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Report No. 63-3893

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EVALUATION OF PHOSPHATE FINISHES ON STEEL AS UNDERCOATS FOR ARMY PAINT SYSTEMS

By

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Approved by:

G. C. Hanson

A. C. HANSON Laboratory Director

26 November 1963

DA Project No. 1-A-0-24401-A-110-05

AMC Code No. 5025.11.84205

Rock Island Arsenal Rock Island, Illinois

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ABSTRACT

When this study was initiated a list of 78 commercially available phosphate coatings was compiled. After investigation of the make-up and and factors of application involved in these products, it was discovered that only 41 had enough distinct differences to warrant their use in these comparative tests.

Fifty different sets of prepared specimens were used in this study. Forty-one commercial phosphating products, 3 commercial wash primers and 4 phosphating process solutions in use at the Rock Island Arsenal were used. Also included was a diluted commercial phosphoric acid rust remover and metal conditioner and one control set comprised only of cleaned steel.

All phosphated panels were processed in accordance with directions supplied by the manufacturer. Six panels from each of the 50 sets of panels prepared were coated with one of the 9 different systems of organic coatings. The prepared panels were scored and mounted on outdoor exposure racks. This report covers the results of 8 years of South (at 45 degrees) exposure at Rock Island Arsenal. A previous R.I.A. Report No. 54-2753, dated 24 August 1954, covers details of preparation of the phosphate coated panels.

RECOMMENDATIONS

Results obtained after 8 years exposure at Rock Island Arsenal establish the absolute superiority of zinc phosphate coatings over iron or manganese phosphate coatings.

Wash primers were found to be superior to both the iron and mangamene phosphate coatings as substrata for paint application. It is recommended that where maximum paint durability and freedom from rusting is desired, zinc phosphate coatings should be applied. Where structures are of such a nature that application of these coatings is impossible, the use of the wash primers is recommended. Results of these tests indicate that iron phosphates should be used only in circumstances where properties other than long term protection under paint is required.

EVALUATION OF PHOSPHATE FINISHES ON STEEL AS UNDERCOATS FOR ARMY PAINT SYSTEMS

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EVALUATION OF PHOSPHATE FINISHES ON STEEL AS UNDERCOATS FOR ARMY PAINT SYSTEMS

OBJECT

To make a comparative study of various commercial phosphate coatings on steel as undercoats for Army paint systems. To evaluate these coatings as undercoats for various paints and paint systems in outdoor exposure. To grade zinc, iron and manganese phosphate coatings in regard to corresion resistance, when used under these organic coatings.

INTRODUCTION

When Government specification requirements included the application of phosphate coatings as pretreatments prior to painting, a number of phosphate coating materials manufacturers complained that the requirements imposed were too stringent. Close scrutiny of the phosphate coatings was engendered during World War II, primarily by the severe atmospheric conditions encountered in the South Pacific Theater of the war. It was, therefore, necessary to provide Army material with the best possible protective coatings. This resulted in the application of the phosphate coatings as paint bases and the initiation of the U.S. Army Specification 57-0-2c, "Finishes, Protective, for Iron and Steel Parts," dated 11 December 1943. Paint bonding coatings were covered in this specification under Type II, Class C. This specification, as amended 16 January 1945, specified that "Articles finished with a Type II, Class C coating shall have a minimum coating weight of 150 mgs. per square foot.

This specification requirement was subsequently included in the joint Army-Navy Specification Jan-C-490; dated 21 August 1947, "Cleaning and Preparation of Ferrous Metal Surfaces for Organic Coatings. This specification was superseded by the Military Specification MIL-C-490, dated 7 May 1954, "Cleaning and Preparation of Ferrous and Zine Coated Surfaces for Organic Protective Coatings. In this specification (p. 3, Section 3.11) it was stated that "the minimum coating weight shall be 150 mg, per square foot for spray processes and 300 mgs per square foot for dip processes." These requirements were considered much too high by some manufacturers of the so-called iron phosphate coating processes. They contended that 40 mgs per square foct was a sufficient requirement for a Type II, Class C phosphate coating. They claimed products that they had developed converted the surface layer of steel into a fine crystalline uniform film of insoluble iron phosphate, and that these films produced a nonconducting bond between the metal and the topcoat of enamel or lacquer and

were sufficient in that role. Samples were requested from manufacturers who had participated in these discussions. Outdoor exposure and salt spray tests failed to substantiate some of the claims for iron phosphate coatings. Many of the coatings could not meet the requirements as to proper weight and salt spray resistance. Regardless of the interim progress reports of these tests, the succeeding Specification, TT-C-490, dated 30 March 1961, included the reduced requirement for the Type II coating (iron phosphate). The 40 mg/sq ft minimum requirement prevails for both the spraying or dipping process.

In the ensuing tests it was found that sodium dihydrogen phosphate or ammonium dihydrogen phosphate plus a wetting agent were the principal ingredients of these products.

The result of these preliminary tests of iron phosphate coatings was a decision to evaluate all the known iron, zinc and manganese phosphating products on the market. After considering the descriptive literature of 78 allegedly different commercial products, it was decided, in order to avoid apparent duplication of the formulations, to include only 41 of these products. For comparison purposes, panels treated with 3 wash primers, 4 production phosphating solutions at Rock Island Arsenal, a dilute phosphoric acid metal treatment and an uncoated set of steel panels were included in the tests (Chart I). This brought the total to 50 sets.

PROCEDURE

In order to evaluate accurately the commercial phosphate coating products used as prepaint treatments, standard procedures had to be established and strictly adhered to. Failure to consider any one of the procedure details of the submitted samples could nullify the results and, therefore, produce peor evaluation of the coatings under test.

Samples and their accompanying literature were received from the manufacturers. The test pieces were 4" x 12" x 1/16" FS 1020 cold rolled steel panels. All panels were prepared from one lot of steel. The sets of panels were alphabetically marked and numbered within each set.

There were many and varied preliminary treatments recommended prior to phosphating. The prephosphating treatments considered were acid-cleaning, alkali cleaning, emulsifiable solvent, emulsion alkali, vapor degreasing, solvent spray and wiping, silica sandblasting, steel grit blasting and electroderusting. It was necessary to decide which of these methods would be most satisfactory for all phosphating procedures recommended by the respective manufacturers. Tests have shown that trichloroethylene vapor

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degreasing followed by the electrolytic alkaline derusting process produces a surface free of oil and rust. This two step procedure of prephosphating treatment was chosen.

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The trichlorcethylene vapor degreasing method was selected because of its simplicity and the thoroughness with which it removes oils and grease from different shaped articles. The solvent used was purchased under Federal Specification O-T-634, "Trochloroethylene, Technical Grade, Type II," dated 8 October 1945.

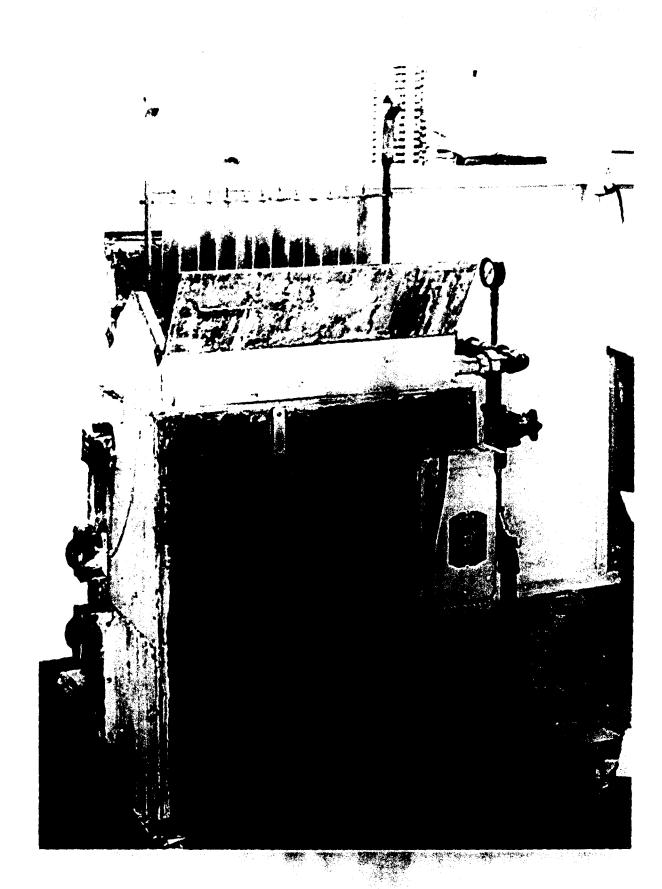
To insure exact and reproducible prephosphating surfaces the electrolytic alkaline derusting process was used. This process not only provides a final removal of any residual oils and soils, but also removes undetectable rust spots, scale and discolorations. This insures an absolutely clean steel surface. In order to accomplish this end, the panels were subjected to ten second cathodic and five second anodic current reversal cycles in the electroderusting solution for a period of 20 minutes. Careful attention was given to the removal of the panels from the solution near the end of the five second anodic cycle. This technique eliminated any possibility of withdrawing the panels with electrodeposited metal contaminants which may have deposited during the cathodic cycle. The current density used was 60 amp/sq ft. The electroderusting solution consisted of 3 pounds of the following mixture per gallon of solution and was used at 130°F.:

25% tetra sodium salt of ethylene diamine tetra acetic acid. 25% sodium cyanide. 50% sodium hydroxide.

Model 50-F-75G spray phosphating cabinet (Figure 1), manufactured by the Pioneer Pump & Manufacturing Company of Detroit, Michigan, was used for those coatings which were to be applied by spray. This cabinet was equipped with thermostatic controls. In the instances where hand spraying was indicated a Model 19 spray gun, which was manufactured by the Binks Manufacturing Company of Chicago, Illinois, was used.

Special steel tanks with 15 gallon capacities were used for the immersion phosphatizing processes. These tanks were equipped with thermostatically controlled steam coils for temperature control.

Panels were racked vertically in the specially designed holder (Figure 1). Once the panels were racked, they remained



SPRAY PHOSPHATING CABINET

RIA Neg. No. 4389

Figure 1

so during the entire processing in the vapor degreaser, alkaline electroderusting solution, the phosphating spray cabinet or the steel tank for immersion phosphating. The use of these racks excluded any further handling of the individual panels.

Table I shows the various products which were used in this project. In this table are listed the product or process (in code, type of phosphate coating, method of application and recommended conditions for proper operation. In the column of pointage, the number indicates the volume of 0.1 N sodium hydroxide, in millimeters, required to neutralize 10 ml of the solution using phenophthalein as the indicator. This end point is considered the total acidity of the phosphating solution.

After each phosphating treatment was applied, the treated panels were painted using the paint systems shown in Table IV. In order to eliminate variability of the dry paint film thickness, a semi-automatic paint spraying apparatus was designed by the author. (Figure 2)

During application of the paint, the panel held by magnets to the conveyor, travelled perpendicularly to the spray gun affusion at a distance determined by the rate of speed of the conveyor and the fan of the spray. The line pressure and orifice opening and the viscosity of the coating material were controlled to provide the designed dry film thickness. As the ganel approached the vicinity of the spray gun, the operator pressed the trigger and held it until the parel passed beyond the spray pattern of the gun. The pattern of the fan of the paint spray was regulated in order to cover the entire width of the panel with an even distribution of paint. After all adjustments were regulated to give a dry film thickness within a telerance of 0.05 mil, all the panels of one group were coated. A General Electric Thickness Gage, Type DO-41 was used in the thickness measurements.

It is impossible to reasure accurately the thickness of a phosphate coating due to the crystallinity and softness of the coating. It was, therefore, imperative that a definitely known amount of paint be applied on these phosphated surfaces. In order to accomplish this end, it was necessary to prepare trial panels in the paint spraying operation. To do this, clean bare steel panels of the same sheet steel stock were coated, baked and thickness measurements made. From these known results the necessary adjustments were made, such as distance of the gun from the panel, the line pressure, nozzle pressure and opening and viscosity of the coating

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TABLE

PRODUCTS AND PROCESSES OF PHOSPHATE CO

		Type of	Method	Materia
		Phosphate	of	
	Code	Coating	Application	State
	U	Iron	Spray	Powder
	D	Iron	Spray	Powder
	AE	Iron	Spray	Powder
	AQ	Zinc-Iron	Immersion	Liquid
	K	Iron	Spray	Powder
	H	Iron	Spray	Powder
	G	Iron	Spray	Powder
	AB	Zinc-Iron	Immersion	Powder
	B	Iron	Spray	Liquid
o	ÅD	Zinc	Immersion	Liquids
	<u>M</u>	Iron	Spray	Powder
	AK	iron	Spray	Powder
	N	Wash Primer	Hand Gun Spray	Liquid
	AM	Manganese	Immersion	Liquid
	F	Iron	Spray	Powder
	A	Iron	Spray	Powder
	B	Iron	Spray	Powder
	AP	Manganese	Immersion	Liquid
	W	Zinc	Immersion	Liquid
	R	Iron	Spray	Liquid
	ÅG	Zinc	Immersion	Liquid
•	BB	Manganese	Immersion	Powder
ü	AS	Zinc-Iron	Immersion	Powder
63-3893	AU	Iron	Hand Gun Spray	Liquid

<u>3</u> I

COATINGS USED FOR COMPARATIVE STUDY

ia]	ls Required	Phospha Con		
	Make-up	Point-	Temper-	Time
	Lbs/100 Gals	age	ature F.	Min.
r	12.5	-	170	1-1/2
r	12.5	-	160	3
r	12.5	-	180	2
đ	46	28	200	25
r	12.5	_	170	2
r	20	_	170	2
r	12.5	-	190	
r	20	-	200	1 3
d	24	-	160	3
	39.6			
ds	14.2	27	200	5
r	25	-	160	3
r	12.5	55	170	1-1/2
d	As Rec.	-	Room	-
d	35	24	205	15
r	9	-	170	1
r	12.5	-	165	1
r	12.5	-	165	1
d	115	60	200	20
d	25	-	190	5
	37.50			
d	8.28	-	180	
d	179.2	25	205	20
r	27.3	30	200	45
r	38	-	200	10
. d	As Rec.	-	Room	-

TABLE

Code	Type of Phosphate Costing	Nethod of Application	Ma St
Code	Coating	Application	5
1			
J	Zinc	Spray	Po
AC	Zinc-Iron	Immersion	Po
AF	Zinc-Iron	Immersion	Po
0	Iron	Spray	Po
OP	Wash	Hand Gun	L
X	Zinc	Immersion	L
L	Iron	Spray	Po
Т	Zinc-Iron	Spray	Po
I	Iron	Spray	Po
AT	Iron	Spray	Po
AI	Iron-Zinc	Immersion	L
AV	Iron	Hand Gun Spray	L
AH	Ircn	Spray	P
AN	Zinc	Immersion	Po
Q	Wash Primer	Hand Gun Spray	L
S	Iron	Hand Gun Spray	
Y	Zinc-Iron	Spray	L
V .	Zinc-Iron	Spray	L
AR	Iron	Spray	L
AJ	Zinc	Immersion	P
AL	Zinc	Immersion	L
Z	Zinc	Immersion	L
AO	Zinc	Immersion	L
С	Manganese	Immersion	P
A A	Zinc	Immersion	L

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1

<u>**E**</u> (Continued)

late ria	ls Required	Phosphat C	conditions	
	Make-up	Point-	Temper-	Time
itate	Lbs/100 Gals	age	ature F.	Min.
-		tanihi dan		
vowde r	35	24	160	2
vowder	33	20	160	5
'owder	50	20	200	5
'owder	45	10	165	1
jiquid	As Rec.	-	Room	1
liquid	174	-	200	5
vowder	12.5	-	190	1-1/2
Powder	54	-	160	5
Powder	12.5	-	170	2
Powder	9	-	160	2
Liquid	17.2	15	130	5
Liquid	As Rec.	-		-
Powder	30	30	160	2
Powder	50	22	190	5
Liquid	As Rec.	-	Room	-
Liquid	As Rec.	-	Room	-
Liquid	19	20	130	1
Liquid	18	25	180	2
Liquid	247.5	10	190	2
Powder	35	25	180	20
Liqui	18	2 5	200	20
Liquid	39.6	-	140	2
Liquid	3 0	25	Room	30
Powder	27.3	30	195	45
Liquid	36	30	200	30

TABLE

PAINT COATINGS AND SYSTEMS USED IN

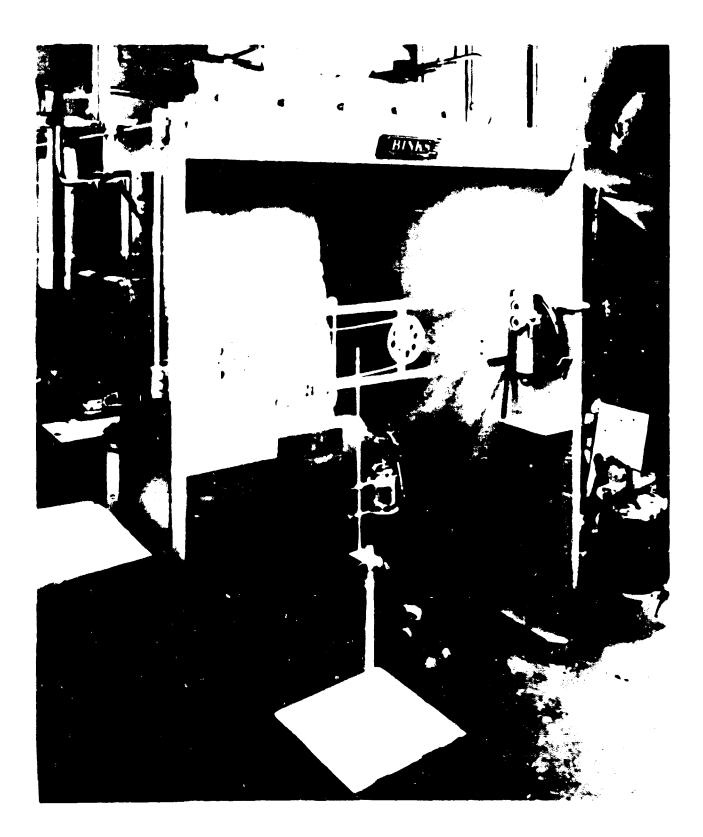
	System No.	Panel No.	Coating (<u>lst Coat</u>)
	1	1-6	MIL-E-10687
	2	13-18	MIL-L-11195
	3	25-30	MIL-P-11414
œ	4	37-42	MIL-P-11414
	5	49-54	MIL-P-11414
	6	61-66	TT-P-636
	7	73-78	TT- P-636
	8	85-90	TT-P-6 36
63-	9	97-102	TT-E-485 b
63-38{ 3			

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EII

IN COMPARATIVE PHOSPHATE COATINGS TESTS

Dry Film Thickness	Coating (2nd Coat)	Dry Film Thickness				
1 mi1 ± 0.1	None					
1 m11 ± 0.1	None					
0.7 mil + 0.1	+ MIL-E-10687	1 mil ± 0.1				
**	+ MIL-L-10182	••				
••	+ MIL-L-11195	••				
1 mil ± 0.1	+ MIL-E-11237	••				
"	+ USA-3-174	**				
"	+ TT-E-489	**				
"	None					



EQUIPMENT USED IN THE SPRAY PAINTING OF THE VARIOUS ARMY PAINTS

RIA Neg. No. 7014 63-3893

Figure 2

material. Using this technique, the thickness of the dry film could be obtained to within plus or minus 0.05 mils.

For each set of panels, it was necessary to make different adjustments, since the viscosity and composition of the different paints varied. The variation in viscosity was due to the difference in the paint material as received from the manufacturers. Tests whowed that the tolerances in the pigment-vehicle ratio could vary the dry-film thickness of different paints, which produced the same viscosity readings. Also, the broad tolerance limit allowed in spraying viscosity is, in a No. 4 Ford Cup, 17-25 seconds in most enamel specifications. This alone is a variable, which must be considered, when setting up the adjustments listed above in the operation of the semi-automatic paint spraying equipment.

It was discovered that the General Electric thickness gage used in this test was not accurate and reproducible in the 0.0 to 1.0 mil range. To overcome this deficiency, tests by this author proved that by using a 1.0 mil nonmagnetic shim and setting the indicator at 1.0 mil on the scale the readings were accurate and reproducible. This adjustment was made as follows: a 1.0 mil nonmagnetic shim was used on a similar bare steel panel under the electrode and the meedle set at 1.0 mils. When the electrode and shim were transferred to a steel panel, which had been sprayed with one of the organic coatings, the reading was noted. The difference between this reading and 1.0 mil was the actual film thickness of the applied coating.

After the phosphating products and the various paints and paint systems had been applied, the ranels were allowed to air-dry for 72 to 96 hours. The panels were then edged with an aluminum pigmented tung oil modified phenolic resin varnish to minimize edge corrosion. The panels were then scored with a sharp scribing tool which cut a line parallel to the left side, one inch from the edge and running to points 2 inches from the top and 1-1/2 inches from the bottom. This score line was cut through the paint and phosphate coat into the basis metal. Six panels from each of the 9 paint systems were placed in specially constructed racks for outdoor exposure (Figures 3, 4 and 5).

In order to give a clear picture of the progress of the outdoor exposure for each of the samples submitted for test, charts were made. A chronological account is presented for all samples. Charts I through XII show the results of observations beginning at 1 year and each 6 months thereafter until 8 years had elapsed.

For one year the panels were held in position by wooden crossbars. This was revised, using procelain insulators, as shown in Figures 4 and 5.

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OUTDOOR EXPOSURE AREA

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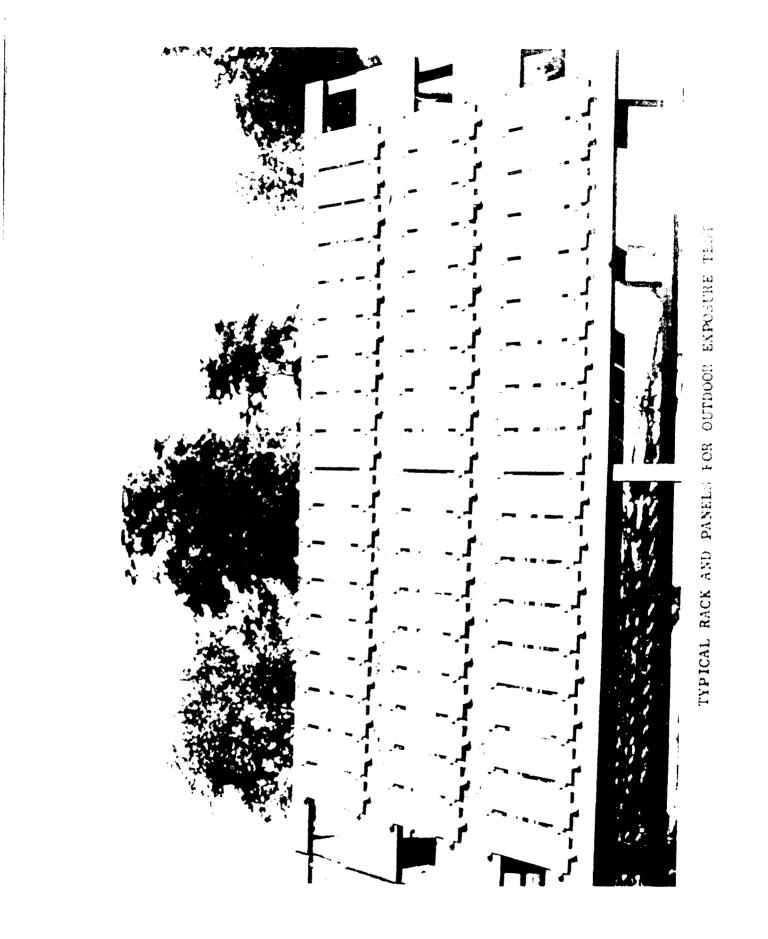
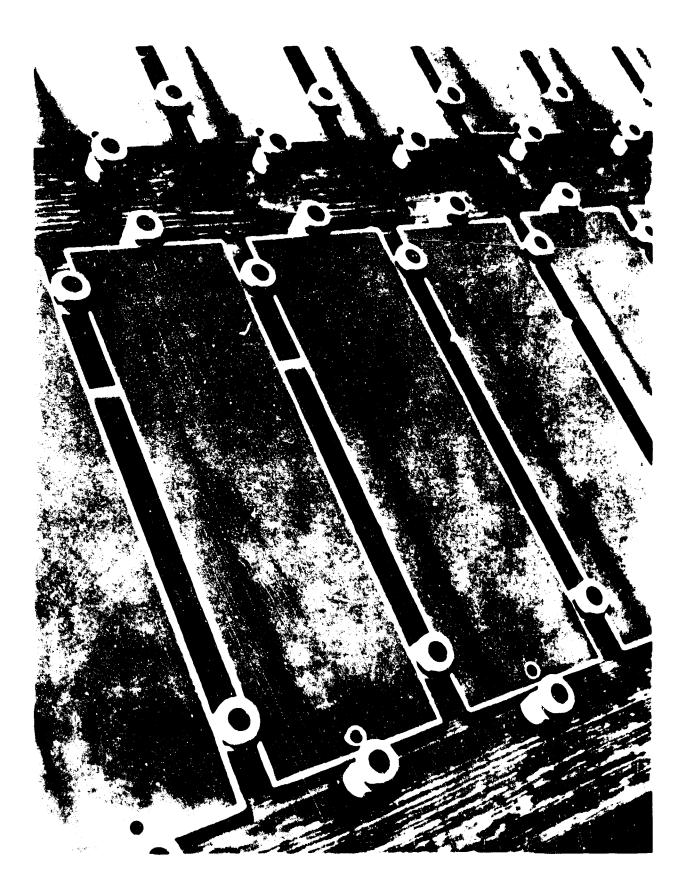


Figure 4

12

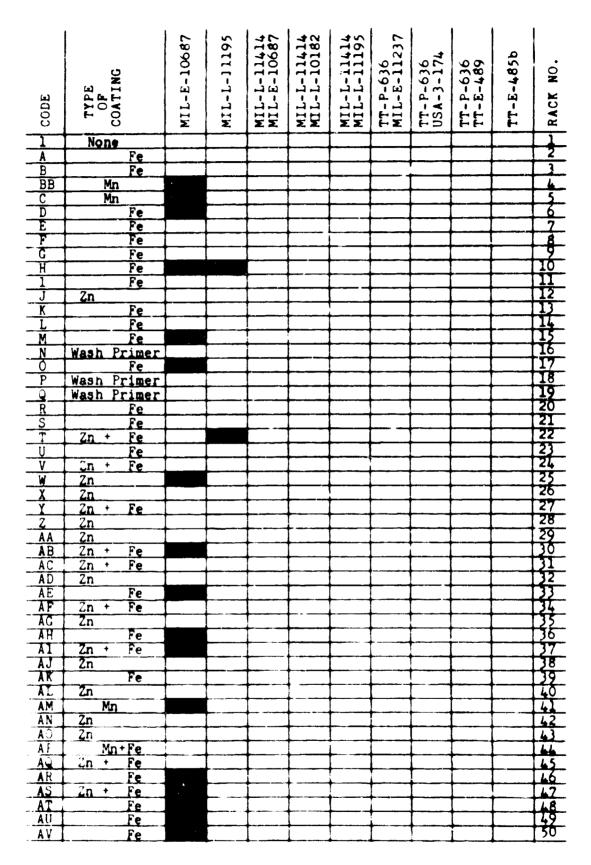
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CLOSE-UP OF EXPOSURE RACK SET-UP

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Figure 5



1 YEAR OUTDOOR EXPOSURE (Filled Areas Indicate the Failures)

CHART I

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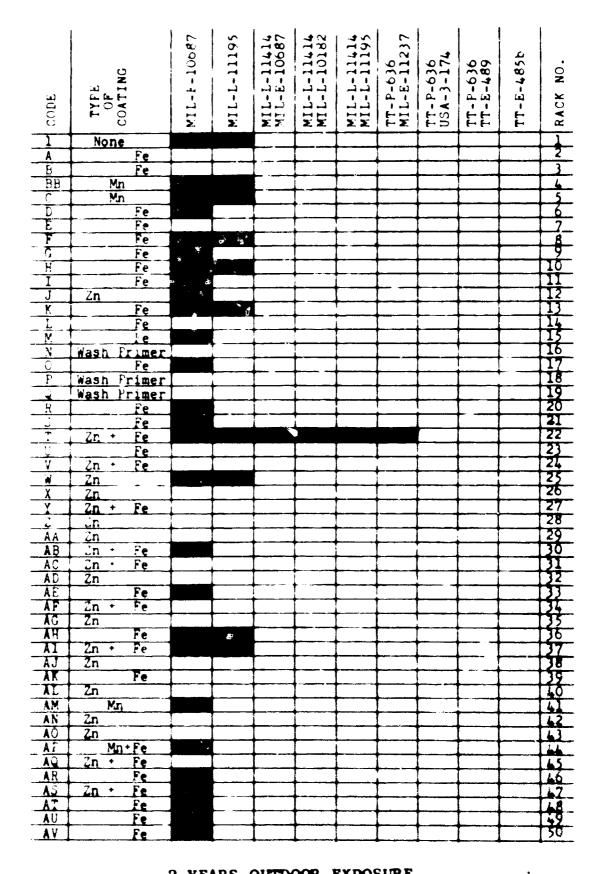
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CODE	TYPE OF COATING	MIL-E-10687	MIL-L-11195	MIL-L-11414 MIL-E-10687	MIL-L-11414 MIL-L-10182	MIL-L-11195	TT-P-636 MIL-E-11237	TT-P-636 USA-3-174	TT-P-636 TT-E-489	TT-E-485b	L N- RACK NO.
	None										
	Fe Fe			h							2
- 88	Mp										1
Ċ	Mn Mn										5
D	Fe										6
	Fe Fe Fe Fe Fe										6 7 8 9
- 5	Fe										-8-
-Ř	Fe										16
	Pe										10 11 12
	Zn										12
<u> </u>	Fe Fe		<u> </u>								13
M	Fe										115
N	Fe Fe Fe Wash Primer Fe Wash Primer										16
<u>- 0</u>	Fe Prince										17_
- <u>r</u>	Wash Primer Wash Primer										18-
R	Fe										20
S	Fe										21
<u> </u>	Wash Primer Fe Zn + Fe Zn + Fe Zn + Fe	, i									18 19 20 21 22 23 24 25 26 27 28 29 30
											23
	Zn Zn										25
X	Zn										26
<u> </u>	2n + Fe										27
-2	Zn										28
- <u>AA</u> 	$\frac{2n}{2n}$ + Fe										
ÂČ	2n Fe 2n 2n 2n Fe 2n 2n 2n 2n 2n Fe 2n Fe										11
AD	2n										32
AE	Fe 2n + Fe										22
J K L M N O P Q R S T U V V V V V V V V V V V V V V V V V V											狂
AH	Zn Fe										36-
	Zn + Fe										37
AJ	Zn										<u>78</u> 39
	Fe Zn										32
AJ AK AL AM AN AO	Mn										10
AN	Zn										12
AOI	2n 2n										43
AP AQ AR AS AT	<u>Mn+Pe</u> Zn + Fe										44
AR	2n + Fe Fe										45
AS.	2n + Fe										<u>16</u> 17
TA	<u> </u>										48
	<u>Fe</u>				I						49
	Fe						1				50

1-1/2 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate the Failures)

CHART II

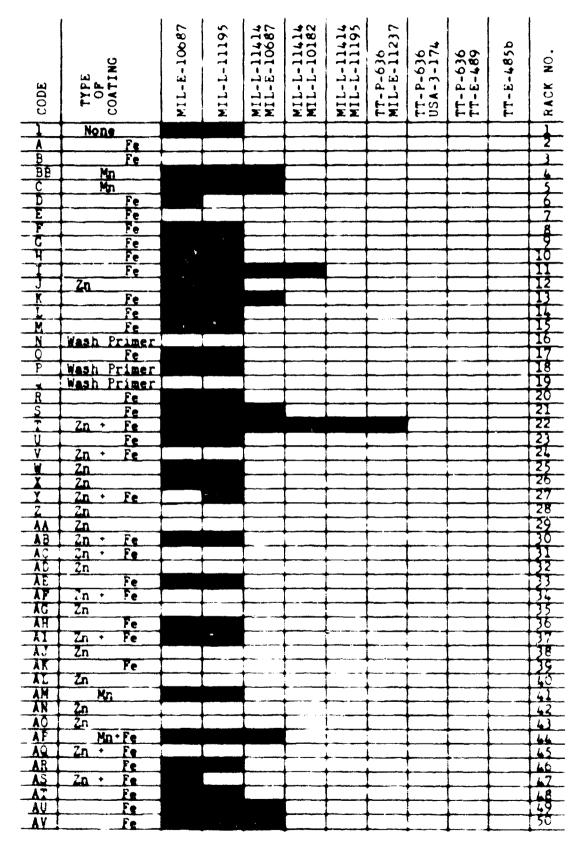
Neg. No. 4614 63-3893



2 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate the Failures)

CHART III

Neg. No. 4615 63-3893



2-1/2 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate the Failures)

CHART IV

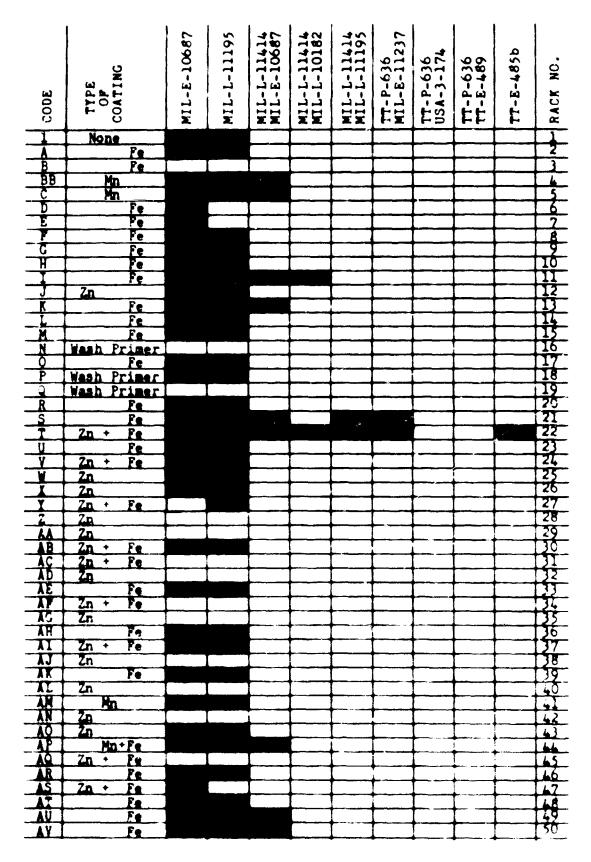
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CODE	TYPE OF COATING	MIL-6-10687	MIL-L-11195	MIL-L-11414 MIL-E-10687	MIL-L-11414 MIL-L-10162	MIL-L-11414 MIL-L-11414	TT-P-636 MIL-E-11237	TT-P-636 USA-3-174	TT-P-636 TT-E-489	TT-E-485b	- RACK NO.
00	F OS	2	Σ	I I I N N	ΣX	ΣΣ	T T	TT. US/	ÉÉ	TT.	RA
1	None			L							
<u> </u>	Fe Fe			.							$\frac{2}{3}$
BB	Mn		_				<u> </u>				
Ç	Mn Mn										4
<u>D</u>	Fe			•							6
A B B B C D E F C H I J K L M N O P I K L M N O P I X X X X Z AA A B AC AE AC AE AC	Fe Fe Fe Fe Fe Fe	i								·	6 7 8 9 10 11
<u> </u>	Fe										Ţ.
<u>H</u>	Fe Fe										10
Ĵ	Zn										112
K	Fe Fe Wash Primer Wash Primer Wash Primer Fe Fe Zn + Fe Fe Zn + Fe Fe Zn + Fe										12
- L	Fe Fe		٠								14
<u>N</u>	Wash Primer										it
<u> </u>	Fe										117
<u> </u>	Wash Primer Wash Primer										17-19
R	Fe										20
<u> </u>	Fe					1					20 21 22 23 24 25 26 27 28 29 30 31 31 32
	<u>Zn + Fe</u>					,					22
	Zn + Fe		_							·····	122
1	Zn Fe Zn Zn Zn Fe Zn Fe Zn Zn Zn Fe Zn Fe		•								25
- X	Zn Zn • Fe										26
	<u>Zn · Fe</u> Zn										28
AA	Zn	+									29
<u>AB</u>	Zn · Fe Zn · Fe		c								30
	2n · Fe										131
ÂÊ	Fe					·····					33
AF	<u>ln</u> · Fe In										34
- Î H	En Fe					+					35
AI	<u>In + Fe</u> In				+				🛉		26
A.	<u>2n</u>										36
$-\frac{\Lambda \Lambda}{\Lambda T}$	Fe Zn	 				·	+				129
AM	Mn	·· · ·									40
AN	2n										42
AI AC AL AM AN AO AF AQ AR	Zn Mn+Fe					ļ					63
- 72	2n · Fe	• >									44
AR	Fe	1					+				45 40 47
AS	<u>In · Fe</u> Fe										17
AU	Fe		÷۴			+					48 29 50
AY	Fe										30

3 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate the Failures)

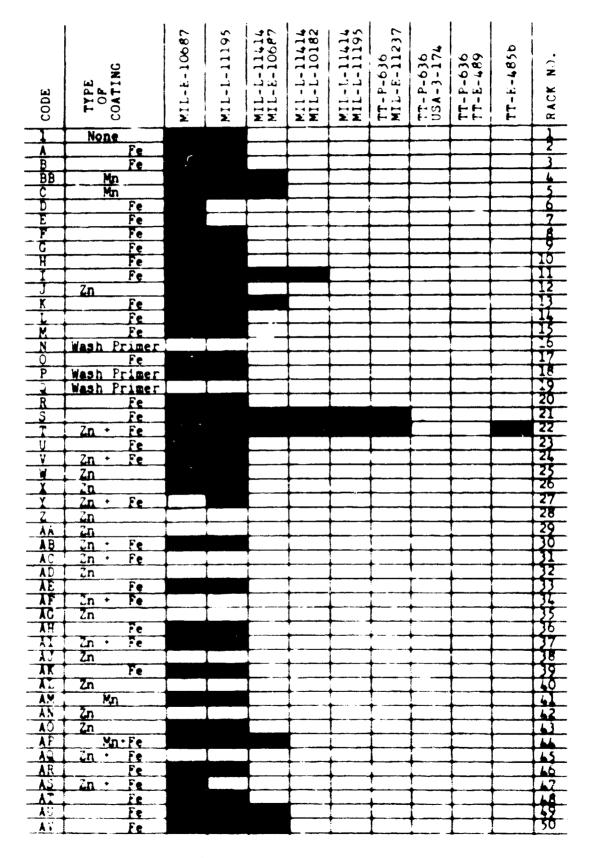
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Neg. No. 4617 63-3893



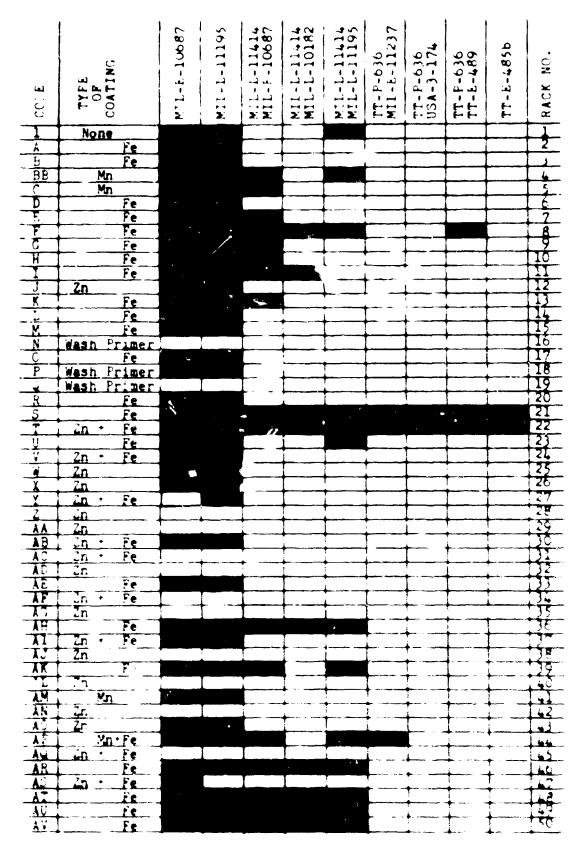
3-1/2 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate the Failures)

CHART VI



4 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate Failures)

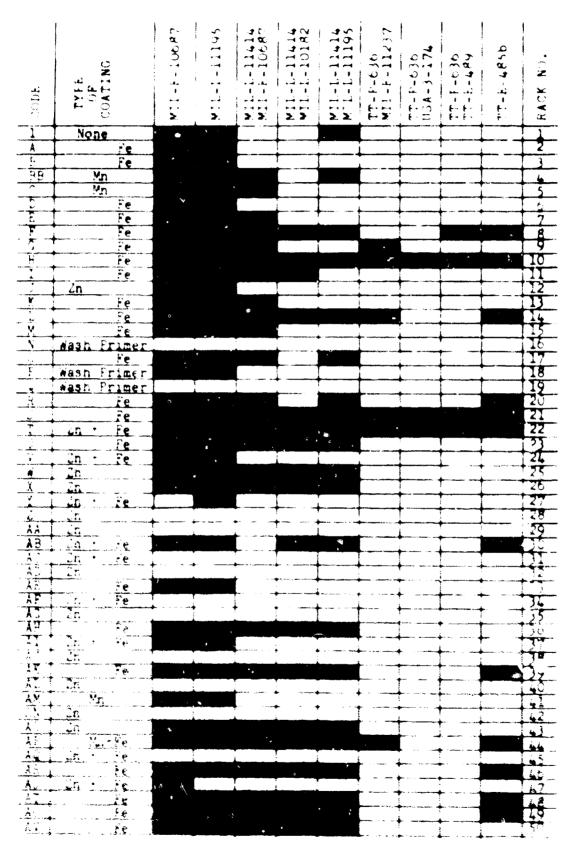
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4-1/2 YEARS OUTDOOR TXPOSURE (Filled Areas Indicate Failu s)

CHART VIII

Neg. No. 4620 63-3893

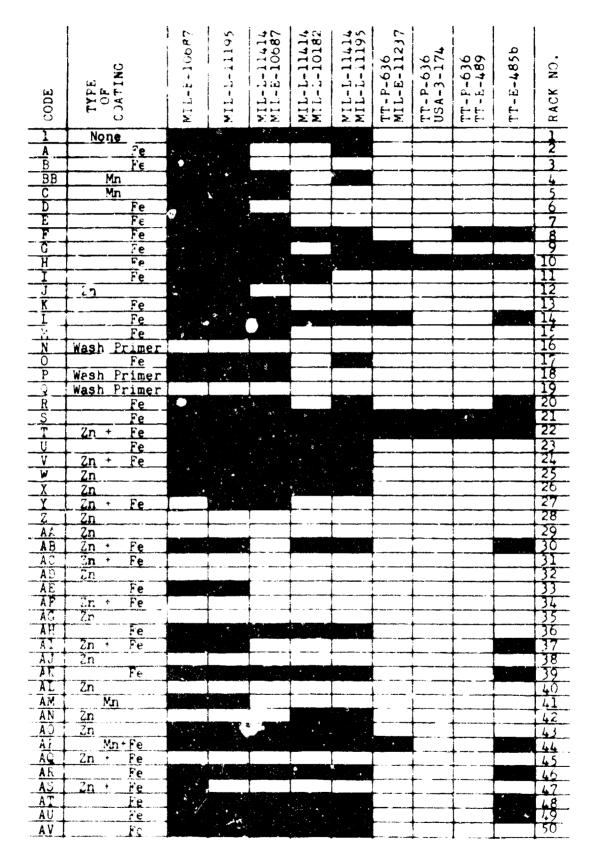


5 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate Failures)

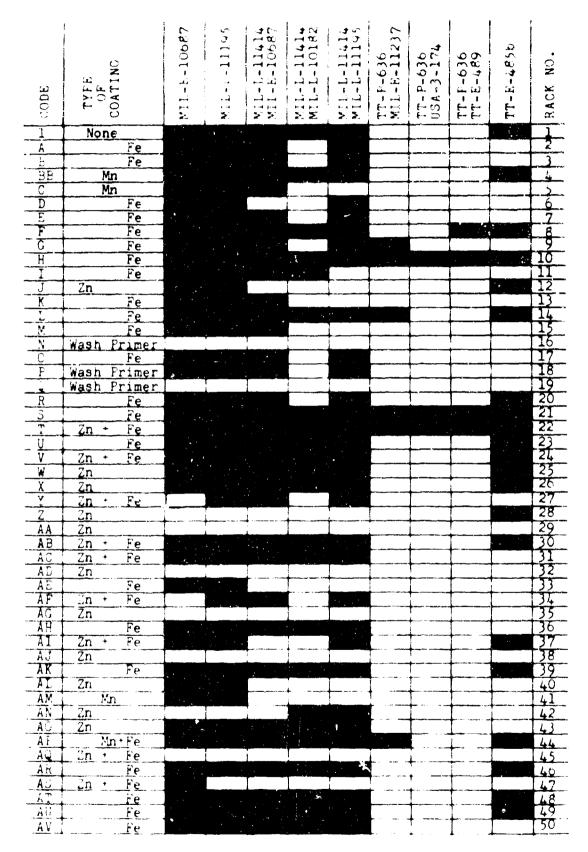
CHART IN

Neg. No. 4021 63-3893 . .

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6 YEARS OUTDOOR EXPOSITE (Filled Areas Indicate Failure)



7 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate Failures)

Neg. No. 4623 63-3893

CODE	TYPE OF COATING	MIL-E-10687	MIL-L-11195	MIL-L-11414 MIL-E-10687	MIL-L-11414 MIL-L-10182	MIL-L-11414	TT-P-636 MIL-E-11237	TT-P-636 USA-3-174	TT-P-636 TT-E-489	TT-E-485b	RACK NO.
	None				<u>.</u>						
	Fe Fe										2
BB	Ma re										4
0	Mn Mn										5
Ď	Fe	$[\mathcal{M}_{i}] = [\mathcal{M}_{i}]$									6
E	Fe										7
	re Fe Fe										8
<u> </u>	Fe Fe							L			10
- <u>H</u>	Fe				• • • • • • • • •				·		11
	Zn										12
K	Fe										13
L	Fe Fe										14
M	Fe					 				ļ	15
N	Wash Primer			1				 			16
0 P	Fe Wash Primer	6 B				•		 	<u> </u>		18-
- <u>-</u>	Wash Primer			1				<u> </u>	<u> </u>		18
R	Fe										20 21 22 23 24 25 26
S	Fe										21
T	Zn + Fe										22
<u> </u>	Fe				•			 			
V	Zn + Fe Zn										25
Y	Zn							1 · · · · · · · · ·			26
- Y	Zn + Fe							1	t		27
Z	Zn				• <u> </u>						28 29 30
AA	Zn										29
AB AC	Zn + Fe Zn + Fe	0	n de la composition de la comp			1	L			ļ	30
AC AD	<u>Zn + Fe</u> Zn		· · · ·	γ	Ċ			<u> </u>	 	}	3-
AD	<u> </u>							•	<u> </u>		111
AE AF	Fe Zn + Fe	Ì				1			<u> </u>		34
AC	Zn	1									35
AH	Fe			<u>, 1 – 4 – 4</u>							36
<u> </u>	Zn + Fe										37
AJ AK	Zn Fe		L						<u> </u>		38
-ÂL	Zn			· · · ·	1			+	 		40
AM	Mn			·····	<u>+</u>		t		<u>+</u>		41
AN	Zn								1		42
A0	Zn		•								43
AP	<u>Mn+Fe</u>						·				44
AQ	Zn + Fe										45
AR AS	<u> </u>									A	46
AT	Fe							t	t		47
AU	Fe										49
AV	Fe			÷ .		•					50

8 YEARS OUTDOOR EXPOSURE (Filled Areas Indicate Failures)

DISCUSSION AND RESULTS

It is virtually impossible to measure accurately the thickness of a phosphate coating. Reasonable accuracy is obtained, however, when measuring a fine and tightly built phosphate coating. Even in this instance, however, measurements vary over a given surface. Crystalline coatings present a poor surface upon which to use any contact type of film thickness gage, because the friable crystals break down under the applied pressure. This gives a low false reading. If one were to measure a paint film over a phosphate coating, one would obtain a composite reading equal to the combined paint film thickness and the mean thickness of the phosphate Some readings would be taken directly above the coating. apices of the phosphate crystals under the paint. The heavier and thicker the phosphate coating, the greater would be the potential error of measurement.

In order to avoid any possibility of error and to exactly reproduce the applied paint film, it was imperative to design and build a paint application mechanism for this project, Figure 2. Using this equipment, the paint film thickness can be controlled to within a \pm 0.05 mils tolerance. Also, in order to obtain this paint film thickness accuracy, clean steel (unphosphatized) panels were sprayed with the specific coating material, baked and thickness measurements taken. When all the adjustments were corrected and the proper paint film thickness was attained, 12, 24 or even 36 panels could be sprayed. Without exception there was no measureable wariation in the film thickness of the paint from the first panel to the last panel in such series.

As mentioned in the introduction, the phosphate coating weight and thickness are important. The most ideal coatings are those which have a moderate thickness and are composed of very fine crystals, tightly packed. Rock Island Arsenal Laboratory Report No. 57-383, "The Influence of Grit-Blasting on the Rate of Coating Formation in Room Temperature Phosphatizing Solutions,"⁽²⁾ shows that "phosphate crystals of great size impart a high coating weight, but a poor protective coatdng."

Specification TT-C-490 requires that zinc compounds shall be used in the Type I solution formulations. Forty mgs/sq ft after proper immersion time, implies that the content of the coating is iron phosphate only. Conventional iron phosphate coatings cannot easily surpass the 40 mgs/sq ft amount and they impart only moderate protection in outdoor exposure. It has been found that the iron phosphate prepaint treatments are weak against creeping and filiform corromion and are, therefore, undesirable. (Charts I thru XII). There are two considerations to keep in mind when evaluating progress in this exposure test, namely, the appearance of rust spots on the panel surface and the widening of the scored line (Figure 6 panels B and C respectively.) When two or more panels in a group of 6 panels shows 5 or more rust spots, however minute, the group is considered as having failed. Likewise when a scored line shows rust creepage to or beyond 1/8 of an inch on either side of the original score, the group of which this panel is a member is considered as having failed.

The first inspection was made 3 months after the initial exposure. At that inspection there was no apparent breakdown. (Figure 5).

During the second 3 month period, rust appeared for the first time. This rust appeared on group 13-18 in Set T.

By the end of the first year of exposure some discoloration had developed slong the scored lines. This condition was most pronounced on panel groups numbered 1 through 6 and 13 through 18 which were coated with only one coat of lusterless paint (Chart I). There was no evidence of blistering on any panel.

The portsity of the organic finishes (lusterless) used in the 1-6 and 13-18 groups is not conducive to good corrosion resistance. Neither do phosphate coatings afford a great deal of protection by themselves. If a thin phosphate coating has a good semi-gloss organic top coating, the protection will be much greater than when a lusterless organic coating is used.

One-coat systems of MIL-E-10687 Enamel, Lusterless, Quick-Drying (for Ammunition), dated 4 Feb. 1953, (Group 1-6) and MIL-L-11195A, Lacquer, Listerless, Hot Spray, dated 5 Jan. 1953 (Group 13-18) (Table II) were used in these tests. One can better compare results when using a single, porcus organic coating than when using a 2-coat system as the specification usually requires. The use of a poor organic coating makes it simpler to evaluate the part that phosphate coatings play in corrosion resistance. Figure 6 is a photographic closeup of 4 different types of developments in these tests after 5 years of outdoor exposure. Panel A is typical of a test panel with a good phosphate base coating with a good paint system. Parwl B shows a typical panel which had a poor phosphate base with a poor paint coating. Rust has formed over the whole surface. Panel C is typical of a steel panel which had been treated with a poor phosphate coating, but was finished with a good paint system. There was no rust on the surface, but rust creepage under the paint coat



ABCDGood PhosphatePoor PhosphatePoor PhosphateGood PhosphateGood PaintPoor PaintGood PaintPoor Paint

COMPARATIVE TESTS ON PHOSPHATE COATINGS

from the scored line due to the inability of the phosphate coating to help the exposed steel resist rust. Panel D is typical of a test panel which had a good phosphate treatment and a poor paint top-coat. The panel surface and scored line appear in good condition due to the good phosphate coating under a poor paint coating.

The results of these tests demonstrate the Superiority of zinc phosphate over iron phosphate coatings. By the use of various paints and paint systems it is possible to detect more easily the loss of protection and the degree of resistance depending on the companion organic coating. The fact that in some sets, certain groups failed quite early in this exposure and other groups in the same sets with another paint system are still intact after several years of exposure, demonstrates that there is some definite relationship between types of phosphate coatings and paint formulations.

Where the best paint systems (Groups 61-66, 73-78 and 85-90) were used, only 13 (8.7%) groups out of 150 had failed after 5 years of exposure and 23 (15.3%) out of 150 after 8 years. This can be compared to results obtained where the weaker paint systems (Groups 25-30, 37-42 and 49-54) were used. At the end of 5 years and 8 years the total failures were 61 (40.7%) and 109 (72.7%) respectively, cut of a possible 150. This resistance must be credited to the paint system. Where the organic coatings are inferior, the burden of protection is shared earlier by the phosphate coating.

Tible III shows the paints and paint systems and how many sets out of the 50 sets have failed under the specific coatings by the end of the 5th and 8+h year of exposure

The 4 prepaint treatments are shown in Table IV and Figure 7, which indicates the progress of each of the treatments over the 8 year span of the test. At the end of the 8 year period. 7 of the 20 zinc phosphate treatments are still intact. Of the 22 iron phosphate treatments all have failed. Of the 4 manganese phosphate treatments, all have failed. Of the 3 wash primers 1 is still intact.

All of the test specimens resisted the outdoor exposure for the firs' 3 months. There was one failure during the second 3 months, and a total of 4 failures at the end of 9 months of outdoor exposure. These 4 failures included 2 zinc and 2 manganese phosphating product treatments. By the end of the first year, 5 zinc, 10 iron and 3 manganese based phosphating products had failed. The wash primer treated sets were still intact, Chart I.

TABLI

5 AND 8 YEAR BOX SC

	Panel	Materials	Out of 5	
	No.	Specification	5 Years	8 Years
	1-6	MIL-E-10687	37	42
	13-18	MIL-L-11195	37	43
	25-30	MIL-L-11414) MIL-E-10687)	23	37
ä	37-42	MIL-L-11414) MIL-L-10182)	17	37
	49-54	MIL-L-11414) MIL-L-11195)	21	35
6 3 -				

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LE III

SCORE OF PAINT SYSTEMS

d	Panel		No. of Sets Failed Out of 50		
	<u>No.</u>	Specification	5 Years	8 Years	
	61-66	TT-P-636) MIL-E-11237)	6	8	
	73-78	TT-P-636) USA 3-174)	3	10	
	85-90	TT-P-636) TT-B-489)	4	5	
	97-102	TT-E-485b	12	23	

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TABLE

PREPAINT IREATMENT CHRONOLOGY OF CASUALTIES T

Number of Failures by Type of Netal Treatment

	Metal	Trea	a tmeni	t.			
YKAR	Zn	Ye	Yn	? ?	UT	1	2
1	5	10	3	0	0	17	2
2	6	16	4	0	1	27	10
3	8	18	4	1	1	31	30
4	10	22	4	1	1	37	35
5	10	22	4	1	1	37	37
6	10	22	4	1	1	38	37
7	10	22	4	1	1	3 9	40
8	13	22	4	2	1	42	43
Туре	of Tre	atm	Int				No. (
Zn =	Zinc 1	Phos	phate	trea	tød		1
Fe =	Iron						4
Mn = Manganese Phosphate treated							
WP =	Wash 1						
VI •	No tro	bat me	at				

TOTAL =

31

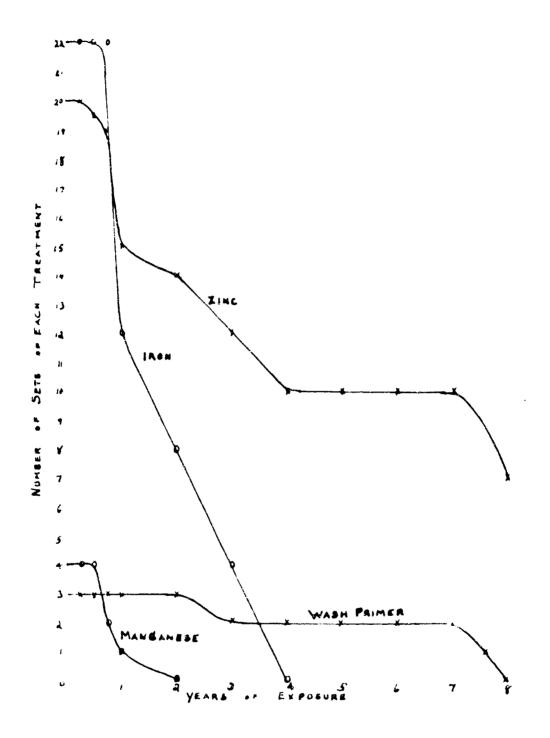
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<u>s iv</u>

NTS AND PAINT SYSTEMS THROUGH 8 YEARS OF EXPOSURE

		ber of Systems			Systems #9)	8	
2	3	4	5	ĥ	7	8	9
2	0	0	0	0	0	0	0
0	1	1	1	1	0	0	0
0	9	2	2	1	0	0	0
5	9	3	2	2	0	0	1
7	23	17	21	6	3	4	12
7	28	20	25	6	3	4	13
0	33	21	32	6	3	4	21
3	37	37	35	9	10	5	23
of Type							
20							
22 4							
3							
1							

30



DURABILITY OF 4 TYPES OF PHOSPHATE COATINGS UNDER PAINT SYSTEMS

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At the end of one year and 3 months, all four of the manganese phosphated sets had failed. The iron phosphated sets began to succemb to the elements during the 4th quarter of the first year and continued to fail at a very rapid rate. Of the 22 iron phosphated sets there were 11 failures by the end of 1-1/2 years. Five additional failures developed during the next half year. Two more failures occurred during the next six months. At the end of three years there were 18 failures out of the 22 original sets. Three more failures occurred during the three year, 9 month inspection that the remaining iron phosphated sampleswere recorded as failures.

Of the 3 wash primers only one failure had cocurred just prior to the 2-1/2 year inspection. The panels coated with the two remaining wash primers were still intact after 7 years of outdoor exposure. It was during the 8th year of exposure that the second set of panels with a wash primer began to show signs of failure.

One group of a set of zinc phosphated panels had failed by the end of the first six months. At the end of nine months exposure: the second group of zinc phosphated panels had developed rust. After 1 year of exposure five zinc phosphated sets had failed. As can be seen by Charts I through XII, there were no further changes in the zirc phosphated sets until after h-1/2 years of cutdoor exposure. By the end of 2 years six had failed, 2-1/2 years eight had failed, 3 years eight had failed and 3-1/2 years there were tep failures. This number remained unchanged through the 5th complete year. Therefore, by the end of 5 years of exposure, exactly 50% of the zinc phosphate treated sets were completely intact against rusting, blistering and peeling. This situation remained static through the 7th year. During the 8th year three more sets failed, leaving seven sets still intact through 8 complete years of exposure.

The fact that all 22 of the iron phosphated sets failed by the end of three years and nine months and the manganese phosphated sets at the end of one year and three months confirms the superiority of the zinc phosphate coating as a protection per se, and as a pretreatment to painting.

Since the above data is derived from the observations of failure of any one group of panels, it should be pointed out that as samples were failing within groups, after an initial failure, other groups within the same set were beginning to show weakness. It would require innumerable charts and tables to indicate this chronologically for each group and/or type of paint. These data can be found in Charts I through XII and Figure 7.

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Comparative evaluations definitely should be made on the basis of surface rust resistance in the first two single organic coatings finishes. These two coatings should be considered as standard because of their poor protection properties and would be first to be expected to fail. The phosphate coatings beneath these paint coatings can be evaluated accurately, since the organic coating slows down the corrosion and makes detection more obvious. However, a similar observation can be made on the other paint systems coatings No. 3 through No. 9. There were 2 paint primers used in these tests. One was a lacquer-formulated primer purchased under the Federal Specification MIL-L-11414, "Primer, Lacquer, Rust Inhibiting," dated 2 December 1952, and the other an enamel primer purchased under the Federal Specification TT-P-636b, "Primer Coating, Synthetic, Wood and Ferrous Metal," dated 23 February 1954. As can be seen in Charts I through XII, the paint systems where the lacquer primer is used are inferior to the enamel primer systems. Even the one coat system, using the material purchased under the Federal Specification TT-E-485b, "Enamel, Semi-Gloss, Rust-Inhibiting," (Coating No. 9) proved to be superior to the lacquer primer systems (Coatings No. 3, 4 and 5). See Table IV.

A composite check on the above mentioned paint systems (Groups 1-9) can readily be made by a glance at Table V.

TABLE V

FAILURE ACCORDING TO GROUPS

Coating No. 1 and 238	failures
Coating Nos. 3, 4 and 528	failures
Coating Nos. 6, 7 and 8 6	failures
Coating No. 912	failures

As far as determining whether a specific paint or paint system is more complimentory to a zinc, iron or manganese phosphate undercoat, these tests show that there are no discernible patterns. In this instance it can be seen that the enamel formulations and systems (Coatings 6, 7, 8 and 9) are longer lasting over phosphate coatings, than systems which contain lacquer formulations (Coatings 3, 4 and 5). It should be noted that Coating No. 9 is superior to Coatings No. 1 and No. 2. This undoubtedly is due, mainly, to the difference in the continuity of a semi-gloss coating and lusterless coatings. A semi-gloss paint film is much less porous than a lusterless paint film. Also, the No. 9 coating is formulated with rust inhibiting pigments. It should be noted at this point that in coatings Nos. 3, 4 and 5 in Table II, all were coated with the lacquer primer (MIL-L-11414), but No. 4 was top-coated with a gloss lacquer. There were only 21 failures out of 50 sets in No. 4, while No. 3 and No. 5 had 33 and 32 respectively at the end of 7 years. The latter two coatings had paint systems containing only lusterless coatings.

Although these tests were not initiated to determine the durability of organic coatings, nevertheless, it has been shown that various paint systems can team up with various phosphate coatings to enhance their overall resistance to weather.

Elsewhere in this report, it has been stated that in 10 instances out of 20, where zinc phosphate was used as the prepaint treatment, no ill effects have been noted on the test samples after 5 years of exposure and 7 still remain in excellent condition at the end of 8 years of exposure. The 22 samples of iron phosphate have all failed, the 4 samples of manganese phosphate failed early in the test, and 2 of 3 samples with wash primer (preprimer treatment) failed only after the exposure time approached the 8 year mark.

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