets, the general procedure was to prepare, coat, and cure the panel. These cured panels were then subjected to high (>600°F) and low (-100°F) temperatures for a day, and placed overnight in the humidity chamber at high (80% RH) humidities and temperatures (120°F). This was generally sufficient to produce considerable rusting on the uncoated panel back surface. The conditioning

was conducted using the MRC oven, and the ATS heating/cooling (-120 to 650°F) and the Blue M humidity (15 to 180°F; 5 to 98% RH) chambers. Adhesion test dollies were bonded to the coatings and cured overnight. The adhesion tests were conducted the next day using ASTM D4541-85. The conditioning history, the measured test results, and observations were recorded on a specially designed form to provide complete documentation for each specimen. After testing, the specimens were generally again subjected to the high and low temperature and high humidity conditioning cycle, and again tested. Some of the more interesting specimens have been through hundreds or more of these conditioning cycles.

The fabrication and testing of these panels was designed to evaluate a number of different parameters and variables. These include:

- o The initial condition of the panels
- o The surface preparation and degree of cleaning
- o The use of commercial inhibitors
- o Primer and undercoat materials
- o Top coat materials (commercial, MRC-blended/filled aluminum and zinc paints)
- o Application techniques

In addition to these specimens with permanent coatings, several "replenishable" concepts were briefly evaluated. These are coatings whose thicknesses are expected to wear away as a function of time, but which can be easily/cheaply recoated and replenished several times a year.

The major development activities focused on the formulation of aluminum flake-and zinc powder-filled silicone resin blends. A number of formulations of each of the paint types were developed and tested. Table 14 summarizes these formulations.

Table 14. MRC-Formulated Silicone Resin Blended Paints

Designation	GE SR-240	DC-6-2230	DC-808	DC-805	ZO	Al Flake	Zn Powder
MARB-33-O	10.0	20.0			0.15	7.4	
MARB-42.5-0	10.0	10.0			0.15	7.4	
MARB-33-2	5.0	25.0			0.15	7.4	
MARB-15	10.0	20.0			0.15	2.7	
MARB-47	10.0	20.0			0.15	13.5	
MARS-33-O		20.0	10.0		0.15	7.4	
MARS-43-O		10.0	10.0		0.15	7.4	
MARS-33-3	30.0				0.15	7.4	
MARS-47-2		20.0		10.0	0.15	13.5	
MARS-47-2a		25.0		5.0	0.15	13.5	
MZRB-60-O	10.0	20.0			0.15		22.2
MZRB-60-2	5.0	25.0			0.15		22.2
MZRB-33	10.0	20.0			0.15		7.4
MZRB-47	10.0	20.0			0.15		13.5
MZRB-80	10.0	20.0			0.15		60.0

Notes: Formulation given in parts by weight

GE SR-240 is 50% by weight solids and 50% Toluene

DC-6-2230 is 50% by weight solids and 50% Xylene

DC-808 is 50% by weight solids and 50% Xylene

DC-805 is 50% by weight solids and 50% Xylene

ZO is Zinc Octoate (8% Zinc), Aluminum Flake is MD-7100, Zinc Powder is #201

The following paragraphs summarize the objectives and results obtained from each specimen fabrication and test series. The detailed thermal history and test result documentation of selected test panels are contained in Appendix D.

**Set 11:** The basic objective was to evaluate the adhesion performance of the MRC aluminum-filled silicone resin (MARB-33-O) to a corroded ferrous substrate which was only prepared with a Xylene wipe. This would provide worst case adhesion data. The A516-70 steel panel was wiped with Xylene solvent and allowed to dry. The coating was applied by knife blade (ASTM D823-87) which produced a 2 to 2.5 mil dry thickness. The coating was cured at room temperature for three days and then pull tested. The two tests gave reasonable adhesion strengths of 150 to 210 psi. (See Photograph 31).

**Set 12:** The objectives were to evaluate coating adhesion as a function of surface preparation. The substrates were A516-70 panels as received from the distributor. The panels were prepared using several different techniques:

- o none
- o simple solvent wipe
- o wire-brush and solvent wipe
- o solvent wipe and glass bead blast
- o glass bead blast and wire brush
- o clean with hydrochloric acid (HCl)
- o clean with HCl and wire brush
- o clean with HCl and glass bead blast
- o clean with HCl, glass bead blast, and wire brush

The coating materials were Bar-B-Que Black (a commercially available high temperature paint) which showed good performance during the Phase I program, and Zinc Clad II (portions of which were top-coated with a blended silicone resin). The specimens were cured at room temperature for one week. The center sections of some of the panels were coated with a blend of GE SR-240/DC-6-2232 silicone resins and cured at 600°F. Some of the specimens underwent thermal cycling before being tested. The general conclusions were that although high adhesion strengths could be obtained on dirty surfaces, these strengths degraded over time due to the growth of new corrosion under the coatings. Other conclusions reached were that the unfilled silicone resin blend became soft at high temperatures, and the Zinc Clad II provided good cathodic protection but can potentially wear away. (See Photograph 32).

**Set 13:** The objective was to continue evaluating coating performance as a function of substrate preparation. Five different top coat materials were used and applied either directly to the steel or over a Zinc Clad II undercoat layer. These included:

- o MRC Aluminum-Filled Silicone Resin Blend (MARB) over Sherwin-Williams Zinc Clad II (ZCII)
- o MARB directly applied to the substrate
- o Sherwin-Williams Zinc Clad I (ZCI)
- o ZCII directly applied to the substrate
- o Sherwin-Williams Hi Heat (SWHH) over ZCII
- o SWHH directly applied to the substrate
- o Nybco Bar-B-Que Black (BBQ)

Three panels of each system were fabricated. Two of the panels were cleaned with hydrochloric acid and then wire brushed. One of these panels was then selected for scrape tests and the other for adhesion tests and thermal cycling. The third panel was prepared by washing with a detergent and wiping until no additional rust (i.e., red color) came off the surface. After several cycles, the MARB/ZCII specimens failed in the ZCII. The MARB coatings applied to the steel showed good performance, and the coating applied to the HCl-cleaned surface looks very promising. Both the ZCI and ZCII specimens showed marginal performance with coating cohesive failures. The SWHH coatings did not show good adhesion characteristics. The BBQ Black had reasonable adhesion but was easily abraded and was found to be unsuitable. Our overall conclusions were that the MARB appeared to have high potential as a coating material. It performed well on both acid-cleaned, and solvent-wiped steel surfaces. (See Photographs 33 through 47).

**Set 16:** The objectives of this specimen series were to evaluate commercially available cleaners, inhibitors, and a combination of topcoats and primers. The topcoats/undercoats evaluated were MARB, MARS (a different MARB formulation), MZRB (MRC Zinc-Filled Silicone Resin blends), three Sheffield high temperature paints (Red Hot, #326, and Pot Belly Black). The inhibitors studied were Brulin 835 and Sharp 104. The cleaners used were Kano X-Rust and VSI-421. Both of the cleaners are phosphoric acid compounds. The following discusses the specimen fabrication and findings as a function of topcoat material: (See Photographs 48 through 53).

**MARB:** Six specimens were fabricated. Two were cleaned with VSI-421 and the residue removed by wire brushing and an MEK wipe. One specimen was coated with Brulin 835 inhibitor and the other with Sharp 104. These were then coated with MARB. The Brulin specimen showed high strengths. The Sharp specimen failed in the inhibitor layers (but had high psi values). Two specimens were wiped with MEK and glass beaded to produce a clean surface. One was coated with Pot Belly Black (PBB) as a primer, and the other coated with Sharp 104. Both were then coated with MARB. The PBB had high strength with the failures being in the PBB. The Sharp specimen began flaking at MARB/Sharp interface after removal from the humidity chamber. The last specimen was cleaned with VCI-421 and the residue removed by glass beading and an MEK wipe. It was then coated with Sharp 104 and MARB. It also began flaking after removal from the humidity chamber. One specimen was cleaned with Kano X-Rust and the residue removed by glass beading and an MEK wipe. A layer of Brulin 835 inhibitor was applied and then coated with a layer of MZRB40. This is an MRC silicone resin blend which is filled with 40% by weight zinc powder. It was then coated by the MARB. It was hoped that the zinc-filled material would provide good cathodic protection for the steel, while the aluminum-filled MARB provided good abrasion resistance. A portion of the coating failed after curing. It is believed that a portion of the substrate was not properly cleaned which led to the failure. Our general conclusion is that the MARB coating does not work as well over primers and inhibitors. The Brulin appears to be a better choice for an inhibitor than the Sharp material.

**MARS:** Six specimens were fabricated. Three specimens were prepared with an MEK wipe and glass beading. Two of these were coated with PBB and one with Brulin 835. These were then coated with the MARS paint. The PBB

primer specimens showed good performance. The Brulin specimen failed at the Brulin/steel interface. One specimen was cleaned with VCI-421 and then glass beaded and MEK wiped to remove the residue. It was then coated with Sharp 104 and MARS. It began flaking after removal from the humidity chamber. Two specimens were cleaned using Kano X-Rust and the residue removed using glass beading and an MEK wipe. One of these was then coated with Brulin 835 inhibitor. Both of them were then coated with a layer of MZRB40. These were then coated with the MARS. The dual layer which was coated directly onto the steel showed very high adhesion strengths. The Brulin specimen began flaking immediately after cure. Our observations suggest that the MARS formulation has potential but is not as high performing as the MARB material.

**MZRB27:** Two specimens were coated using this 27%-by-weight zinc powder-filled silicone resin blend. Both were cleaned using X-Rust. One was coated with Brulin 835 and one with Sharp 104. These were then coated with the MZRB. Both showed poor performance with failures at the inhibitor/steel interfaces.

**MZRB40:** Three specimens were coated using this 40%-by-weight zinc powder-filled silicone resin blend. Two of these were cleaned using X-Rust. One was coated with Brulin 835 and one with Sharp 104. These were then coated with the MZRB. Both showed failures at the inhibitor/steel interfaces. The third specimen was cleaned with the X-Rust and the residue then cleaned by glass beading and an MEK wipe. The steel was then directly coated with MZRB40. This specimen has exhibited very high performance and strength values. We concluded that the MZRB formulations have promise and needed further evaluation.

**Sheffield Paints:** Similar specimens were coated with commercially available paints (Red Hot, #326, and Pot Belly Black). X-Rust, VCI-421, and glass beading were used as cleaning techniques. The panels were either directly coated or first coated with one of the two inhibitors. They were then coated with the paints. The specimens using the inhibitors showed poor performance. The panels without the inhibitors showed better performance and will be further evaluated.

**Inhibitors:** Control panels were fabricated and tested which consisted of panels cleaned by various methods and then coated with the Brulin 835 and Sharp 104 inhibitors. Adhesion tests were not performed on these specimens. Visual observations indicate that rust began forming on the specimens.

**Set 17:** The objectives of these specimens were to investigate a variety of cleaning techniques which focussed on mechanical removal systems. The methods involved using "conversion coatings" to modify the rust such that the residue could then be wire brushed off of the surface leaving a relatively clean surface for coating. All of the specimen had a MARB topcoat. Primers and undercoats that were used were ZCII, PBB, and BBQ. The Brulin 835 inhibitor was used (although in some cases it was wire brushed off before coating or priming). The "conversion" materials used were Neutra-Rust, BBQ, and PBB. The cleaning techniques used were HCl acid, VCI-421, MEK wipe, and wire brushing. Most of the specimens showed poor performance. It was very difficult to wire-brush the "conversion" materials off of the steel. In addition,

the surfaces were not clean enough for the use of a ZCII undercoat. Most of the concepts failed in the ZCII or at the ZCII/steel interface. These cleaning techniques have no potential and were not pursued. (See Photographs 54 through 76).

- Set 18: These specimens were the first to use the controlled substrate corrosion panels. These panels were thoroughly cleaned with HCl and neutralized using sodium bicarbonate. They were then allowed to grow a uniform light rust on the surface. The panels were then used to look at both permanent and "replenishable" coating. The following paragraphs summarize the fabrication and testing. (See Photographs 77 through 87).

**Permanent:** The panels were cleaned using an MEK wipe until no rust was observed on the cleaning cloth. The specimens were then coated with one of the following coatings: MARB, MARS, MZRB27, MZRB40, MZRB27/MZRB40, Red Hot, #326, PBB, or BBQ. The MARB, MARS, MZRB40, Red Hot, and #326 showed excellent strengths and performance. The MZRB27, PBB, and BBQ showed evidence of rusting under the coating. The MZRB27/ MZRB40 showed relatively low strengths. These panels suggested that lightly rusted areas of the ducts could possibly be solvent wiped and coated with several of these silicone resin paints. The results were encouraging and these materials needed additional investigation using differing cleaners.

**Replenishable:** The concept of this type of coating is to coat the ducts with a cheap material which would inhibit any corrosion but would gradually wear away and need to be reapplied several times a year. The panels were cleaned either with an MEK wipe or by wire brushing followed by an MEK wipe. The materials investigated included: IDA-Kote, Neutra-Rust, MS-122, McLube 358, McLube 362, Lubribond HT, Lubribond 320, Esnalube 382, and Everlube 812. Most of these materials contain MoS<sub>2</sub> and are used as lubricants and mold release agents. None of these materials looked promising. Rusting was evident on the panels after conditioning at high and low temperatures. These concepts were not pursued any further.

- Set 19: This set of specimens consists of six panels which were cleaned using the preliminary organic acid cleaners. One of the panels was only cleaned using an MEK wipe as a control. One half of each panel was coated with MARB and the other half with MZRB27. These specimens were cycled and tested numerous times with no failures. All of the adhesion tests showed very high strengths (>1050 psi). These results confirmed that the MRC-developed MARB and MZRB coatings would survive the duct environments when applied to clean surfaces. These materials were carried to next phase of the program, optimized and tested in conjunction with the consultant chemical cleaners.

### 2.9.3 Coating Optimizations and Evaluations

The results of the coating development studies indicated that the aluminum flake and zinc powder-filled silicone resin blends could survive the harsh duct environments. Several sets of tests and studies were performed to optimize these materials, initiate supporting long-term tests, and support the development of the cleaner materials/techniques. The following paragraphs summarize the objectives and results obtained from each specimen fabrication and test series. The detailed thermal history and test result documentation of selected test panels are contained in Appendix D.

- Set 20:** This set consisted of 6 panels (8601, 8602, 8603, 8701, 8702, and 8703). The first three were lightly rusted and cleaned as well as possible using gel #2. The next three had residue rusting which was documented on vu-foil transparencies. Two thin coats of MARB-33 were sprayed on each panel and the panels were slowly oven cured at 480°F. Long-term aging tests showed early failure over extensively rusted areas. No failures were seen on the wellcleaned and coated areas. (See Photographs 88 through 93).
- Set I-21:** This set consisted of 6 panels (8801, 8802, 8803, 8901, 8902, and 8903). The first three cleaned with acid, allowed to rust and glass-bead blasted. They were then coated with MARB using a brush and cloth wipe. The second three were acid-cleaned, allowed to rust and cleaned with organic acid cleaner 11F3 and then brushed with three thin layers of MZRB-80. These panels were then used to evaluate accelerated aging techniques. (See Photographs 94 through 96).
- Set 21P:** This set consisted of 13 panels (9001 through 901). These were preliminary panels used to plan for set 21. The panels were cleaned using a variety of techniques including hydrochloric acid, solvents, and several cleaning gels. The coatings included single and bi-layer MARB and MZRB coatings and commercial paints. (See Photographs 97 through 109).
- Set 21:** This set consisted of four panels (9014, 9015, 9016, and 9017) designed to optimize the aluminum flake and zinc powder filler loading content. All of the panels were cleaned using gel #3. The aluminum filler content ranged from 15 to 47%. It was determined that the 40% aluminum flake-filled material was the best selection. The zinc powder contents ranged from 33 to 80%. Although the 50% zinc powder-filled material showed good corrosion protection, it was sensitive to mudcracking and curing difficulties and was therefore dropped from further consideration. (See Photographs 110 through 113).
- Set 22P:** This set consisted of three panels (9100, 9200, and 9300) for preliminary applications used to plan for set 22. All of the panels were cleaned using gel #4. The coating compared the original and improved MARB formulations with coatings using only Dow Chemical silicone resins. (See Photographs 114 through 120).
- Set 22:** This set consisted of sprayed coatings of MARB-45 on three steel panels (9101, 9102, and 9103) composed of different steel alloys (A516, A203 and A285). The substrates all had had the mill scale previously removed using 50% HCL in October and had mild rusting. The rust was removed by gel #4 in 5 to 15 minutes. The specimens were then rinsed, thoroughly dried, and coated with MARB-45. Within an hour the panels were sprayed with up to four swipes with a air-spray paint gun. Each coat was dried for one hour before overcoating. The specimens were cured for 1 hour @ 480°F. Extensive testing was performed. The measured adhesion strengths of 500 psi were low and were attributed to using a MARB-45 formulation made in October for use in the duct test patch application. The coating material is suspected of having seen too much aging, diluting, and filtering. (See Photographs 121 through 126).
- Set 22R:** This set dealt with repairing damaged/deteriorated panels from set 20. The repair was accomplished by removing the loose MARB, cleaning the rusted

steel with gel #4, and respraying with a new coat of MARB-45. The completely cleaned areas which were repaired showed no deterioration after 11 aging events. Those areas which had deteriorated MARB (deliberately left on) which were then oversprayed with MARB, failed by flaking. The results suggest that a complete cleaning job must be completed before adequate repair can be accomplished. (See Photographs 127 through 129).

Set : This set (panels 9301, 9302, and 9303) used a new MARB formulation (MARB-DC-40) containing a flexible resin. It is as effective in adhesion as the MARB blend. (See Photograph 130 and 131).

Set 24: This set consisted of panels (9401 through 9406) designed to test the effectiveness of ferrophosphate corrosion inhibitors. The tests suggested that drying the substrates with hot air is as effective as these inhibitors. (See Photographs 132 through 137).

The following paragraphs describe the results of specific studies performed while optimizing the aluminum flake, and zinc powder-filled silicone resin-blended coatings.

**Binder Optimization Studies:** A resin blend study was performed on a steel test panel (Series I - Panel SB-7) completely cleaned with hydrochloric acid. The resin blends contained no filler materials and were designed to optimize the ratio between the two selected baseline components (GE SR-240 and DC-6-2230). The baseline MARB formulation has 1 part of GE SR-240 for every 2 parts of DC-6-2230. The DC-6-2230 is a solid silicone without any plasticizers which has a good high temperature capability. The addition of the GE SR-240 (which also has high temperature capability) provides a plasticizer which allows for better low temperature flexibility while maintaining the high temperature capability. The flexibility, and therefore low temperature capability can be theoretically increased by adding GE SR-240. The hardness can be theoretically increased by reducing the SR-240 concentration. The blends consisted of the following concentrations of each resin:

Blend #	1	2	3	4	5	6	7
GE SR-240	15.0	0.0	2.0	10.0	5.0	7.5	13.0
DC 6-2230	0.0	15.0	13.0	5.0	10.0	7.5	2.0
Zinc Octoate	0.08	0.08	0.08	0.08	0.08	0.08	0.08

The panel was brushcoated with strips of each of these formulations with wet thicknesses of about 3.5 mils. The panel was cured at 480°F and then subjected to hot and cold temperature and high humidity cycling. Scratch tests were conducted to evaluate coating resistance and hardness. The results of the severe testing indicated that all of the resin blends cracked and crazed when exposed to the test environment. The best performing resin after the testing was the original standard MARB blend. This 1 to 2 ratio appears to be the optimum blend between the two silicone resins.

The results of the testing generated deep concern over the binder cracking. A cracked or crazed coating could potentially fail catastrophically and would certainly allow the development of underlayer corrosion. Microscopic observation of the zinc powder-filled coatings also showed this cracking. However, none of the aluminum filled specimens have ever showed this effect. It is

hypothesized, that the leafing action of the aluminum stabilizes the binder materials and precludes such cracking effects. These results suggested that any coating outer layer ought to have an aluminum flake filler.

**Weight Loss Determinations:** (Test Series IP-2, Panels 1 through 4) Four panels were prepared to determine coating weight loss due to high temperature-induced effects such as outgassing and shrinkage deterioration. The panels were first completely cleaned by first using hydrochloric acid and later using one of the consultant-formulated cleaners. The edges of the panel were also cleaned and coated to prevent corrosion of the metal which could cause mass gain due to the substrate oxidation and mass loss from corrosion flake off. The coating materials that were tested were standard MARB, the baseline silicone resin blend with no filler, and Sherwin Williams Hi Heat Enamel paint. The original intent behind this testing was to determine the time required for degradation of the coating, assuming that it would only stand up for a short period of time. However, this was short-sighted since the panels have been tested for many weeks without any significant failures. The MARB-coated panel that has been tested the longest still looks good.

The panels were subjected to temperatures of 600°F in the oven over sixteen (16) times. The panels were weighed after each temperature cycle and the results are presented in Figure 6. Another useful result that came from these tests was an estimate of the approximate time required for full cure. This is believed to occur when the weight loss stops. The MARB-painted panel weight appeared to stabilize after about five oven cycles. The plain resin blend continues to lose weight throughout the 16 heating cycles. The Sherwin Williams paint (non-siliconized) blistered after the first exposure to heat. The blisters were left intact for the duration of testing. The Sherwin Williams paint stabilized at the same time as the MARB, which was five cycles. In addition to minimizing the paint stabilization time, the aluminum flake seems to assist in the polymer cross-linking and prevents coating mass loss. (See Photographs 138 through 141).

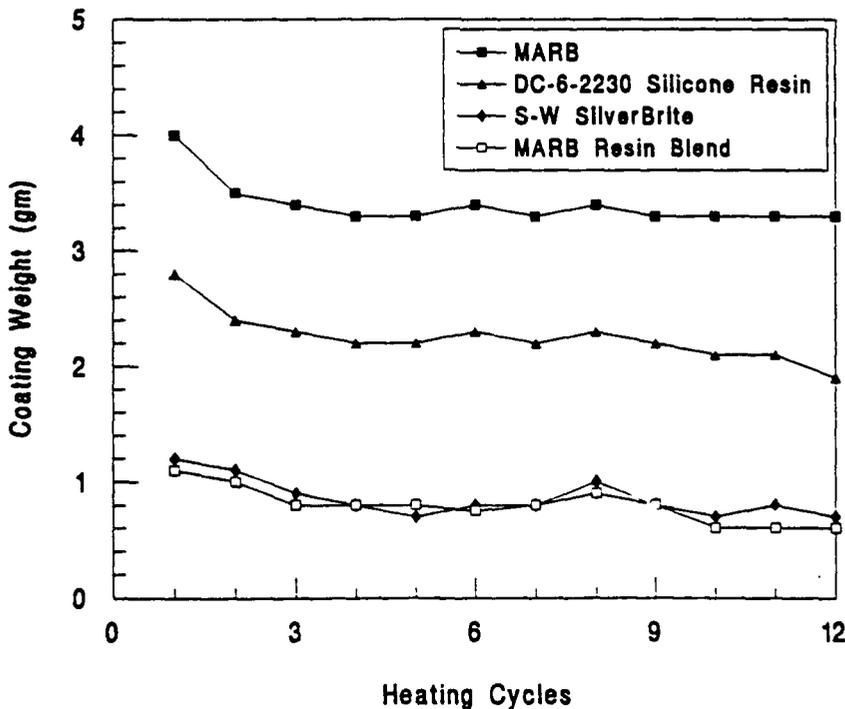


Figure 6. Coating Mass Loss Versus Heating Cycle

### Aluminum Paint Studies:

#### o Optimization Studies (Test Series I, Panel AS-4)

A panel was fabricated to initiate the optimization of the aluminum flake-filled paint. It compared the original MARB formulation against a primer/topcoat system. All of the formulations contained 33% by weight aluminum flake. The exact formulations were previously presented in Table 14. A panel was completely cleaned with hydrochloric acid. Four stripes were applied. The first area was coated with the standard MARB-33-O material. The second area was coated with MARS-33-3 which is a high temperature blend which may be appropriate for a topcoat. The third area was coated with MARB-33-2 which is a low temperature blend which may be appropriate for a primer. The fourth area was coated with a two layer primer/topcoat system. The primer coatings failed after exposure to high temperatures. Areas of thick coating exhibited lifting and cracking as well as rusting caused by the brush strokes texture. The controlled standard MARB-33-O layer performed the best. Conclusions from these results were that the original, standard formulation is the optimum, and that thin, uniform coating layers are desired. This can be achieved by spraying several thin coating layers. (See Photograph 142).

#### o Failure Mode Studies (Test Series 20, Panels 8701 Through 8703)

Specimens were cleaned except for small pin head sized rust locations. These rust locations were recorded on a transparency. Two thin coats of MARB paint were brushed onto the panel and cured. The panels were subjected to the temperature and humidity cycling and artificially aged. The rust was initially slow to appear, but rapidly increased its effect. As the rust expanded, it blistered the paint up from the substrate. The region around the rust blister is relatively unaffected due to cathodic protection, which may make repair relatively easy. If the paint is abraded from the high velocity flow in the ducts, the paint should come off in flecks or as a dust. (See Photographs 91 through 93).

Failure has also occurred on the specimens prepared by brushing, inside the valleys of the brush strokes. While there has been indication of rust, the paint at this stage of testing has not begun to peel or blister. This problem can be easily solved by using a compressed air spray gun to evenly apply the paint to the surface. The panels that have been sprayed hold up when applied to thoroughly cleaned substrates.

Zinc Paint Studies (Test Series 21, Panels 9015 and 9016): Two sets of experiments were conducted to investigate optimized zinc-filled paints. In the first set, the zinc concentration was varied. The original standard baseline was MZRB-33 (refer to Table 14 for the details of the formulations). This material is 33% by weight zinc powder and is a direct analog to the MARB-33-O formulation. This paint was applied to the interior of the ducts during the first duct application. At that time it was apparent that there was not enough zinc in the paint, since it did not cover well. HCl-cleaned steel panels were coated with different formulations containing higher concentrations of zinc, namely:

- o 47% by weight zinc (MZRB-47)
- o 60% by weight zinc (MZRB-60-O)
- o 80% by weight zinc (MZRB-80)

The 60% zinc formulation had good spreadability when brushed on the plate with a uniform thin coat and is now considered to be the zinc baseline paint. One of the potential problems with the zinc paint is that most of the coatings eventually begin to craze and crack. These cracks do not reach to the substrate interface but is very undesirable. Because the aluminum flake-filled paints do not exhibit this cracking, it is believed that the leafing action of the flake prevents the "mud cracking" during thermal cycling. It may be desirable or necessary to topcoat any zinc paint with an aluminum paint. (See Photographs 111 and 112).

The second experiment involved the investigation of a two layer zinc paint system. The concept used a primer layer which has a high filler content for increased cathodic protection. The GE SR-240/DC-6-2230 ratio was maintained at 1/2 in order to retain good low temperature flexibility (and therefore act as a strain isolation layer). This material is MZRB-80. The topcoat was formulated to have good high temperature hardness at the loss of low temperature flexibility. The material is MZRB-60-2. A panel was cleaned with hydrochloric acid and four areas were painted. The first area was coated with MZRB-60, the best single layer paint. The second and third areas were coated with the primer and topcoat formulations, respectively. The fourth area had the two layer system, primer covered with topcoat. The panels were cured and thermally cycled. The panel was subjected to both scratch hardness tests and microscopic examination. The results were that the MZRB-60 exhibited good performance, the primer layer showed some cracking, and the topcoat looked good where applied in a thin (< 1 mil) layer. The two layered system showed very heavy cracking. The conclusions reached from the zinc paint tests include:

- o Uncured zinc paints are crumbly and have low cohesive strength.
- o Thick (>1 mil), cured zinc paints exhibit almost immediate cracking.
- o Thin (<1 mil), cured zinc paints cracked after a number of thermal cycles.
- o Coatings with high zinc concentrations (>60%) are easy to apply but exhibit heavy cracking.
- o Coatings with low zinc concentrations (<40%) produce an uneven coat which permits substrate rusting under the light spots and crazes after thermal cycling.
- o Additional development work is required to produce a useable, acceptable zinc powder-filled paint.

**Application Studies:** Preliminary paint application studies were initiated which addressed both the aluminum flake and zinc powder-filled paints. The application techniques studied were brush, wipe, and spray. Most of the laboratory applications have been performed using a brush. This is simple and easy to use, however coating thickness is an issue and brush strokes generated valleys. The experiments show that these valleys become points for moisture penetration and subsequent undercoat corrosion. Brushing is not an acceptable application method for the ducts. The results and conclusions for wiping are the same as for brushing. Wiping produces a more uniform thickness (strokes are not a problem), but the thickness cannot be well controlled and the leafing properties seemed to be degraded. Spray painting is by far the best technique for applying the coating. Issues which will be addressed in the next phase of the program include:

- o Air versus airless spraying
- o Paint viscosity
- o Filtering and filler uniformity
- o Coating thickness
- o Number of coats/applications

## 2.9.4 Comparison of Performance of MARB and Commercially Available Paints

MRC MARB4 is a specially formulated silicone resin protective coating containing a 40% by weight leafing aluminum flake concentration. The documented high and low temperature resistance is due to the high volume content of selected silicone resin types and the humidity resistance is dependent on the leafing action of the aluminum flake. The beneficial effect of the leafing can be lost by excessive mixing and improper application. This problem is being mitigated through the addition of an organoclay suspension and leafing property stabilizer. The coating requires a slow heating schedule to attain its full potential.

This coating (Panels 9605 and 9605A - ambient cured) was directly compared to an older mix (Panels 9604 and 9604A) which was prepared four months ago and four commercial paints which included:

- o Sherwin Williams B59S3 (Silver-Brite Hi-Heat Resisting Aluminum Paint - good to 400-700°F) - Panel 9601 and 9601A (ambient cured).
- o Sherwin Williams B59S8 (Silver-Brite Hi-Heat Silicone Alkyd Aluminum Paint - good to 500-1000°F) - Panel 9602 and 9602A (ambient cured).
- o Sheffield #392 (Industrial Siliconized Super-Hot Aluminum Paint - good to 1600°F) - Panel 9603 and 9603A (ambient cured).
- o Sherwin Williams Hi-Heat Spray Enamel (Aluminum Paste-Filled Silicone Paint - good to 700°F) - Panel 9606 and 9606A (ambient cured).

The coatings were sprayed onto panels of A516-70 low carbon steel. The panels had been thoroughly cleaned with a 50% HCl solution in water, neutralized with sodium bicarbonate, and quickly force air dried. The cleaned panels exhibited little flash rusting, but were further cleaned with the standard MORC cleaner for 15 minutes on both sides, then water rinsed, and thoroughly air dried. Twelve panels were sprayed on both front and back (two with each coating material). The commercial coatings were sprayed with two layers. The coatings were tack free within fifteen minutes after application of the first layer, but the second coats were not applied until after 30 minutes of drying time and then hung to dry for 24 hours. The total thicknesses ranging from to 0.7 to 1.2 mils. A Mikrotest 0-4 mil dry film thickness gauge was used. The four MRC MARB-4 panels were sprayed on both sides with total thicknesses of 1.0 to 1.5 mil. The MARB material which had been prepared over four months ago was difficult to apply in a uniform coating since it was very viscous. The front side of the panel was smoothed using a brush after the spray application. One panel of each type coating was set aside without any heat curing for further study. The other six panels were cured by a slow heating ramp cycle. The oven reached 480°F (250°C) within one hour and was held at that temperature for one hour. Table 15 documents the fabrication of the twelve panels. After curing and cool down, scrape hardness tests were conducted on the cured panels using the balanced beam and the results are summarized in Table 16. The MARB4 panel exhibited the highest performance. The commercial paints showed a decreased resistance to scraping.

The panels were environmental conditioned through exposure to high and low temperature extremes, and high humidity. The conditions included temperature and humidity variation cycles during one month of 30 events. An event is defined as an excursion from and a return to ambient conditions and is longer than one hour in duration. The extremes ranged from -120°F (-84°C) to 750°F (400°C) and humid cycles between 115 and 140°F. Visual observations have been made and recorded with photographs. (Note: there was a major drip on the front side of panel 9601A - a Sherwin Williams paint. We do not believe that this compromised the comparison, since the

Table 15. Sample Fabrication Summary

Number	Material	Thickness (mils)	Cure
9601	Sherwin Williams B59S3	0.8	Yes
9601A	Sherwin Williams B59S3	0.8	No
9602	Sherwin Williams B59S8	1.2	Yes
9602A	Sherwin Williams B59S8	1.2	No
9603	Sheffield #392	0.7	Yes
9603A	Sheffield #392	0.7	No
9604	MRC MARB4 (old)	1.5	Yes
9604A	MRC MARB4 (old)	1.5	No
9605	MRC MARB4	1.0	Yes
9605A	MRC MARB4	1.0	No
9606	Sherwin Williams Hi-Heat Spray	0.9	Yes
9606A	Sherwin Williams Hi-Heat Spray	0.9	No

Table 16. Scrape Hardness Test Results

Panel	Weight (gm)				
	20	50	100	200	300
9601	O	L	ML	MH	H
9602	L	M	M	H	VH
9603	O	L	M	H	VH
9604	O	O	L	ML	H
9605	O	O	O	O	VL
9606	ML	M	MH	MH	VH

Notes: O = No marring  
 VL = Very little marring  
 L = Light marring  
 M = Medium effect  
 H = Heavy scratch  
 VH = Very heavy scratch

rear side of the panel was also sprayed without any dripping, and has exhibited the same degree of failure). Adhesion tests were conducted after 6, 27, and 44 events. A summary of the visual observations and adhesion results are contained in Table 17. Failure area and adhesion strength as a function of events are plotted in Figure 7. No adhesion strength results are reported for those panels which exhibited coating failure over 50% of the panel front surface area.

Panels 9601 and 9606 began failing within two weeks after initiation of the cycling with observations of rusting, flaking/blistering, and discoloration which continued to worsen as the conditioning proceeded. Panels 9602 and 9603 began exhibiting failures over significant areas after 27 events. After 44 events the new MARB panel (9605) finally began exhibiting rusting along the edges. The adhesion strength was still measured at greater than 1000 psi at that time. The MARB has performed considerably better than the selected commercial paints under these preparation and testing conditions. (See Photographs 143 through 173).

Table 17. Preliminary Visual Failure and Adhesion Test Results

After 6/27/44 Events			
Panel	Material	% Area Failed	Adhesion Strength (psi)
9601	SW B59S3	0/85/90	1000/450/---
9602	SW B59S8	0/50/50	1000/500/---
9603	Sheffield #392	0/30/40	1000/500/400
9604	MRC MARB4 (old)	0/10/50*	1000/750/1000
9605	MRC MARB4	0/0/10**	1000/600/1000
9606	SW Hi-Heat Spray	0/90/95	550/---/---

\*Brushed Side  
 \*\*Edge Only

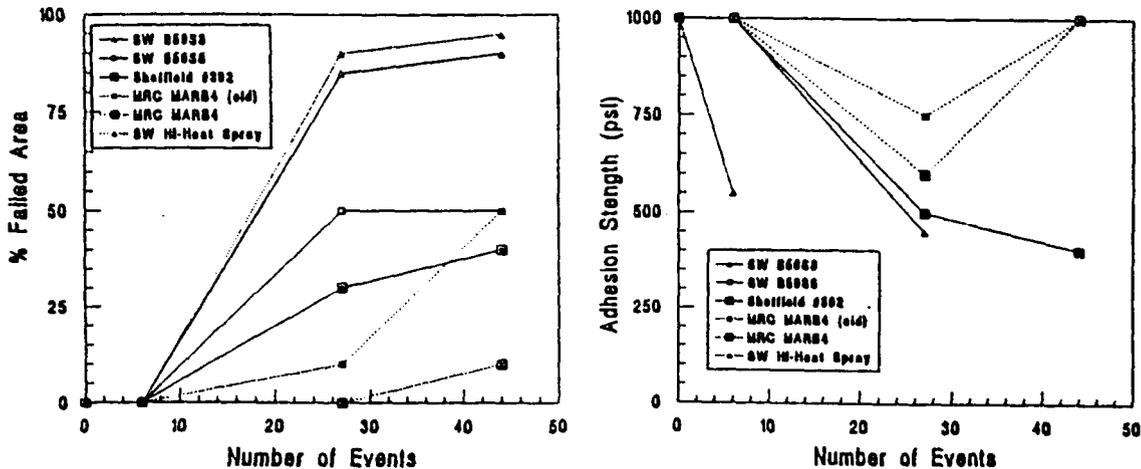


Figure 7. Failed Area and Adhesion Strength Versus Number of Events

### 2.9.5 Advanced Concepts

Throughout the period of coating development, it was desired that a zinc-filled material could be developed since increased cathodic performance could be obtained. However, the simple zinc powder-filled materials always exhibited cracking after the thermal cycling. This was attributed to the fact that zinc powder was used than zinc-flake. We believe that the aluminum flake provides a leafing action which inhibits coating cracking. Attempts to locate a supplier of zinc flake was unsuccessful. However a number of materials have been fabricated which contained both aluminum flake and zinc powder and which have shown excellent thermal resistance and corrosion protection.

The new coating formulation is called MAZRB-60 and contains 10% aluminum flake, 60% zinc powder, and 30% silicone solids. It can be used as a single coating material sprayed on in several thin coats, or as an overcoat to zinc powder-filled MZRB-60. The formulations are presented in Table 18.

The first samples of this concept were fabricated during Set 26A. Panel 9607 was acid cleaned and then sprayed with 0.88 to 1.0 mil thin coat. After eight thermal cycling events, testing gave adhesion levels of over 1050 psi. After 15 events, there was no sign of any coating failure. After 101 thermal cycling events, the adhesion was still measured to be above 950 psi. The thermal cycling has shown little to no effect on the adhesion strength of the coating.

A second set of panels, was fabricated using a multi-layered concept. These are Set 26B, panels 9611, 9612 and 9613. Three coating layers were sprayed on these panels. The first coat was 0.2 - 0.5 mils of MAZRB with 33% zinc mixed with aluminum flake-filled MARB 30. The second coat was 0.2 to 0.4 mils of MARZB' with 33%/wt zinc powder mixed with MARB 40. The top was 0.1 to 0.3 mils <33% zinc powder mixed with standard MARB 47 formulation. There was a 1/2 hr drying period between spray coats and the panels were allowed to dry 18 hrs before a slow heat up period to the cure of 1 hr @ 480°F. That afternoon the panels saw a 1 hr at 650°F event and showed no effect. These panels saw -120 to +790° F without negative effect.

Table 18. Advanced Concept Formulations

Constituents	MZRB-60	MAZRB-60
Silicone resin, GE SR-240	5.0	5.0
Solvent @ 50% Toluene	(5.0)	(5.0)
Silicone resin, DC 6-2230	10.0	10.0
Solvent @ 50% Xylene	(10.0)	(10.0)
Metal drying agent, Zinc Octoate	0.15	1.5
Zinc powder 60% #201	23.0	30.0
Aluminum flake 10% MD-711		5.0
Total Solids	38.0	50.0

## 2.10 Duct Test Patch Applications

MRC visited the AEDC three times for the expressed purpose of applying coating test patches in the ducts. In addition, the AEDC performed a "long time" cleaner test for MRC. All of this work has taken place in the "B" header, although the cleaner has been tested in several other locations including the "A" header.

The first patch application was performed in early July 1990. It was decided to attempt the patch after the Program Review held at the AEDC in late June. The progress made in the coating development and the initial work with the cleaner prompted the decision to quickly try out the system. Little work in optimizing the cleaner had been accomplished at that time. MRC attempted to clean two patch areas, one for the MARB and one for the zinc-filled coating. The cleaner could only be applied for a short period of time (about 1 hour). This was insufficient time for the cleaner to work. At that time, the cleaner was in a liquid form and required constant wetting. (It was then modified into a gel form). Because of the time constraints, the coatings were applied over a still rusty surface. The aluminum-filled test patch showed signs of rust blooming under the coating surface after about nine months on the ducts. This is indicative of the superior performance shown by the MARB material since it was applied to a very poorly prepared surface. MRC was therefore encouraged by the results of this test patch. MRC was, however, discouraged by the performance of the cleaner.

Additional formulation modifications were made to the cleaner and a second cleaning/patch application attempt was made in November 1990. This attempt was spread over several days and again produced poor results. It appeared that a layer of previously applied paint was under the area that MRC was attempting to clean. Various areas in the "B" header were examined using coarse carborundum sandpaper. Previously applied paint was found in all areas examined, leading us to believe that a significant fraction of the "B" header and possibly a good portion of the ducting may still contain this paint. If so, the cleaner will not work without the paint being removed by water blasting or some combined mechanical/cleaner technique.

In an attempt to demonstrate and validate the survivability of the MARB coating, a third test patch application was attempted at the beginning of March 1991. An area was cleaned using a belt sander which still left various small spots of corrosion in the pits. These appeared to be mill scale. There was no visible orange rust left. A layer of rust dust lay over most of the area which cleaned off using our cleaner. The cleaner was rinsed off and after a delay an inhibitor was applied. Flash rusting may have already occurred but was not observed by MRC personnel since the surface was still protected by a film of water. The MARB coating was sprayed on and around the "cleaned" area by the AEDC. The coating may have been thicker than typically applied in our laboratory. In spite of the problems, we believe that this test is superior to the original patch and will exhibit significantly better survivability. Recent examinations by AEDC personnel, indicate that the coating is surviving and that there is no evidence of corrosion growth under the coating. However, the duct system has not been fully exercised since the patch application, and no high temperature conditions have been used. Therefore the coating has not been stressed nor has the coating been fully cured. The AEDC should examine this test patch over the next several years on a regular basis to evaluate its performance.

The AEDC performed a test of the cleaner at MRC's request. After the laboratory tests of the cleaner as a function of temperature and time, it was believed that several applications of the cleaner over 12 to 24 hours might produce a good cleaning performance on the B-Header corrosion. MRC provided the AEDC with sufficient cleaner material and the instructions shown in Table 19. The results of this cleaning test were negative. As indicated in the previous sections, the B-Header corrosion is believed to be extremely tough and difficult to remove because of the temperature-time history which it has been exposed to over a long period of time.

Table 19. Duct Wall Cleaning Test Procedures

1. Select a portion of the duct wall for test cleaning.
2. Shake the MORC gel until all "top" liquids are incorporated.
3. Apply the MORC to the wall using a spatula with one pass. The recommended thickness is 0.125 inch.
4. Squeegee off after 4 to 8 hours. Record the condition of the surface and any signs of surface rust softening.
5. Apply another 0.125 inch thickness of MORC and allow to stand overnight.
6. Remove the gel/rust residue with a pressurized water hose. If the gel is black and hard, it has been effective, but has dried out. A reapplication of the gel will quickly soften the residue material which can then be washed off.
7. Record the condition of the surface. If the wall is not yet clean, but the gel has softened the rust and shown some effectiveness, reapply another layer of MORC and reexamine after 4 to 8 hours.

Note: The gel may require some mechanical help to be most effective in the duct. Suggest that a portion of the test patch use the following: After step 4, reapply a layer of gel using a 3M-type green plastic scrubber which has been dipped into the gel. Gently massage the gel into the duct surface. Then precede to step 5 and add additional gel.

### 2.11 Other Potential Duct Solutions

During the Phase I program, we evaluated a wide range of potential solutions, surface preparation techniques, and materials. These included such extreme ideas as rebuilding the ducts with stainless steel, incorporating an inner stainless steel, ceramic, or other non-corroding material liner, modifying the surface through ion implantation, and electroplating a metal coating. Surface preparation techniques which were reviewed included washing, mechanical cleaning, blasting, and chemical cleaning. We investigated enamels, ceramics, organics, fluorocarbons, and inorganics. The results of the investigations rejected the "far out" solutions as being difficult to implement and "out-of-scope" since they were not polymer coating solutions.

We have considered other surface preparation and cleaning techniques which would cut through the current tough corrosion. These would include chemical techniques to convert the current into FeO. In other words, it may be possible to grow a corrosion which the MORC cleaner will attack and which will provide access to the ferrous surface.

Bonded tiles composed of lightweight materials (similar to the space shuttle tiles) might be applicable. The material selected would need to be tough, produce no outgassing, and not rapidly deteriorate under the duct environment. It may be possible to arc-spray a metal coating which would provide the necessary corrosion protection. Unfortunately, these techniques generally require a blasted surface.

## SECTION III

### PHASE III PLANNING

Two products were developed by MRC during the AEDC contract: a rust remover and a high temperature paint. The advantage to the high temperature paint is that MRC owns the paint formulation outright. However, it may be difficult to successfully market the aluminum paint for use by the general public since it is expensive and there are other suitable, less expensive, name brand, high temperature paints available on the market.

The more marketable item of the two is the rust remover since it may be marketed in both industrial and consumer markets. It is unique in its low toxicity level, unlike other products presently on the market. It has the added advantage of being considerably less reactive to dermatologically-sensitive potential users. This product removes rust at least as well as, and in some cases better than, existing commercial rust removers. All of the components in the rust remover composition are easily diluted and washed away with common tap water.

There appear to be three feasible ways to initiate a market pursuit making the rust remover available for industrial and consumer use. These are:

- (1) License the technology and patent to an existing company which already has a suitable production facility and a marketing/sales organization in place.
- (2) Invest in the production, marketing and sale of the product with MRC capital.
- (3) Seek investment capital to fund production, marketing and sale of the product.

Option (1) is straight forward and would require neither an outright investment by MRC nor a search for investment capital. Options (2) and (3) would require an investment to initiate and sustain the operation for at least the first year. General estimates have been made on the magnitude of the investment required to establish a production facility and a small-scale marketing/sales operation. Our best estimate is that a minimum investment of \$300,000 to \$350,000 would be required.

Based upon bulk chemical prices for chemicals of superior purity, we estimate the raw materials cost to be in the range of \$15 to \$20 per gallon when produced in 50-gallon batches. To estimate a production cost and a cost to the supplier or distributor, it is assumed that the rust remover would be supplied in 8 ounce and 16 ounce plastic recyclable containers. Using this basis, the following values were computed:

Item	8 Ounce	16 Ounce
Raw Materials (based on \$18/gallon)	\$1.13	\$2.25
Package and Label	0.40	0.50
Shipping (24 items/box)	--	0.70
Shipping (48 items/box)	0.35	--
	<hr style="width: 50%; margin: 0 auto;"/>	<hr style="width: 50%; margin: 0 auto;"/>
Total Principal Base Cost:	\$1.88	\$3.45

Overhead costs have not been included in these estimates since market size is unknown at this point. For purposes of this report, a value of \$0.25/item for the 8 ounce containers and \$0.50/item for the 16 ounce containers has been used. Therefore, the production costs for the

rust remover could be \$2.13 for the 8 ounce container and \$3.95 for the 16 ounce container. We have further assumed that the rust remover would be sold at a mark-up of 50% over principal costs. This translates to a selling cost to the supplier or distributor of \$3.20 and \$5.18 respectively. Further assuming that the suggested selling price to the consumer would involve a 50% mark-up above these prices, the final selling price of the rust remover to the end user would be \$4.75 to \$5.00 for the 8 ounce container and \$7.50 to \$8.00 for the 16 ounce container.

Considering the greater effectiveness and low toxicity of this rust remover, these prices are in the general range of existing competitive products:

Product		Cost
Duro Naval Jelly	8 ounces	\$4.00
Duro Naval Jelly	16 ounces	6.00
Lime & Rust Remover	8 ounces	4.00
MRC Rust Remover	8 ounces	4.75
MRC Rust Remover	16 ounces	7.50

The values reported above for the MRC rust remover are estimates. Depending upon market size, the prices presented could be lower than estimated and be closer to those of competitive products. An estimate of minimum investment costs are included in Table 20.

A qualified market survey has not been conducted from which a reliable projection could be generated on the market size for the MRC rust remover. A low-end estimate has been made, however, based upon conversations with experienced marketing analysts. It has been assumed that there are some  $6 \times 10^6$  households in the U.S.A. which would purchase a product of this type once a year. It is reasonable, based upon other products, to assume that with adequate marketing efforts at least one-tenth of the rust-remover market would be captured by the MRC product. This translates to \$400,000 to \$500,000 gross profit in the first year of sales. This estimate is to be considered only as a gross estimate of the immediate prospect for the MRC rust remover.

**Table 20. Estimate of Costs**

This estimate is based on a company that is staffed by two sales persons who work on a small salary plus commission, two minimum wage workers and a full time employee who performs the functions of office manager, book keeper, secretary and receptionist. It is assumed that the start up period (business operates at a loss) will be 1 year. A loan or investment will include start up costs and one years operating expenses.

**Outside Services Startup Expenses:**

Attorney (Articles of Incorporation)	\$ 2000.00
Marketing Consultant	\$ 5000.00
Accountant	\$ 1000.00
Chemists	\$ 2000.00
	-----
	\$10000.00

**Facility Startup Expenses:**

Desk	3 @ \$ 400.00	\$ 1200.00
Chair	3 @ \$ 300.00	\$ 900.00
Table	8 @ \$ 150.00	\$ 1200.00
File Cabinet	3 @ \$ 130.00	\$ 390.00
FAX Machine		\$ 1500.00
Computer for Word Processing		\$ 1500.00
IBM Typewriter		\$ 600.00
Laser Printer		\$ 1200.00
Telephone Connect	3 x \$70.75	\$ 212.00
Hunting System	3 x \$ 30.00	\$ 90.00
Phone Jack Installation	2 hours	\$ 139.00
Electric Hookup		\$ 5.00
Electric Deposit		\$ 200.00
Water		No Fee
Burglar Alarm Installation		\$ 1000.00
Architect		\$ 2000.00
Contractor		\$ 2000.00
Carpenter		\$ 3500.00
Electrician		\$10000.00
Equipment Hookup		\$ 2000.00
Miscellaneous		\$ 2000.00
High Shear Mixer		\$ 1000.00
		-----
		\$32636.00
<b>Total Start Up</b>		<b>\$42636.00</b>

**Table 20. Estimate of Costs (Continued)****Monthly Expenses:**

Facility Rental 3500 sq.ft. @ \$1.00 / sq.ft.	\$ 3500.00
Telephone	\$ 600.00
Electric	\$ 400.00
Water	\$ 100.00
Reproduction Machine Lease	\$ 370.00
Reproduction Machine Supplies	\$ 100.00
Drinking Water Cooler	\$ 21.00
Janitorial	\$ 150.00
Carpet Cleaning	\$ 27.00
Air Conditioning Service	\$ 20.00
Air Conditioning Maintenance Agreement	\$ 20.00
Postage Meter	\$ 24.00
Postage	\$ 200.00
Express Mail	\$ 200.00
Burglar Alarm System	
Monthly Service Charge	\$ 70.00
Payroll and Payroll Accounting	\$ 50.00
Quarterly Accounting	\$ 750.00
Magazine Advertising	\$ 2000.00

**Employee Wages**

Multi Function Office Manager (\$ 30,000/yr)	\$ 2500.00
Laborer (2 @ 4.35/hr. x 160 hrs.)	\$ 1392.00
Sales Person (2 @ \$ 30,000 + Commission)	\$ 5000.00
Chemist/Tech Manager (1/2 time @ 42,000/yr)	\$ 3500.00

**Employee Benefits (35% of Employees Salary)**

Multi Function Office Manager (.35 x \$ 2500.00)	\$ 875.00
Laborers (0.35 x \$ 1392.00)	\$ 487.00
Sales Persons (0.35 x \$ 5000.00)	\$ 1750.00
Chemist/Tech Manager (0.35 x \$ 3500)	\$ 1225.00

**Insurance**

Fire, Liability and Product Liability (\$ 5000/yr)	\$ 417.00
Workman's Compensation (\$ 5000/yr)	\$ 417.00
	-----
	\$31586.00

Total Start Up Plus First Months Expenses	\$74222.00
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## SECTION IV

### CONCLUSIONS AND RECOMMENDATIONS

The following summarizes the accomplishments, results, and conclusions drawn from this SBIR Phase II effort. The results of this program indicate that the AEDC duct corrosion problem cannot be solved with a polymer coating system unless abrasive blasting is used to remove the current corrosion. MRC has developed a coating material which would provide the required protection and survivability. However, an acceptable cleaning and surface preparation technique could not be developed for the B-Header. An organic acid cleaner was developed which has significant commercial potential, but which could not produce a satisfactory surface in the duct system. The corrosion in the B-Header is extremely tough and not easily "grown" or simulated in the laboratory.

- o MRC has formulated an aluminum-filled silicone resin coating (MARB) which is expected (based upon laboratory testing) to survive the duct environments for an acceptable period of time. The binder formulation was optimized with specimens applied to HCl-prepared panels. The aluminum flake content was optimized on specimens prepared with HCl and organic-acid cleaners. The coating has been applied and evaluated on a variety of prepared steel surfaces including detergent wash, solvent, wire brush, glass bead blast, HCl-cleaned, water-blasted, and the MRC organic acid cleaners. The specimens have been conditioned by many environmental cycles (-120°F, 600°F, and high humidity). (The cycling parameters were reviewed by the AEDC support contractors and found to be an acceptable simulation of the duct conditions). Adhesion and scratch tests have been conducted to determine the quality and condition of the coating and to determine the degradation as a function of time/accelerated aging. There is no major difference in performance as a function of surface preparation as long as the surface is "clean". Similar performance is obtained on surfaces prepared with HCl, organic-acid cleaner, water blasting, and glass bead blast. Tests have shown the cohesive strength of the MARB to be superior to commercial products. MRC developed a technique for repairing damaged areas of the aluminum-filled paint.
- o A chemical cleaner has been successfully formulated which has proven to be very effective in our laboratory studies. The cleaner will remove the rust from all three types of steel used in our program. Its performance (time to clean) is dependent upon the thickness and type of rust. The cleaner dissolves the FeO layer which is located between the steel surface and the higher oxide layers such as Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub>. In order for the cleaner to effectively work, it must come in contact with the FeO layer. The corrosion in the B-Header appears to be too thick and tough for the cleaner to soak through. A cleaning technique combining the cleaner with a mechanical method might be effective.
- o Water blasting was evaluated as a technique for removing the duct corrosion. Water blast systems are available which can remove the corrosion. However, these systems are slow, labor intensive, large, and expensive. These factors eliminate them as potential cleaning candidates.

As in any research program, many of the results and conclusions are based on tests and experiments which did not work, thereby eliminating or reducing the number of candidate materials and techniques. The following briefly summarizes studies that we conducted and the (mostly negative) results:

- o **Multi-layer coatings were evaluated. The inner coats included zinc ethyl silicates and high temperature paints. The outer layers included aluminum and zinc-filled silicone resin blends (both commercial and MRC formulated). We attempted to design a multi-layered silicone resin coating system which used zinc powder near the steel interface and which was flexible to adhere well to the expanding/contracting steel. This was coated with a more rigid, harder aluminum-filled layer to protect against abrasion. These concepts continually failed at the interface between the two coating layers.**
- o **Zinc ethyl silicates were evaluated but showed cohesive failures after thermal/ environmental cycling.**
- o **Zinc-filled silicone resins were evaluated, but exhibited cracking when exposed to low temperatures. This was not true of the aluminum-filled coatings. Because the zinc was in powder form and the aluminum in flake form, we believe that the aluminum provides a leafing action which reduces the potential for cracking. Zinc flake is no longer being produced in this country.**
- o **Several commercial cleaners were evaluated. All were slow performing and left an undesirable residue.**
- o **An innovative cleaning technique was postulated and attempted. This involved coating the corroded steel panel with a "conversion coat" or some other material which was then mechanically removed, hopefully leaving a clean surface. This technique did not work.**
- o **We evaluated a number of "replenishable" concepts. This coating is an inexpensive material which would inhibit any corrosion but would gradually wear away and need to be reapplied at known time intervals. None of the candidate materials showed promise.**
- o **Failure mode studies have been conducted on the MARB. Specimens were prepared with pin head sized rust locations. As the rust expanded, the paint blistered and finally failed. The failed paint should come off in flecks or as dust.**

## REFERENCES

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## APPENDIX A

### Specifications For Cleaner and Coating Materials

#### MATERIAL SPECIFICATION

#### PAINT, ALUMINUM, COLD/ HEAT-RESISTING -80°C to +360°C (-112°F to +680°F)

This specification is proposed for use by the United States Air Force and is available for use by all Departments and Agencies of the Department of Defense.

#### 1. SCOPE

1.1 **Scope:** This specification covers a composition of aluminum pigmented paint which, when cured as required, serves as a protective coating to prevent rusting of low carbon steel that is subjected to cycling of humidity and temperatures between -80 and +360°C and tangential air flow exposure.

1.2 **Classification.** Cold/heat-resisting aluminum paint covered by this specification shall be of the following compositions, as specified (see 6.2):

Composition G - General use (see 6.2).

Composition L - Limited use (see 6.4).

#### 2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

#### SPECIFICATIONS

##### FEDERAL

TT-P-320 - Pigment, Aluminum; Powder and Paste for Paint.  
- Thinner, Solvent; Xylene

PPP-P-1892 - Paint, Varnish, Lacquer, and Related Materials; Packaging, Packing, and Marking of.

##### MISSION RESEARCH CORPORATION (MRC)

MS-Draft - Rust Remover, Gel, Organic Acid Type.

#### STANDARDS

##### FEDERAL

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

FED-STD-313 - Symbols for Packages and Containers for Hazardous Industrial Chemicals and Materials.

##### MRC PROCESS

PS-9101 - Organic Gel and Rust Cleaner Acceptability

PS-9102 - Aluminum protective coating paint

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

**2.2 Other Publications.** The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

**AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)**

- D93 - Flash Point by Pensky-Martens Closed Tester.
- D480 - Testing Aluminum Powder and Paste.
- D562 - Consistency of Paints Using the Stormer Viscosimeter.
- D1014 - Exterior Exposure Tests of Paints on Steel.
- D1200 - Ford Viscosity Cup.
- D1210 - Fineness of Dispersion of Pigment-Vehicle Systems.
- D1310 - Flash Point, °F (Fisher-Tag Open Cup).
- D1475 - Density, g/cc Average at 77°F.
- D1640 - Drying, Curing, or Film Formation of Organic Coatings at Room Temperature.
- D2196 - Viscosity, cps (avg. at 77°F), Brookfield RV-T Spindle #3 @ 2 RPM.
- D2369 - Volatile Content of Paints.
- D2698 - Pigment Content of Solvent-Type Paints by High Speed Centrifuging.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103).

(Technical society and technical association specifications and standards are generally available from reference libraries. They are also distributed among technical groups and using Federal agencies.)

**3. REQUIREMENTS**

**3.1 Material.** The paint furnished under this specification shall consist of ingredients conforming to the quantitative requirements specified in table 1 when tested in accordance with 4.3.

TABLE 1. Quantitative requirements.

Characteristics	Requirements	
	Minimum	Maximum
Pigment, percent by weight of paint	35	48
Drying time, hours:		
Set to touch, air dry	0.5	1
Dry hard, air dry	2	6
Full hardness, baked at 250°C	0.5	1
Flash point, °C	38	--

**3.2 Composition.**

**3.2.1 Pigment.** The pigment shall be a leafing type aluminum powder conforming to type I or type II, class A of TT-P-320.

**3.2.2. Vehicle.** The vehicle shall be a silicone resin blend together with driers, if necessary, and volatile aromatic or aliphatic-type solvents to meet the requirements of this specification. (Toluene and Xylene are recommended).

**3.2.2.1 Solvent (composition L only).** The solvent portion of composition L shall conform to the following requirements when tested as specified in 4.3.2 (see 6.4).

- (a) Aromatic compounds with eight or more carbon atoms except ethyl benzene: 8 percent maximum.
- (b) Toluene: 20 percent maximum.
- (c) Solvents with an olefinic or cyclo-olefinic type of unsaturation: negative test.
- (d) Ketones: negative.
- (e) Total of (a) + (b): 20 percent maximum.

**3.3 Qualitative requirements.** The paint shall meet the following qualitative requirements:

**3.3.1 Condition in container.** A freshly opened full container of the paint tested as specified in 4.3.4 shall be free from grit, skins, lumps, abnormal thickening, or gelling and shall show no more pigment settling than can be reincorporated by hand to a smooth homogeneous state.

**3.3.2 Storage stability.**

**3.3.2.1 Partially full container.** A three-quarter filled, closed 8-ounce glass jar of the paint shall show no skinning when tested as specified in 4.3.5.1. After aging as specified in 4.3.5.1, the paint shall show no livering, curdling, hard caking, or tough gummy sediment. It shall mix readily to a smooth uniform state, and any skin formed shall be continuous and easily removed.

**3.3.2.2 Full container.** A full quart can of the paint shall show no gas pressure in the can, no skinning, hard dry caking, or tough gummy sediment when tested as specified in 4.3.5.2. The paint shall mix readily to a smooth uniform state.

**3.3.3 Brushing properties.** The brushing properties when tested as specified in 4.3.6 shall produce a smooth uniform film consistency free from runs, sags, or streaks.

**3.3.4 Spraying properties.** The paint, tested as specified in 4.3.7, shall spray in all respects as required herein and shall show no running, sagging, or streaking. The dried film shall show no dusting or mottling and shall present a smooth uniform finish free from solvent eyes.

**3.3.5 Heat resistance.** Films of paint prepared and tested as specified in 4.3.8 shall show no cracking, blistering, flaking, or peeling. When cut as specified in 4.3.8 the paint shall adhere tightly to the metal.

**3.3.7 Toxicity.** The paint shall contain no benzene (benzol), methanol, chlorinated compounds, hydrolyzable chlorine derivatives, or other ingredients which are deemed toxicologically hazardous under normal conditions of usage.

**3.3.11 Flash point.** The material shall have a minimum flash point of 38°C when tested in accordance with 4.3.10.

#### **4. QUALITY ASSURANCE PROVISIONS**

**4.1 Responsibility for inspection.** Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable

for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

**4.2 Quality conformance inspection.** Quality conformance testing of individual lots shall consist of all the tests of 4.3.

**4.2.1 Sampling and inspection.** Sampling and inspection shall be in accordance with method 1031 of FED-STD-141.

**4.3 Test procedures.**

**4.3.1 Test conditions.** The routine and referee testing conditions shall be in accordance with section 7 of FED-STD-141 except as otherwise specified herein. The dry film thickness shall be measured after the coated panel has been baked at  $204 \pm 2^\circ\text{C}$  for 15 minutes.

**4.3.2** The tests listed in Table II shall be conducted in accordance with FED-STD-141 or as required by this specification.

TABLE II. Tests.

Test	FED-STD-141	ASTM	Paragraph	Requirement
Sampling Al Powder		D 480		Table 1
Flash Point		D 1310		Table 1
Viscosity		D 2196		Table 1
Viscosity		D 1200		Table 1
Dispersion		D 1210		
Drying, Curing at RT		D 1640		Table 1
Total solids		D 2369		Table 1
Pigment content		D 2698		Table 1
Isolation of vehicle		D 2698		
Vehicle solids		D 2698		Table 1
Consistency		D 562		Table 1
Drying time:				
Set to touch	4061		4.3.5.1	Table 1
Dry hard	4061		4.3.5.1	Table 1
Full hardness			4.3.5.2	Table 1
Condition in container	301		4.3.6	3.3.1
Storage stability:				
Partially full container	3021		4.3.7.	3.3.2.1
Full container	3022		4.3.7.2	3.3.2.2
Brushing properties	4321		4.3.8	3.3.3
Spraying properties	433		4.3.9	3.3.4
Heat resistance:				
Hot rolled steel			4.3.8.1	3.3.5
Cold rolled steel			4.3.8.2	3.3.5
Weather resistance		D 1014	4.3.14	3.3.9
Flash point ( $^\circ\text{C}$ )		D 93	4.3.17	3.3.12

### 4.3.3 Drying times.

4.3.3.1 Air drying. Draw down a film of the paint on a glass panel using a 0.038 mm applicator (0.076 mm gap clearance) and determine air drying time under referee conditions in accordance with Method 4061 of FED-STD-141 for compliance with Table I.

4.3.3.2 Full hardness. Determine full hardness by spraying a film of paint to a dry thickness of 0.1 mm on a steel panel that has been solvent cleaned with the aliphatic naphtha-ethylene glycol monoethyl ether mixture in accordance with Method 2011 of FED-STD-141. Air dry 30 minutes and then bake at  $240 \pm 15^\circ\text{C}$  for 1 hour. Determine compliance with full hardness requirement of Table I. The film shall be considered to have reached full hardness when it is tested to ASTM D-3363.

4.3.4 Condition in container. Determine package condition on acceptance testing in accordance with method 3011 of FED-STD-141 and observe for compliance with 3.3.1. Evaluate pigment settling or caking by proceeding as in method 3011 of FED-STD-141 but do not stir or shake.

### 4.3.5 Storage stability.

4.3.5.1 Partially full container. Determine skinning after 48 hours in accordance with method 3021 of FED-STD-141 and observe for compliance with 3.3.2.1. Reseal and age for seven days at  $60^\circ\text{C}$  and observe for compliance with 3.3.2.1.

4.3.5.2 Full container. In accordance with method 3022 of FED-STD-141, allow a full standard quart can of the paint to stand undisturbed for one month and then examine the contents. Evaluate pigment settling or caking as in 3.3.1

4.3.6 Brushing properties. Apply the paint as packaged using a 2-1/2 inch brush and observe for brushing properties in accordance with method 4494 of FED-STD-141 except make the drawdown a minimum of 254 mm long on clear plate glass. The 0.102 mm strip shall not make contact with the next thicker strip at any point within the 140 mm central portion of the blade path.

4.3.7 Spraying properties. Spray the paint as packaged or thinned with not more than five percent by volume of thinner conforming to TT-T-306 on a steel panel to a dry film thickness between 0.05 and 0.10 mm and observe for spraying properties in accordance with Method 4331 of FED-STD-141 for compliance with 3.3.4. For referee test use automatic application in accordance with Method 2131 of FED-STD-141.

### 4.3.8 Heat resistance.

4.3.8.1 Hot rolled steel. Select ten panels which are completely free of loose mill scale and have been cut to 76 mm by 152 mm from 14 gauge as rolled commercial quality low carbon steel conforming to ASTM A203. Solvent clean with the aliphatic naphtha-ethylene glycol monoethyl ether solvent mixture of method 2011 of FED-STD-141. Spray the paint (one or two coats as necessary) on all eight panels to a total dry film thickness between 0.05 to 0.10 mm and air dry for at least 24 hours. Place the panels in an oven on a rack or holder so that no part of the panels is in direct contact with the bottom or sides of the oven and then subject to the following heating schedule:

	Temperature, °C
8 hours (first day)	200
16 hours (overnight)	260
8 hours	320
16 hours	380
8 hours	440
16 hours	500
8 hours	550
16 hours	600
8 hours	650

Remove the panels from the furnace for as short a period of time as possible and inspect (except for the knife test) for compliance with 3.3.5 at the end of each heating period.

Remove two panels at the end of the 260°C, 320°C, 500°C heating periods for use in the salt spray test (see 4.3.11). At the conclusion of the heating schedule remove the remaining two panels, allow to cool in air, cut with a knife blade, and observe for compliance with 3.3.5.

4.3.8.2 *Cold rolled steel.* Spray the paint to a dry film thickness between 0.05 and 0.10 mm on two 76 mm by 152 mm steel panels that have been solvent cleaned as in 4.3.10.1. Apply the paint to the side of the panel that has been flat polished as described in note 1 to method 2011 of FED-STD-141. Air dry 30 minutes and then bake at  $202 \pm 2^\circ\text{C}$  for 1 hour. Cool to room temperature. Place the panels on a rack as in 4.3.8.1 and heat for 24 hours in an oven that has been previously raised to a temperature of  $550 \pm 4^\circ\text{C}$ . Remove from the oven, cool, and inspect for compliance with 3.3.5 performing the knife test as in 4.3.8.1.

4.3.9 *Toxicity.* The manufacturer shall certify that the paint contains no benzene (benzol), methanol, chlorinated solvents or other ingredients which are deemed to be toxicologically hazardous under normal conditions of usage.

4.3.10 *Flash point.* Flash point determination is made in accordance with ASTM D 93. The flash point shall be in accordance with 3.3.12.

4.4 *Inspection of preparation for delivery.* Sample packages and packs and the inspection of packaging, packing, and marking for shipment and storage shall be in accordance with the requirements of Section 5 and the documents specified therein.

## 5. PREPARATION FOR DELIVERY

(The preparation for delivery requirements specified herein apply only for direct Government procurements. For the extent of applicability of the preparation for delivery requirements of referenced documents listed in section 2, see 6.5.)

**5.1 Packaging, packing, and marking.** Paint shall be furnished in 1-quart or 1-gallon multiple friction closure type cans or in 5-gallon lug can or steel pails as specified (see 6.2). Packaging shall be Level A, B, or C as specified (see 6.2); packing Level A, B, or C as specified (see 6.2) and marking shall be in accordance with PPP-P-1892.

**5.2 Special markings.** In addition to the markings required by PPP-P-1892, interior containers shall be identified in accordance with FED-STD-313 and change(s) thereto. Each container shall also be marked or labeled with the following statement:

**"CAUTION - USE ONLY WITH ADEQUATE VENTILATION.  
AVOID CONTACT WITH SKIN AND/OR PROLONGED BREATHING OF VAPOR"**

**5.3 Material Safety Data Sheets.** A copy of the Material Safety Data Sheet, DD Form 1813, of FED-STD-313 shall be attached to each shipping container and palletized load for each shipping destination.

## **6. NOTES**

**6.1 Intended use.** The paint covered by this specification is intended for use on duct walls, boilers, superheater headers, and similar high temperature applications. It is also intended for painting military equipment such as personnel heaters, rocket launchers and other components, where operating temperatures preclude the use of conventional paints.

**6.2 Ordering data.** Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Size of container required (see section 5).
- (c) Level of packaging and packing required.

**6.3** The paint should be purchased by volume, the unit being one United States liquid gallon of 231 cubic inches at 20°C.

**6.4 Air pollution requirements.** Air pollution characteristics of composition L paint shall be in conformance with air pollution Rule 66; Los Angeles County Air Pollution Control District.

**6.5 Sub-Contracted material and parts.** The preparation for delivery requirements of referenced documents listed in Section 2 do not apply when material and parts are procured by the contractor for incorporation into the equipment and lose their separate identity when the equipment is shipped.

## **MRC PROCESS AND TEST STANDARD 9102 (for a MRC developed Protective Coating)**

This process standard outlines the procedures recommended by Mission Research Corporation (MRC) for substrate cleaning, coating application and qualification testing of MRC developed rust removing gel, a commercial corrosion inhibitor, and aluminum pigmented silicone paints.

### **1. SCOPE**

**1.1 Scope.** The procedures included in this standard covers the processing instructions for substrate cleaning and application and testing of MRC's Aluminum Resin Blend (MARB) protective coating. This MRC developed protective coating withstands cycling of humidity, tangent air flow, exposure between -80°C and +360°C and prevents rusting of steel substrates when applied and properly cured as directed in this document. An MRC developed rust cleaning gel, a commercial corrosion inhibitor and zinc primer application are included

**1.2 Classification.** Gel cleaner, corrosion inhibitor and coatings for steel substrates covered by this specification shall be of the following compositions, as specified in 3.1:

- Composition G - Organic gel rust remover and substrate cleaner.
- Composition C - Commercial corrosion inhibitor in rinse water.
- Composition MAZRB - Zinc filled primer for fast rusting substrates.
- Composition MARB4 - Standard 40% filled coating formulation.

**1.3 Testing.** Batch testing follows ASTM procedures as described in Section 4.4 for the general and physical properties outlined. Qualification testing also includes health and safety issues as described in 4.4.

### **2. APPLICABLE DOCUMENTS**

**2.1 Issues of documents.** The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

### **SPECIFICATIONS**

#### **FEDERAL**

- TT-P-320 - Pigment, Aluminum; Powder and Paste for Paint.
- PPP-P-1892 - Paint, Varnish, Lacquer, and Related Materials; Packaging, Packing and Marking of.

#### **MRC**

- DRAFT2 - Paint, Aluminum, Cold/ Heat-Resisting (-80 to +360oC).
- DRAFT1 - Rust Remover, Gel, Organic Acid Type

### **STANDARDS**

#### **FEDERAL**

- FED-STD-141 - Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling, and Testing.
- FED-STD-313 - Symbols for packages and containers for Hazardous Industrial Chemicals and Materials.

**2.2 Other Publications.** The following documents form a part of this standard to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

**AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)**

A516	Pressure Vessel Plates, Carbon Steel, for Moderate and Lower-Temperature Service. Grade 70, High Strength
A203	Pressure Vessel Plates, Alloy Steel, Nickel. Grade E, Intermediate Tensile Strength.
A285	Pressure Vessel Plates, Carbon Steel. Grade C, Intermediate Tensile Strength
D 16	Paint and Related Products, Standard Definitions of Terms Relating to.
D 609	Paint and Related Products, Preparation of Steel Panels for Testing.
D 610	Rusting on Painted Steel Surfaces, Evaluating Degree of.
D 661	Cracking of Exterior Paints, Evaluating Degree of.
D 823	Producing Films of Uniform Thickness of Paint and Related Products on Test Panels. Test Method A.
D1186	Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to a Ferrous Base. Test Method A.
D2197	Adhesion of Organic Coatings by Scrape Adhesion
D2246	Finishes on Primed Metallic Substances for Humidity - Thermal Cycle Cracking.
D2485	Evaluating Coatings Designed to be Resistant to Elevated Temperatures During Their Service Life. Method A.
D3276	Standard Guide for Painting Inspectors-Metal Substrates.
D3630	Determining Constituents Classified as Hazardous Contained in Protective Coatings.
D3924	Determining VOC of Paints and Related Coatings.
D4414	Measurement of Wet Thickness by Notch Gages. Procedure A
D4417	Surface Profile of Blast Cleaned Steel, Field Measurement of. Method B.
D4541	Pull-Off Strength of Coatings Using Portable Adhesion Testers. Annex A1

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

### **3. REQUIREMENTS**

**3.1 Materials.** The following compositions are a part of this standard to the extent noted.

Composition G consists of organic acids which effectively removes rust and coatings by under film attacking and/or dissolving. MRC can supply this cleaner in plastic containers.

Composition C is a commercial corrosion inhibitor which effectively prevents flash rusting and can be painted over (unlike Sodium Nitrite inhibitors). A Qualified Products List (QPL) commercial source is Sharp Corp.

Composition MAZRB is a variation of MARB4 containing zinc powder and is most effective as a thin primer coat to prevent undercoat rusting.

Composition MARB4 is a leafing aluminum filled, extreme temperature range, protective coating. The paint furnished under this standard shall consist of ingredients conforming to the quantitative requirements specified when tested in accordance with 4.4. The generic type is a high and low temperature hard silicone resin binder blend with the characteristics of a outstanding protective coating when properly applied and cured as a rust corrosion barrier from the detrimental effects of humidity cycling, abrasive air flow, and temperature extremes from -100 to +650° F. May be used in air flow steel ducts of low-carbon steel susceptible to rusting.

3.2 Definitions. ASTM D16 and D3276 governs the terms and inspector requirements. The Bechtel manual may be used for field inspection procedures.

#### 4. PROCEDURES.

4.1 Surface Preparation. ASTM A516, A203, and A285 steel is acid cleaned of all rust and mill scale to a white profile. Steel with a tightly bound mill-scale coating must be cleaned with HCL or, for A203, Aqua Regia acids. Rust can be removed with composition G. The organic gel is time and temperature dependent in cleaning capability. Light rust is dissolved in 5-15 minutes, medium rusting is removed in 1 to 4 hrs, and heavy rusting is attacked after access to the substrate in 8 hrs, loosening by getting under the layer, with reapplication as required to remove the rust. Rerusted coated panels are cleaned by high pressure (minimum 20,000 psi) water jet blasting with composition C, a corrosion inhibitor, in the final rinse. The clean substrate is dried quickly and thoroughly by forced air before coating. ASTM D609 and D4417 (using the Method B comparator) describes the surface required for the substrate.

4.2 Coating Application. Spray application is recommended to a maximum of 0.5 mils dry (at 50% solvent this may be 1.0 mil wet) for the first coat and 1.0 mil dry for additional coats to a maximum dried buildup of 5.0 mils with one hour time elapse between coats. The following sprayers are recommended:

De Vilbiss MBC, tip FX, cap 704  
Graco 205-162, orifice .013,PSI

D823 describes the method in producing coating films on substrates. The wet thickness is determined with D4414 gauge.

Shelf Life should exceed 2 months. If the material is gelling it can not be reconstituted and should be discarded. Do not shake or the leafing action could be altered.

Coverage	600 ft <sup>2</sup> /gal
Tack-free Time	10 minutes
Dry time	see cure schedule

Remove all volatile solvents before heat curing.

4.3 Curing. The recommended curing schedule for the coating is one hour air drying followed by a slow (up to 12 hours) heat ramping to 487°F maximum held for one hour. This ensures the high temperature and ablative resistance to abrasion and deterioration.

Recommended duct wall cure: 1-2 hours minimum between layers for overnight drying with a slow (up to 1 day) heatup rate to 450°F. Hold for one hour and cool slowly. The airflow must be clean, free from contaminants and of a low velocity.

**4.4 Testing.** The physical properties will be tested:

The VOC of composition MARB4 is determined by ASTM D3924

D3630 determines that hazardous constituents are not present in the coatings.

**Cured Properties:** The dry film thickness, is determined by the banana gage described in ASTM D1186

Adhesion to steel, scrape	Balanced Beam	2000 g	ASTM D2197
Adhesion to steel	Elcometer	>1000 psi	ASTM D4541
Resistance to environment,	Humidity cycles in chamber		ASTM D2246
	Hot and Cold Chamber		ASTM D2485

**4.5 Repair.** The recommended repair of failed coating sections less than 1 square foot is to remove all coating and adjacent area by plastic wheel (3M Scotch) on a drill motor. Clean to a white surface, brush and solvent clean of all contaminates. The degree of failure is determined by D610 and D661.

# MATERIAL SPECIFICATION

## RUST REMOVER, GEL, ORGANIC ACID TYPE

This specification is recommended for use by the United States Air Force and is available for use by all Departments and Agencies of the Department of Defense.

### 1. SCOPE

**1.1 Scope.** This specification covers a mixture of organic acids specifically formulated for removing corrosion and deteriorated coatings from duct walls by adhering, dissolving and undermining the rust layer.

**1.2 Classification.** The formulation consists of organic chemical acids specifically selected to clean rust by undermining and lifting the rust from the substrate. The gel covered by this specification shall be of the following composition:

Composition G - General use (see 3.1).

Composition E - Extended strength (see 3.2.1).

### 2. APPLICABLE DOCUMENTS

**2.1 Issues of documents.** The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

#### STANDARDS

##### FEDERAL

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

FED-STD-313 - Symbols for Packages and Containers for Hazardous Industrial Chemicals and Materials.

##### MISSION RESEARCH CORPORATION

MRC-PS 9101 - Organic Gel and Rust Cleaner Acceptability, Process for.

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

**2.2 Other Publications.** The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

##### AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

D610 - Rusting on Painted Steel Surfaces, Evaluating Degree of.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

### 3. REQUIREMENTS

**3.1 Material.** The gel furnished under this specification shall consist of ingredients conforming to the quantitative requirements specified in table I when tested in accordance with 4.3.

TABLE 1. Quantitative requirements.

	Minimum	Maximum
Organic Acids	10	20
Silica, (SiO <sub>2</sub> ), %/wt of vehicle solids	8	15

#### 3.2 Composition.

**3.2.1. Ingredients.** The active ingredients shall be composed of organic acids selected for their capability to effectively remove rust but not be detrimental to humans or the environment. The formulations may also contain smaller quantities of surfactants and/or wetting agents to aid the gel solution in penetrating the rust.

**3.2.2. Vehicle.** The vehicle shall be water at a ratio which along with silica forms a gel for the active ingredients to remain on a vertical surface without appreciable sag and meet the requirements of this specification.

**3.3 Qualitative requirements.** The gel shall meet the following qualitative requirements:

**3.3.1 Condition in container.** A freshly opened full container of the gel tested as specified in 4.3.3 shall be free from grit, skins, lumps, abnormal thickening, or dilution separation and show no more settling than can be reincorporated by hand or shaking to a smooth homogeneous state.

#### **3.3.2 Storage stability.**

**3.3.2.1 Partially full container.** A three-quarter filled, closed 8-ounce glass jar of the gel shall show no skinning when tested as specified in 4.3. After aging as specified in 4.3.3.1, the gel shall show no curdling, hard caking, or tough gummy sediment. It shall mix readily to a uniform gel state. Addition of water may be required.

**3.3.2.2 Full container.** A full glass or plastic quart jar of the gel shall show no gas pressure in the container, no skinning, hard dry caking, or tough gummy sediment when tested as specified in 4.3.3.2. The gel shall mix readily to a smooth uniform state.

**3.3.3 Application properties.** The process properties when tested as specified in 4.3.4 shall produce a smooth uniform film.

**3.3.4 Spraying properties.** The gel, tested as specified in 4.3.5, shall spray, using an airless application device, in all respects as required herein and shall show no running, sagging,

or streaking. The gel shall show no separation and shall present a smooth uniform finish free from uncovered areas. The gel shall adhere to the metal in a vertical position without running or sagging.

3.3.5 Toxicity. The gel shall contain no ingredients which are deemed toxicologically hazardous under normal conditions of usage, as described in 4.3.7.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Quality conformance inspection. Quality conformance testing of individual lots shall consist of all the tests of 4.3 with the exception of storage stability (see 4.3.5).

4.2.1 Sampling and inspection. Sampling and inspection shall be in accordance with method 1031 of FED-STD-141.

#### 4.3 Test procedures.

4.3.1 Test conditions. The routine and referee testing conditions shall be in accordance with the attached MRC Standard.

4.3.2 Condition in container. Determine package condition on acceptance testing in accordance with method 3011 of FED-STD-141. Evaluate separation of components if evident and stir in additional water as necessary. Shaking is acceptable.

#### 4.3.3 Storage stability.

4.3.3.1 Partially full container. Determine skinning after 48 hours and observe for compliance with 3.3.2.1. Reseal and age for seven days at 40°C and observe for compliance with 3.3.2.1.

4.3.3.2 Full container. In accordance with method 3022 of FED-STD-141, at ambient temperature, allow a full standard quart glass or plastic jar of the gel to stand undisturbed for one month and then examine the contents. Evaluate water separation or filler settling or caking.

4.3.4 Application Brushing properties. Apply the gel as packaged using a brush or plastic/wood applicator and observe for coverage properties.

4.3.5 Spraying properties. Spray the gel as packaged to a film thickness between 2.5 mm and 3.5 mm and observe for spraying properties, in compliance with 3.3.4.

4.3.6 Cold rolled steel. Apply the gel to a wet layer thickness between 2 and 3 mm on two steel panels that have been solvent cleaned. Mill-scale must be removed. Apply the gel to the side of the panel that has been rusted as described in note 1. After 30 minutes minimum, remove to see effectiveness of rust removal. Inspect for compliance with 2.1.

**4.3.7 Toxicity.** The manufacturer shall certify that the gel contains no benzene (benzol), methanol, chlorinated solvents or other ingredients which are deemed to be toxicologically hazardous under normal conditions of usage.

**4.4 Inspection of preparation for delivery.** Sample packages and packs and the inspection of packaging, packing, and marking for shipment and storage shall be in accordance with the requirements of Section 5 and the documents specified therein.

## 5. PREPARATION FOR DELIVERY

(The preparation for delivery requirements specified herein apply only for preparation for delivery requirements of referenced documents listed in section 2, see 6.5.)

**5.1 Packaging, packing, and marking.** Gel shall be furnished in 1-quart or 1-gallon screw lid plastic containers. Packaging shall be Level A, B, or C as specified (see 6.2) and marking shall be in accordance with PPP-P-1892.

**5.2 Special markings.** In addition to the markings required by PPP-P-1892, interior containers shall be identified in accordance with FED-STD-313 and change(s) thereto. Each container shall also be marked or labeled with the following statement:

"CAUTION - USE ONLY WITH ADEQUATE PRECAUTIONS. SEE MSDS.

**5.3 Material Safety Data Sheets.** A copy of the Material Safety Data Sheet, DD Form 1813, of FED-STD-313 shall be attached to each shipping container for each shipping destination.

## 6. NOTES

**6.1 Intended use.** The gel covered by this specification is intended for use on duct walls, boilers, superheated headers, and similar high temperature applications. It is also intended for military equipment such as personnel heaters, rocket launchers and other components, where conditions preclude the use of conventional phosphoric, chloric or caustic rust removers.

**6.2 Ordering data.** Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Size of container required (see section 5).
- (c) Level of packaging and packing required.

**6.3** The gel should be purchased by volume, the unit being one United States liquid gallon of 231 cubic inches at 20oC.

**6.4 Pollution requirements.** Pollution characteristics of composition gel shall be in conformance with air, waste and water pollution Rule 66; Los Angeles County Air Pollution Control District.

**6.5 Sub-Contracted material.** The preparation for delivery requirements of referenced documents listed in Section 2 do not apply when materials are procured for incorporation by the contractor and lose their separate identity when shipped.

## MRC PROCESS STANDARD 9101

### MRC-PS 9101 - Organic Gel and Rust Cleaner Acceptability, Process for.

This process standard is to be used by Mission Research Corporation (MRC) and the Arnold Engineering Development Center (AEDC) for cleaning rust coated low carbon steel by an organic gel application method.

#### 1. SCOPE

**1.1 Scope.** This specification covers the processing instructions for MRC's Organic acid Rust removing gel Compound (MORC) formulation, with a recommendation to use SHARP Corp Corrosion Inhibitor 104. Included are the materials, supplies, equipment and storage of these products in preparing carbon steel substrate for future coating. This MRC cleaner removes difficult rust from steel when properly applied as directed and tested per the specification. Water blasting to remove loosened rust or may be used in conjunction with the gel method but is not in the scope of this outline. Paint coating and coating repair is also not included in this standard.

**1.2 Classification.** Gel cleaning and corrosion inhibition protection for steel substrates covered by this standard shall be of the following compositions, as specified:

Composition G - Organic gel rust remover and substrate cleaner.  
Composition I - Commercial corrosion inhibitor in rinse water.

**1.3 MRC Standard for Rust Cleaner Acceptability.** The Cleaner, Rust Gel, Organic Acid shall be deemed acceptable according to the test procedures in 3.4.

#### 2. APPLICABLE DOCUMENTS

**2.1 Issues of documents.** The following documents, of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

##### SPECIFICATION, MATERIAL

MRC - Rust Remover, Gel, Organic Acid Type

**2.2 Other Publications.** The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

##### AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

A516 Steel, Low-Carbon, Type 70.  
A203 Steel, Nickel alloy, Low-Carbon, Type C.  
A285 Steel, Low-Carbon, Type A.  
B117 Salt Spray (Fog) Testing, Standard Method of.  
D610 Rusting on Painted Steel Surfaces, Evaluating Degree of.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

### 3. REQUIREMENTS

**3.1 Material.** An amorphous filled organic acid compounded gel of MRC formulation. The gel furnished under this standard shall consist of ingredients conforming to the quantitative requirements specified in table I when tested in accordance with 3.3. The generic type is a low toxicity gel with characteristics of an outstanding rust removing capability when properly applied and removed. May be used in air flow steel ducts of low-carbon steel susceptible to rusting.

**Surface Preparation:** When evaluated by ASTM D 610 as a rusted painted surface. Mechanical scratching may be required for undermining effectiveness. Water blasting may be effective to advance the deterioration process to allow reapplication of gel to do its job in a reasonable time period. Add corrosion inhibitor in final rinse or dry thoroughly before immediate coating.

**Application:** Spatula the gel evenly and with one pass on the vertical surface to a recommended thickness of 1/8". Excess is not needed and wasteful, but reapplication is effective.

#### General Properties:

Shelf Life                      Shake before using      12 months covered  
 Coverage                      Two applications          400 ft<sup>2</sup>/gal  
 Time/ recommended:      12 hours maximum before reapplication.  
 Overnight drying can be removed with a light reapplication.  
 Temperature range for fastest effectivity is 70 ± 15°F.

Table 1. Quantitative Formulation Requirements

Material		g/cc
Tap water	2500 ml	1.00
Organic Acid 1	316 g	
Organic Acid 2	316 g	
Organic Acid 3	1.6 g	
Water Soluble Solvent	474 ml	1.13
Amorphous Filler	253 g	
Surfactant	63 ml	1.40

A Braun Handblender may be used in a 3000 ml glass beaker for a single gallon quantity.

### 3.3 Quality Control Testing

3.3.1. Apply gel for 15 minutes on medium rusted low carbon steel.

3.3.2. Apply gel on vertical, heavily rusted steel panel for 4 hours

Test	Requirement
Look for slumping Look for cleaning	No vertical displacement >50% required cleanliness

**3.3.3. Shake and check for excess or lack of water:**

Test	Requirement
Shake	Water incorporation required.
Thicken	As required to pass slump test.
Thinning	Add water in small quantity to maintain spraying consistency.

**3.4 Testing the cleaner**

3.4.1 The test shall be carried out on low carbon steel, cleaned of mill scale and oils, 2.0 - 4.0 mm thick Pressure Vessel Steel A516 or equivalent which have been rusted according to ASTM B117-85 in a salt-spray chamber operating at 30°C and using a 50 weight percent sodium chloride solution of 7.0 pH, etc.

3.4.2 The gel shall be applied to a thickness of 2.5 - 3.5 mm at 55 - 80°F by brush, spatula, roller or spray application in order to provide a uniform gel film thickness across the plate.

3.4.3 The gel shall be allowed to contact the rust layer for 30 to 60 minutes at 25°C (77°F) and removed with pressurized water and cleaned with a medium bristle brush under the running water.

3.4.4 The panel shall be immediately dried with an air hose and inspected.

3.4.5 The rust removing solution is acceptable if 50% or greater of the original rust has been removed.

**4.0 Identification**

The substrate shall be identified and the condition recorded as to the coating, scale, rust remnants and surface. The environmental conditions and time of application to include temperature, humidity, air flow and effectiveness shall be recorded.

## Duct Wall Cleaning Recommendations

These recommendations outline and define the procedure for removal of loose rust, coatings, mill scale and/or  $Fe_3O_4$  rust which has built up on the duct surfaces over time and because of environmental conditions.

- o Apply a liberal (up to 1/8" thick) amount of gel.
- o Squeegee off after 4-8 hours which will remove loose rust and soften the under layer.
- o At difficult areas, to expose the steel, scratch or impact the tightly adhered material so the gel can be effective faster in lifting off the coating or  $Fe_3O_4$ .
- o Apply gel overnight at 60-80 °F and remove in the morning with a pressured wand hose. If the gel is black and hard, it has been effective but has dried out due to a drying air flow and cold < 50°F or, warm > 90°F temperature extremes and a reapplication of gel will quickly soften the residual material. If colored, the excess may be removed before reapplication.
- o Reapply as necessary over residual difficult to remove spots. The gel is apparently time dependent and temperature sensitive.
- o Any rerusting is easily removed in 5-15 minutes and easily rinsed off with water containing Sharp 104 rust inhibitor and/or force air dried.

**APPENDIX B**  
**Rust Analysis Report**

Technical Report

Prepared for: Mark West, Dave Newander  
Mission Research Corp.

August 28, 1991

Title: Analysis of Surface Scale, Panel X-1

Prepared by:

  
Dr. Ralph Kafesjian

May 25, 1991

ANALYSIS OF SURFACE SCALE, PANEL X-1

Section 7 of Panel X-1 was selected for analysis of the surface oxide layers. It was hoped that this would provide some guidance for modification of the rust removal formulation. Photomicrographs of the surface at 10X and 25X are shown in Fig. 1. Even at low magnification, the heterogeneous nature of the surface is evident.

The Scanning Electron Micrographs (SEM) show similar features. See Fig. 2. Energy Dispersive X-ray (EDXA) analyses for the areas labeled A through D in Fig. 2. are as follows:

AREA	A	B	C	D	Overall
Fe/O ratio	1.9	3.6	2.2	3.4	2.8

Actual values for the oxides are as follows:

Oxide	FeO	Fe <sub>2</sub> O <sub>3</sub>	Fe <sub>3</sub> O <sub>4</sub>
Fe/O ratio	3.5	2.33	2.64

The EDXA analyses data are shown in Fig. 3 - Fig. 7. Chlorine, silicon, and manganese are shown in the overview analysis (Fig. 3) while Areas A and B show no chlorine, again demonstrating the surface variability.

A polished cross section through the scale was prepared using vacuum epoxy potting and standard metallographic techniques. The results are shown in Fig. 8. Even with the vacuum epoxy potting, some of the brittle scale layer tends to be plucked during the polishing operation as shown in the low magnification micrograph at 25X. The various oxide layers become evident as shades of grey in the lower micrographs at 250X. Attempts to identify these layers with EDXA were not successful. This is probably due to their limited extent.

Caplan, et al. discuss the air oxidation of Fe-C alloys at high temperatures and show some structures similar to these

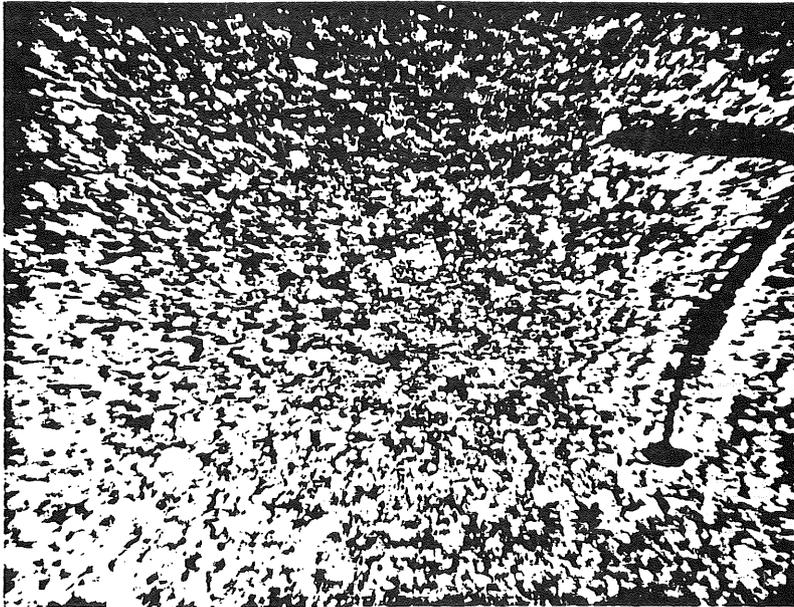
results. (Caplan, D., G.I. Sproule, R.J. Hussey, and M.J. Graham, Oxid. of Metals, 13, 255-273, 1979). See Fig. 5 in the attached reference.

In particular, Caplan et al. note that below 570° C a two-layered scale of  $Fe_3O_4$  and  $Fe_2O_3$  generally formed with  $Fe_3O_4$  next to the metal. Above 570° C an additional layer of wustite ( $FeO$ ) was present next to the metal. It has also been reported that above 400° C, magnetite ( $Fe_3O_4$ ) is oxidized to  $-Fe_2O_3$  (hematite).

Attempts to classify the scaled material based on its magnetic character using a magnet and a nonmagnetic steel were not successful. All the particles of rust were attracted by the magnet while only a dust was retained on the plain steel. A successful separation would allow a more detailed analysis to be readily made. Evaluation of the rust remover formulations on the separated fractions could also provide useful information to guide optimization.

Another approach to this evaluation is to treat the polished scale cross-section using the formulations as etchants. Then, efficacy could be estimated from the degree of etching observed on the various layers. A short trial using this approach was made. The polished section shown in Fig. 9 was etched for 30 seconds with formulation #1\*. The etched section is shown in Fig. 10 at 200X. The darker oxide layer at the metal surface,  $Fe_3O_4$ , was substantially attacked while the  $Fe_2O_3$  shows little or no reaction. The metal surface itself was also etched slightly. This observation tends to substantiate the "lifting" action of this rust remover composition as opposed to a simple dissolution mechanism.

\*See K.G. Mayhan Notebook



10X

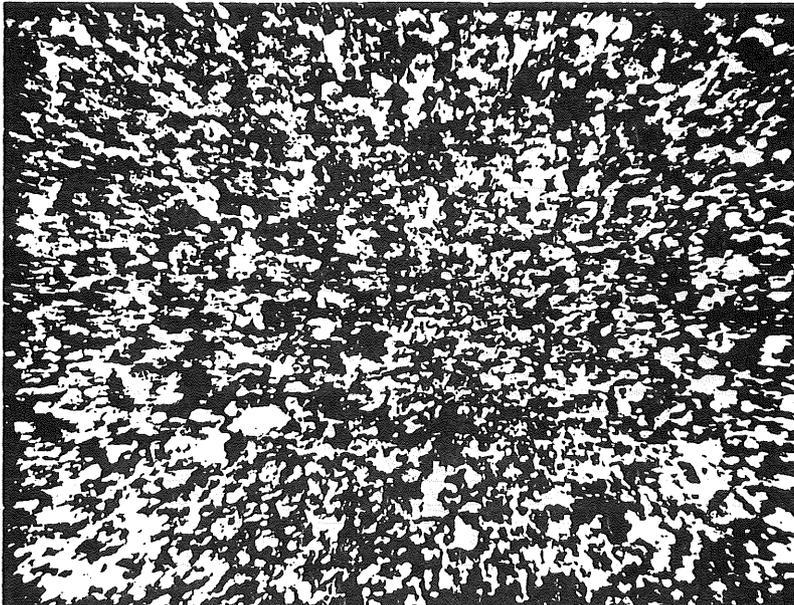


Figure 1. Surface of Panel X-1, Section 7

25X

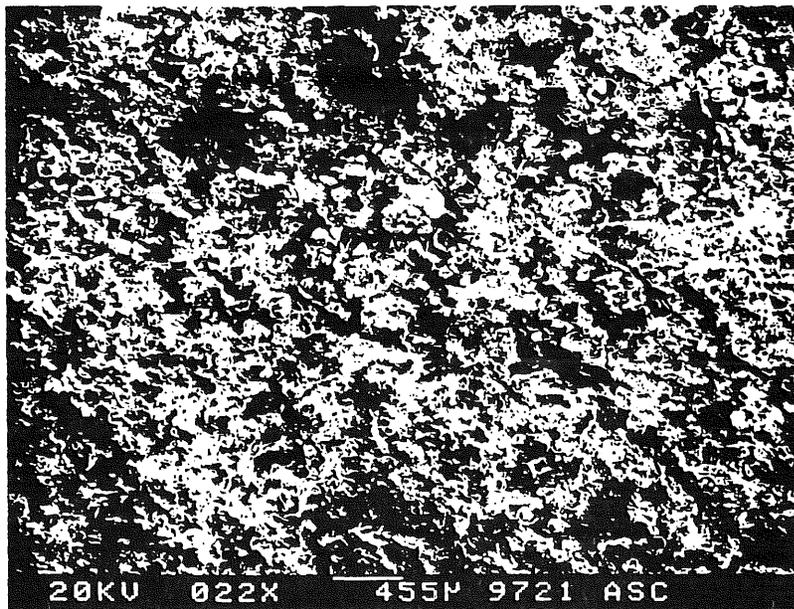
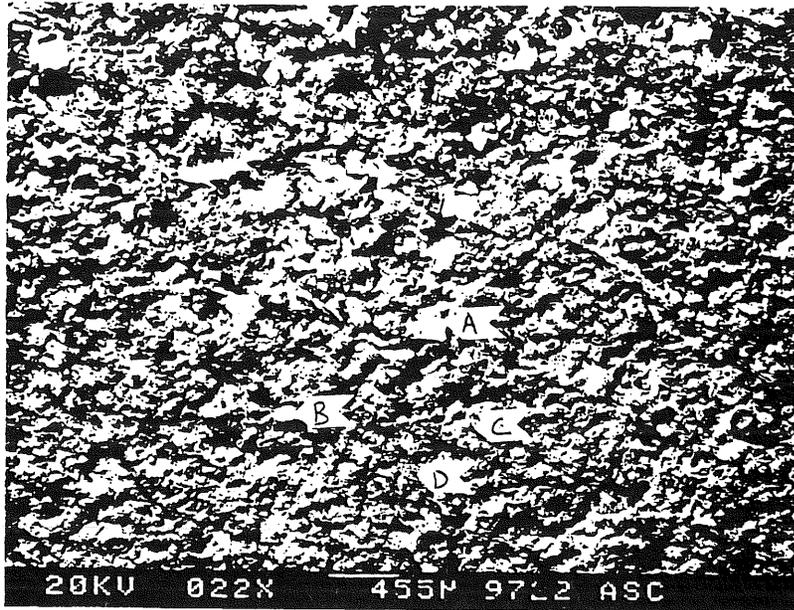


Fig. 2 Scanning Electron Micrographs of the Surface Scle at 22X

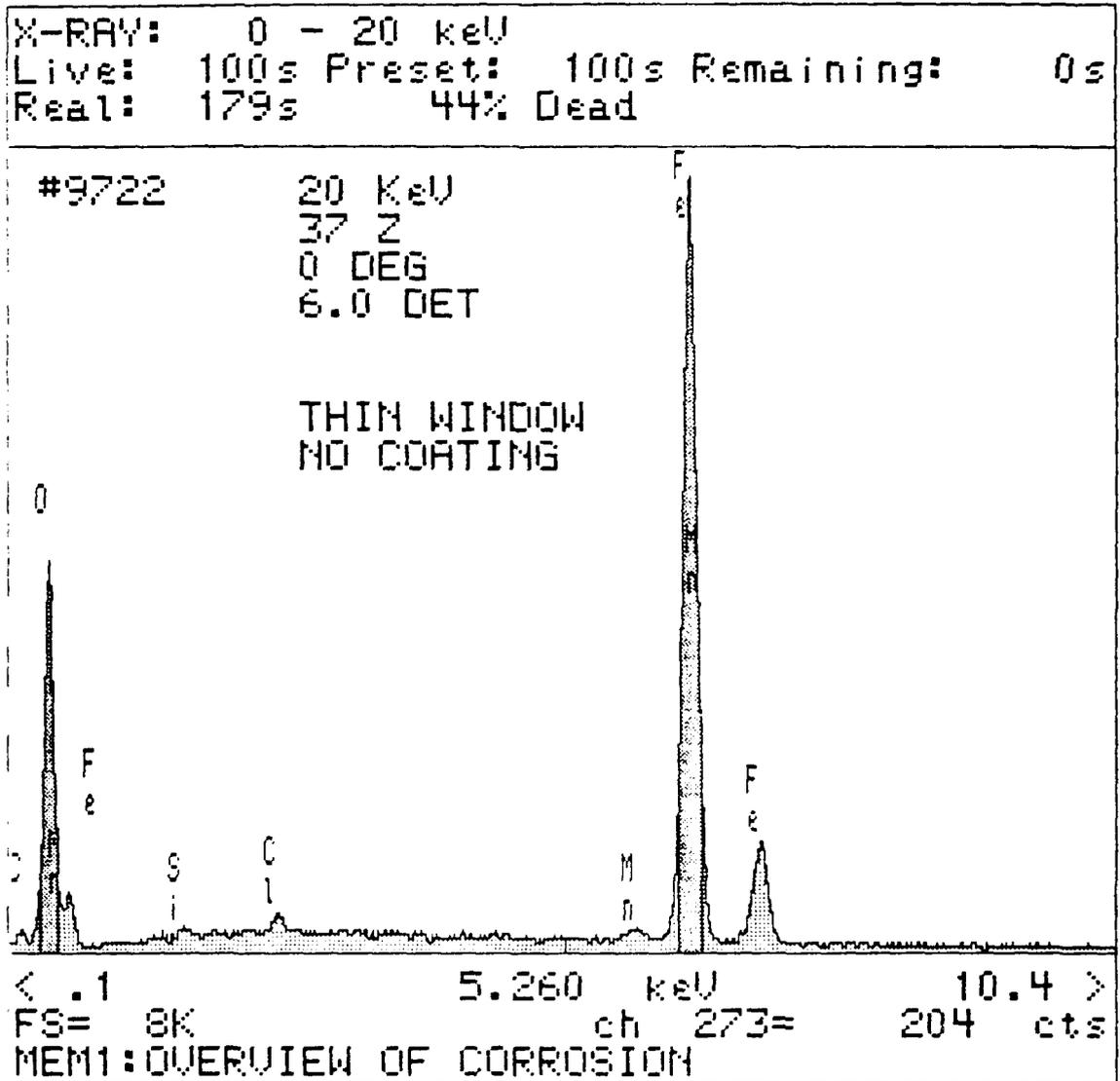


Figure 3.

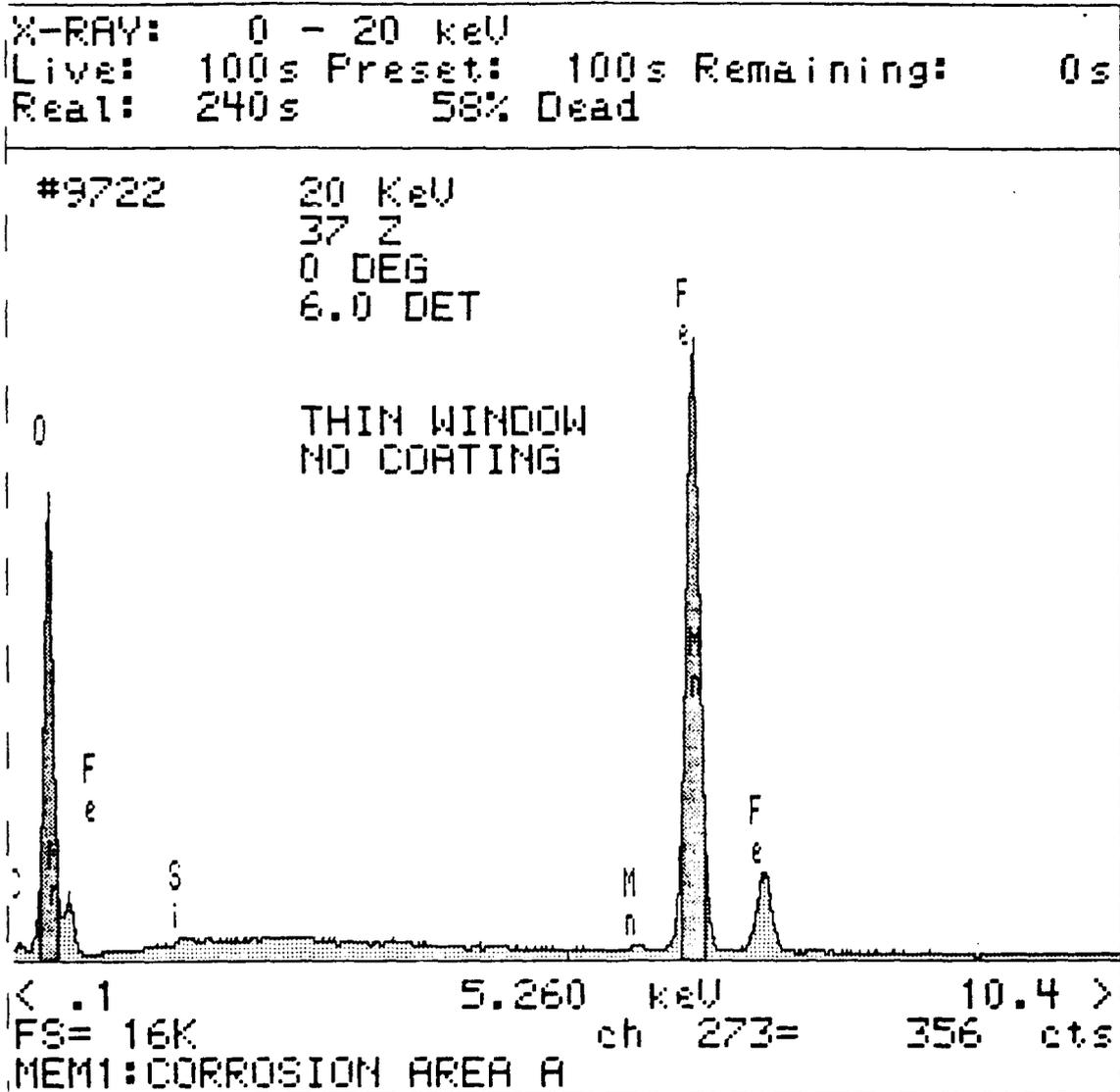


Figure 4.

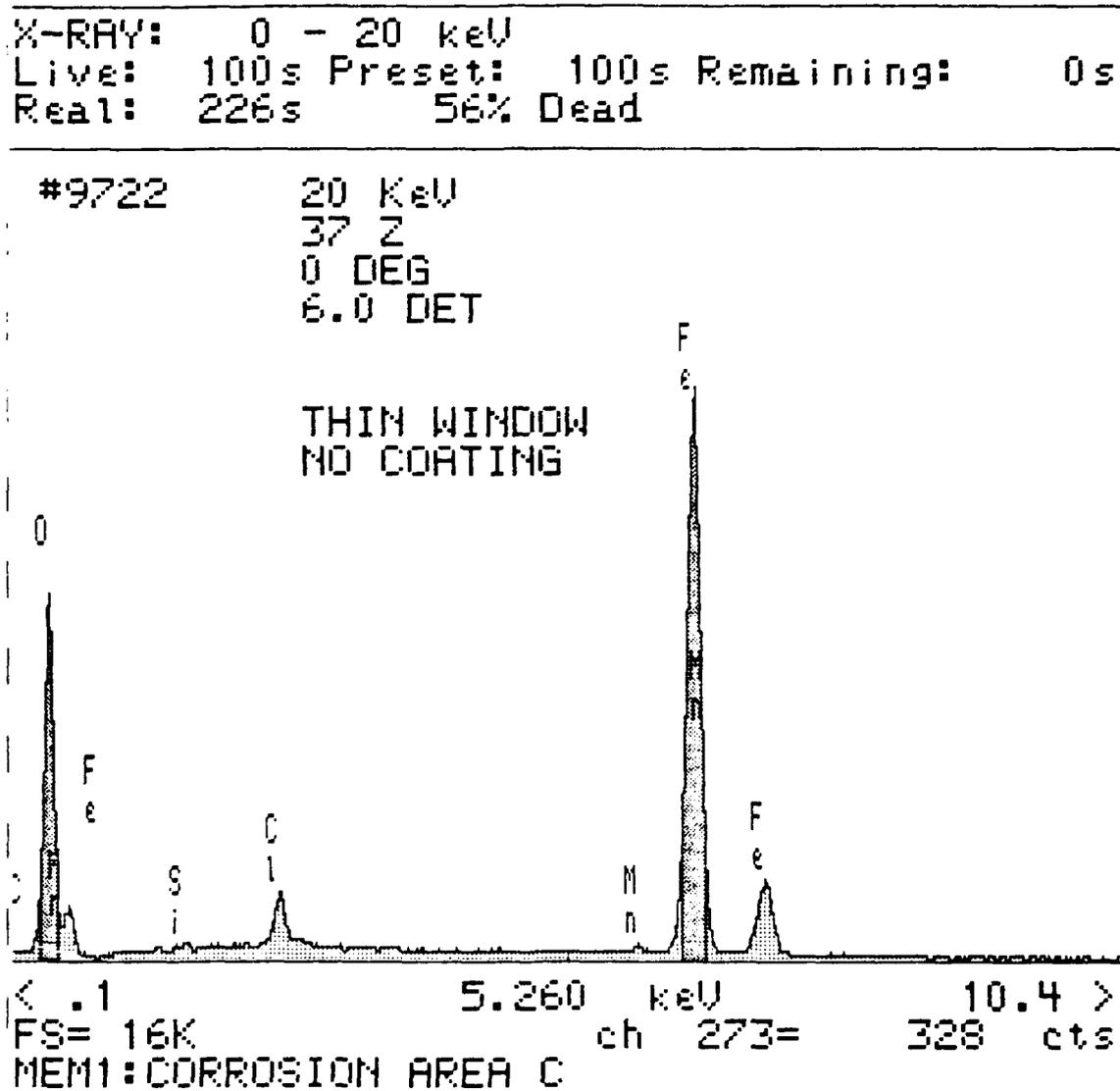


Figure 5.

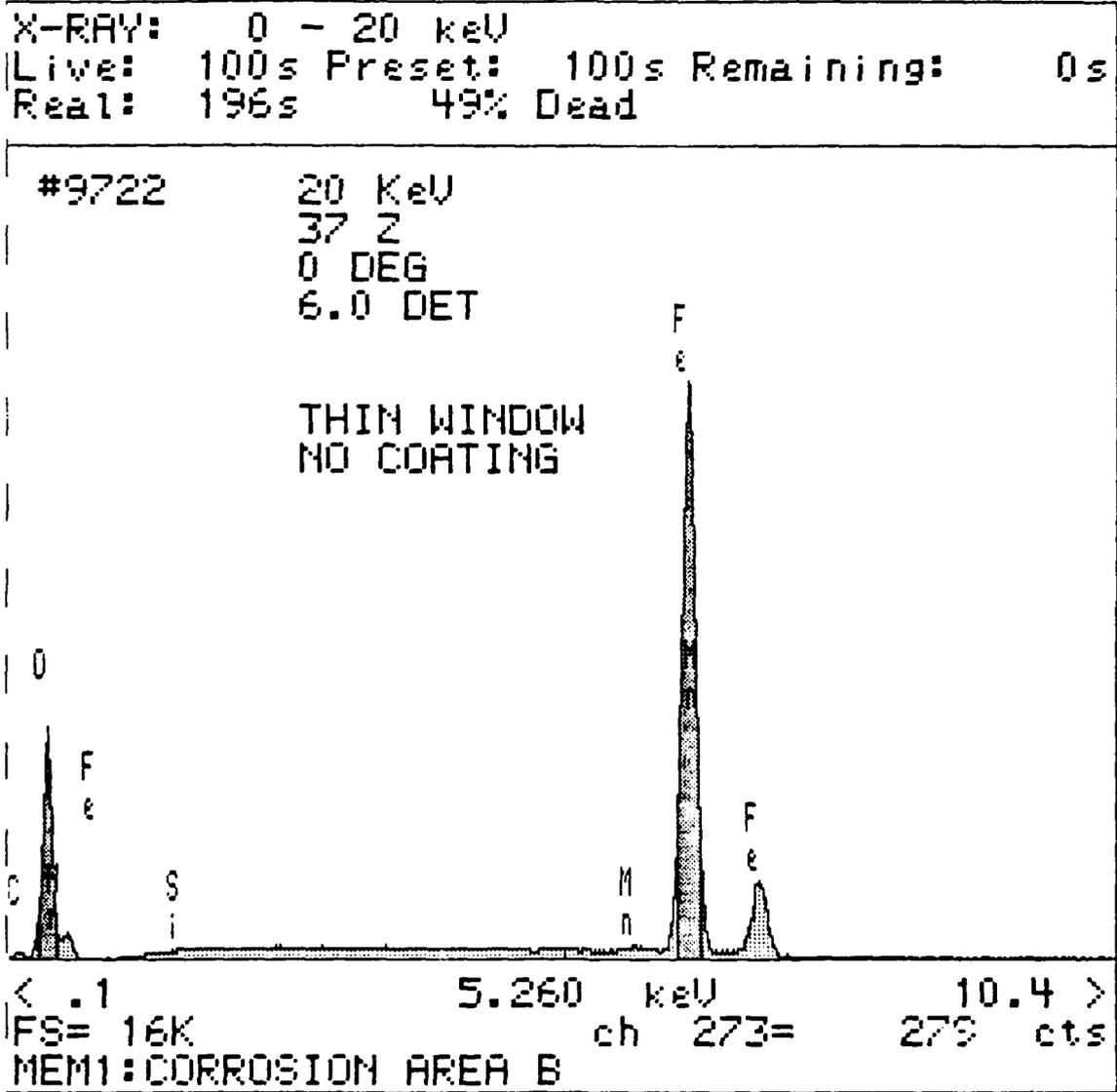


Figure 6.

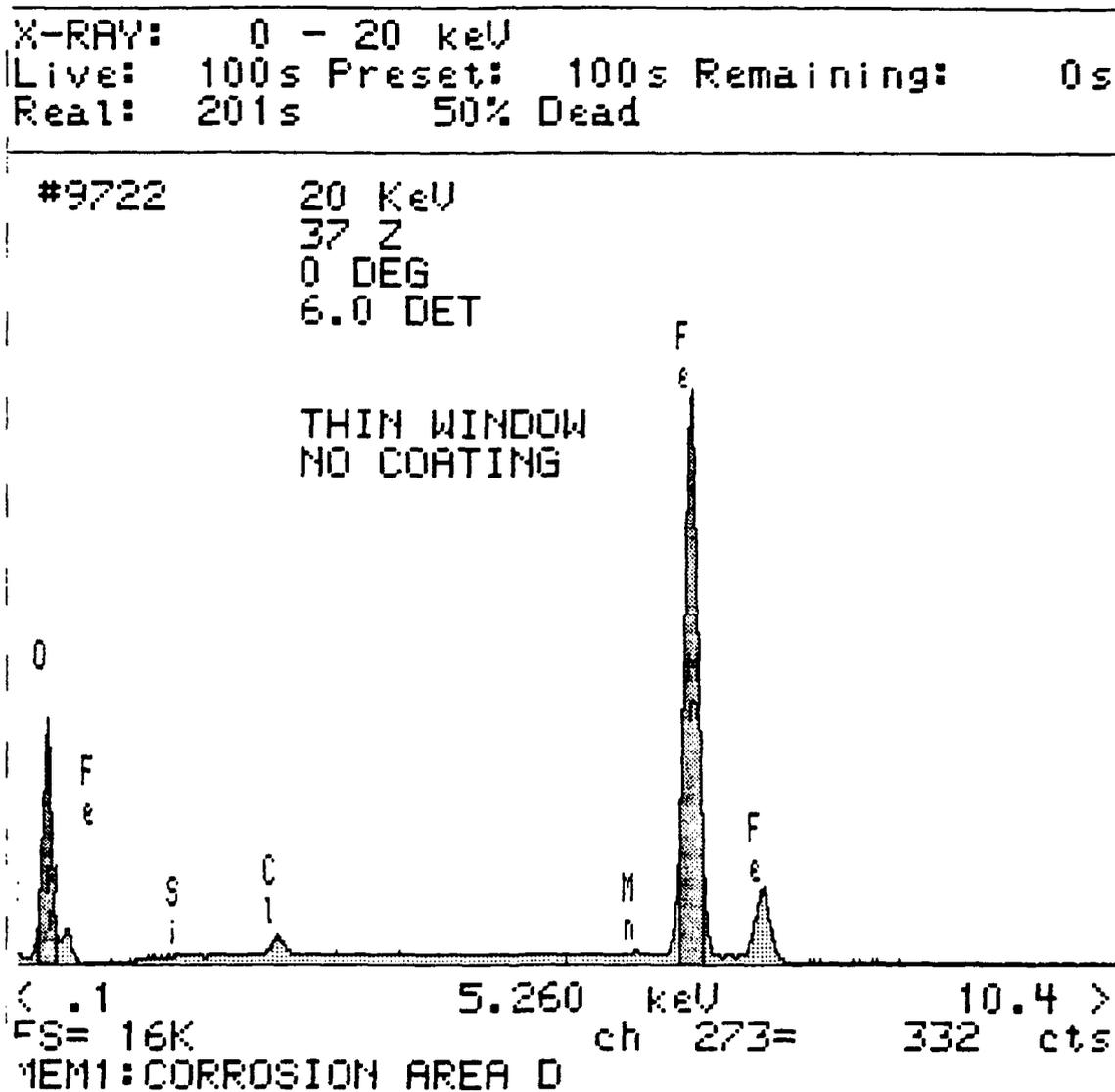
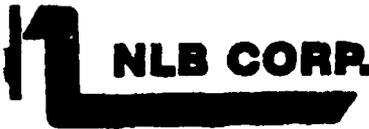


Figure 7.



Manufacturer of the  
National Liquid Blaster

530 Beck Road • Wixom, Michigan 48096  
Telephone (313) 824-8555  
Fax (313) 824-0908 Telex 234334

TO: Mission Research  
3505 Cadillac, Bldg H  
Costa Mesa, CA 92627

Attn: Mr. Dave Newlander

QUOTATION

REPRESENTATIVE: J. Blackmon

INQUIRY NO: Verbal

QUOTATION NO: 10006

DATE: November 15, 1991

TERMS: TBA DELIVERY: 5-6 Weeks

POB: Wixom, MI

VALID UNTIL: 90 Days

ITEM NO.	QTY.	DESCRIPTION	AMOUNT
	1	NLB Model 20256 waterjetting unit rated for 20,000 psi at 21 gpm. Unit is mounted on an industrial skid and driven by a Cummins NT-855-T engine. - See attached specifications.	\$95,870.00
2.	2	MGV20-850 multi gun valves at \$835.00 each	\$1,670.00
.	1	SRH20-77 SPIN NOZZLE®	\$870.00
4.	2	S-7-0002-M cutting nozzles at \$32.50 each	\$65.00
3.	1	DM6443-20 SPIN JET® Stack & Pipe Cleaner 20K	\$13,620.00
.	6	NP20-1/2-50F hose, 20K working pressure at \$1,080.00 per section.	\$6,480.00
7.	1 Lot	Couplers	\$930.00
ADD SALES TAX IF APPLICABLE			

SIGNED *Joseph P. Blackmon* NLB 107613

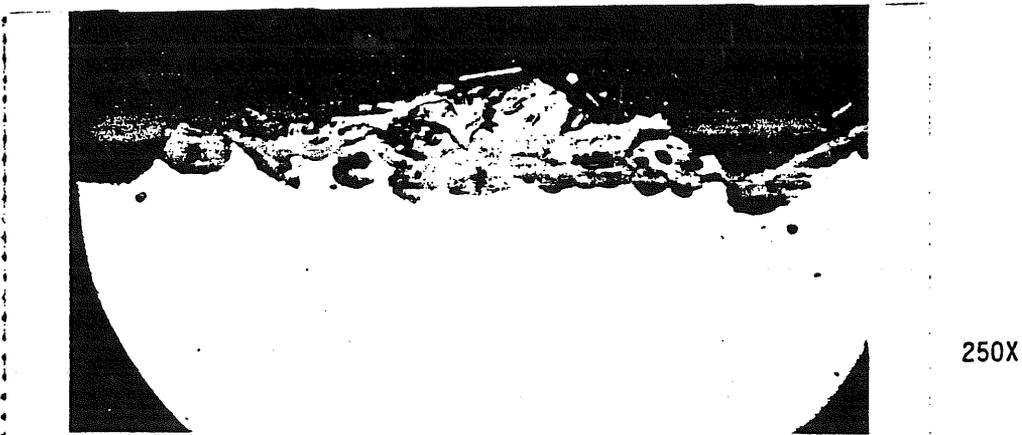
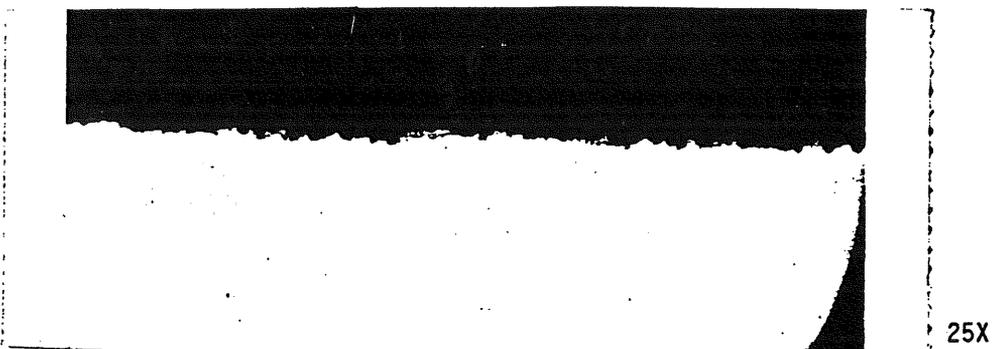


Fig. 8 Section thru oxide layer on Panel X-1Section 7, typical regions. Note layer structure and variable penetration of base metal below.

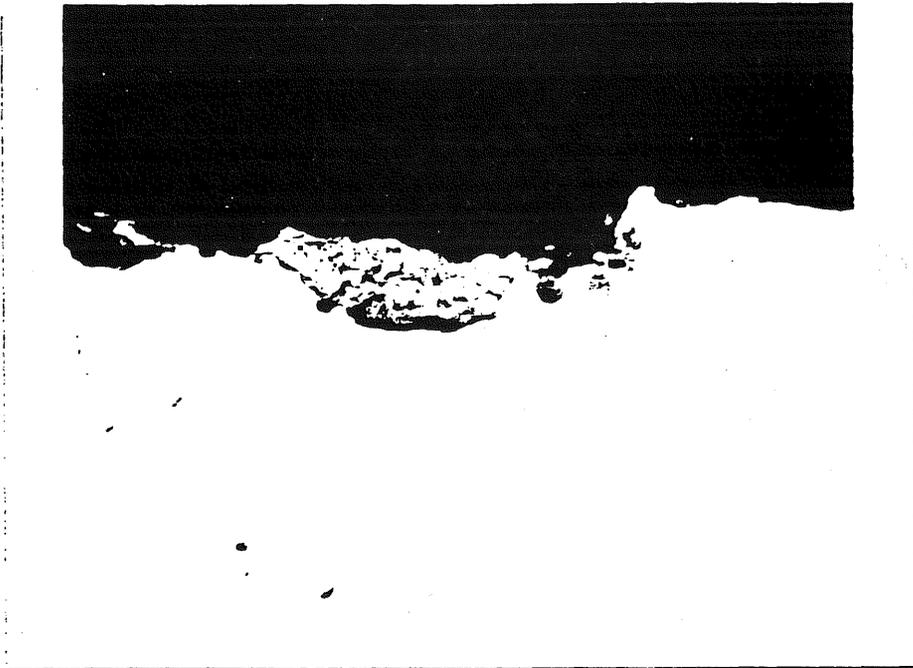


Fig. 9 Polished Section of Panel X-1, Section 7 prior to etching, 200X

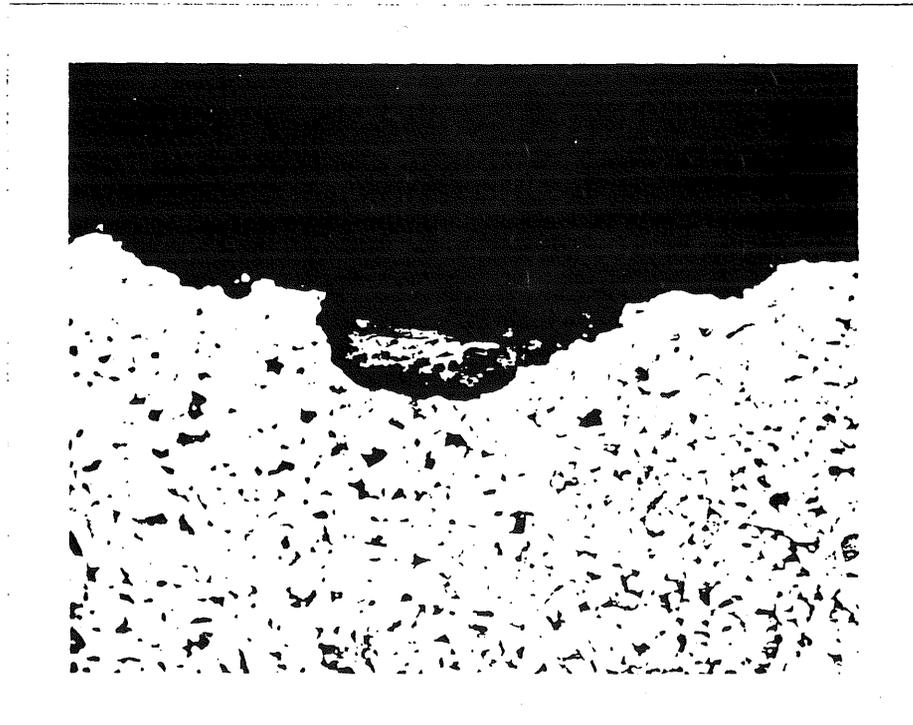


Fig. 10 Polished Section of Panel X-1, Section 7 after 30 Second etch at room temperature with Formulation #1

## APPENDIX C

## Water Blasting Quote

SPECIFICATIONS  
 NLB MODELS 20256D, 15256D, & 13256D  
 INDUSTRIAL ENGINE DRIVE

PUMP - MODEL 20256 QUINTUPLEX PUMP WITH THE FOLLOWING CONTINUOUS DUTY RATINGS:

A CYLINDER

NLB MODEL 20256D	7/8" PLUNGERS - 21 GPM @ 20,000 PSI
NLB MODEL 15256D	1" PLUNGERS - 28 GPM @ 15,000 PSI
NLB MODEL 13256D	1 1/8" PLUNGERS - 33 GPM @ 13,000 PSI

FEATURES - FORGED STAINLESS STEEL FLUID CYLINDER WITH DRILLED PASSAGES TO MINIMIZE TURBULENCE. NO INTERSECTING BORES, TO MINIMIZE STRESS CONCENTRATIONS. SELF-ADJUSTING PACKING ASSEMBLY. FORGED STAINLESS STEEL STUFFING BOXES (DESIGNED TO ALLOW FOR INTERCHANGEABILITY BETWEEN PLUNGER SIZES). SPECIAL HEAT-TREATED ALLOY STEEL CRANKSHAFT WITH LARGE TIMKEN ROLLER BEARINGS RATED FOR OVER 150,000 HOURS OF MAXIMUM OPERATION - CONNECTING ROD BEARINGS - BABBITT LINED AND INTERCHANGEABLE - NO INTERNAL THREADS TO CORRODE OR GALL.

ENGINE - 335 H.P., NT853-P CUMMINS DIESEL. ENGINE INCLUDES ROCKFORD HEAVY-DUTY CLUTCH POWER TAKE-OFF UNIT AND ALL NECESSARY OPERATING GAUGES. OPTION OF 3406T, 6 CYLINDER CATERPILLAR DIESEL.

FEATURES - SIDE MOUNTED PANEL WITH OIL, WATER, TAC AND HOUR METER, START AND STOP SWITCH - EXHAUST MUFFLER.

BATTERY - HEAVY DUTY 12 VOLT BATTERY ENCLOSED IN A WEATHER TIGHT STEEL BOX.

THROTTLE CONTROL - ADJUSTABLE, PRESSURE OPERATED.

FEATURES - AUTOMATICALLY CONTROLS ENGINE SPEED TO A PRE-SET LEVEL AND BACK TO IDLE WHEN USED WITH DUMP VALVE.

FUEL TANK - HEAVY-DUTY STEEL 175 GALLON TANK.

FEATURES - THEFT PROOF FILL PORT AND FUEL LEVEL GAUGE.

PUMP DRIVE - V-BELT DRIVE - COMPLETELY SHROUDED WITH BELT GUARD.

PIPING - PUMP DISCHARGE MANIFOLD FOR PRESSURE GAUGE, RELIEF VALVE, WATER DISCHARGE CONNECTION, ETC.

LUBRICATOR - FIVE PLUNGER FORCE FEED POSITIVE PACKING AND PLUNGER LUBRICATOR.

FEATURES - USES REGULAR LUBRICATING OILS, PRESSURE FORCES OIL IN AND AROUND PLUNGERS AND PACKING - OIL FLOWS THROUGH THE STUFFING BOX AND OIL GLAND RING - CLOSED SYSTEM PREVENTING DIRT AND OTHER CONTAMINATES FROM ENTERING THE ROD BOX - ALLOWS FOR EXTENDED PLUNGER AND PACKING LIFE.

6/91

SAFETY POP-OFF VALVE - NLB RUPTURE DISC SAFETY BLOW-OFF - DISC PRE-SET TO RUPTURE AT 5% OVER RATED OPERATING PRESSURE OF UNIT - COMPLETELY SHROUDED TO PROTECT AGAINST "JET STREAM" DURING POP-OFF.  
FEATURES - PROTECTS PUMP FROM OVERLOADING, RELIEVES 100% OF VOLUME IF OPERATING PRESSURE IS EXCEEDED.

PRESSURE RELIEF BY-PASS VALVE - 20,000 PSI.  
FEATURES - ALLOWS PRESSURE TO BE REGULATED BY OPERATOR - HIGHER PRESSURES WITH LOWER VOLUMES MAY ALSO BE OBTAINED WITHOUT LOADING THE MOTOR DOWN - ALLOWS USE OF SEVERAL LANCES.

PRESSURE GAUGE - 30,000 PSI - OIL FILLED PRESSURE SNUBBER.  
FEATURES - ABSORBS HYDRAULIC SHOCKS - MORE ACCURATE PRESSURE READINGS - LONGER OPERATING LIFE.

WATER SUPPLY TANK - 250 GALLON CAPACITY - AUTOMATIC FLOAT CONTROL VALVE - EPOXY COATED TO ELIMINATE RUSTING. TANK FURNISHED WITH TWO FILTERS - ONE ON INLET TO TANK AND ONE AT OUTLET TO PUMP.  
FEATURES - TANK PROVIDES IDEAL SUCTION CONDITIONS FOR PUMP MAXIMUM EFFICIENCY, NUMBER 10 MICRON FILTER INSURES CLEAN QUALITY WATER TO PUMP.

SKID - COMPONENTS MOUNTED ON HEAVY-DUTY 5'6" WIDE X 18' LONG STEEL SKID.

TRAILER - OPTIONAL - EQUIPPED WITH THREE AXLES, HEAVY-DUTY FENDERS, PINTO HITCH, OVER-THE-ROAD EQUIPMENT PROVIDED WITH ELECTRIC BRAKES, SAFETY CHAINS AND TAIL LIGHTS, SIZE 7'5" WIDE X 21'2" LONG.

PAINT - EACH UNIT PRIMED AND PAINTED MACHINE ORANGE AND BLACK - INDUSTRIAL GRADE ENAMEL.

MANUALS - TWO SERVICE MANUALS PROVIDED WITH EACH UNIT.  
FEATURES - SAFETY, OPERATING, MAINTENANCE, AND TROUBLE SHOOTING GUIDES.

FACTORY TESTED - ALL EQUIPMENT THOROUGHLY TESTED AT NLB'S FACTORY PRIOR TO SHIPPING.

ACCESSORIES - NLB HAS VARIOUS ACCESSORIES FOR USE WITH ABOVE EQUIPMENT FOR VARIOUS APPLICATIONS.

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**FEATURES  
NLB MODEL 20256  
QUINTUPLEX PLUNGER PUMP**

**GENERAL DESCRIPTION**

THE NLB 20256 IS AN ADVANCED-DESIGN QUINTUPLEX PUMP WITH EXCEPTIONAL FLEXIBILITY AND VERSATILITY TO EFFICIENTLY MEET THE REQUIREMENTS OF A WIDE VARIETY OF PUMPING SERVICE. THE UNIT IS OF SIMPLIFIED, RUGGED DESIGN TO MEET THE HEAVY DUTY REQUIREMENTS OF CONTINUOUS DUTY OPERATION AND TO MINIMIZE MAINTENANCE.

**FEATURES**

**DESIGN RATINGS** - CONTINUOUS DUTY - PRESSURES UP TO 20,000 PSI AND VOLUME RATINGS UP TO 33 GPM.

**LIQUID END** - THE STRAIGHT LINE NON-INTERSECTING BORE DESIGN ELIMINATES STRESSES INHERENT WITH INTERSECTING BORE PUMPS AND MAXIMIZES VOLUMETRIC EFFICIENCY.

EACH BORE IN THE CYLINDER CONTAINS A HOLLOW CONICAL SHAPED (CENTERLESS) SUCTION VALVE AND A DISCHARGE VALVE. THESE STAINLESS STEEL VALVES ARE POSITIONED AT EACH END OF A COMMON VALVE.

**MATERIALS OF ALL PRESSURE RETAINING PARTS** - ASTM GRADE STAINLESS STEEL.

**PLUNGERS** - THREADED TO CROSSHEAD EXTENSIONS OUTSIDE THE FRAME HOUSING TO SIMPLIFY ASSEMBLY AND MINIMIZE COST TO REPLACEMENT PLUNGERS. A 60 RC HARD COLMONOY COATED ALLOY IS FUSED TO THE PLUNGER SURFACE, THEN GROUND TO AN 8 MICRO-INCH RMS MIRROR FINISH FOR LONG PACKING AND PLUNGER LIFE.

**POWER FRAME** - ASTM GRADE CAST IRON WITH GENEROUS OIL RESERVOIR AND PROVISIONS FOR ADDING OIL HEATER AND THERMOSTAT. FULL CYLINDRICAL CROSSHEAD BORES ARE CAST WITH SUFFICIENT EXTRA THICKNESS TO ALLOW FOR OVERBORING AND INSTALLATION OF LINERS.

**CRANKSHAFT** - ALLOY FORGED STEEL SELECTED FOR TOUGHNESS AND HIGH ENDURANCE LIMIT. REVERSIBLE IN POWER FRAME FOR DRIVE INPUT ON OPPOSITE SIDE.

**MAIN BEARINGS** - TAPERED ROLLER BEARINGS DESIGNED FOR 150,000 HOURS AVERAGE LIFE.

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CONNECTING ROD BEARINGS - CARRY LOADS IN COMPRESSION ONLY. RODS ARE THE "MARINE TYPE" (BOLTED CAPS AT CRANK END). CRANK BEARINGS ARE THE SPLIT, REPLACEABLE, SHIM-ADJUSTED TYPE (BABBITTED BRONZE). PIN BEARINGS ARE BRONZE.

CROSSHEAD - FULL PISTON TYPE, EXTRA LONG FOR MAXIMUM GUIDING ACTION AND LARGE BEARING AREAS. OPERATES IN FULL CYLINDRICAL GUIDES ACCURATELY BORED IN THE FRAME. ENTIRE CYLINDRICAL SURFACE IS LUBRICATED, WHICH, IN COMBINATION WITH LARGE BEARING AREA, REDUCES WEAR TO NEGLIGIBLE PROPORTIONS.

CROSSHEAD EXTENSIONS - THREADED INTO CROSSHEAD FOR EASE OF REPLACEMENT. STAINLESS STEEL.

CROSSHEAD EXTENSION DEFLECTORS - NEOPRENE RUBBER, LOCATED IN GROOVES IN EXTENSIONS.

CROSSHEAD EXTENSION SEALS - PRESS FIT INTO SEAL CAP. RUBBER LIP TYPE.

CROSSHEAD PINS - SLIP-FIT INTO CROSSHEAD, LOCKED WITH A SET SCREW, ALLOY STEEL, CARBURIZED, HARDENED AND ACCURATELY GROUND.

POSITIVE POWER END LUBRICATION - THE SIMPLE AND SURE SCOOP-GRAVITY SYSTEM USED ON NLB POWER PUMPS FOR DECADES PROVIDES POSITIVE LUBRICATION TO CROSSHEAD AND ROD BEARINGS.

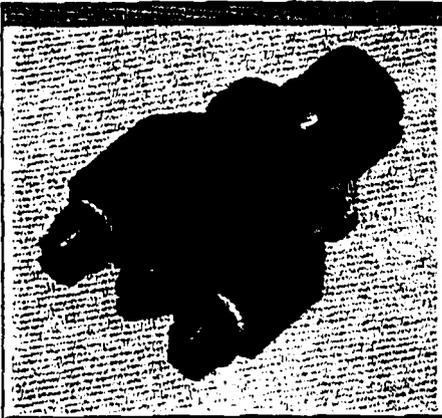
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NEW FROM NLB CORPORATION . . .

# SPIN - NOZZLE®

## Model No. SRH 20-77

### Self-Rotating Lance Head



NLB Corporation introduces its latest breakthrough in high pressure waterjetting technology. The SPIN-NOZZLE®, model no. SRH 20-77 is a self-rotating jetting nozzle that affixes to any 20,000 psi high pressure cleaning lance.

The SRH 20-77 features a specially designed and balanced body that houses two (2) standard jetting nozzles in an offset pattern. When the lance is activated, the SPIN-NOZZLE rotates by the reaction energy of the high pressure water. No external driver is needed to propel the nozzle.

The NLB SPIN-NOZZLE self-rotating head turns an ordinary high pressure lance into a sophisticated waterjetting tool. Paint, rust, scale and heavy product build-up is quickly removed. And, the SPIN-NOZZLE is capable of dramatic gains in productivity when compared to conventional cleaning lances.

#### SPIN-NOZZLE® Model No. SRH 20-77 VOLUME CAPACITY (GPM)

NOZZLES	10,000 psi	15,000 psi	20,000 psi
two # 2	6.40	7.84	9.08
two # 1.5	4.00	4.98	5.64
two # 1	2.00	2.44	2.82

(Actual volume output will be higher due to water lubrication.)

#### OPERATIONAL SPECIFICATIONS

INLET CONNECTION: 9/16 Inch Autoclave  
 OVERALL LENGTH (with nozzles): 5.0 inches  
 DIAMETER: 3.0 inches  
 WEIGHT: 2 lb. - 13 oz.  
 SPEED: 5,000 rpm @ 20,000 psi (approx.)  
 MAX. WORKING PRESSURE: 20,000 psi



*The Industry leader in high pressure water-jet technology*

National Liquid Blasting Corporation  
 29930 Beck Rd. Wixom, MI 48096

(313) 624-6855 TLX 234334 FAX (313) 624-0908

SPIN NOZZLE® is a registered trademark  
 of NLB Corporation. Patent pending.

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