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INTRODUCTION

In the field of paint technology, two current problems brought about the recognition of a research need. The first, which has been of interest in the industry for many years, is the desire to accurately predict long-term durability of paint films in a short time. Progress made in this area has been through the use of accelerated testing conditions, which have made possible the prediction of relative durability of a film in about 500 test hours. Shortening of this time requirement is still a widespread goal in testing.

The second subject became one of concern more recently, as did most questions of environmental impact. With the ban of lead compounds from household surface coatings by the FDA, a barrage of studies ensued, and pressure began mounting for the elimination of lead compounds as components from all paint. Red lead oxide being one of the best known and most effective rust inhibitive pigments used in undercoats, the fact that its continued use appears in jeopardy has made the search for a suitable substitute a pressing responsibility.

Kewish[1], in his work with latex paint on wood, has asserted that x-ray diffraction appears to be of value in following changes due to weathering of paint films. He has also suggested that the various components of a paint behave independently of one another, to some degree. On this basis, it would seem that a very thin film of paint could be subjected to accelerated test methods and monitored by x-ray diffraction to give a rapid indication of long-term durability of the paint.

The goal of this study was to test the thin film theory as a rapid means of evaluating paint durability, using as test subjects primers thought to have value as substitutes for a long oil primer containing red lead oxide.

SCOPE

The scope of this project is two-fold:

(1) It is designed to perfect the application of x-ray diffraction to thin films of paint to monitor pigment changes that rapidly occur in the film.

(2) The secondary aim is to determine the best possible pigment substitute in lieu of lead pigments. This pigment substitute will have to be compatible with films of existing red lead pigments presently on bridges that need only spot repair.

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[1] Kewish, 1974, unpublished

METHODOLOGY

The step-wise program taken from the original research proposal is given below. Deviations from this program were only those dictated by data obtained, and such changes are subsequently described with the results which prompted them.

Program:

A. Obtain samples of substitutes for red lead in oil from various manufacturers. It may also be desirable to have a few specific formulations prepared by local manufacturers.
B. For each test paint establish what film thickness will allow significant corrosion of the steel substitute in the weather-o-meter in about 24 hours.
C. Expose similar films at 45° south and examine periodically for corrosion.
D. For each test paint, establish what thickness of film on steel will show significant corrosion in salt spray tests in about 24 hours.
E. With films on glass, significantly thicker than those used in the above tests but still measurable by X-ray techniques, determine the rate of erosion in the weather-o-meter.
F. Expose similar films at 45° south and determine the rate of erosion.
G. Conduct weather-o-meter, salt spray and 45° south exposure tests of the most promising paint(s) at usual film thickness.

TESTING EQUIPMENT AND CONDITIONS

Instruments and equipment used in this study are listed below, along with the standard operating conditions used, where applicable:

A. Salt Fog Cabinet

Conditions: Controlled temperature of $135 \pm 8^{\circ}F$ ($57.2 \pm 4.4^{\circ}C$) and salt concentration of 18%.

B. Weather-O-Meter

Conditions: Weather-o-meter exposure was conducted at a black panel temperature of $145 \pm 9^{\circ}F$ ($62.8 \pm 4.9^{\circ}C$), with a water spray lasting 18 minutes at 20 ± 3 psi (137.8 ± 20.8 kPa) for each 102 minutes of continuous ultraviolet light. Relative humidity was maintained at $85 \pm 5\%$.

C. Outdoor Exposure Racks

Conditions: Tilted 45° from normal to horizon, facing south.

D. Paint Sprayer - conventional type

E. Scanning Electron Microscope - AMR 1000 Model, equipped with Ortec Energy Dispersive X-Ray System

F. X-Ray Diffractometer - Phillips Wide Range Goniometer, XRG-3000 Generator, and Modular Data Measuring System

G. Biddle Gauge - standardized using precision paint shims.

RESULTS

Each step in the program is listed again below, followed by its results.

A. Obtain samples of substitutes for red lead in oil from various manufacturers.

Primers were requested for use in place of the oil/alkyd type primer containing red lead that is now in use, under specification AASHTO M 72. Of the original primers submitted, one had a latex vehicle, one was an oil-base enamel, and one was an epoxy ester. All others are oil/alkyd, similar to, or compatible with, red lead meeting specifications mentioned. Although the epoxy ester vehicle could not be used over existing red lead, it was included in order to provide supplemental information on the erosion test, on which the vehicle has an important influence.

Several of the primers contained chromates, another pigment group that has subsequently encountered mounting disfavor due to possible carcinogenic effects. It is possible that the use of chromates, as well as lead, will be restricted or even prohibited in the future. None of the primers originally submitted contained Busan II-M-I, or Nalzin SC-I, two of the newer environmentally safe pigments proposed for corrosion resistance. Another rust-inhibitive group, the molywhites, was represented only in a latex primer. Therefore, experimental primers using each of these three components were prepared at our request. A total of sixteen primers was accepted for study. Their inhibitive pigments and vehicle types are listed with code letters in Table I. Testing of Primer P was discontinued in the project, due to poor performance.

B. For each test paint, establish what film thickness will allow significant corrosion of the steel substrate in the weather-o-meter in about 24 hours.

No test paint allowed significant corrosion after 24 hours in the weather-o-meter, except in cases where portions of the panel were blatantly bare before weathering. Even coatings in the range of 0.2 to 0.3 mils showed no visible rusting, provided the films were fairly continuous.

C. Expose similar films at 45° south and examine periodically for corrosion.

After two weeks' outdoor exposure, paints H, J and E had rust on panels of 0.3 mils thickness or less. Three paints showed rust at a thickness of 0.5 mils or more. These included C, two panels of 0.5 mils thickness; E, one panel of 0.7 mils thickness; and N, one panel at 1.0 mils thickness. The fact that rust was visible to the eye showed no real correlation with intensity changes measured by x-ray diffraction. See Table IIA. The red lead panels are not shown on IIA, due to the fact that an interfering pigment peak prohibited a substrate reading. Further weathering caused rusting of panels in the order shown in Table IIB.

D. For each test paint, establish what thickness of film on steel will show significant corrosion in salt spray tests in about 24 hours.

As shown in Table III, this step involved the application of test paints at several thicknesses each, diluted when necessary with the appropriate thinner, to insure coverage as even as possible. After 24 hours in the salt fog, each panel was rated by 1" zones, according to the scale given below.

A = No rust or slight edge rust only
B = Isolated rust spots or rust spread from edges
C = Speckled appearance
D = Heavy rust

These ratings were converted to a numerical figure (A = 3), which was expressed as a per cent of the best possible rating, and this score used to denote whether or not significant corrosion had taken place. If five zones rated B or below, resulting in a numerical percentage of 85, then roughly half of the surface area of the panel showed some corrosion damage. It can be seen that all coatings of less than 0.5 mil fell below this score, except two of the standard red lead panels and the O panel of 0.4 mil thickness.

Those thicknesses from 0.5 to 1.0 mil showed the widest range of scores, from 97 for a 1.0 mil panel of Primer H, to 0 for a 0.5 mil panel of Primer C. Ratings of thicknesses above 1.0 mil ranged from 39 (1.9 mils Primer E) to 97 or above for seven panels showing little or no damage.

The best panels of each group rating 70 or above were replaced for a second 24 hours in the salt fog, where they showed performances of varying quality. See Table IV. Some scores were drastically diminished, while the thickest panels of red lead, B, I, and L, showed more moderate declines. The epoxy ester primer (0) was remarkably stable considering the low thicknesses applied.

E. With films on glass, significantly thicker than those used in the above tests but still measurable by x-ray techniques, determine the rate of erosion in the weather-o-meter.

Figure I shows 10 of the primers after weather-o-meter exposure, on glass plates. Since the red lead primer standard failed to adhere to the glass, no comparisons could be made between its resistance to erosion and that of the other primers. Primer I also flaked off the glass. See Figure 2.

F. Expose similar films at 45° south and determine the rate of erosion.

After 23 weeks, the average thickness loss on 12 primers ranged from 0 to 0.5 mils. Primer P showed a loss somewhat higher than the other primers, an average of 0.9 mils. See Table V. Primers G, I, J, and K showed swelling of the paint film.

G. Conduct salt spray, weather-o-meter, and 45° south exposure tests of the most promising paints at usual film thickness.

These tests were conducted on all primers, since the performances of thin films were not certain to be representative of the true durability of a paint. The coatings were applied by conventional spray gun, at thicknesses easily sprayed, giving good coverage. The resulting panels showed more gradual declines than brushed panels.

1. Salt Fog Results - After 72 hours of exposure, only a 3.0 mil red lead film and a 2.5 mil film of Primer G rated above 85, as rated according to the scale described in Step D. See Table VI, and Figures 3 and 4.

2. Weather-O-Meter Results - All panels except white exhibited marked fading in the first 250 hours of exposure. The order of failure (rusting) of panels coated with approximately 2.5 mils of paint was as follows, with failure due to flaking indicated by *: P, E*, Red lead*, J, at 650 hours; A, C, D*, F*, I, K, L, M, N, O, at 750 hours; B, G, H at 800 hours.

3. 45° South Exposure Tests - After 28 weeks' exposure, 2.5 mil panels of primers C, J, N showed failure by rusting. Primer P was cracked over the entire surface, and primer A showed heavy mildew growth. Primer O had faded from dark brown to a very light tan. Other panels showed little change in appearance.

DISCUSSION OF THIN FILM THEORY

During the course of this project, a substantial body of knowledge on the nature and behavior of a thin film of paint has been collected. Such generalized findings have bearing on the total program, and are perhaps even more important than the numerical data.

In their book X-Ray Absorption and Emission in Analytical Chemistry, Liebhafsky and his coworkers explain the determination of film thickness by x-ray spectroscopy over a range of coating thickness of .0004 inches to .006 inches. However, the examples cited deal with vacuum evaporated or plated films of a single metallic element onto a substrate metal. The effect of collisions of monochromatic x-rays with atoms of these surface metals would be predictable in this case, because absorption or diffraction of impinging x-rays is highly dependent on atomic number of the specimen. Therefore, if x-rays penetrate the coating and strike the substrate metal, the substrate intensity should generally increase with a decrease in coating thickness; on the other hand, intensity from the plating metal shows increase with increasing thickness, up to the critical depth of penetration of the x-rays.

The difference between this type of specimen and a thin film of paint on steel can be demonstrated with the aid of a scanning electron microscope. In Figure 5 of a film of paint on sand-blasted steel, note the nonhomogeneity of the coating at a microscopic level. The difference in pigment particle sizes may be easily seen, and the fact that these particles contain elements of different atomic number also alters the effect on x-rays. Thus, each impinging x-ray would strike a different "mini-environment" and would be absorbed or diffracted to a different degree. The first point to be noted in x-ray studies of paint films is the fact that even a well-mixed paint is not truly homogeneous on a sub-visual level. This causes difficulty in the duplication of x-ray test results even when the same can of paint is used on the same day. The optimum result in this case would be that the composite effect of all collisions on the x-rays would be representative for a given paint. However, the limited usefulness inherent in this average of dissimilar subjects finds a simple analogy in the mean of apples and oranges.

In addition to the consideration of the homogeneity of a coat of paint, the question arises for a thin film study of the minimum thickness necessary for continuity. In the light micrographs (Figures 6 and 7), a comparison is made of a 3.0 mil coating of red lead on steel to a 0.5 mil coating, the latter figure falling below a theoretical critical thickness required in some x-ray measurements. Note the obvious areas in the 0.5 mil coating where pigment is absent. In this case, the results of an accelerated corrosion test could not be considered representative of the effectiveness of an inhibitive pigment, since in certain areas no pigment is available to protect the steel.

The third and final drawback of a thin film paint study on steel substrates is the question of vehicle integrity. Dilution being the only method available for getting films of relative continuity (i.e., those which appear continuous to the naked eye) at thicknesses below 1 mil, considerations of porosity and adhesion of the film to the substrate come to the forefront. If the oil/alkyd portion of the paint is diluted below the usual consistency of paint for spreading, the barrier effect of the vehicle is lessened. This detrimental effect is not necessarily of the same magnitude for the same dilution in vehicles employing different oils and resins; therefore, the use of high dilutions might give an unfair comparison of test sugjects.

Taking into account the problems of maintaining homogeneity, film continuity, and vehicle integrity, it would appear that satisfactory comparisons of different paint systems would be extremely difficult by the thin film technique using x-ray diffraction. However, if a system shows itself to perform well in ordinary laboratory and field tests, a thin film technique could be useful in monitoring product quality from one batch/job to another. In this case, the pigment and vehicle type would remain the same, and only the relative amounts would vary. The performance in this application lends itself much better to evaluation by the theory in question.

DISCUSSION OF NUMERICAL DATA

The three major weaknesses in the thin film theory are obviously reflected in the specific tests of the work program. For example, in part B, there exists an ideal thickness of each paint which would result in corrosion of the substrate in the weather-o-meter in 24 hours. However, a practical method for applying paint at a thickness below 0.2 mils has not been found. Therefore, the bias due to discontinuity of films and alterations in vehicle behavior due to dilution would make a 24-hour weather-o-meter test of little value.

Part C is affected by the varying concentrations of paint ingredients at a given site of examination, and by varying x-ray absorption characteristics due to particle size and atomic number of the elements. The wide range of thicknesses used also makes the films susceptible to the effects discussed in the previous paragraph. The fact that rusted panels were not recognized as a separable group from the data (Table II) is due to these effects on all panels, and also to other events registering simultaneously in the results.

The presence of rust causes background counts to increase, the subtraction of which would decrease net peak intensities of both the substrate and the pigment peaks. However, the fact that certain pigments are chalking out of the binder makes them more available to diffract x-rays, and thereby increases the peak intensities of these pigments. As a general trend, it will be noted that those panels showing an increase in the substrate peak/pigment peak intensity ratios gave consistently poor performances in all tests. This change would indicate that less pigment is present on the panels, therefore, more substrate metal is exposed.

A decrease in the substrate peak/pigment peak intensity ratio probably indicates that the binder is being eroded, thereby leaving pigment more exposed to x-ray bombardment. A stable ratio would either indicate no changes in the film, or more likely a counterbalancing of the two effects mentioned. In any case, both good and poor performers showed a decrease or no change in this ratio, which precludes trending of these results. The eight panels which had visible rust had no common response, as well as no categoric difference from the unrusted panels. If the diffractometer were picking up changes before rust becomes visible, one would expect that the rusted panels would show agreement among themselves, although some panels which appear to be uncorroded might also match the group.

In part D, the salt fog test is also affected by dilution and discontinuity. Those paints showing a good performance for a thicker film, followed by a sharp decline as thickness decreases, probably are affected to the greatest degree by dilution. Those paints doing poorly at all thicknesses in 24 hours would suffer from deficiencies as rust inhibitors. On the contrary, paints showing only a gradual decline with additional weathering show predictable behavior of good rust inhibitive primers. Widely varying behavior by zones on a given panel would be the best evidence of continuity difficulties, although a consistently low rating at a minimum thickness might also reflect this effect.

Since the red lead primer, to which the other paints were compared, did not adhere to the glass, part E has little meaning. Delamination from this glass surface is not necessarily an evidence of poor performance, since a primer is not ordinarily applied to a surface with that degree of smoothness. The adherance of red lead to a steel surface with good anchor pattern is not questioned.

In part F, the similarity of results obtained on erosion of all primers indicate that corrosion of the substrate is an important factor in the lasting integrity of a film. Only Primer P showed an erosion figure significantly removed from the main body of results. It appears that a vehicle that is relatively impervious to erosion by outdoor conditions is relatively common, and that corrosion is a more prominent cause of failure.

Part G shows the behavior of the paints at ordinary thicknesses. From the salt fog data (Table VI), it can be seen that corrosion occurred more gradually at these thicknesses, probably due to the fact that coverage was even. Primer G gave a remarkable performance in this group, compared to the thin film performance in Table III. This is further evidence that, at least in some products, the damage to performance properties by dilution and inadequate coverage make a thin film non-representative of product quality.

Further proof of the importance of the vehicle in paint durability is given in the comparisons of performance and physical test results (Table VII) of paints containing the same inhibitive pigment. It will be noted from Table I that primers A, M, N, and O offer zinc chromate as their rust inhibitive pigment. Primer O has an epoxy ester vehicle, while the others are all modifications of an alkyd type paint. A quick review of the performance of these paints shows that Primer O gave a much better performance at thin applications than the others, even though it ranked lowest physically in density and solids content. Of the three alkyds types, however, Primer A had the highest weight/gallon and percent nonvolatiles, and also gave a slightly better performance overall than M and N.

Another group which points out vehicle effects is composed of B, G, and J. In the exposure tests results, paints G and B outperformed paint J. This is unusual indeed, if pigment composition is considered. Paint G contains Nalzin, and has a lower solids content than either B or J. Primer B contains red iron oxide in a thixotropic (gel) vehicle. Primer J, which contains both Nalzin and red iron oxide, at an intermediate nonvolatile content level, performs inferiorly to the other two. In this case, it will be noted that the density of Primer J was the lowest of the three paints. All these considerations point to a substantial vehicle effect on paint performance.

The outdoor exposure of these ordinary thicknesses at 45° south is also very severe for these test paints, since a primer depends heavily on a topcoat for its own protection. The red lead standard has chalked and faded markedly in one year of exposure, although no significant rust has occurred. Considering the fact that a primer is designed to prepare the substrate for further coats, performance of test paints on being exposed alone is very good as a group. The facts that Primer C has rusted, that N and A have mildewed, and that P has shown cracking are certainly consistent with their poor performance in other portions of this project.

Weather-o-meter exposure does not seem to be the best indication of corrosion resistance of a primer, since flaking occurs before rusting in many cases, and the exposed steel becomes the site for rust. Such behavior is not necessarily an indication of how a primer will perform after being topcoated, and no conclusions can be drawn.

CONCLUSIONS AND RECOMMENDATIONS

Little value was found in the use of thin films of paint on steel to monitor corrosion resistance. Therefore, no implementation of such a testing scheme is planned.

It is also evident from the combined test results that none of the primers tested approached the performance of the red lead primer in corrosion resistance. As a group, the zinc chromate primers offer no clear-cut advantage over primers containing non-toxic pigment. It is therefore our opinion that a system utilizing Nalzin SC-I pigment would be the best substitute of the paints tested in areas where an oil/alkyd system without lead pigment is required. It is further recommended that great care be exercised in the choice of a vehicle. Vehicle contribution to total performance could be studied far better if a scheme standardizing content of like pigment were used in a variety of vehicles.

APPENDIX A

TABLES

TABLE I

TYPES OF PIGMENTS & CORRESPONDING VEHICLES

CODE LETTER	INHIBITIVE PIGMENT	VEHICLE TYPE
A	Zinc Chromate	Modified Alkyd
B	Red Iron Oxide	Modified Alkyd
C	Molywhite 212	Latex
D	Zinc Phosphate	Chlorinated Rubber
E	Zinc Phosphate w/Iron Oxide	Oil Alkyd
F	Zinc Phosphate w/Iron Oxide	Oil Alkyd
G	Nalzin SC-1	Oil Alkyd
H	Zinc Chromate/Iron Oxide Basic Lead Silico Chromate	Synthetic Resin
I	Busan 11 -MI/Iron Oxide	Oil Alkyd
J	Nalzin SC -1/Iron Oxide	Oil Alkyd
K	Molywhite 101/Iron Oxide	Oil Alkyd
L	Titanium Dioxide/Yellow Iron Oxide	Oil Enamel
M	Zinc Chromate/Iron Oxide	Alkyd
N	Zinc Chromate	Synthetic Resin
O	Zinc Chromate	Epoxy Ester
P	Micaceous Iron Oxide	Chlorinated Rubber

TABLE II A

EFFECTS OF TWO WEEKS' OUTDOOR EXPOSURE AT 45° SOUTH ON X-RAY INTENSITIES

Panel #	Original Thickness (mils)	Unweathered $\frac{I_s}{I_F}$ [1]	$\frac{P_s}{B_s}$ [2]	$\frac{P_F}{B_F}$ [3]	Weathered $\frac{I_s}{I_F}$	$\frac{P_s}{B_s}$	$\frac{P_F}{B_F}$	$\Delta\frac{I_s}{I_F}$	$\Delta\frac{P_s}{B_s}$	$\Delta\frac{P_F}{B_F}$	Rust Present
A	2.0	0.2	2.6	10.3	0.3	3.2	9.6	+0.1	+0.6	-0.7	
2	0.7	0.9	3.0	3.7	0.6	3.6	4.2	-0.3	+0.6	+0.5	
3	0.1	3.7	3.5	2.5	2.4	2.5	1.7	-1.3	-1.0	-0.8	Yes
B 1	1.6	0.1	1.1	2.3	0.1	1.1	2.5	0.0	0.0	+0.2	
2	0.5	1.9	2.1	1.9	1.6	2.3	1.8	-0.3	+0.2	-0.1	
3	0.4	2.9	2.4	1.6	4.9	2.7	1.3	+2.0	+0.3	-0.3	
C 1	2.5	0.2	3.1	14.3	0.2	3.8	16.5	0.0	+0.7	+2.3	
2	0.9	0.2	3.3	15.2	0.2	3.2	17.0	0.0	-0.1	+1.8	Yes
3	0.6	1.3	3.5	3.6	1.1	3.2	3.8	0.2	-0.3	+0.2	Yes
D 1	2.3	0.0	1.3	5.0	0.0	1.3	6.5	0.0	0.0	+1.4	
2	1.2	0.3	2.6	6.8	0.3	3.0	9.5	0.0	+0.4	+2.7	
3	0.8	0.3	2.7	6.6	0.3	3.0	7.6	0.0	+0.3	+1.0	
E 1	1.8	0.1	1.3	3.9	0.1	1.4	4.1	0.0	+0.1	+0.2	
2	0.7	0.4	2.5	5.0	0.4	2.3	4.8	0.0	-0.2	-0.2	Yes
3	0.3	1.0	2.9	3.0	1.2	3.2	2.9	+0.2	+0.3	-0.1	Yes
F 1	2.6	0.1	1.1	2.0	0.0	1.0	5.2	-0.1	-0.1	+0.4	
2	0.7	0.8	2.3	2.9	0.6	2.3	3.3	-0.2	0.0	+0.4	
3	0.5	1.0	2.2	2.4	1.5	2.7	2.3	+0.5	+0.5	-0.1	
G 1	2.5	0.1	2.9	14.6	0.1	3.0	18.7	0.0	+0.1	+4.1	
2	0.8	1.0	3.7	4.5	1.0	3.6	4.6	0.0	-0.1	+0.1	
3	0.5	1.0	3.6	4.5	0.9	3.1	4.4	-0.1	-0.5	-0.1	
H 1	0.9	0.7	1.3	1.2	0.4	2.1	4.0	-0.3	+0.8	+2.8	
2	0.4	2.2	1.2	1.3	1.0	2.9	3.1	-1.2	+1.7	+1.8	
3	0.2	3.2	1.1	1.3	1.7	3.1	2.3	-2.5	+2.0	+1.0	Yes

- continued -

TABLE II A (continued)

Panel #	Original Thickness (mils)	Unweathered $\frac{I_s}{I_F}$ [1]	Unweathered $\frac{P_s}{B_s}$ [2]	Unweathered $\frac{P_F}{B_F}$ [3]	Weathered $\frac{I_s}{I_F}$	Weathered $\frac{P_s}{B_s}$	Weathered $\frac{P_F}{B_F}$	$\Delta \frac{I_s}{I_F}$	$\Delta \frac{P_s}{B_s}$	$\Delta \frac{P_F}{B_F}$	Rust Present
I 1	2.8	0.0	1.1	4.6	0.1	1.1	2.1	+0.1	0.0	-2.5	
2	0.9	0.1	2.0	9.0	0.1	2.0	10.7	0.0	0.0	+1.1	
3	0.4	1.3	2.9	2.6	0.4	2.8	2.5	+0.1	-0.1	+1.1	
J 1	2.5	0.2	1.6	3.8	0.4	1.6	2.5	+0.2	0.0	-1.3	
2	1.2	0.7	2.7	4.1	1.7	2.4	2.2	+1.0	-0.3	-1.9	
3	0.3	1.0	3.0	4.0	4.1	3.3	1.9	+3.1	+0.3	-2.1	Yes
K 1	1.4	0.3	2.2	5.2	0.3	2.2	5.8	0.0	0.0	+0.6	
2	1.0	0.9	2.9	3.5	0.8	2.8	3.6	-0.1	-0.1	+0.1	
3	0.5	1.0	3.0	3.3	0.9	3.1	3.7	-0.1	+0.1	+0.4	
L 1	2.3	0.0	1.4	24.8	0.0	1.7	31.9	0.0	+0.3	+7.1	
2	0.8	0.5	3.4	9.8	0.5	3.5	9.6	0.0	+0.1	-0.2	
3	0.7	0.3	3.5	13.0	0.4	3.8	13.0	+0.1	+0.3	0.0	
M 1	1.1	1.6	2.7	2.2	1.5	2.7	2.4	-0.1	0.0	+0.2	
2	0.6	2.6	2.8	2.0	2.2	3.0	2.1	-0.4	+0.2	+0.1	
3	0.4	8.8	3.4	1.3	5.0	3.3	1.3	-3.8	-0.1	0.0	
N 1	4.6	0.1	1.6	5.6	0.1	1.7	6.2	0.0	+0.1	+0.6	
2	2.3	0.3	2.8	6.0	0.3	2.9	6.1	0.0	+0.1	+0.1	
3	1.0	0.4	2.6	5.0	0.5	2.8	4.5	+0.1	+0.2	-0.5	Yes
O 1	1.4	0.1	1.2	3.7	0.1	1.2	3.9	0.0	0.0	+0.2	
2	1.0	0.2	1.5	3.3	0.3	1.5	3.2	+0.1	0.0	-0.1	

[1] Ratio of substrate (steel) peak intensity (I_s) to peak intensity of major pigment peak in film (I_F)
[2] P_s/B_s is the peak to background ratio for the substrate
[3] P_F/B_F is the peak to background ratio for pigment in film

TABLE II B

LENGTH OF OUTDOOR EXPOSURE
CAUSING APPEARANCE OF RUST

Panel #	Original Film Thickness (mils)	Outdoor Exposure (weeks)					
		2	3	4	5	8	10
A 1	2.0						
2	0.7				X		
3	0.1	X					
B 1	1.6						
2	0.5					X	
3	0.4				X		
C 1	2.5			X			
2	0.9	X					
3	0.6	X					
D 1	2.3						
2	1.2						X
3	0.8						
E 1	1.8						
2	0.7	X					
3	0.3	X					
F 1	2.6						
2	0.7						
3	0.5						
G 1	2.5						
2	0.8						
3	0.5			X			
H 1	0.9						
2	0.4						
3	0.2	X					
I 1	2.8						
2	0.9						
3	0.4			X			

- continued -

TABLE II B (continued)

Panel #	Original Film Thickness (mils)	Outdoor Exposure (weeks)				
		2	3	4	5	8
J 1	2.5					
2	1.2			X		
3	0.3	X				
K 1	1.4					
2	1.0					
3	0.5					
L 1	2.3					
2	0.8			X		
3	0.7			X		
M 1	1.1					
2	0.6					
3	0.4					X
N 1	4.6			X		
2	2.3		X			
3	1.0	X				
O 1	1.4					
2	1.0					
Red Lead 1	3.0					
2	1.9					
3	0.5					
4	0.3					
5	0.2				X	

TABLE III

APPEARANCE OF BRUSH-PAINTED PANELS
AFTER 24 HOURS' SALT FOG EXPOSURE

Code	Thickness (mils)*	Appearance Score	Average Ratings by Zones 1	2	3	4	5	6	7	8	9	10	11
Red Lead	3.0	100	A	A	A	A	A	A	A	A	A	A	A
	1.9	100	A	A	A	A	A	A	A	A	A	A	A
	0.5	94	A	A	A	A	A	A	A	A	B	A	B
	0.3	85	B	B	A	A	A	A	A	B	A	B	B
	0.3	82	B	B	B	A	A	A	A	A	B	B	B
	0.2	52	D	C	B	B	C	B	C	B	C	C	C
A	1.6	100	A	A	A	A	A	A	A	A	A	A	A
	0.4	39	C	C	C	C	C	C	B	C	C	C	B
	0.1	0	D	D	D	D	D	D	D	D	D	D	D
B	1.8	100	A	A	A	A	A	A	A	A	A	A	A
	0.5	88	B	D	A	A	A	A	A	A	A	A	A
	0.4	64	D	D	B	B	A	B	A	B	A	A	A
C	1.5	88	A	A	A	B	A	B	A	B	A	B	A
	0.5	15	D	C	C	D	D	C	C	C	D	D	D
	0.5	0	D	D	D	D	D	D	D	D	D	D	D
D	1.7	94	B	A	A	A	A	A	A	B	A	A	A
	0.8	82	B	B	B	A	A	A	A	A	B	B	B
	0.7	82	B	B	A	A	B	A	A	A	B	B	B
E	1.9	39	D	D	B	D	B	D	A	D	B	B	B
	0.7	64	B	B	C	B	B	B	B	B	B	B	B
	0.2	0	D	D	D	D	D	D	D	D	D	D	D
F	2.4	67	B	B	B	B	B	B	B	B	B	B	B
	0.6	79	B	C	A	B	A	A	A	A	A	A	D
	0.4	12	C	D	C	D	D	D	D	C	D	C	D
G	3.4	97	B	A	A	A	A	A	A	A	A	A	A
	0.8	61	B	B	C	B	B	A	B	C	B	C	B
	0.4	61	B	B	B	A	C	A	B	B	D	C	B
H	1.0	97	A	B	A	A	A	A	A	A	A	A	A
	0.4	94	A	B	A	A	A	A	A	A	A	A	A
	0.2	39	B	C	B	C	C	C	C	C	C	C	C

* 1 mil = 0.0254 mm

- continued -

TABLE III (continued)

Code	Thickness (mils)*	Appearance Score	Average Ratings by Zones 1	2	3	4	5	6	7	8	9	10	11
I	3.0	97	A	B	A	A	A	A	A	A	A	A	A
	0.8	70	C	A	C	A	C	A	C	A	C	A	A
	0.3	45	B	C	A	C	C	D	B	D	B	B	C
J	2.6	42	A	A	B	A	D	C	D	C	D	C	D
	1.0	30	B	D	C	D	D	B	D	B	D	B	D
	0.3	9	D	C	D	D	D	D	D	D	D	B	D
	0.15	9	C	D	C	D	C	D	D	D	D	D	D
K	1.4	94	B	B	A	A	A	A	A	A	A	A	A
	0.2	91	B	A	A	A	A	A	A	B	A	A	B
	1.0	91	A	D	A	A	A	A	A	A	A	A	A
L	2.7	100	A	A	A	A	A	A	A	A	A	A	A
	0.9	91	B	B	A	B	A	A	A	A	A	A	A
	0.7	91	C	D	C	D	D	D	D	C	D	C	D
M	1.1	85	B	B	B	A	B	A	B	A	A	A	A
	0.4	97	B	A	A	A	A	A	A	A	A	A	A
	0.3	18	D	C	C	C	B	D	C	D	D	D	D
N	5.6	76	C	B	A	B	B	A	B	A	B	A	B
	2.5	52	C	B	B	B	B	C	B	C	C	C	B
	1.0	39	C	C	C	C	C	C	C	C	C	C	C
O	0.5	94	B	B	A	A	A	A	A	A	A	A	A
	0.4	91	B	A	B	A	A	A	A	A	A	B	A
	0.2	79	B	B	B	A	A	A	A	B	B	B	B

* 1 mil = 0.0254 mm

TABLE IV

APPEARANCE OF BRUSH-PAINTED PANELS
AFTER 48 HOURS' SALT FOG EXPOSURE

Code	Thickness (mils)*	Appearance Rating	Average Ratings by Zones 1	2	3	4	5	6	7	8	9	10	11
Red Lead	3.0	100	A	A	A	A	A	A	A	A	A	A	A
	1.9	88	B	A	B	A	B	A	B	A	A	A	A
	0.5	42	D	D	D	B	B	B	B	B	B	C	C
A	1.6	33	C	C	C	D	D	D	C	B	C	C	B
B	1.8	76	C	B	B	A	B	A	A	A	B	B	B
C	0.5	3	C	D	D	D	D	D	D	D	D	D	D
D	1.7	33	C	C	C	C	C	C	C	C	C	C	C
G	3.4	33	C	C	C	C	C	C	C	C	C	C	C
H	1.0	42	C	C	B	B	C	C	C	C	B	C	C
	0.4	24	D	D	C	C	C	C	C	C	C	C	D
I	3.0	88	B	B	B	A	A	A	A	A	A	B	A
K	1.4	55	C	C	B	B	B	B	C	B	C	B	B
	1.0	45	C	D	B	B	B	C	C	B	C	B	C
	0.2	42	C	D	C	B	C	B	C	B	C	BQ	C
L	2.7	88	B	A	B	B	B	A	A	A	A	A	A
	0.9	27	D	C	C	C	C	C	C	C	C	C	D
	0.7	9	D	D	D	D	D	D	D	D	C	C	C
O	0.5	84	B	B	B	A	B	A	B	A	B	A	A
	0.4	80	B	A	B	A	B	A	B	A	B	A	B
	0.2	70	B	B	B	B	B	B	B	B	B	B	B

* 1 mil = 0.0254 mm

TABLE V

AVERAGE THICKNESS LOSS BY EROSION (on glass)
AFTER 23 WEEKS' OUTDOOR EXPOSURE

Code Letter	Thickness Loss (mils)*
Red Lead	0.4
A	0.4
B	0.4
C	0.2
D	0.1
E	0.3
F	No thickness change
G	Coating Swelled (0.3 mil)
H	0.2
I	Coating Swelled (0.2)
J	Coating Swelled (0.4)
K	Coating Swelled (0.3)
L	0.5
M	0.3
N	0.5
O	0.1
P	0.9

* 1 mil = 0.0254 mm

TABLE VI

APPEARANCE OF SPRAY PAINTED PANELS AFTER SALT FOG EXPOSURE

		APPEARANCE RATINGS		
CODE LETTER	MILS *	17 HOURS' EXPOSURE	51 HOURS' EXPOSURE	72 HOURS' EXPOSURE
Red Lead	3.0	100	97	95
A	3.4	100	83	78
B	1.8	100	86	66
C	3.5	64	--	--
D	3.0	100	75	64
E	3.25	100	80	71
F	3.25	100	80	71
G	2.5	100	92	88
H	1.0	100	90	80
I	3.5	100	80	69
J	2.5	88	70	59
K	2.75	100	81	73
L	2.7	100	79	73
M	1.6	100	70	49
N	4.0	39	--	--
O	2.4	100	80	71
P	3.1	43	--	--

* 1 mil = 0.0254 mm

TABLE VII

PHYSICAL TEST RESULTS

Code Letter	Density lb/gal.* (ASTM D-1475)	% Nonvolatile Matter (ASTM D-2369)	% Solids by Volume	Dry To Touch (min) (ASTM D-1640)	Dry Hard (hrs.) (ASTM D-1640)
A	12.1	71.5	47	5	0.4
B	12.7	78.1	60	45	24
C	11.6	59.9	42	5	0.3
D	11.1	63.2	27.5	5	0.8
E	12.7	88.1	75	390	23
F	12.7	80.7	59	330	22
G	12.3	57.7	63	360	23
H	10.3	64.5	39	15	3.5
I	11.2	76.1	58	90	5.5
J	11.2	73.9	58	60	5.5
K	11.6	77.3	57	330	23
L	11.9	73.2	52	60	5
M	10.3	69.2	46	60	23
N	11.0	68.5	47	20	3.5
O	10.5	56.6	36.4	12	0.5
Red Lead	24.0	93.4	93	270	30

* 1 lb/gal. = 0.1199 kg/l

APPENDIX B

FIGURES

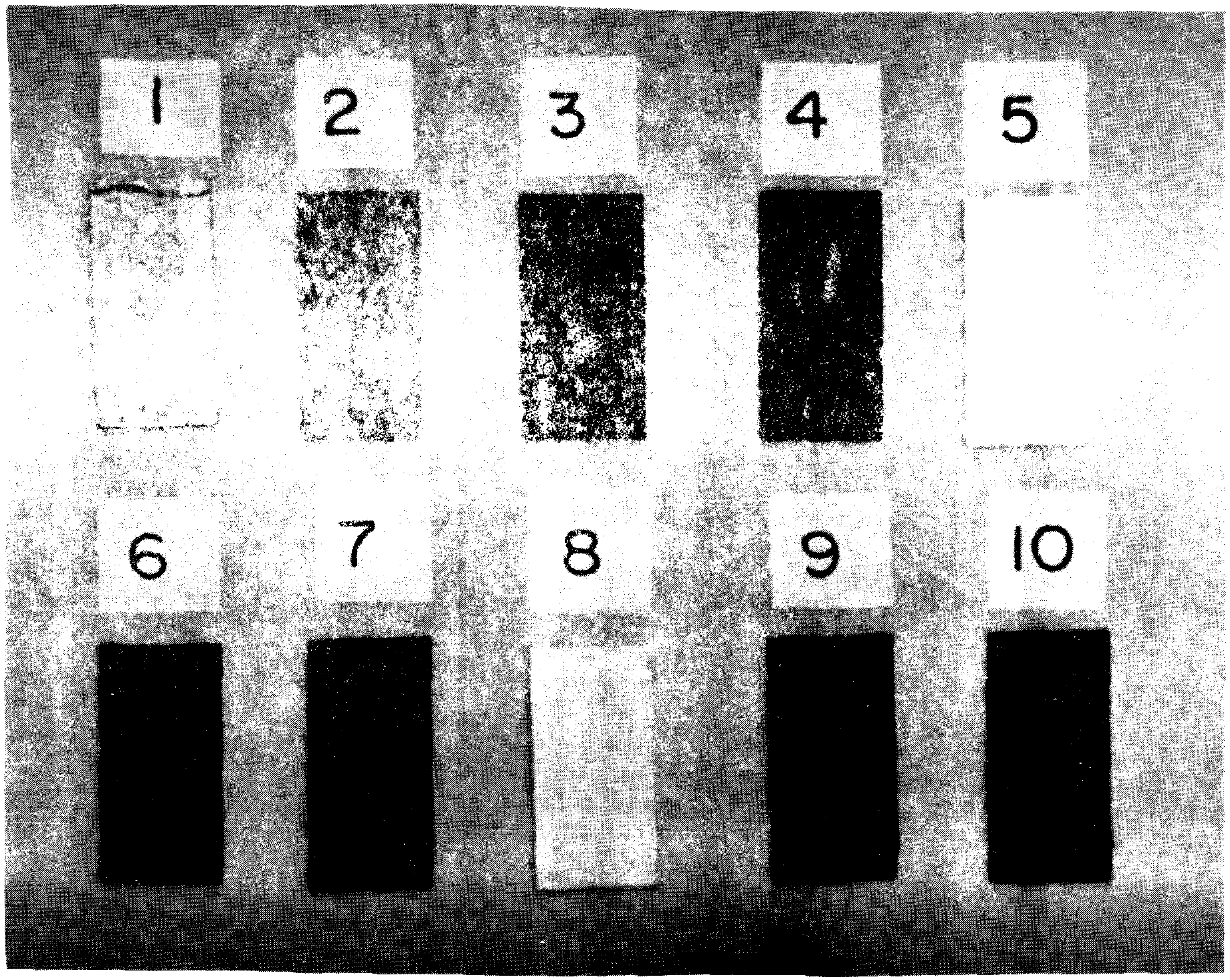

Figure 1. Glass Plates after Weather-o-meter exposure. (l-r top): Red Lead, A, B, H, C, (l-r bottom): N, M, O, L, P

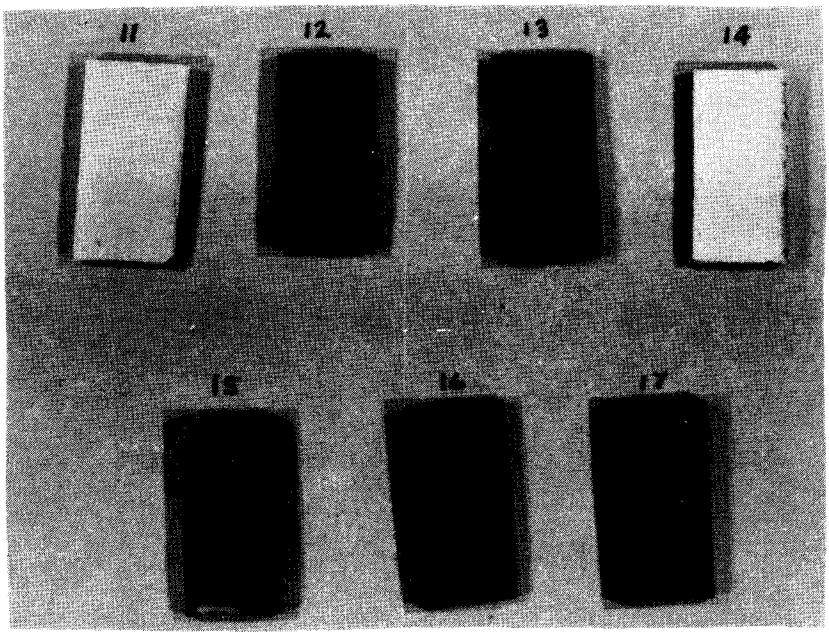

Figure 2. Glass Plates After Weather-O-Meter exposure, in the numerical order shown: D, E, F, G, I, J, K.

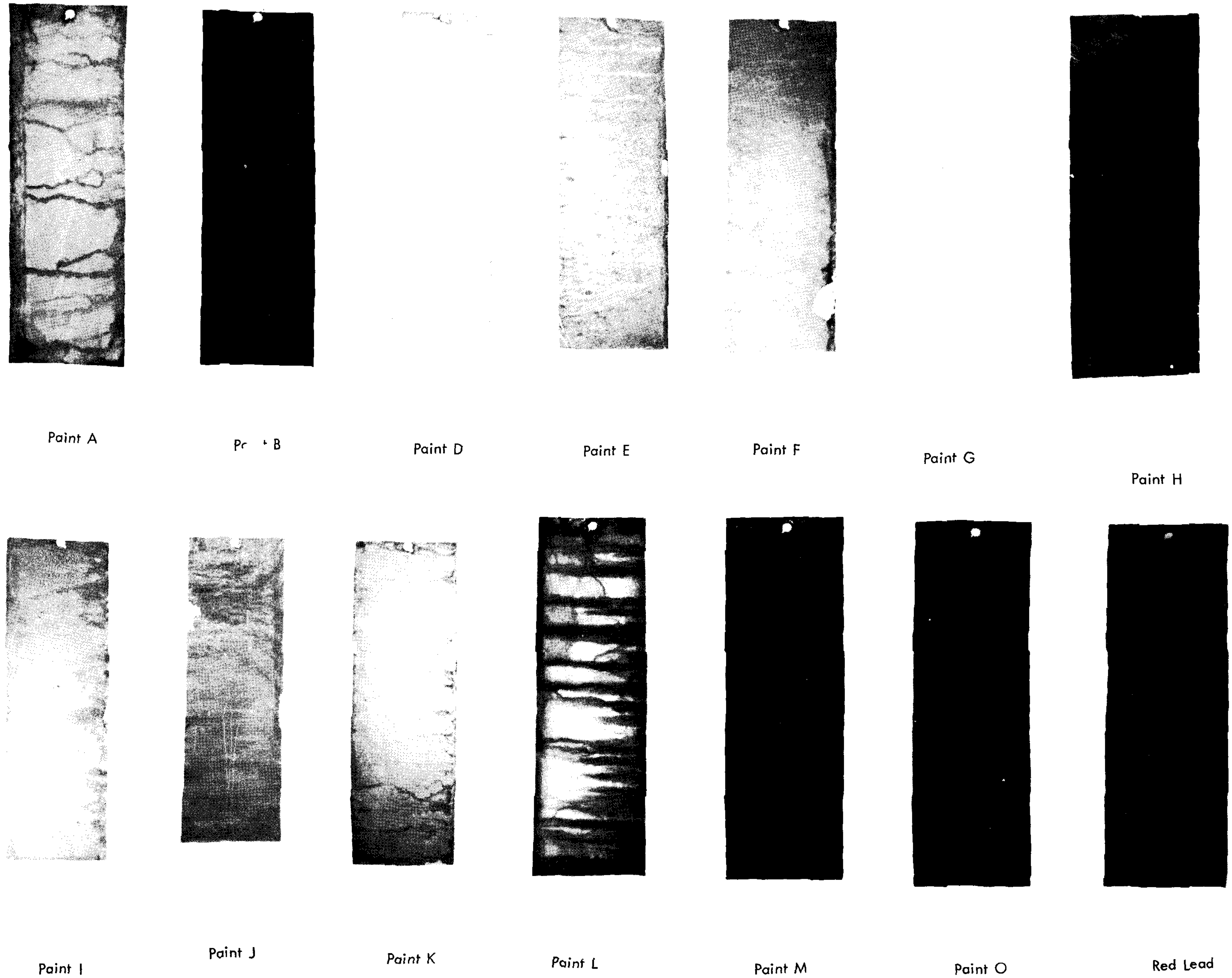

Figure 4 Appearance of Panels after 72 Hours Salt Fog Exposure

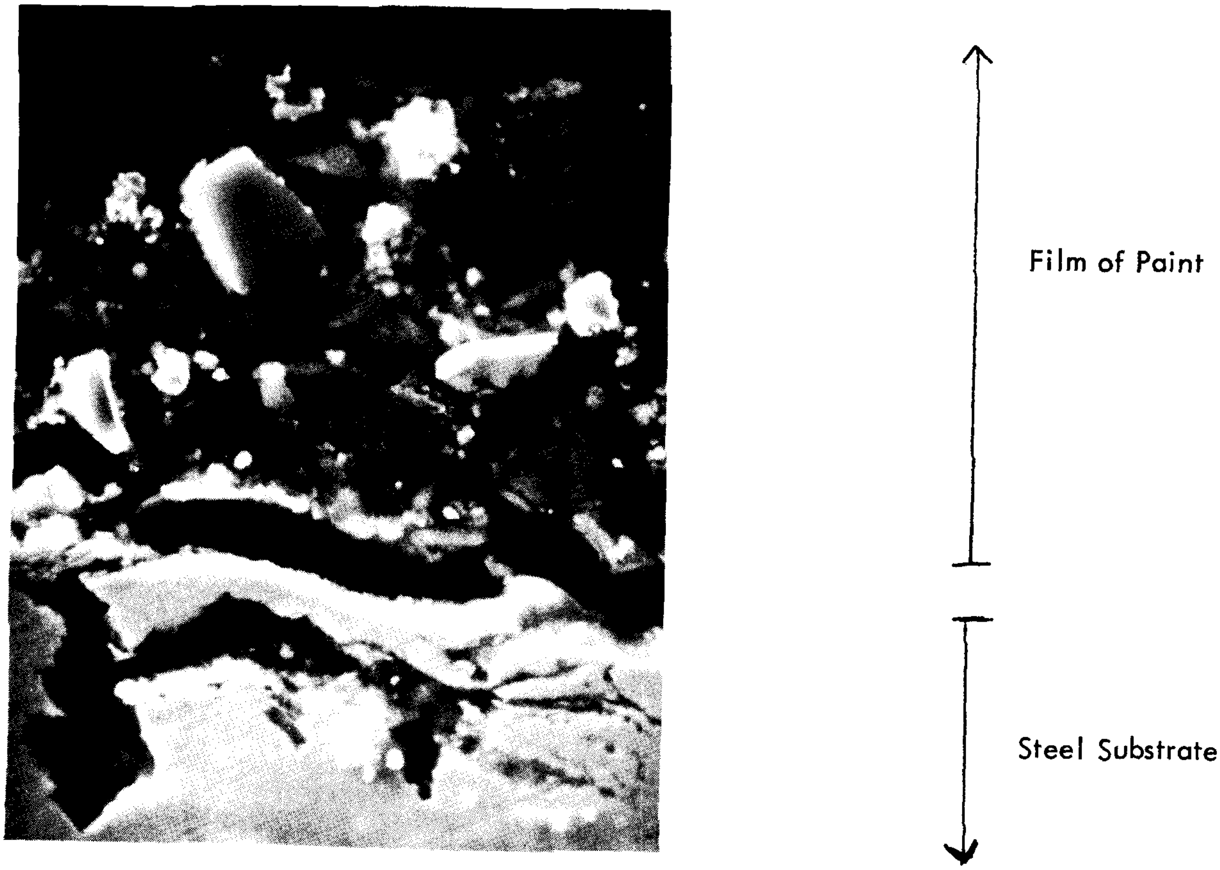

Figure 5: Cross section of a film of paint on steel, showing pigment particles (X2000)

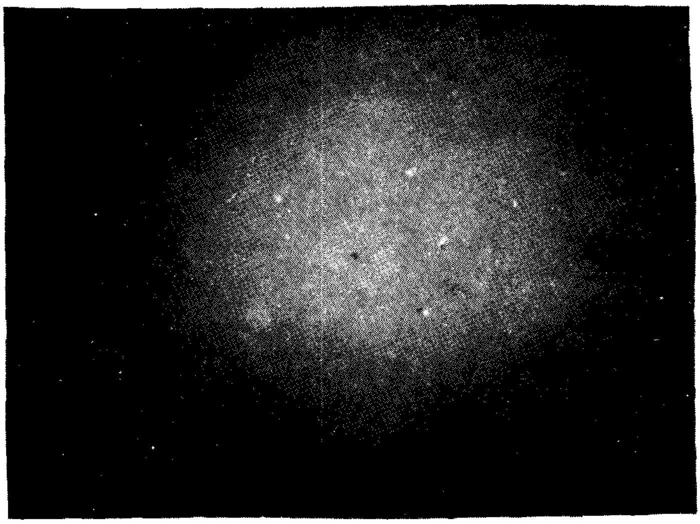

Figure 6: Coverage Provided by a 3.0 mil Film of Red Lead Paint (X25)

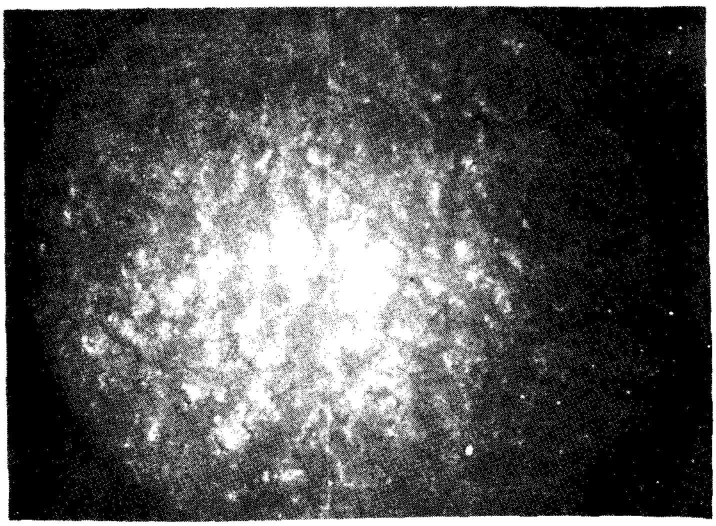

Figure 7: Coverage Provided by a 0.5 mil Film of Red Lead Paint (X25)