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DEVELOPMENT OF AN ALKALINE-INHIBITED EPOXY PRIMER  
FOR MAGNESIUM ALLOYS

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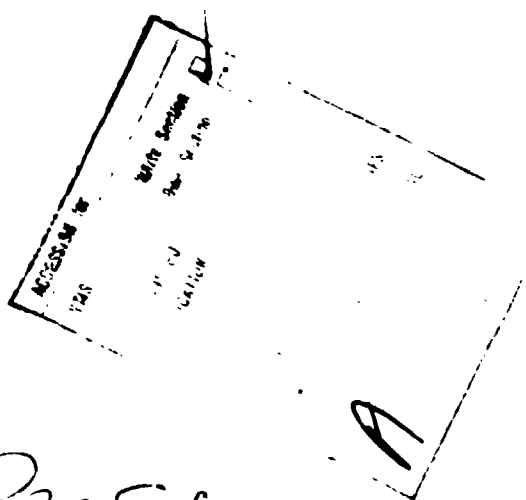
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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br><b>Current primer coatings, using metallic chromates as the sole corrosion inhibitors, have not provided adequate protection for magnesium alloys on major aircraft components. However, it has been found that such metals can be protected by an epoxy formulation similar to specification MIL-P-23377 if a specific ratio of magnesium oxide to strontium chromate is substituted for the existing inhibitive filler. Salt-spray tests, with and</b> |   |  |

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without sulfur dioxide additions, have been used to demonstrate the effectiveness of this material on wrought and cast magnesium alloys. In addition, other laboratory tests have verified that this coating is capable of meeting the performance requirements of current tactical aircraft.

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## I N T R O D U C T I O N

Both wrought and cast magnesium alloys have gained increasing usage on naval aircraft because of their high strength-to-weight ratios. Such alloys are found on the AV-8A (compressor housing, wheel wells), A-7B (vertical stabilizer), F-8 (vertical stabilizer), and H-46 (control levers) aircraft. More extensive use of these materials is limited, however, as they are anodic to most other structural alloys and are subject to galvanic corrosion when contacted with dissimilar metals. Current primer coatings, using metallic chromates as the sole corrosion inhibitors, have not provided adequate environmental protection. In order to reduce the vulnerability of aircraft that contain major magnesium components, the Naval Air Development Center was tasked by reference (a) to develop an epoxy primer capable of providing a "quick fix" solution to this problem.

Due to funding limitations, it was determined that modification of the MIL-P-23377 epoxy primer presently qualified for exterior aircraft usage would be the most cost-effective approach to take. A search of current literature was conducted to find alternate materials with the ability to passivate magnesium alloys. Reference (b) indicated that the electro-chemical potential for magnesium in an aqueous solution was highly dependent upon the pH of that solution. In a neutral or acidic environment, magnesium reacts with atmospheric moisture, which it reduces with the evolution of hydrogen, and is dissolved in the form of  $Mg^+$  and  $Mg^{++}$  ions. However, in an alkaline environment (particularly at pH's between 8.5 and 11.5), the metal reacts to form an adherent layer of  $Mg(OH)_2$ ; and the reaction quickly reaches equilibrium. Another by-product of this reaction,  $MgO$ , is not as thermodynamically stable as  $Mg(OH)_2$  in the presence of water. It is normally hydrated according to the reaction  $MgO + H_2O \rightarrow Mg(OH)_2$ . The formation of the hydroxide film by either means is highly dependent on such factors as the solution purity and the composition of the particular alloy to be protected. If the solution in question is that existing at the moist primer/substrate boundary layer exposed by a "break" in a coating system, then the validity of this theory will most likely be shown by empirical means.

## P R O C E D U R E

## SELECTION OF MATERIALS

A total of four metallic chromates and four alkaline compounds were chosen as potential inhibitors (see Table I). Their selection was partly based on such factors as solubility and alkalinity in water. The solubility, generally indicative of the leaching rate from a coating film, was taken from reference (c). The alkalinity was experimentally determined as the pH of a saturated solution of each material in distilled water with the use of a Beckman pH meter, Model SS-3. The availability and applicability to various alloy types were other factors considered.

## SALT-SPRAY TESTS

Table II is a breakdown of the epoxy primer formulation contained in MIL-P-23377C. Except for the inhibitive fillers, all of the formulations used in this effort are identical to it. In each of the succeeding tables, the candidate primers are designated by the respective parts-by-weight of each component inhibitor.

TABLE I

INHIBITIVE FILLERS

| <u>Metalllc Chromates</u> | <u>Solubility in Water</u> | <u>pH*</u> |
|---------------------------|----------------------------|------------|
| barium chromate           | 0.00034 gm/100 ml at 16°C  | 9.2        |
| cobalt oxochromate        | ~ 0                        | 6.6        |
| strontium chromate        | 0.12 gm/100 ml at 15°C     | 9.1        |
| zinc chromate             | ~ 0                        | 6.8        |
| <u>Alkaline Compounds</u> |                            |            |
| aluminum oxide            | 0.0001 gm/100 ml at 29°C   | 10.2       |
| calcium hydroxide         | 0.185 gm/100 ml at 0°C     | 12.5       |
| magnesium hydroxide       | 0.0009 gm/100 ml at 18°C   | 10.7       |
| magnesium oxide           | 0.00062 gm/100 ml at 20°C  | 11.3       |

\* pH of a saturated water solution at 25°C

TABLE II

MIL-P-23377C EPOXY PRIMER FORMULATION

|                    |                                  | <u>parts-by-weight</u> |
|--------------------|----------------------------------|------------------------|
| Inhibitive filler  | strontium chromate <sup>1</sup>  | 24.0                   |
| Extending pigments | titanium dioxide <sup>2</sup>    | 4.6                    |
|                    | diatomaceous silica <sup>3</sup> | 5.8                    |
|                    | magnesium silicate <sup>4</sup>  | 11.0                   |
| Vehicle            | epoxy resin <sup>5</sup>         | 27.3                   |
|                    | polyamide resin <sup>6</sup>     | 15.0                   |
| Solvents           | methyl isobutyl ketone           | 25.2                   |
|                    | toluene                          | 22.1                   |
|                    | xylene                           | 3.7                    |

- 1 - X2865: Hercules Inc.
- 2 - R966: E. I. duPont de Nemours & Co., Inc.
- 3 - Celite 266: Johns-Manville Products Corp.
- 4 - SF-399: Whittaker, Clark & Daniels, Inc.
- 5 - Epon 1001: Shell Chemical Co.
- 6 - Versamid 115: General Mills Chemicals, Inc.

The total, as in the control formulation (Table II), is always equal to 24. Each was screened for the ability to inhibit the corrosion of magnesium alloy panels in a salt-spray environment.

The salt-spray panels were prepared as follows. Each primer was sprayed over one side of a pretreated magnesium panel to a dry-film thickness of 0.7 to 0.9 mils (18 to 23 microns). After 60 minutes air-dry, a topcoat of MIL-C-81773 polyurethane enamel was sprayed over the primer to an additional dry-film thickness of 1.3 to 1.7 mils (33 to 43 microns). One day later, each coated panel was scribed in a figure "X" down to the bare metal substrate with a sharply pointed instrument. Finally, after an additional six days of curing time at ambient temperatures, the coating system was ready for testing.

Two different salt-spray environments were employed. These included a standard 5% sodium chloride concentration (Method 6061 of Federal Test Method Standard (FTMS) No. 141a) and an identical salt concentration incorporating sulfur dioxide ( $SO_2$ ) as a contaminant. In each test, the panel specimens were exposed at a rack angle of six degrees from the vertical. If, during the course of the test, the topcoat lifted or separated from any candidate primer because of underlying corrosion, that panel was removed from the salt-spray cabinet.

All of the test panels exposed to one or both of the salt-spray environments constituted a "series". In the first (Table III), second (Table IV), and third (Table V) series of tests, primers made with various inhibitor combinations were exposed for periods of 14 to 28 days on QQ-M-44 wrought magnesium alloy with MIL-M-3171C, Type VIII pretreatment (Iridite 15). In the fourth series (Table VI), the optimum inhibitive primer (Formulation 7510) was compared directly to the MIL-P-23377 control formulation for a period of 28 days on QE-22A-T6 cast magnesium alloy with Iridite 15 and Dow 17 pretreatment. Post-test photographs of the panels exposed during the third and fourth series of tests are shown in Figures 1 through 8, as indicated on the corresponding tables. (Note that each panel was removed from the salt-spray cabinet when evidence of topcoat failure was observed.)

#### EVALUATION OF OPTIMUM INHIBITIVE PRIMER

In order to determine whether or not the change in inhibitive fillers had an adverse effect on the physical properties of the original MIL-P-23377 epoxy primer, Formulation 7510 was directly compared with that material in a comprehensive laboratory evaluation. Those tests taken directly from specification MIL-P-23377 are described in the applicable paragraph listed in Table VII. Such properties include condition in container, odor, fineness of grind, drying time, surface appearance, adhesion, water resistance, and lubricating oil resistance. The following additional properties were also included.

#### Pot Life

A No. 2 Zahn viscometer cup was vertically submerged within the unthinned, admixed coating and then raised above the fluid surface to allow the contents

TABLE III

FIRST SERIES OF SALT-SPRAY TESTS

Test Conditions: 14 days, 5% salt concentration, 6° rack angle

Materials: B - barium chromate  
 C - cobalt oxchromate  
 S - strontium chromate  
 Z - zinc chromate

Substrate: QQ-M-44 magnesium alloy; MIL-M-3171C, Type VIII pretreatment

| <u>Inhibitive Fillers</u><br><u>(parts-by-weight)</u> | <u>Salt Spray</u> |                | <u>SO<sub>2</sub>/Salt Spray</u> |                |
|---|-------------------|----------------|----------------------------------|----------------|
|   | <u>Blistering</u> | <u>Lifting</u> | <u>Blistering</u>                | <u>Lifting</u> |
| 24 B  | 1 day             | 2 days         | 12 days                          | -              |
| 1 C, 23 S*  | 1                 | 7              | 1                                | 7              |
| 24 S (MIL-P-23377)                                    | 4                 | -              | 2                                | 11             |
| 24 Z  | 4                 | -              | 6                                | 14             |

\* level recommended by supplier of cobalt oxchromate



T A B L E I V

SECOND SERIES OF SALT-SPRAY TESTS

Test Conditions: 14 days, 5% salt concentration, 6° rack angle

Materials: AO - aluminum oxide  
 CH - calcium hydroxide  
 MH - magnesium hydroxide  
 MO - magnesium oxide  
 S - strontium chromate

Substrate: Q-M-44 magnesium alloy; MIL-M-3171C, Type VIII pretreatment

| Inhibitive Fillers<br>(parts-by-weight) | Salt Spray        |                | SO <sub>2</sub> /Salt Spray |                |
|---|-------------------|----------------|-----------------------------|----------------|
|   | <u>Blistering</u> | <u>Lifting</u> | <u>Blistering</u>           | <u>Lifting</u> |
| 4 AO, 20 S                              | 1 day             | 4 days         | 9 days                      | -              |
| 8 AO, 16 S                              | 5                 | 14             | 7                           | -              |
| 12 AO, 12 S                             | 7                 | 14             | 14                          | -              |
| 4 CH, 20 S                              | 2                 | 7              | 14                          | -              |
| 8 CH, 16 S                              | 9                 | -              | 12                          | -              |
| 12 CH, 12 S                             | 5                 | -              | 12                          | -              |
| 4 MH, 20 S                              | 1                 | 2              | 12                          | -              |
| 8 MH, 16 S                              | 5                 | -              | 7                           | -              |
| 12 MH, 12 S                             | 2                 | 7              | 2                           | -              |
| 4 MO, 20 S                              | 5                 | -              | 2                           | -              |
| 8 MO, 16 S                              | 5                 | -              | 7                           | -              |
| 12 MO, 12 S                             | 9                 | -              | -                           | -              |

T A B L E V

THIRD SERIES OF SALT-SPRAY TESTS

Test Conditions: 28 days, 5% salt concentration, 6° rack angle

Materials: MO - magnesium oxide  
 S - strontium chromate  
 Z - zinc chromate

Substrate: QQ-M-44 magnesium alloy; MIL-N-3171C, Type VIII pretreatment

| Figure No. | Inhibitive Fillers (parts-by-weight) | Salt Spray |         | SO <sub>2</sub> /Salt Spray |         |
|------------|--------------------------------------|------------|---------|-----------------------------|---------|
|            |                                      | Blistering | Lifting | Blistering                  | Lifting |
| 1          | 24 S (MIL-P-23377)                   | -          | 5 days  | -                           | 6 days  |
| 1          | 2 MO, 22 S                           | -          | 4       | -                           | 7       |
| 1          | 3 MO, 21 S                           | -          | 3       | -                           | 5       |
| 2          | 4 MO, 20 S                           | -          | 4       | -                           | 10      |
| 2          | 5 MO, 19 S                           | -          | 28      | -                           | -       |
| 2          | 6 MO, 18 S                           | -          | -       | -                           | -       |
| 3          | 8 MO, 16 S                           | -          | 14      | -                           | 14      |
| 3          | 12 MO, 12 S                          | 14 days    | -       | -                           | 14      |
| 3          | 16 MO, 8 S                           | -          | 10      | 7 days                      | 10      |
| 4          | 20 MO, 4 S                           | 3          | 7       | 3                           | 14      |
| 4          | 24 MO                                | 1          | 7       | -                           | 10      |
| 4          | 24 Z                                 | -          | 4       | -                           | 7       |
| 5          | 2 MO, 22 Z                           | -          | 3       | -                           | 4       |
| 5          | 3 MO, 21 Z                           | -          | 3       | -                           | 4       |
| 5          | 4 MO, 20 Z                           | -          | 5       | -                           | 3       |
| 6          | 5 MO, 19 Z                           | -          | 5       | -                           | 5       |
| 6          | 6 MO, 18 Z                           | -          | 6       | -                           | 5       |
| 6          | 8 MO, 16 Z                           | -          | -       | -                           | 14      |
| 7          | 12 MO, 12 Z                          | -          | 14      | -                           | 14      |
| 7          | 16 MO, 8 Z                           | 14         | -       | 7                           | 14      |
| 7          | 20 MO, 4 Z                           | -          | 10      | 7                           | 10      |
| 4          | 24 MO                                | 1          | 7       | -                           | 10      |

TABLE VI

FOURTH SERIES OF SALT-SPRAY TESTS

Test Conditions: 13 days, 5% salt concentration, 6° rack angle

Materials: MD - magnesium oxide  
S - strontium chromate

Substrate: QE-22A-T6 cast magnesium alloy

| Figure<br>No. | Inhibitive Fillers<br>(parts-by weight) | SALT SPRAY        |                |                   |                |
|---------------|---|-------------------|----------------|-------------------|----------------|
|               |   | Iridite 15*       |                | Dow 17            |                |
|               |   | <u>Blistering</u> | <u>Lifting</u> | <u>Blistering</u> | <u>Lifting</u> |
| 8             | 24 S (MIL-P-23377)                      | -                 | 5 days         | -                 | 10 days        |
| 8             | 6 MD, 18 S<br>(Formulation 7510)        | -                 | 4              | -                 | 28             |

\* MIL-M-3171C, Type VIII pretreatment

SALT SPRAY

SALT/SO<sub>2</sub> SPRAY

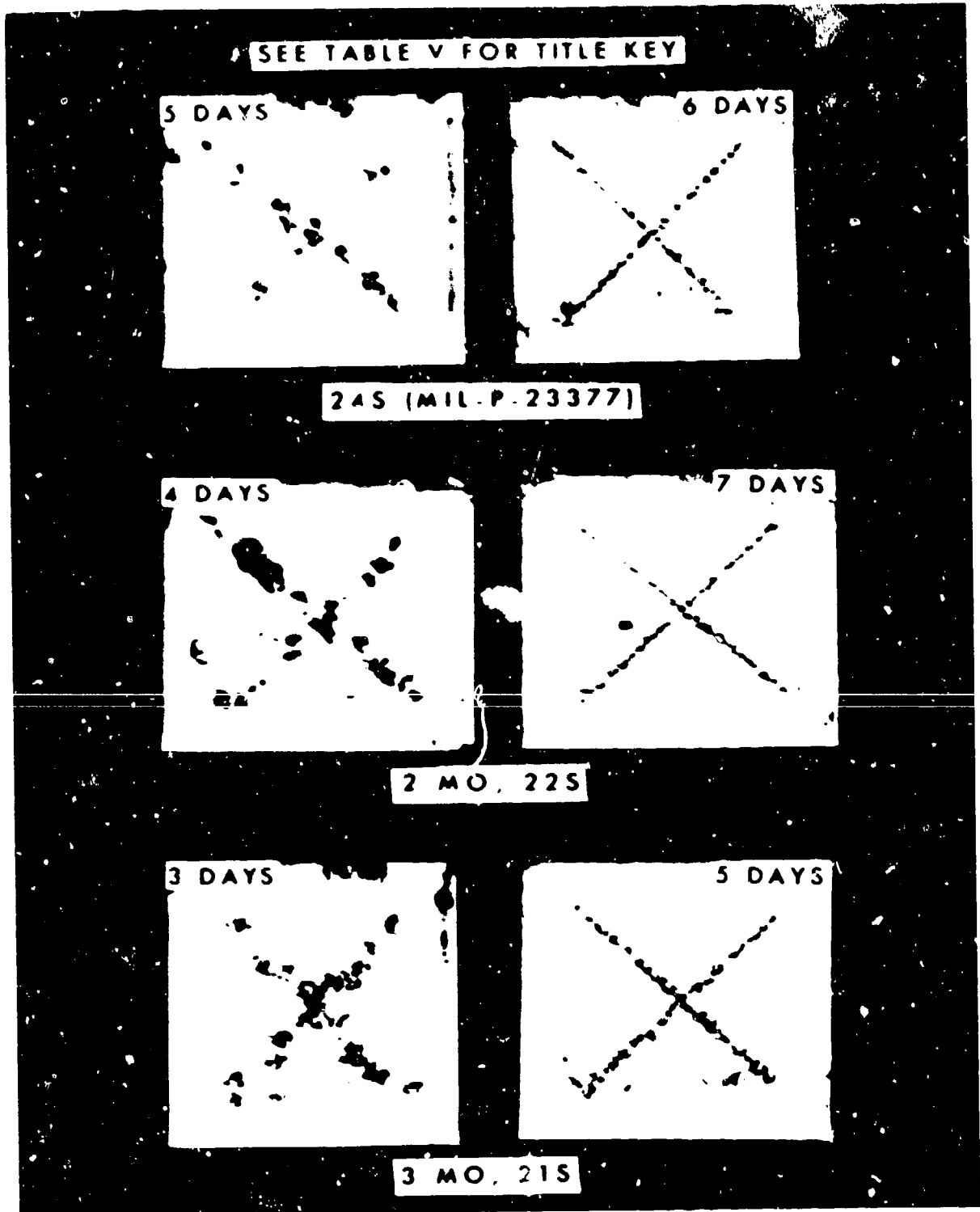


FIGURE 1 THIRD SERIES OF SALT SPRAY TESTS

SALT SPRAY

SALT/SO<sub>2</sub> SPRAY

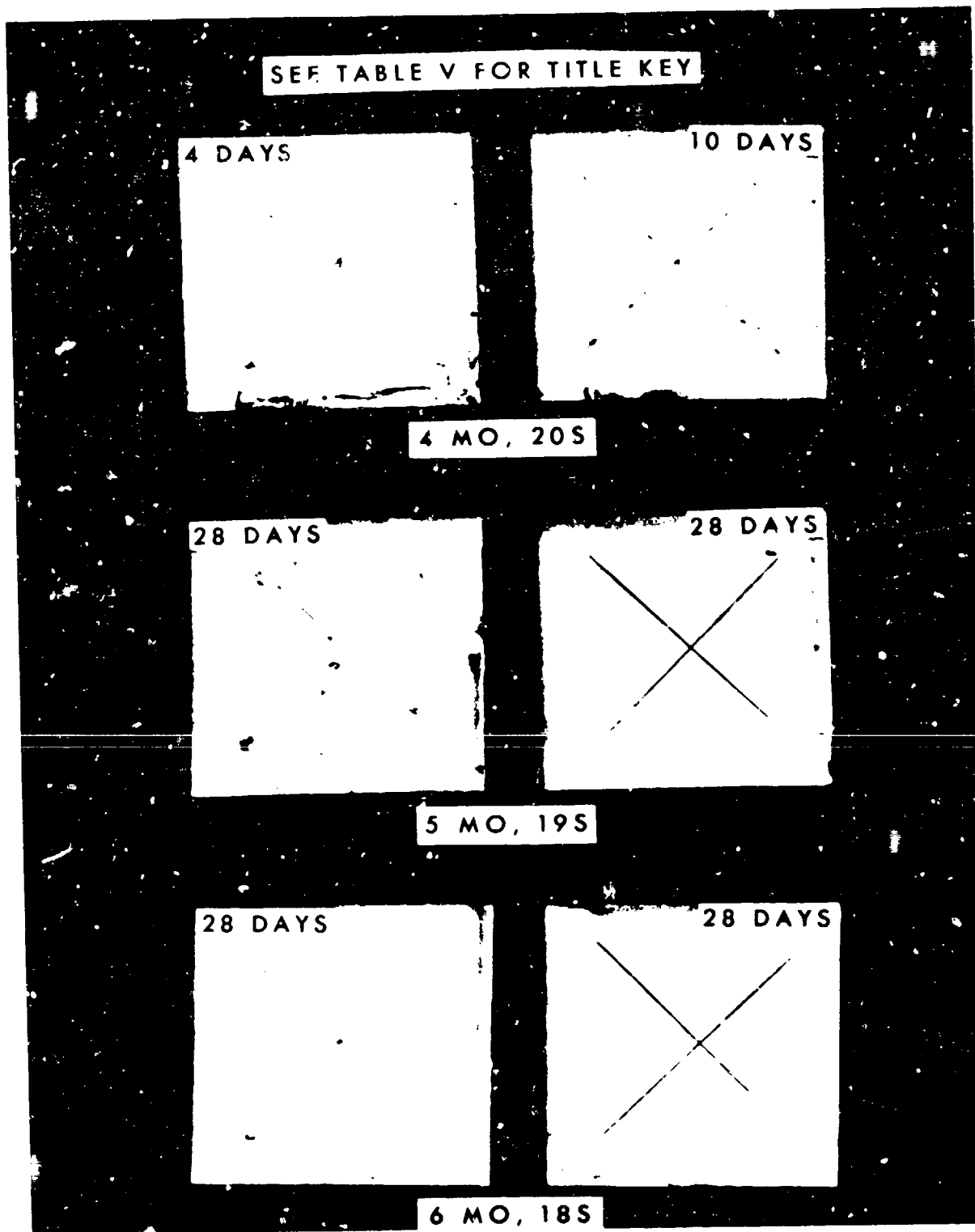


FIGURE 2 THIRD SERIES OF SALT SPRAY TESTS

SALT SPRAY

SALT/SO<sub>2</sub> SPRAY

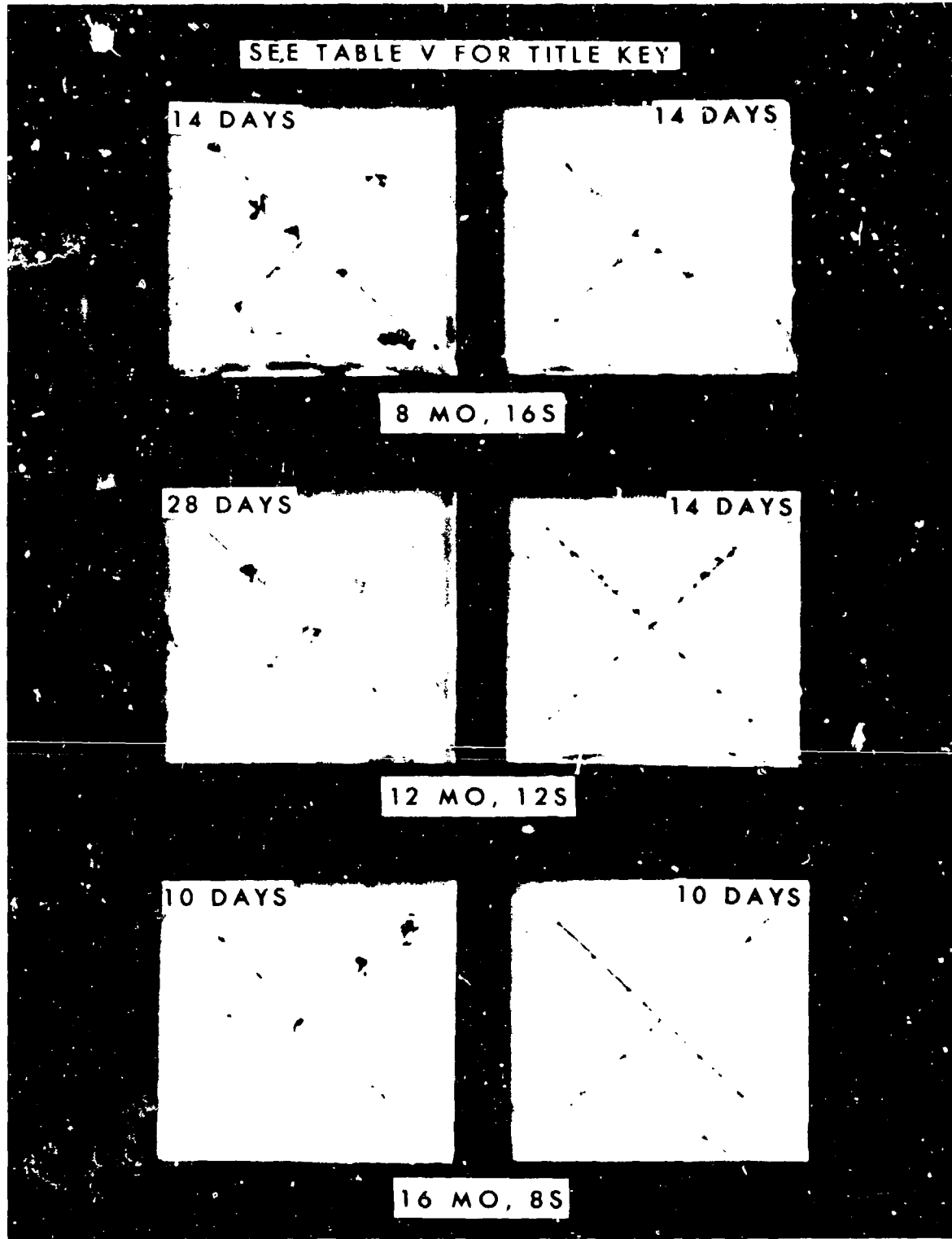


FIGURE 3 THIRD SERIES OF SALT SPRAY TESTS

SALT SPRAY

SALT/SO<sub>2</sub> SPRAY

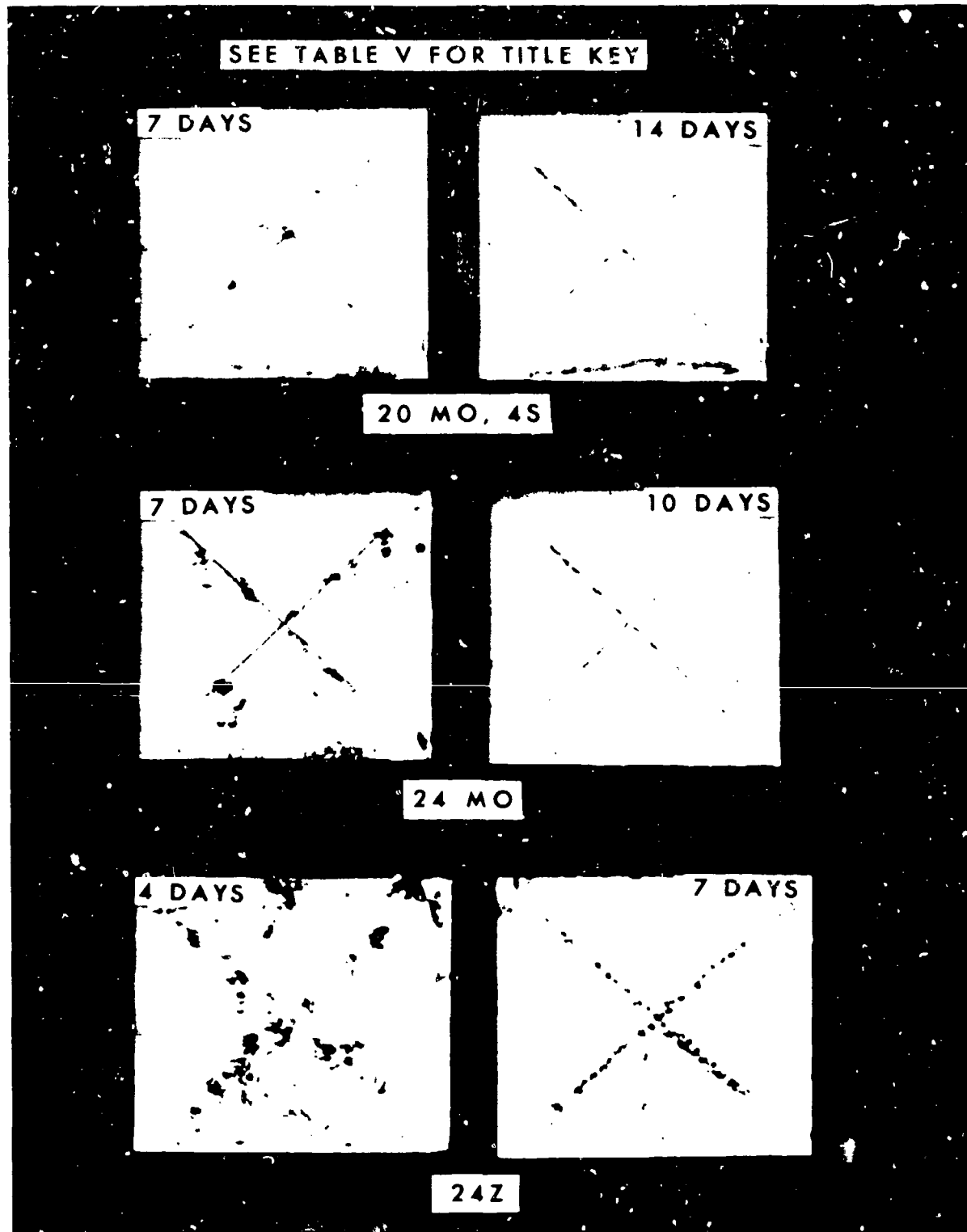


FIGURE 4 THIRD SERIES OF SALT SPRAY TESTS

SALT SPRAY

SALT/SO<sub>2</sub> SPRAY

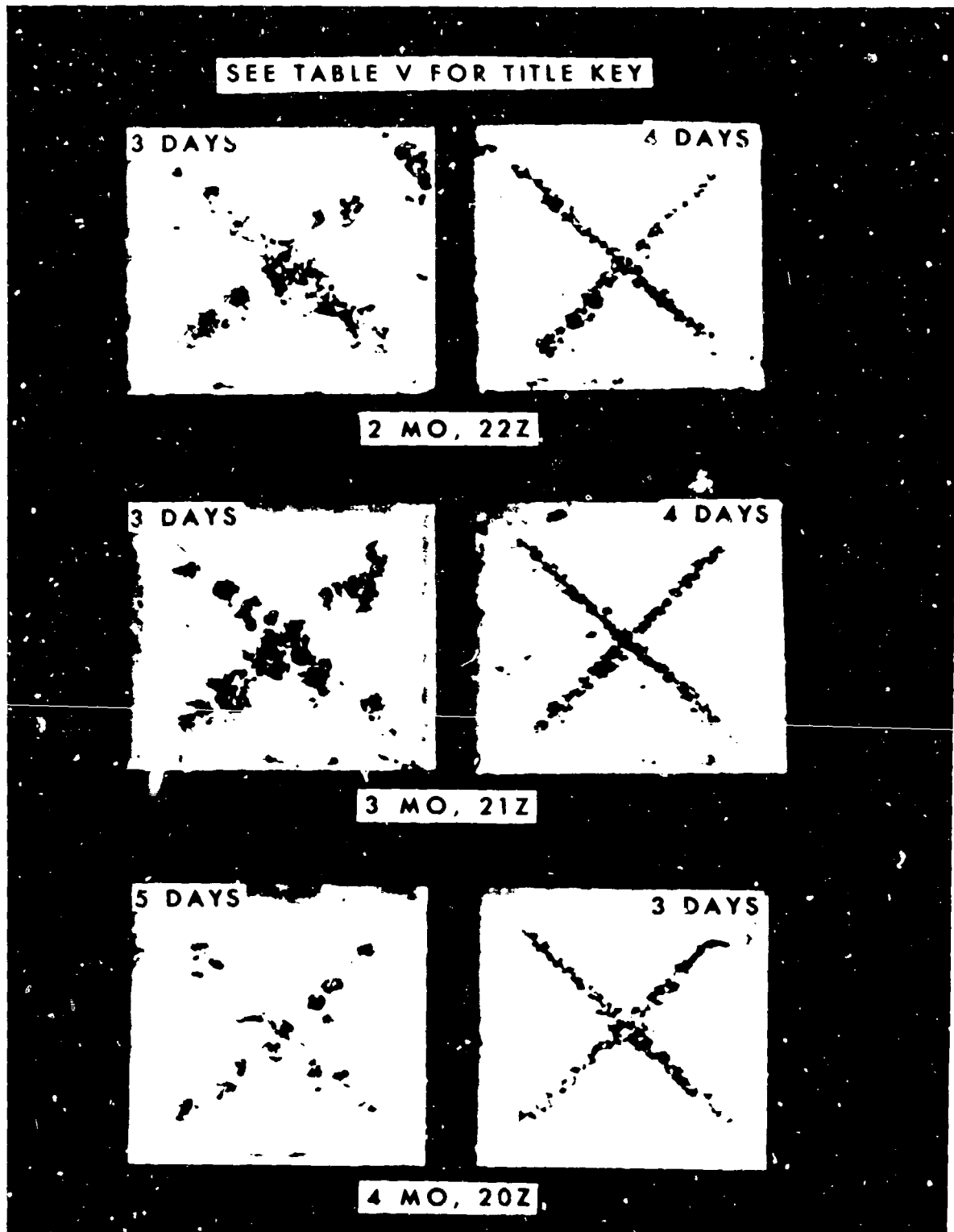


FIGURE 5 THIRD SERIES OF SALT SPRAY TESTS



SALT SPRAY

SALT/SO<sub>2</sub> SPRAY

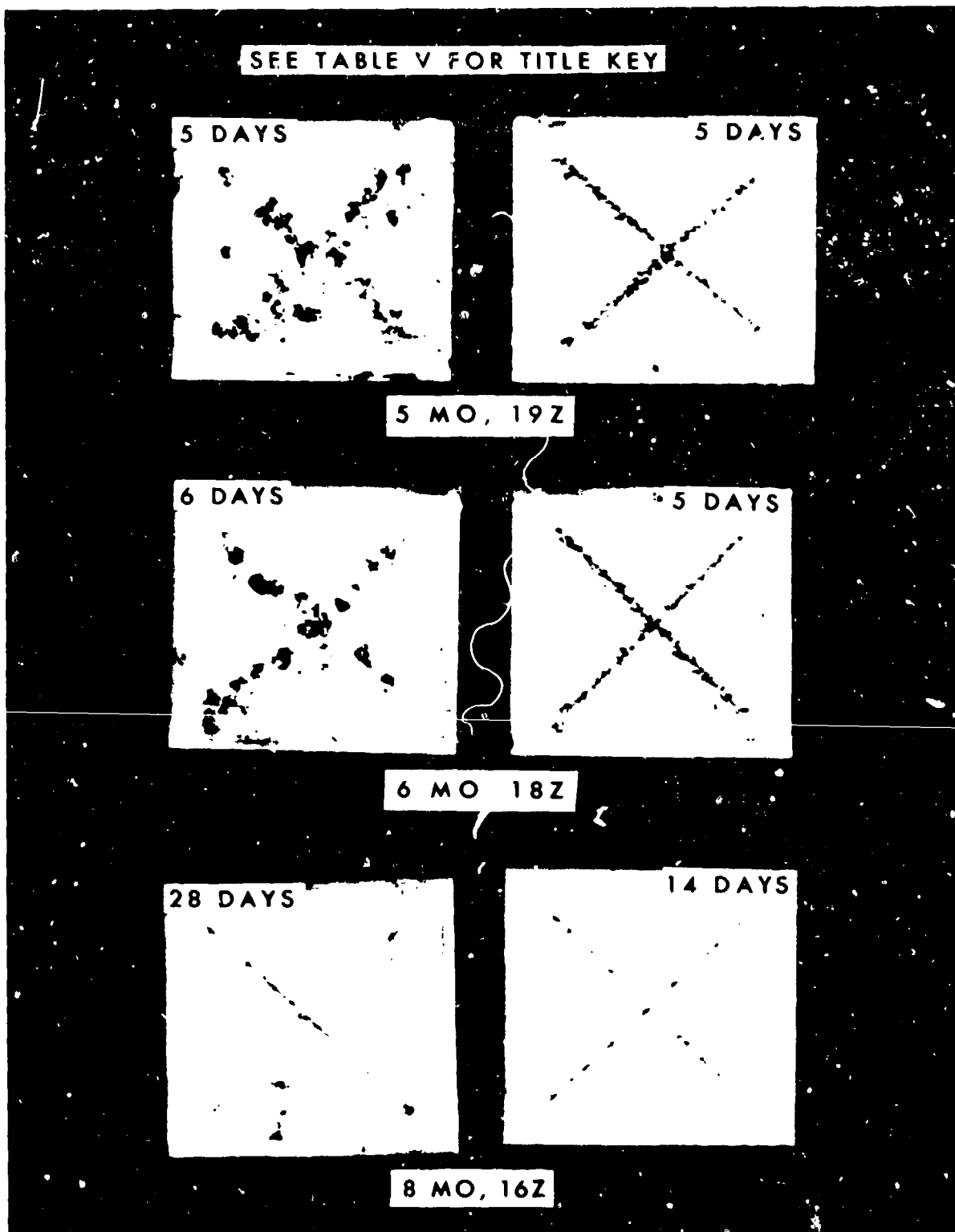


FIGURE 6 THIRD SERIES OF SALT SPRAY TESTS

SALT SPRAY

SALT/SO<sub>2</sub> SPRAY

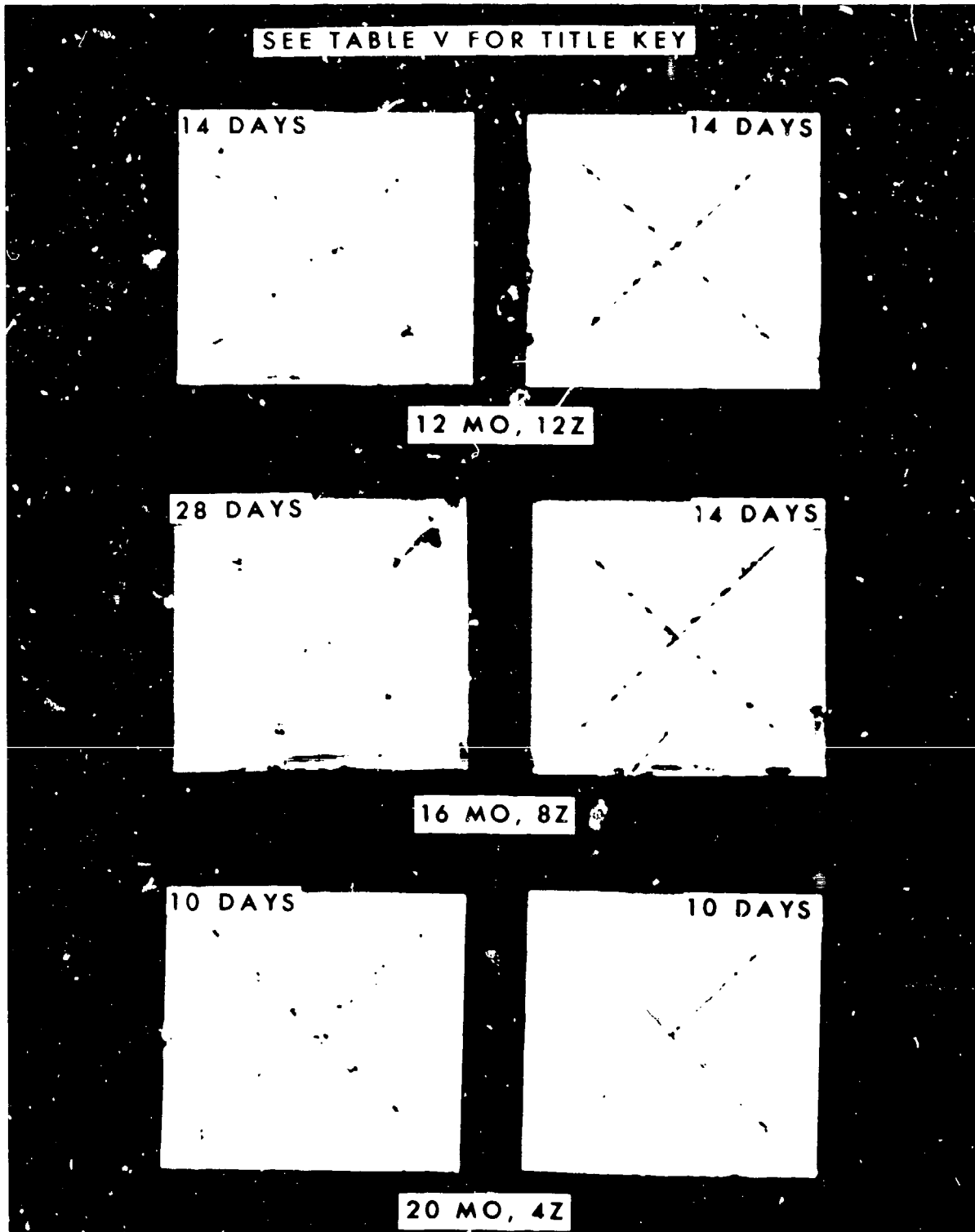


FIGURE 7 THIRD SERIES OF SALT SPRAY TESTS

SALT SPRAY

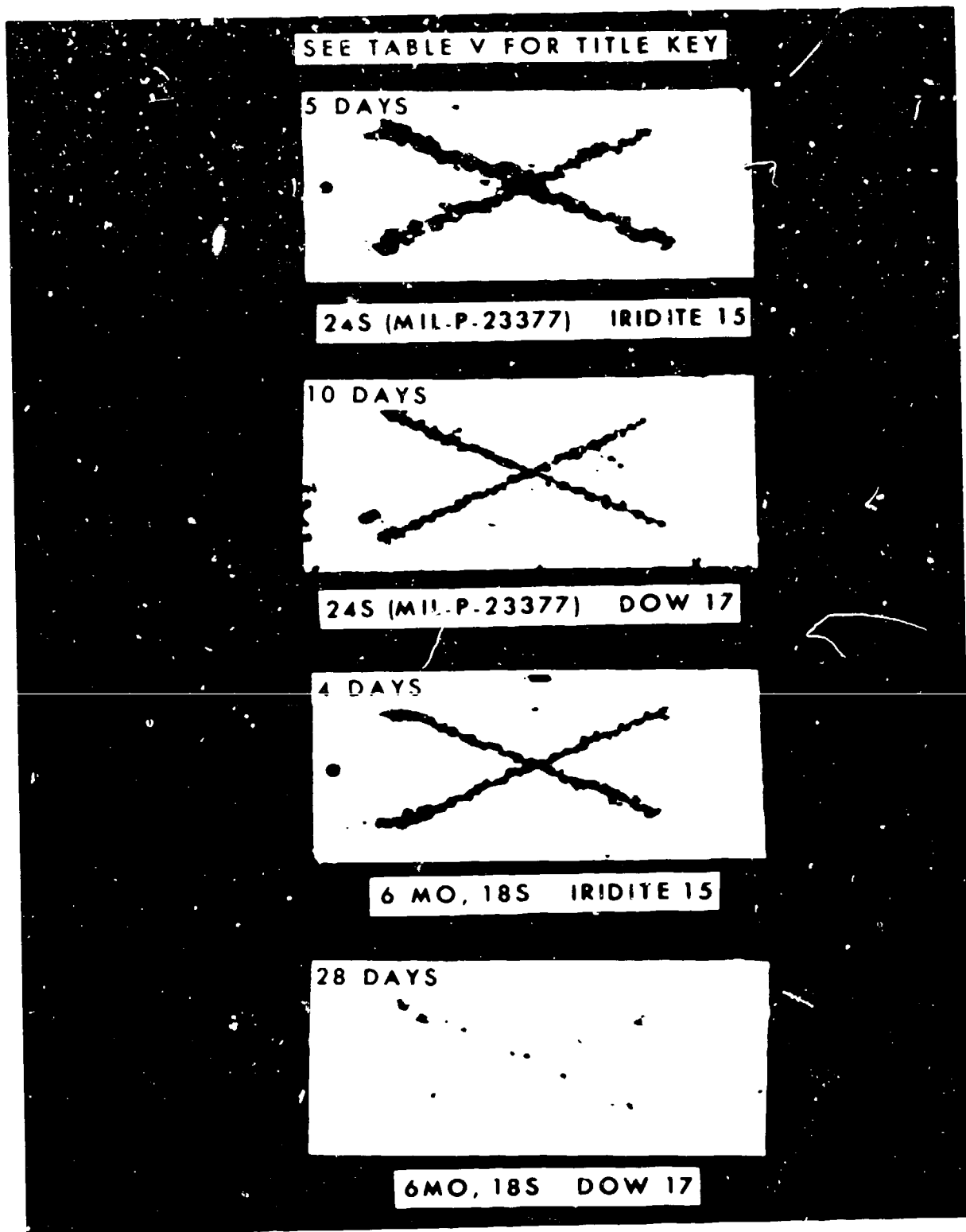


FIGURE 8 FOURTH SERIES OF SALT SPRAY TESTS

T A B L E V I I  
PHYSICAL PROPERTIES OF MIL-P-23377 EPOXY PRIMER VS. FORMULATION 7510

| Paragraph    | Test Property                     | TEST RESULTS        |                       |
|--------------|-----------------------------------|---------------------|-----------------------|
|              |                                   | Control Formulation | NADC Formulation 7510 |
| MIL-P-23377C |                                   |                     |                       |
| 3.5.1        | Condition in container            | satisfactory        | satisfactory          |
| 3.5.2        | Odor                              | satisfactory        | satisfactory          |
| 3.5.5        | Fineness of grind                 | satisfactory        | satisfactory          |
| 3.6.2        | Drying time                       | satisfactory        | satisfactory          |
| 3.6.3        | Surface appearance                | satisfactory        | satisfactory          |
| 3.6.4        | Adhesion (tape test)*             | satisfactory        | satisfactory          |
| 3.6.4.1      | Adhesion (knife test)*            | satisfactory        | satisfactory          |
| 3.7.1        | Water resistance                  | satisfactory        | satisfactory          |
| 3.7.2        | Lubricating oil resistance        | satisfactory        | satisfactory          |
|              | Pot life, initial viscosity       | 17 sec.             | 16 sec.               |
|              | Pot life, viscosity after 6 hrs   | 17 sec.             | 16½ sec.              |
|              | Adhesion to substrate*            | 5 kg. (11.0 lbs)    | 4 kg. (8.8 lbs)       |
|              | Adhesion to topcoat*              | 5 kg. (11.0 lbs)    | 8 kg. (17.6 lbs)      |
|              | Flexibility (mandrel bend)**      | satisfactory        | satisfactory          |
|              | Flexibility (impact elongation)** | 40%                 | 40%                   |

\* Substrate: QQ-M-44 magnesium alloy; MIL-M-3171C, Type VIII pretreatment  
Topcoat: MIL-C-81773 polyurethane enamel

\*\* Substrate: QQ-A-250/5 (0 temper); MIL-A-8625, Type I anodize

of the cup to drain through the bottom orifice. The viscosity was recorded as the time required to completely drain the cup. A coating was considered to have a six-hour pot life, if the viscosity did not increase more than 25% from the initial value after six hours in a closed container.

### Adhesion

Two test panels, conforming to QQ-M-44 wrought magnesium alloy with MIL-M-3171C, Type VIII pretreatment, were required for each primer. The first panel was masked with one-inch (25.4 mm) masking tape along the longer edge, while the second was left bare. Then the test primer was sprayed over both panels to a dry-film thickness of 0.7 to 0.9 mils (18 to 23 microns). After 60 minutes air-dry, the second panel was then masked with the above tape along the longer edge in order to cover the freshly primed surface. Next, a topcoat of MIL-C-81773 polyurethane enamel was sprayed over this panel to an additional dry-film thickness of 1.3 to 1.7 mils (33 to 43 microns). After removing the masking tapes and allowing the test films seven days at ambient temperatures to fully cure, the panels were ready for testing.

The adherence between adjacent surfaces was quantitatively determined with the use of the Gardner Scrape-adhesion Apparatus as defined in Method 6303.1 of FTMS No. 141a. To evaluate the primer to substrate adhesion, the wire loop was placed onto the bare metal surface of the first panel at a 45° angle of incidence and was pushed against the exposed edge of the test primer under increasingly heavier loads until the primer was sheared from the metal beneath. The weight of this load is directly related to the strength of the adhesive bond. The primer to topcoat adhesion on the second panel was evaluated in a similar manner.

### Flexibility

This test required the use of 0.020-inch (0.5 mm) thick clad aluminum alloy panels conforming to QQ-A-250/5 (0 temper) and anodized in accordance with MIL-A-8625, Type I. The test primer was sprayed to a dry-film thickness of 0.7 to 0.9 mils (18 to 23 microns) and was allowed seven days at ambient temperatures to fully cure.

The flexibility was evaluated using two different methods. First, a primed panel was bent, coated side away, over a 0.125-inch (3.175 mm) mandrel as described in Method 6221 of FTMS No. 141a. Visually observed surface cracking was the criterion for failure in this test. Method 6226 of FTMS No. 141a was employed to determine the impact elongation of the primer. This involved dropping a cylindrical weight onto the uncoated side of a primed panel to produce a series of spherical impressions corresponding to various percentages (¼ to 60%) of elongation. The largest impression that exhibited no surface cracking indicated the minimum impact elongation of the material.

## RESULTS AND DISCUSSION

In the salt-spray test series, the results are given as the time required before blistering or lifting of the protective topcoat occurred. The dashes indicate that the listed type of failure did not occur during the entire test period. The difficulty of correlating salt-spray data on a quantitative basis is compounded when that data is taken from more than one test series. For this reason, all of the conclusions herein are based on relative comparisons within each series.

Primers incorporating the individual metallic chromates were exposed during the first series (Table III). Of these, the strontium chromate and zinc chromate filled coatings exhibited the best overall corrosion resistance on the magnesium substrates. In the second test series (Table IV), strontium chromate was used in combination with various levels of the alkaline compounds. The results indicated that the addition of the alkaline fillers significantly improved the performance of the original chromate-inhibited primer, particularly in the SO<sub>2</sub>/salt-spray environment. The magnesium oxide inhibited formulations were the only group to complete the standard salt-spray exposure without lifting. The third series (Table V) was a more intensive study on the use of magnesium oxide with both strontium and zinc chromates. The optimum system occurred at a level of 5 parts-by-weight of magnesium oxide to 18 parts-by-weight of strontium chromate. This system was the only one to withstand 28 days exposure in both environments without any type of failure. By comparison, the MIL-P-23377 primer failed after 5 days in salt-spray and 6 days in SO<sub>2</sub>/salt-spray. The results indicate that the mechanism of alkaline inhibition is highly concentration dependent. Levels above and below the optimum did not perform as well. Post-test photographs of the test panels are shown in Figures 1 through 7, as indicated in Table V. (Note that each panel was removed from the salt-spray cabinet when evidence of topcoat failure was observed.)

In the fourth test series (Table VI), the optimum primer (designated NADC Formulation 7510) was directly compared to the MIL-P-23377 control primer on a cast magnesium alloy. Two pretreatments often used on magnesium, Iridite 15 and Dow 17, were also compared in this series. There was little to choose between the coatings over the Iridite film. Both failed due to corrosion in a short period of time (4 to 5 days). However, definite improvement was noted with the Dow pretreatment as the MIL-P-23377 primer withstood twice the exposure time before failing (10 days) and Formulation 7510 lasted 20 days. Post-test photographs of the test panels are shown in Figure 8.

The substitution of magnesium oxide for part of the strontium chromate filler used in the MIL-P-23377 epoxy primer had only one significant effect on the physical properties of that material. As listed in Table VII, the adhesion to the pretreated magnesium substrate declined slightly, although this was offset by a considerable increase in adhesion to the MIL-C-81773 polyurethane topcoat. In either case, the adhesion is more than adequate for the requirements of current naval aircraft.

## C O N C L U S I O N S

The optimum inhibitive system consists of 6 parts-by-weight of magnesium oxide to 18 parts-by-weight of strontium chromate. When this system was substituted for the existing inhibitive filler in the MIL-P-23377 epoxy primer formulation, considerable improvement in salt-spray resistance was noted. On a pretreated wrought magnesium alloy, the optimized primer (NADC Formulation 7510) withstood 28 days in a standard salt-spray environment without effect while the specification material failed due to corrosion within 5 days. Similar results were also noted on a cast magnesium alloy. The inhibitive properties are highly dependent on the ratio of magnesium oxide to strontium chromate in the primer formulation. Variations above and below the specified level will decrease the corrosion resistance somewhat. Formulation 7510 possesses physical properties identical to those of the MIL-P-23377 standard, with the exception of a slight decrease in its adhesion to the magnesium substrate. The difference, however, is more than offset by a considerable increase in adhesion to the MIL-C-81773 polyurethane topcoat.

## R E C O M M E N D A T I O N S

It is recommended that a service evaluation be conducted on the magnesium components of a tactical aircraft such as the AV-8A Harrier to assess the effectiveness of NADC Formulation 7510 in protecting these areas through a typical fleet deployment.

## F U T U R E P L A N S

An effort will be undertaken to develop improved primers for magnesium alloys by exploiting other unique inhibitive systems beyond the scope of this study. This will include the possibility of using different binders, extenders, etc. to determine the influence of the various component parts on coating performance.

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