

U. S. DEPARTMENT OF COMMERCE

BUILDING
MATERIALS
AND
STRUCTURES

REPORT BMS44

Surface Treatment of Steel
Prior to Painting

by ROLLA E. POLLARD
and WILBUR C. PORTER

NATIONAL
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BUILDING MATERIALS *and* STRUCTURES

REPORT BMS44

Surface Treatment of Steel Prior to Painting

by ROLLA E. POLLARD *and* WILBUR C. PORTER



ISSUED APRIL 8, 1940

The National Bureau of Standards is a fact-finding organization; it does not "approve" any particular material or method of construction. The technical findings in this series of reports are to be construed accordingly.

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Foreword

This report contains the results of a number of investigations covering accelerated laboratory tests designed to determine the relative effect of various surface-treatment processes on plain or galvanized steel to prevent corrosion. The growing demand for structural sheet steel has indicated the need of better protection for the metal through improvement of the durability of paint films to insure longer service life.

A description of the tests and an explanation of their limitations were published in a previous report, *Methods of Investigation of Surface Treatment for Corrosion Protection of Steel*, BMSS.

The present report is the first of a series in which the results of these experiments will be discussed. Comparison of results is limited to the tests themselves, and conclusions are drawn on the basis of factors affected by conditions peculiar to the individual test.

LYMAN J. BRIGGS, *Director*.

Surface Treatment of Steel Prior to Painting

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ABSTRACT

A number of surface-treatment processes are described for both plain and galvanized steel. Among such treatments subjected to accelerated weathering, salt spray, and condensation corrosion tests, the hot-dip phosphate treatments for both plain and galvanized surfaces showed outstanding merit in improving the protective value of paints. Particularly effective protection was obtained when such treatments were used, under severely corrosive conditions, in combination with a primer of the inhibitive type. A phosphate cold wash for galvanized steel and two phosphate-chromate cold-wash treatments for plain steel also appeared to improve paint protection.

I. INTRODUCTION

The increasing use of sheet steel as a structural building material serves to emphasize the need of improved methods for prolonging the life of the material by suitable surface treatment. During the past 2 years the National Bureau of Standards has been investigating this subject as part of a general research program on building materials for low-cost house construction. In a previous publication, report BMS8, Methods of Investigation of Surface Treatment for Corrosion Protection of Steel, the testing program was outlined, the testing methods were described, and the pretreatment processes and paints being tested were listed. The testing program consisted of a number of accelerated laboratory corrosion tests applied to painted panels of plain and galvanized sheet steel, supplemented by outdoor exposure tests of similar materials at Washington, D. C.

Sufficient time has not elapsed as yet for the outdoor exposure tests to produce significant results. The condensation corrosion tests also have been rather slow, results to date being limited largely to paints applied to untreated galvanized-steel panels. However, results have been obtained in a shorter time from the salt-spray and accelerated-weathering tests, and a considerable amount of useful information has been accumulated. It is believed that some of these results may be discussed with profit if comparisons are limited to the tests themselves and conclusions are drawn on the basis of factors affected by conditions peculiar to the individual test.

II. SCOPE

One of the chief objectives of the investigation has been to study the various methods used in attempting to improve the protective value of paints applied to steel. One such method is to treat the surface prior to painting, in order to provide an improved base for the paint; another method is to improve the paint itself. The present report will be concerned chiefly with the first, or pretreatment, method of improvement. This report is limited to the description of the pretreatment processes tested, and to the discussion of the relative merits of such processes as measured by the protective value of paints subsequently applied and then tested in the salt-spray cabinet, accelerated-weathering machine, and condensation corrosion cabinet. Some of the factors affecting

the protective value of the paints themselves will be considered in a later publication.

The panels used in tests of the pretreatment processes covered by the present report were of plain hot-rolled annealed sheet steel or of hot-dip zinc-coated (galvanized) sheet steel.

III. PRETREATMENTS FOR GALVANIZED-STEEL SURFACES

The object of pretreating galvanized steel is to improve the surface for painting. For example, paint adherence can be improved by roughening the surface mechanically or chemically or by the deposition of a film, preferably nonmetallic, which improves the bond between paint and metal. It is desirable that the film formed should be one that prevents reaction between the zinc and the paint vehicle and retards corrosion by reason of its insolubility, its inhibitive action, or its reaction with corrosion products to form an insoluble film.

The following pretreatment processes for galvanized steel, designed to effect some of these improvements, were included in the testing program:

LIST 1.—*Pretreatment processes for galvanized steel*

- A. Untreated hot-dip galvanized surface.
- B. Proprietary phosphate treatment. Cold wash in aqueous zinc-phosphate solution containing free phosphoric acid and an activating agent.
- C. Etching solution. Water containing 8 oz. of copper sulfate per gallon.
- D. Etching solution. Water containing 8 oz. of zinc sulfate per gallon.
- E. Etching solution. Alcohol, 60; toluol, 30; carbon tetrachloride, 5; and hydrochloric acid, 5; parts by volume.
- F. Etching solution. Saturated aqueous solution of nickel sulfate, 10 parts by weight, antimony-potassium tartrate, 0.5 part by weight, neutralized with ammonium hydroxide, made slightly acid with hydrochloric acid, and then diluted to 100 parts by weight with water.
- G. Etching solution. Alcohol, 4; phosphoric acid (85 percent), 1; part by volume.

H. Proprietary phosphate treatment. Hot dip in aqueous zinc-phosphate solution containing free phosphoric acid and an activating agent.

I. Laboratory etching solution. Dilute acetic acid, 4 percent by volume.

J. Proprietary phosphate treatment. Hot dip in aqueous zinc-phosphate solution containing free phosphoric acid and an activating agent.

K. Proprietary oxalate-phosphate treatment. Hot dip in an aqueous solution containing an oxalate and phosphoric acid.

L. Proprietary dichromate treatment. Cold immersion in a sodium-dichromate aqueous solution containing sulfuric acid.

IV. PRETREATMENTS FOR PLAIN-STEEL SURFACES

The adherence of paint to a clean, dry steel surface is generally very good. An important objective to be sought in any pretreatment process, therefore, is to obtain a clean, dry surface free of rust and loose scale, or to deposit a film having equal or superior adherence, which will prevent or retard corrosion underneath the paint.

The following pretreatment processes, which were applied to plain sheet steel for the purpose of attaining some of these objectives, were included in the testing program:

LIST 2.—*Pretreatment processes for plain-steel surfaces*

- Fe. No treatment. Painted over mill scale.
- Fe-A. Pickled in hot aqueous solution of hydrochloric acid (20-percent concentrated acid by volume).
- Fe-B. Proprietary phosphate treatment. Hot dip in an aqueous zinc-phosphate solution containing free phosphoric acid and an activating agent.
- Fe-C. Proprietary chromate treatment. Hot dip in an aqueous chromic-acid solution containing activating agents.
- Fe-D. Proprietary phosphate-chromate treatment. Cold wash in an aqueous solution containing phosphoric acid, phosphates, and chromates.

Fe-E. Proprietary phosphate-chromate treatment. Cold wash in an aqueous solution containing phosphoric acid, phosphates, and chromates—generally used with an inhibitive primer.

Fe-H. Proprietary phosphate treatment. Hot dip in an aqueous zinc-phosphate solution containing free phosphoric acid and an activating agent.

Fe-K. Proprietary oxalate-phosphate treatment. Hot dip in an aqueous solution containing an oxalate and phosphoric acid.

Fe-M. Proprietary phosphate treatment. Cold wash in an aqueous solution of phosphoric acid containing a wetting agent.

Fe-P. Pickled in hot aqueous solution of phosphoric acid (10-percent sirupy acid by volume).

Fe-S. Pickled in hot aqueous solution of sulfuric acid (20-percent concentrated acid by volume).

V. METHODS OF TESTING

After pretreatment, the plain-steel or galvanized-steel panels were brushed, as a rule, with two coats of primer, a week being allowed for drying between application of coats. One half of the surface of each panel was then given a top coat of outside finish paint. Most of the painted panels were scratched diagonally before being tested. All panels were 3 by 6 inches except those for the condensation cabinet, which were 5 by 7 inches.

Galvanized-steel panels are identified in this report by letters representing the surface treatments given in list 1. For the plain-steel panels the letters are preceded by the symbol *Fe*, as shown in list 2. Following each letter is a number identifying the primer with which the panel is painted. For example, designation *A-1* represents an untreated galvanized-steel panel coated with primer 1 out of a group of some 80 primers and paints. Similarly, panel *Fe-A-1* represents a plain-steel sample pickled in hot hydrochloric acid and coated with primer 1.

Panels representing all the pretreatment processes listed above for galvanized-steel surfaces have been tested by salt spray and by accelerated weathering. A number have also

been tested in the condensation corrosion cabinet. Tests of plain-steel panels have been more limited but include salt-spray and accelerated-weathering tests of samples representing all the pretreatment processes listed.

VI. RESULTS OF TESTS

1. SALT SPRAY

The salt-spray test as used in this work consisted in the continuous exposure of painted panels to the fine mist or spray produced by "atomizing" a 20-percent aqueous solution of sodium chloride at 35° C.

The principal factors which accelerate breakdown of paint on galvanized steel in this test seem to be moisture penetration and action of the chloride ion. The moisture, of course, causes corrosion of the metal and promotes reaction between metal and paint or between corrosion product and paint. These reactions probably are accelerated by the chloride ion. The paints which would be expected to stand up best in the salt spray are those which resist moisture penetration or those which contain an inhibitive pigment, such as zinc chromate.

The pretreatments for galvanized-steel surfaces which gave the greatest improvement to the protective value of paints subjected to salt-spray tests were the hot-dip phosphate treatments *H* and *J*. These treatments appeared to be about equal in value and were definitely superior to all others tested. A panel which was given treatment *J* and then covered with primer 8 is shown in *J-8* of figure 1. The surface of the panel was in essentially perfect condition after 365 days in the salt spray. The phosphate cold-wash treatment *B* was found to be slightly inferior to the hot-dip treatments. Panel *B-8*, as shown in figure 1, was still in excellent condition after 104 days in the spray. The acid-dichromate treatment *L*, probably because of its inhibitive action, also gave very good results in this test and was in many cases, almost equal to treatments *H* and *J*. As will be shown later, however, this treatment has some disadvantages in other tests and, therefore, is considered separately. The two left-hand panels in figure 2 show the relative protective value of paint 1, after testing in the salt spray, when applied to an untreated galvanized surface

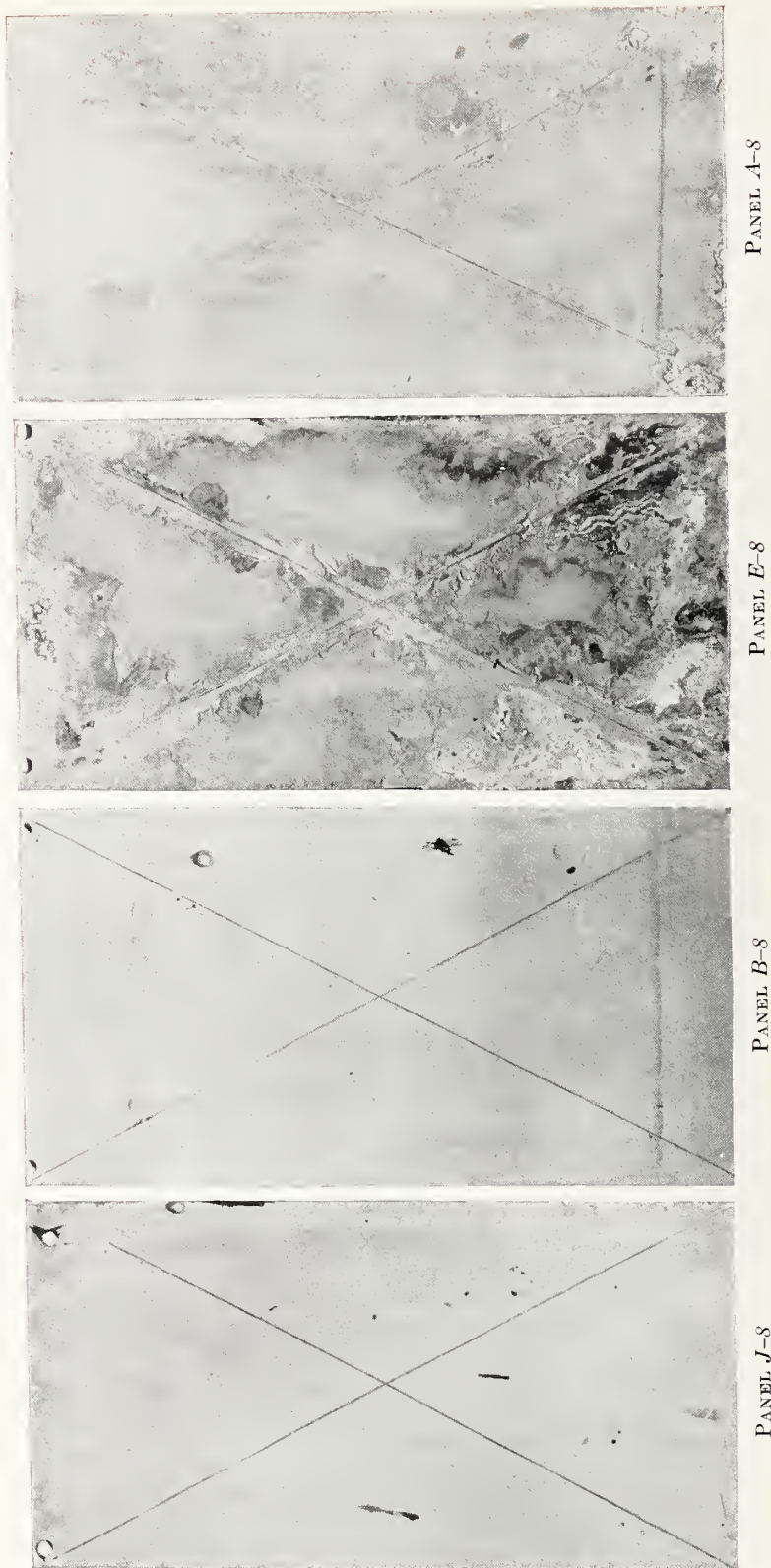


FIGURE 1.—Comparison of salt-spray tests of hot-dip galvanized-steel panels subjected to surface treatments J, B, E, and A prior to painting with two coats of primer 8.

Panel J-8: Hot-dip phosphate treatment (J); salt spray, 365 days' exposure.
 Panel B-8: Cold-wash phosphate treatment (B); salt spray, 104 days' exposure.
 Panel E-8: Alcohol-toluol-acid treatment (E); salt spray, 123 days' exposure.
 Panel A-8: Untreated galvanized surface (A); salt spray, 104 days' exposure.

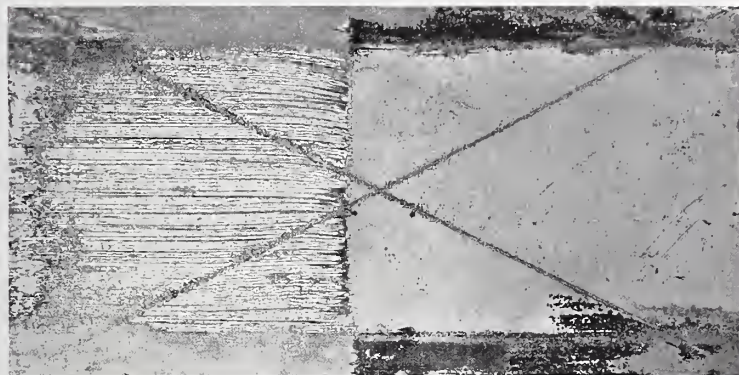
(Panels as shown are approximately two-thirds actual size.)



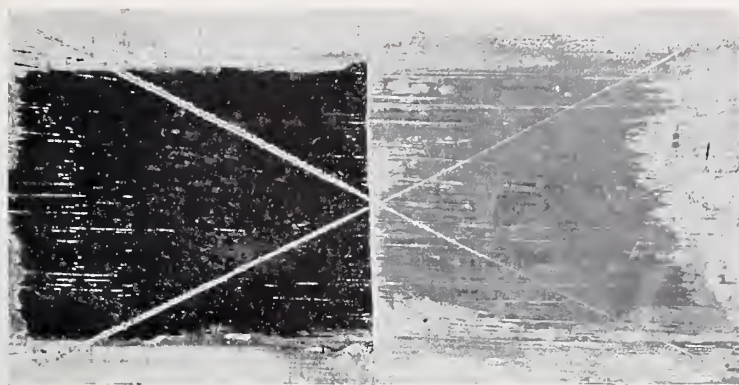
PANEL A-1



PANEL L-1



PANEL A-1-w



PANEL L-1-w

FIGURE 2.—Comparison of salt-spray and accelerated-weathering tests on galvanized steel panels subjected to acid-dichromate treatment *L* prior to painting.

Panel A-1: Untreated galvanized surface (A), two coats primer 1; salt spray, 28 days' exposure.

Panel L-1: Acid-dichromate treatment (L), two coats primer 1; salt spray, 61 days' exposure.

Panel A-1-w: Untreated galvanized surface (A), two coats primer 1 white top coat upper half; accelerated weathering, 8 months' exposure.

Panel L-1-w: Acid-dichromate treatment (L), two coats primer 1 black top coat upper half; accelerated weathering, 6 months' exposure.

(Panels as shown are approximately two-thirds actual size.)

(panel *A-1*) and when applied over treatment *L* (panel *L-1*). It will be noted that panel *L-1* was in much better condition than panel *A-1* after exposures of 61 and 28 days, respectively.

The other pretreatments listed for galvanized-steel surfaces were found to be of questionable value for the improvement of the protective value of paints in the salt-spray test. Treatment *K*, for example, appeared to improve or, rather, prolong the protective value of some of the inhibitive primers but not of other types. In figure 3 the two left-hand panels show the marked improvement obtained with inhibitive primer 5 applied over treatment *K* (panel *K-5*) as compared with the same primer applied to an untreated galvanized surface (panel *A-5*). With noninhibitive primer 6 no improvement was obtained, as shown by panels *A-6* and *K-6*.

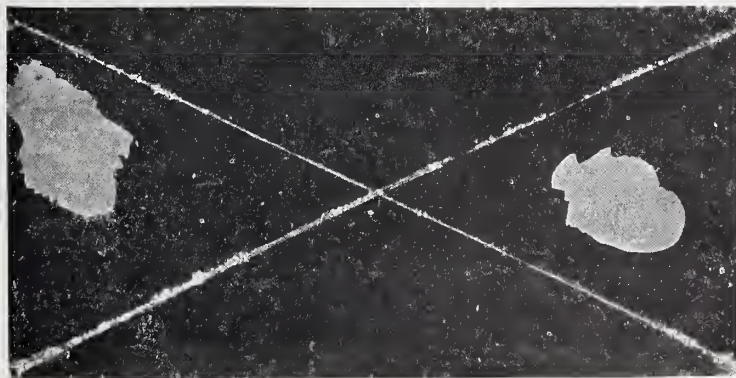
Treatments *C*, *D*, *E*, *F*, *G*, and *I*, classed together as etching solutions, gave no evidence of improvement over comparison panels of untreated material. An example of treatment *E* of this group is shown in panel *E-8* of figure 1 in comparison with the untreated panel *A-8* after exposures of 123 days and 104 days, respectively.

Salt-spray tests of plain-steel panels completed to date include at least two different primers on all the surface treatments listed. In these tests the hot-dip phosphate treatments *Fe-B* and *Fe-H* have been found consistently the best in improving the protective value of paints applied over them. Figure 4 is an example of treatment *Fe-B* (panel *Fe-B-29*) as it appeared after 202 days in the salt spray. It was removed at that time to be photographed and was then returned to the spray. Since there was no change in the appearance of the specimen after the first 40 days of the original exposure, it is probable that the test will continue for a considerable length of time. The phosphate-chromate cold-wash treatments *Fe-D* and *Fe-E* also have given good results in the salt-spray test. Panel *Fe-D-29*, representative of these treatments, appears in figure 4. After the same period of exposure it was almost as good as the panel (*Fe-B-29*) treated by the hot-dip phosphate solution. With other paints, however, this treatment gave slightly inferior results. The hot-dip chromic-acid treatment *Fe-C* showed considerable improvement over

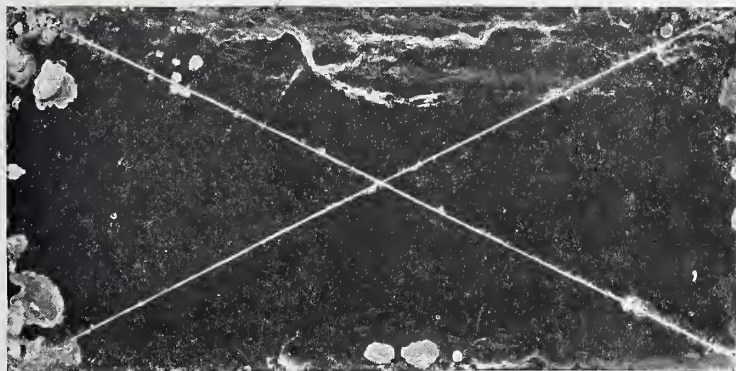
the plain pickled surface treatment *Fe-A*, but was decidedly inferior to the treatments mentioned above, as shown by panels *Fe-C-29* and *Fe-A-29* (fig. 4) as they appeared after 110 and 48 days, respectively. The other treatments (*Fe-K*, *Fe-P*, *Fe-M*, and *Fe-S*) listed for plain steel, showed no advantage over treatment *Fe-A* (pickled surface) in improving the protective value of paint applied over them.

In testing the plain-steel panels comparisons were made with the plain pickled surface (treatment *Fe-A*), because on the original hot-rolled annealed sheet material there was considerable variation in the amount and distribution of the surface mill scale. On one side of the sheet the scale appeared to be tight and fairly uniform, whereas on the other side large areas appeared to have no scale at all. Test results to date have been inconclusive in regard to the relative merit of paint applied over mill scale as compared with a pickled surface. However, in short-time tests under severely corrosive conditions, such as the salt spray, it appears that there are no outstanding advantages in either of these conditions, as is shown in figure 5. This figure shows the results of salt-spray tests on selected panels which had tight, uniform mill scale on the side marked *b* and practically no mill scale on the side marked *y*. There appeared to be no particular advantage in painting over mill scale (panel *Fe-1-b*) as compared with a surface practically free of mill scale (panel *Fe-1-y*). Moreover, the hot-dip phosphate treatment (*Fe-H*) appeared to be equally effective whether applied directly over mill scale (panel *Fe-H-1-b*), on a surface originally free of mill scale (panel *Fe-H-1-y*), or on a pickled surface (panel *Fe-A-H-1*). Other tests, in which direct comparisons of paints on mill scale or scale-free surfaces are being made, have not been completed, and definite conclusions cannot be drawn.

That the presence of initial surface rust has a much greater effect on paint than the presence of mill scale is illustrated in figure 6. The two right-hand panels of figure 6 were exposed outdoors until covered with rust. Panel *R-Fe-5a* was then painted directly over mill scale and rust. This panel was decidedly inferior in the salt-spray test to panel *Fe-5a*, which was painted over unrudded mill scale, and to panel



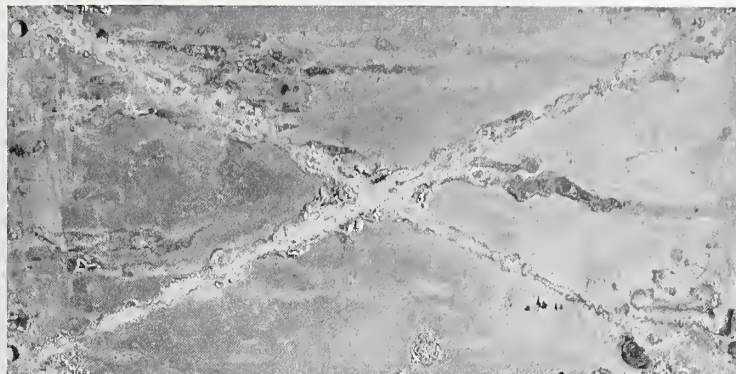
PANEL A-5



PANEL K-5



PANEL A-6



PANEL K-6

FIGURE 3.—Comparison of salt-spray tests of different types of paints on galvanized-steel panels subjected to oxalate-phosphate treatment (K) prior to painting.

Panel A-5: Untreated galvanized surface (A), inhibitive primer 5, salt spray, 104 days' exposure.
 Panel K-5: Oxalate-phosphate treatment (K), inhibitive primer 5, salt spray, 231 days' exposure.
 Panel A-6: Untreated galvanized surface (A), noninhibitive primer 6, salt spray, 70 days' exposure.
 Panel K-6: Oxalate-phosphate treatment (K), noninhibitive primer 6, salt spray, 74 days' exposure.

(Panels as shown are approximately two-thirds actual size.)

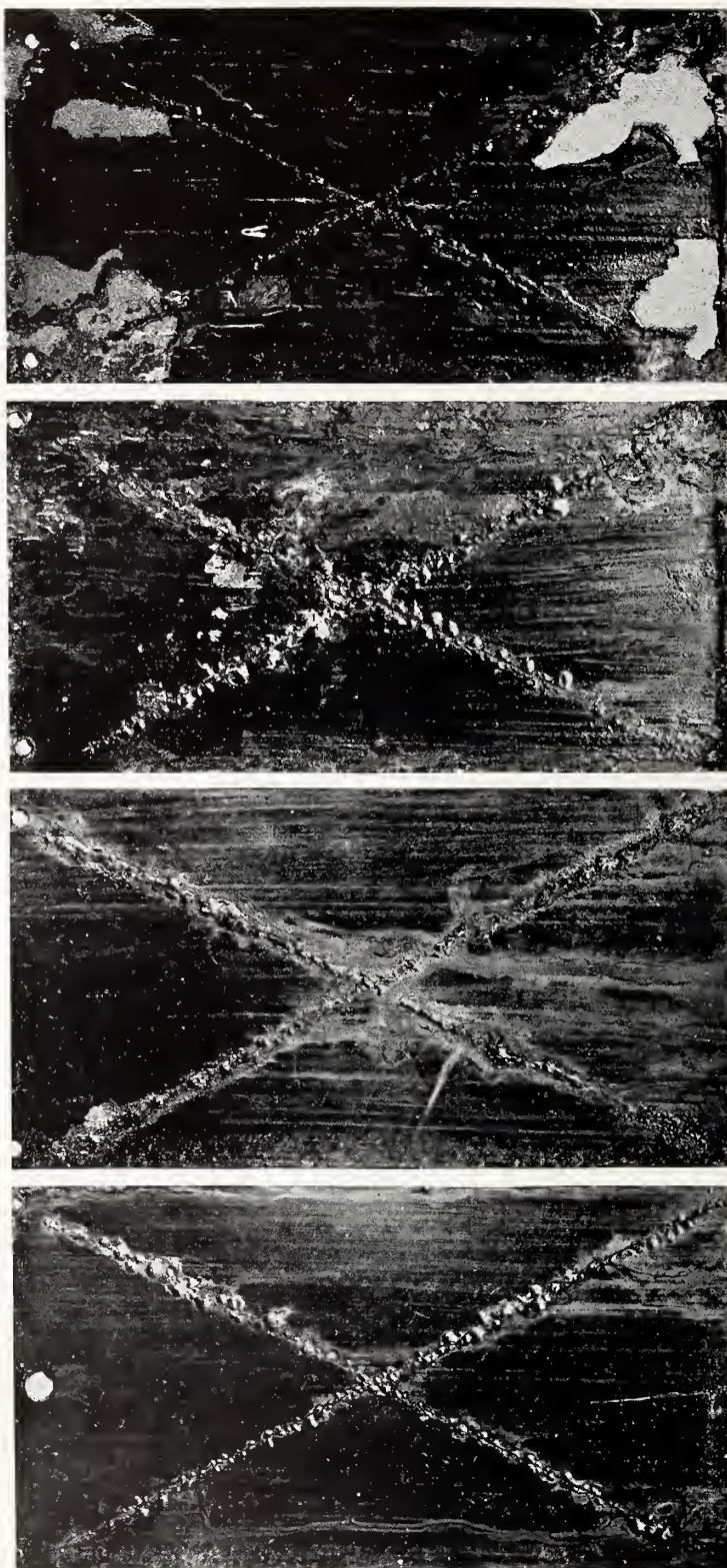


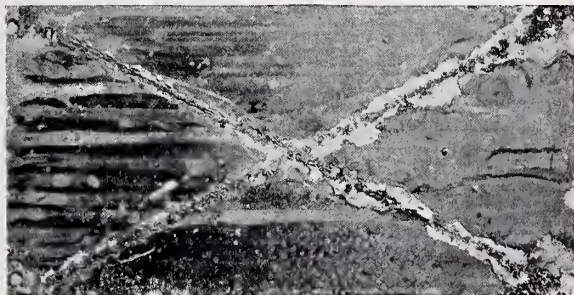
FIGURE 4.—Comparison of salt-spray tests on steel panels subjected to treatments Fe-B, Fe-D, Fe-C, and Fe-A, prior to painting with two coats of primer 29.

Panel Fe-B-29: Hot-dip phosphate treatment (Fe-B); salt spray, 202 days' exposure.
 Panel Fe-D-29: Phosphate-chromic cold wash (Fe-D); salt spray, 202 days' exposure.
 Panel Fe-C-29: Hot-dip chromic acid (Fe-C); salt spray, 110 days' exposure.
 Panel Fe-A-29: Hydrochloric acid pickle (Fe-A); salt spray, 48 days' exposure.

(Panels as shown are approximately two-thirds actual size.)



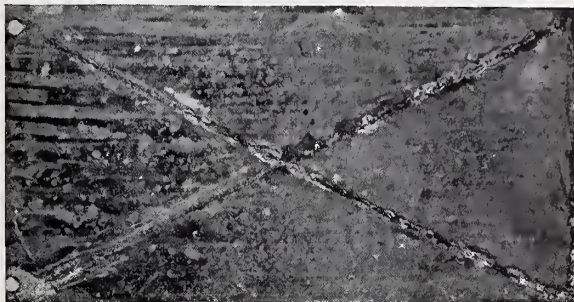
PANEL *Fe-1-b*



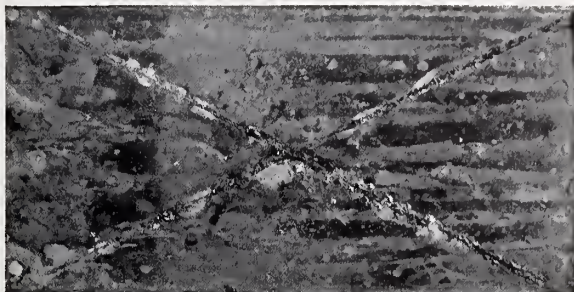
PANEL *Fe-1-y*



PANEL *Fe-H-1-b*



PANEL *Fe-H-1-y*



PANEL *Fe-A-H-1*

FIGURE 5.—Salt-spray tests of steel panels showing effect of mill scale on the protective value of paint.

Panel *Fe-1-b*: Primer 1 over tight mill scale; salt spray, 20 days' exposure.
 Panel *Fe-1-y*: Primer 1 on scale-free surface; salt spray, 20 days' exposure.
 Panel *Fe-H-1-b*: Primer 1 over mill scale after hot-dip phosphate treatment (*Fe-H*); salt spray, 34 days' exposure.
 Panel *Fe-H-1-y*: Primer 1 on scale-free surface after hot-dip phosphate treatment (*Fe-H*); salt spray, 34 days' exposure.
 Panel *Fe-A-H-1*: Primer 1 on pickled surface after hot-dip phosphate treatment (*Fe-H*); salt spray, 34 days' exposure.

(Panels as shown are approximately one-half actual size.)

Fe-A-5a, painted on a pickled surface. The other rusted panel, *R-Fe-E-5a*, was given the phosphate-chromate cold-wash treatment *Fe-E* prior to painting. The treatment evidently removed some of the rust and improved the surface for painting. However, the same treatment was much more effective when applied to a surface with unrusted mill scale (panel *Fe-E-5a*). Superior results were also obtained with the hot-dip phosphate treatment on a scale-free and rust-free surface (panel *Fe-H-5a*). All panels in figure 6 were covered with an aluminum top coat over primer 5a. These panels were not scratched.

2. ACCELERATED-WEATHERING TESTS

In the accelerated-weathering test, painted panels are exposed, alternately wet and dry, to the heat and light of a carbon arc lamp. During a 20-minute cycle the panels are exposed to the light for more than 18 minutes and are heated to about 54° C. They are then suddenly wet by a water spray at 15° to 21° C and are quickly dried off when they come under the light again to complete the cycle. Important factors affecting the breakdown of paint in this test are the action of light and the sudden change in temperature. It is believed that actual corrosion of metal due to moisture under the paint film is relatively unimportant.

The paints which stand up best under the accelerated-weathering test are those which have good adhesion and are opaque to light. Good adhesion is particularly important when primers and covering coats with different coefficients of expansion or different drying characteristics are used. A good test for adhesion of a primer is to use a hard-drying top coat, which, by drying at a rate different from that of the primer, tends to loosen the priming coat from the metal.

Opacity to light is, of course, a property of the paint itself. Adherence of the paint film, however, is markedly affected by the condition of the metal surface. Among the surface treatments listed for galvanized-steel panels, the hot-dip phosphate treatments *H* and *J* stood up best in accelerated-weathering tests, as shown in figure 7 (panel *H-5*). It will be noted that after 12 months' exposure most of the hard-drying top coat on the top half of the panel had

chalked off, but the priming coat remained intact over the entire surface. In contrast, the same primer on the untreated panel *A-5* (fig. 7) was pulled off entirely by the top coat after 6 months and was also peeled on the bottom half in large areas at the edges subjected to abrasion by the vertical slots in which the panel rested during testing. The cold-wash phosphate treatment *B* stood up very well in the accelerated-weathering test, as shown by the appearance of panel *B-5* in figure 7. After 6 months' exposure the top coat pulled the primer off slightly along the scratch, but all other areas were in good condition. Panel *D-5* (fig. 7) is representative of a group of treatments (*C*, *D*, *E*, *F*, *G*, and *K*) in which the improvement over an untreated galvanized surface was very slight or entirely negligible. Treatments *I* and *L* showed no improvement over untreated galvanized surfaces in the accelerated-weathering tests. In figure 2 the two right-hand panels show primer 1 over the acid-dichromate treatment, *L*, as compared with the same primer on untreated galvanized steel. Although a much softer black top coat was used on panel *L-1*, no improvement was observed over the untreated panel *A-1* exposed for a longer time.

Accelerated-weathering tests of steel panels completed to date include only one series in which all the surface treatments listed have been represented. The results of this test (of 9 months' duration) are illustrated in figure 8, in which panel *Fe-B-1* represents the hot-dip phosphate treatments *Fe-B* and *Fe-H*, which again stood up best. These treatments were followed closely by the phosphate-chromate cold washes *Fe-D* and *Fe-E*, represented in figure 8 by panel *Fe-D-1*. The hot-dip chromic-acid treatment *Fe-C* gave slight improvement over treatment *Fe-A* (panels *Fe-C-1* and *Fe-A-1*, fig. 8). The primer used on these panels chalked badly in areas not covered with top coat. The lower parts of the panels therefore appear white except where the chalked material has been rubbed off by handling. The places which appear black should not be confused with the dark areas on the top half of the panels representing spots where the top coat cracked and flaked off and took the priming coat with it.

None of the other surface conditions listed

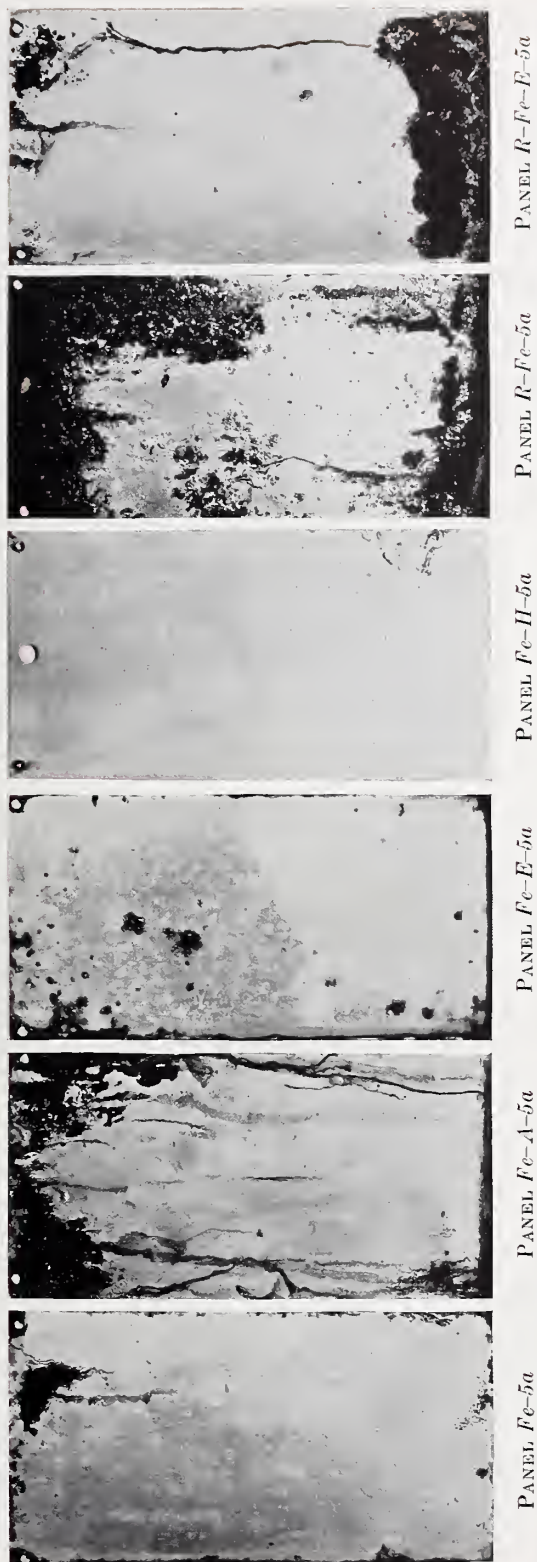


FIGURE 6.—Salt-spray tests of steel panels showing effect of presence of scale and of rust on the protective value of paint.

Panel *Fe-5a*: Primer *5a* and aluminum top coat over mill scale; salt spray, 96 days' exposure.
 Panel *Fe-A-5a*: Primer *5a* and aluminum top coat over pickled surface (*Fe-I*); salt spray, 135 days' exposure.
 Panel *Fe-B-5a*: Primer *5a* and aluminum top coat over mill scale after phosphate-chromate cold wash (*Fe-I*); salt spray, 250 days' exposure.
 Panel *Fe-C-5a*: Primer *5a* and aluminum top coat over mill scale after hot-dip phosphate treatment (*Fe-II*); salt spray, 135 days' exposure.
 Panel *Fe-D-5a*: Primer *5a* and aluminum top coat over rusted mill scale; salt spray, 64 days' exposure.
 Panel *Fe-E-5a*: Primer *5a* and aluminum top coat over rusted mill scale after phosphate-chromate cold wash (*Fe-E*); salt spray, 138 days' exposure.

(Panels as shown are approximately two-fifths actual size.)

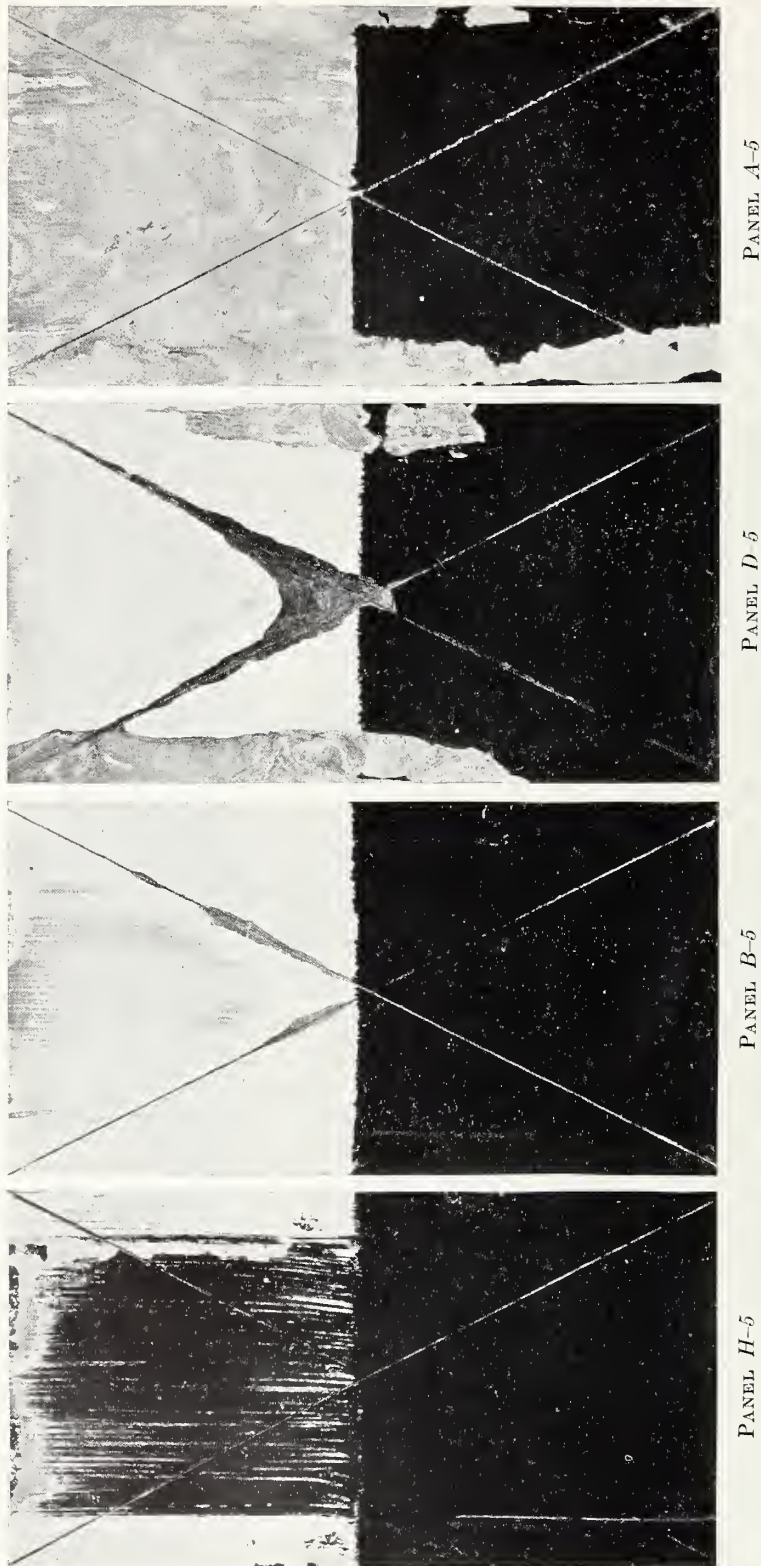


FIGURE 7.—Comparison of accelerated-weathering tests of hot-dip galvanized-steel panels subjected to treatments H, B, D, and A prior to painting with two coats of primer 5.

The top half of each panel is covered with one hard-drying outside white top coat.

Panel H-5: Hot-dip phosphate treatment (H); accelerated weathering, 12 months' exposure.
 Panel B-5: Cold-wash phosphate treatment (B); accelerated weathering, 6 months' exposure.
 Panel D-5: Zinc-sulfate solution treatment (D); accelerated weathering, 6 months' exposure.
 Panel A-5: Unreated galvanized surface (A); accelerated weathering, 6 months' exposure.

(Panels as shown are approximately two-thirds actual size.)

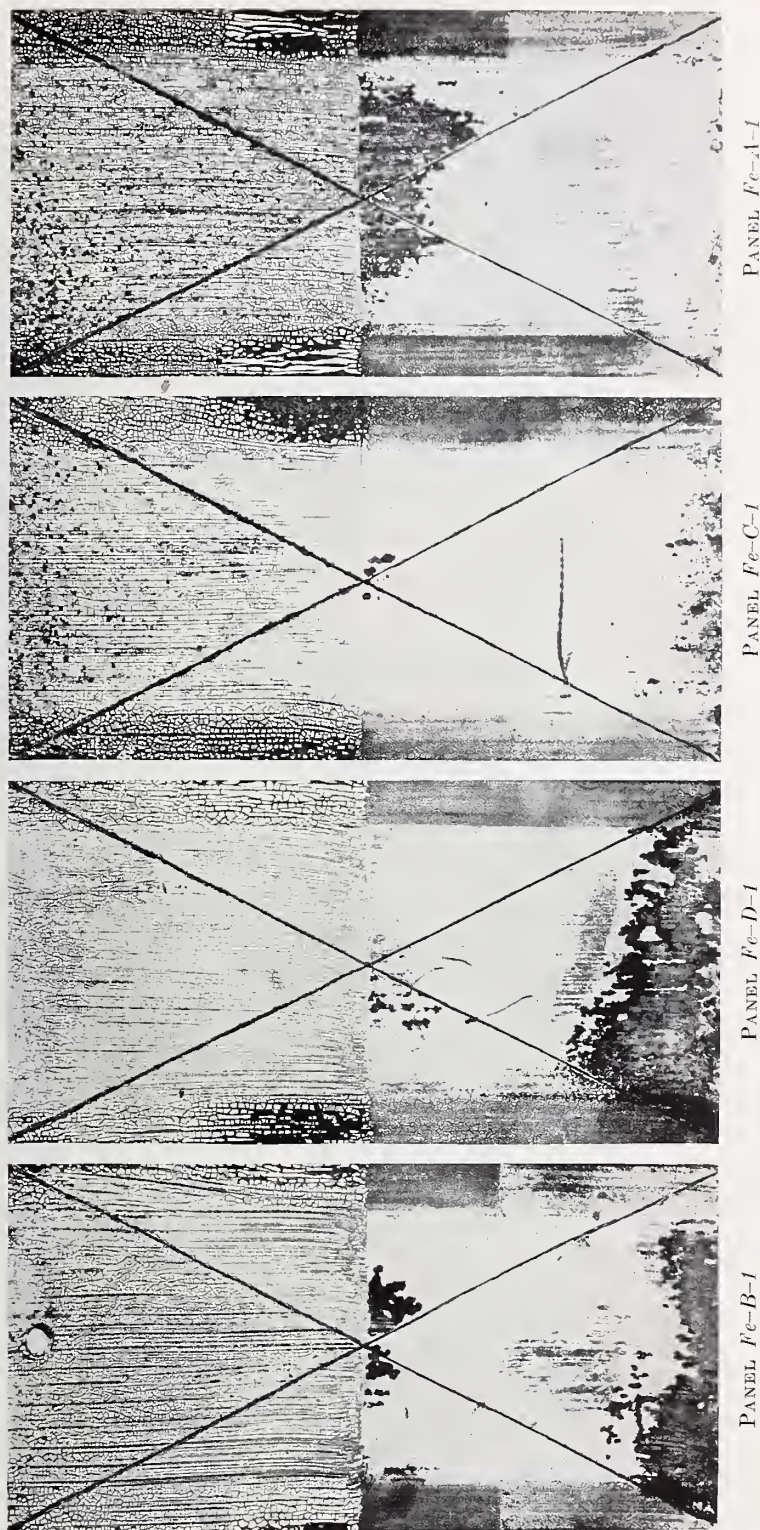


FIGURE 8.—Comparison of accelerated-weathering tests of steel panels subjected to treatments Fe-B, Fe-D, Fe-C, and Fe-A prior to painting with two coats of primer 1.

The top half of each panel is covered with one hard-drying outside white top coat.

Panel Fe-B-1: Hot-dip phosphato treatment (Fe-B); accelerated weathering, 9 months' exposure.
 Panel Fe-D-1: Phosphate-chromate cold wash (Fe-D); accelerated weathering, 9 months' exposure.
 Panel Fe-C-1: Hot-dip chromic-acid treatment (Fe-C); accelerated weathering, 9 months' exposure.
 Panel Fe-A-1: Hydrochloric acid pickle (Fe-A); accelerated weathering, 9 months' exposure.

(Panels as shown are approximately two-thirds actual size.)

for steel panels (treatments *Fe*, *Fe-K*, *Fe-P*, *Fe-M*, and *Fe-S*) has shown any improvement over treatment *Fe-A* (acid pickle) in the one accelerated-weathering test that has been completed. Other tests are in progress.

3. CONDENSATION CORROSION TESTS

The condensation corrosion test was designed to simulate as nearly as possible the conditions that may occur in enclosed spaces, such as the interior of the walls of a house. The test consists essentially in exposing the prepared panels to the corrosive action of moisture condensed from a controlled atmosphere. The panels rest in a nearly horizontal position on the surface of a water tank forming the bottom of an insulated box through which air of controlled temperature and humidity is circulated. A test run includes a series of cycles, each of which embraces a condensation period of approximately 6 hours, an overnight period of 17 hours, during which the panels remain wet, and a final 1-hour drying period. During the condensation period the panels are maintained at a temperature of 15° to 20° C by water circulated through the tank, while the air circulated through the box is saturated at about 35° C. During the overnight period circulation of air is stopped, but the cabinet remains closed and the water tank is maintained at a temperature varying between 10° and 25° C by tap water. During the 1-hour drying period hot water is circulated through the tank and the panels are heated to about 40° C.

The principal factors causing deterioration of paint coatings in the condensation corrosion test are moisture penetration and rapid changes in temperature. Paints that would be expected to stand up best under these conditions are those which resist moisture penetration or contain an inhibitive pigment and have good adhesion. This has been confirmed by the test results to date, in which failures have been due either to corrosion under the paint or to flaking through loss of adhesion.

Condensation corrosion tests so far have been confined to painted galvanized-steel panels. These panels include representatives of treatments *A*, *H*, *J*, and *L*. Of 14 different primers on untreated panels (treatment *A*) all but two have failed after periods varying from

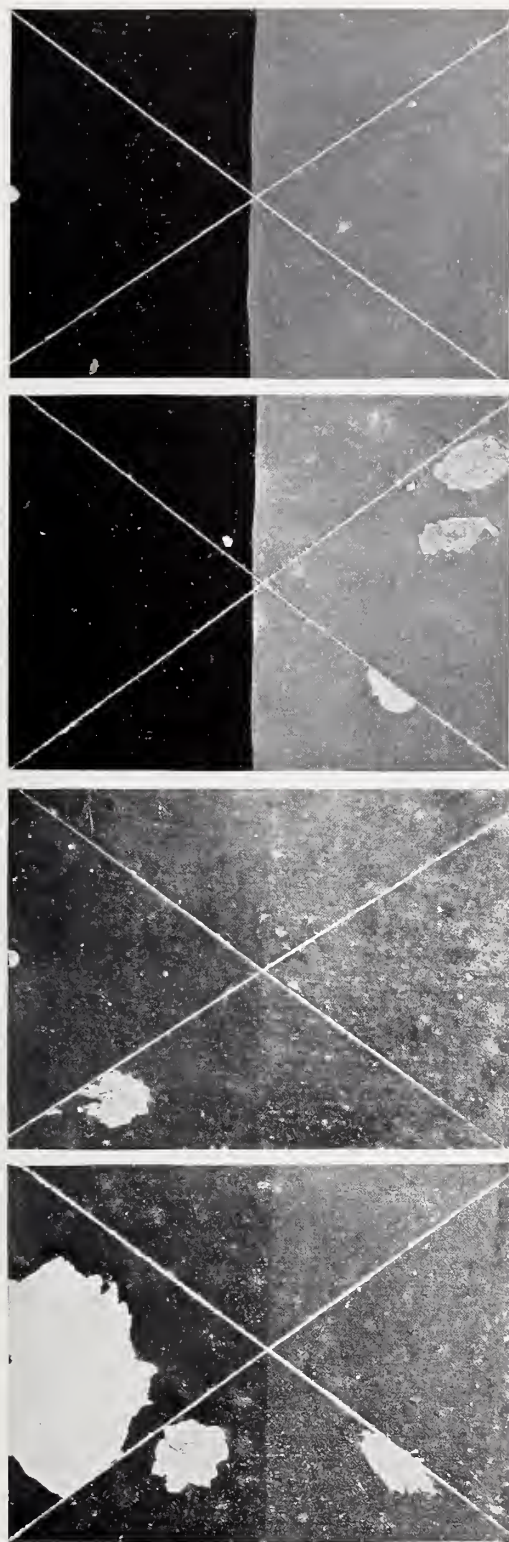
5 to 16 months. Of 14 similar primers applied over each of the hot-dip phosphate treatments *H* and *J*, only one of treatment *H* and one of treatment *J* have failed during this time. Neither of two primers over the acid-dichromate treatment *L* has failed after 16 months' exposure. In figure 9, primers 9 and 14 are shown on untreated galvanized-steel surfaces (panels *A-9* and *A-14*) as they appeared after exposures of 10 and 5 months, respectively. Marked improvement was shown by the same primers over the hot-dip phosphate treatment *H* (panels *H-9* and *H-14*, fig. 9) after exposures of 16 and 15 months, respectively.

VII. DISCUSSION OF RESULTS

As far as the results of accelerated laboratory corrosion tests are concerned, it is evident that surface treatments for plain steel or galvanized steel may be classified in several distinct groups according to their effectiveness in improving the protective value of paints applied over them. For example, surface treatments for galvanized steel (list 1) may be rated by groups in decreasing order of merit as follows:

- Group 1. Treatments *H* and *J*. Best in all tests to date; marked improvement over untreated galvanized steel.
- Group 2. Treatment *B*. Marked improvement, but slightly less than group 1 in all tests.
- Group 3. Treatments *K* and *L*. Improvement marked or negligible, depending on the type of paint and particularly the conditions of testing.
- Group 4. Treatments *C*, *D*, *E*, *F*, and *G*. Improvement slight or negligible, depending on type of paint and conditions of testing.
- Group 5. Treatments *A* and *I*. No improvement.

The hot-dip phosphate treatments *H* and *J* (and cold treatment *B* in less degree) showed outstanding merit in all the accelerated laboratory tests. Improvement in protective value of paints in the accelerated-weathering test may be attributed chiefly to improved adherence. Under severely corrosive conditions, such as the salt spray, the phosphate film deposited by



PANEL H-9

PANEL A-9

PANEL H-14

PANEL A-14

FIGURE 9.—Comparison of condensation corrosion tests on hot-dip galvanized-steel panels subjected to treatments H and A prior to painting with two coats of primers 14 and 9.

The top half of each panel is covered with one soft-drying outside black top coat.

Panel A-14: Primer 14 on untreated galvanized surface (A); condensation corrosion, 5 months' exposure.

Panel H-14: Primer 14 on hot-dip phosphate treatment (H); condensation corrosion, 15 months' exposure.

Panel A-9: Primer 9 on untreated galvanized surface (A); condensation corrosion, 10 months' exposure.

Panel H-9: Primer 9 on hot-dip phosphate treatment (H); condensation corrosion, 16 months' exposure.

(Panels as shown are approximately two-thirds actual size.)

these treatments apparently aids materially in slowing down corrosion underneath the paint. This action is particularly effective when combined with that of an inhibitive pigment in the priming coat. It is possible that the phosphate film acts as a sort of anchor for the inhibitive pigment, which, to be very effective, must be slightly soluble.

The chromate film deposited by treatment *L* apparently has some inhibitive action. Improvement in paint protection as a result of this treatment is particularly marked with paints that have little or no inhibitive value in themselves. However, this film seems to be slightly soluble, and after an initial period of complete protection the paints applied over it may fail rapidly at edges and scratches through dissolution of the chromate film and loss of adhesion. A treatment of this type which is protective in itself for considerable periods under severely corrosive conditions would be very desirable if it could be anchored more securely both to the metal and to the applied paint. Some experiments along the line of combining inhibitive action and adherence in films have been made, but the results to date have not been very promising.

In all the laboratory tests for galvanized steel discussed above, the smooth spangled surface of hot-dip galvanized sheet was used. The test results indicate that good treatments for this material do more than merely roughen the surface to hold paint. The best pretreatments so change the surface chemically as to prevent reaction between metal and paint, increase the adhesion of the applied paint, and prevent or slow down corrosion under the paint film. It is claimed that other types of metallic zinc coatings—such as zinc-sprayed, electro-galvanized, or annealed hot-dip galvanized materials—have acceptable surface characteristics and do not require special treatment prior to painting. Tests on these materials have not progressed far enough as yet to warrant definite conclusions being drawn.

According to the results of the accelerated laboratory tests, surface treatments for plain steel (list 2) prior to painting can be rated in the following decreasing order of merit with regard to effectiveness in improving the protective value:

Group 1. Treatments *Fe-B* and *Fe-H*. Best in all tests to date; marked improvement over plain pickled surface.

Group 2. Treatments *Fe-D* and *Fe-E*. Improvement slightly less marked than group 1 in all tests.

Group 3. Treatment *Fe-C*. Improvement slight or marked, depending on conditions of testing.

Group 4. Treatments *Fe*, *Fe-A*, *Fe-K*, *Fe-M*, *Fe-P*, and *Fe-S*. No improvement compared with plain pickled surface.

These results are in agreement with those obtained on galvanized-steel surfaces. The best results to date, for example, have been obtained with phosphate films produced by the hot-dip processes *Fe-B* and *Fe-H*, closely followed by the phosphate-chromate cold washes *Fe-D* and *Fe-E*. Both types of treatment appear to improve paint adhesion and to slow down corrosion under paint.

The chromate film deposited by treatment *Fe-C* apparently has considerable inhibitive value under severely corrosive conditions (salt-spray test, fig. 4), but its effect on paint adhesion is not marked (accelerated-weathering test, fig. 8).

The nature of the different acids used in the pickling treatments so far tried (treatments *Fe-A*, *Fe-P*, and *Fe-S*) does not seem to influence the results; that is, the pickled surface behaves the same regardless of the acid used. A variation of the phosphoric acid pickle, by which a phosphate film is said to be deposited, is being tested. This treatment, recommended by The Corrosion Committee of the British Iron and Steel Institute (5th Report), consists of an initial pickle in sulfuric acid to remove scale, followed by treatment in hot dilute phosphoric acid containing dissolved iron.

The relative effect of scratches and edges is noteworthy in the tests on both plain- and galvanized-steel panels. Defects, such as staining and blistering, developed along and spread from the scratches. In many cases, however, actual failures, such as peeling or disintegration of paint, seem to have been influenced more by the edges than by the scratches. In view of the fact that no precautions were taken against

edge effect other than to apply approximately the same amount of paint as in other areas, the results obtained by some combinations of pretreatments and priming paints are considered remarkable for both plain- and galvanized-steel surfaces.

VIII. SUMMARY AND CONCLUSIONS

1. GALVANIZED-STEEL PANELS

A number of accelerated-weathering, salt-spray, and condensation corrosion tests have been made on painted galvanized sheet-steel panels. The results of these tests, insofar as they are affected by surface treatment of the material, may be briefly summarized as follows:

(a) The hot-dip phosphate treatments (*H* and *J*) and the cold-wash phosphate treatment (*B*) improved both the adhesion and the corrosion protection of paints under widely varying conditions. The hot-dip treatments apparently were slightly superior to the cold wash.

(b) The acid-dichromate treatment (*L*) apparently had considerable protective value in itself and had an inhibitive action under paints but did not improve their adherence.

(c) The oxalate-phosphate treatment (*K*) prolonged the protective value of inhibitive paints under severely corrosive conditions (salt spray) but had little effect on other types, and did not improve adhesion to any appreciable degree.

(d) All other pretreatments tested were found to have slight or negligible effect in improving the protective value of paints.

(e) Tests of other types of zinc-coated material are being conducted but have not progressed far enough to warrant conclusions being drawn. These materials include zinc-plated, zinc-sprayed, and annealed hot-dip galvanized surfaces.

2. PLAIN-STEEL PANELS

Salt-spray tests of painted steel panels include at least two types of paint for all surface conditions listed. Accelerated-weathering tests include at least one paint for all pretreatments.

The results of these tests may be briefly summarized as follows:

(a) The hot-dip phosphate treatments (*Fe-B* and *Fe-H*) markedly improved the protective value of paints in both tests.

(b) The phosphate-chromate cold-wash treatments (*Fe-E* and *Fe-D*) were only slightly inferior to the hot-dip phosphate treatments in improvement of paint protection.

(c) The hot-dip chromic-acid treatment (*Fe-C*) improved paint protection as compared with a plain pickled surface, but the improvement was not as marked as that obtained with the treatments mentioned above.

(d) All other treatments tested were found to have slight or negligible effect in improving the protective value of paints. This includes various types of plain pickled surfaces but does not include a type of pickling which is said to result in the deposition of a phosphate film.

(e) Tests to determine the effect of mill scale on the protective value of paints in short-time tests have not been completed. However, some of the pretreatments, particularly those mentioned under (a) and (b), have given good results either over mill scale or on scale-free surfaces.

(f) Under severely corrosive conditions (salt-spray test) the protective value of paints applied on rusted surfaces was decidedly inferior to that of paints applied on plain pickled surfaces. Some of the pretreatments apparently were able to remove light rust. However, they improved paint protection much more effectively when used on a rust-free surface.

WASHINGTON, October 2, 1939.

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