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DEPARTMENT OF THE NAVY

NAVAL AIR DEVELOPMENT CENTER WARMINSTER, PA. 18974

AERO MATERIALS DEPARTMENT

REPORT NO. NADC-MA-7160

16 SEP 1971

AN EVALUATION OF THE RECIRCULATING CHROMATE RINSE CONCEPT FOR AIRCRAFT CORROSION CONTROL

NAEC P.O. 1-8027 of 12/17/70

A feasibility study was made of the recirculating chromate aircraft rinse. A preliminary design plan is presented along with test data to support the design conclusions.

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Distribution of this report is unlimited.

SUMMARY

Introduction

Since Naval aircraft are subjected to extremely corrosive environments, water rinsing of the aircraft after missions is highly desirable to remove salt deposits that cause corrosion. However, water rinsing does not entirely eliminate corrosion and the effort can be enhanced significantly by the addition of a corrosion inhibitor to the rinse water. Compounds containing hexavalent chromium, quachrom glucosate in particular, have been found to be effective inhibitors. Unfortunately, hexavalent chromium compounds are highly toxic and rinse waters containing useful concentrations cannot be emptied into waterways or diverted to sanitary sewage treatment plants for purification. A typical water rinse facility generates about 1,000 gallons of effluent water per aircraft, or about 50,000 gallons per day. Preliminary evaluations of industrial chromate treatment processes indicate that the cost of treating this volume of concentrated chromate waste water would be unacceptably high.

Summary of Results

Evaluation of the water rinse environment indicates that only a few possible contaminating materials will be present in the rinse in significant quantities. Much of the investigation was devoted to measuring the effects of these contaminating materials on the chromate inhibitor solution. Materials from the aircraft such as lubricating oils, hydraulic oils and fuel have no significant effect on the chromate's effectiveness. Likewise, gaseous emissions are not expected to affect the rinse, with the possible exception of sulfur dioxide. However, the sulfur content of present jet fuels is very low and it is anticipated that only trace amounts of sulfur dioxide will be dissolved in the recirculating rinse.

Of the constituents normally present in the water used for makeup of the chromate rinse, only the chloride ion has a significant effect on the inhibiting capacity of the chromate. Where the water contains a high concentration of chloride ions, its removal or an increase in inhibitor concentration will be desirable.

Another factor that may effect the life of the rinse is the stability of the inhibitor itself. Effectiveness would be lost if the hexavalent chromium were reduced to trivalent chromium. However, hexavalent chromium is reduced only in the presence of certain types of compounds and it is improbable that these reducing agents will find their way into the rinse.

It is anticipated that the chromate rinse will produce a slight yellow spotting of the aircraft as the chromate dries. These spots will wash off when the aircraft goes through the fresh water rinse, but will be redeposited by the follow-up chromate rinse. If spotting is objectionable, a surfactant can be added to the rinse to control it. However, if a surfactant is used one must be found which is borh non-foaming and compatible with the inhibitor.

The mechanical aspects of a recirculating system are straightforward and the system can be achieved using standard process equipment. The major problem will be to confine the chromate to the spray area. Rain falling on the chromate rinse spray area will pick up an unacceptable concentration of chromate in the effluent. This run-off must be collected. Where the run-off is less than the demand for make-up water, it can be utilized as make-up water, otherwise excess run-off will have to be treated or diluted to acceptable concentrations.

Chromate carried over to the fresh water rinse by the aircraft is another area of concern. It is estimated that the chromate removed from the aircraft by the fresh water rinse will be about six times greater than permissable if the fresh water rinse is to be emptied into the storm sewer. However, simple dilution of this rinse with sanitary wastes to reduce the chromate concentration to acceptable levels may be possible.

Conclusions

A recirculating chromate rinse system presents no major operational or maintenance problems provided stray contamination via run-off of contaminated rain water and drag-out of chromate to the fresh rinse effluent are controlled. It is concluded that the life of the rinse will be excellent, perhaps several months, since materials that are likely to enter have little or no adverse affect on the inhibiting action of the chromate. An actual field evaluation is the only reasonable way to determine the true life of the chromate inhibitor.

Recommendations

It is recommended that a prototype recirculating water rinse system be built as described herein and diagrammed in figure 3. A chromate waste treatment system may ultimately be necessary, but it is recommended that investment in waste treatment facilities be delayed until operational load data is established for rinse bath life and for drag-out of chromate in the fresh rinse. Such data is necessary for proper sizing of the waste treatment plant. Our recommendation is that the prototype plant be operated for six months to establish this data. During this period operation of the rinse without a waste treatment facility can be accomplished by reducing the number of aircraft treated so that the fresh water rinse effluent from non-treated aircraft will dilute the chromate drag-out to an acceptable level.

It is recommended that potassium dichromate be used as the inhibitor rather than quachrom glucosate. The original selection of the quachrom glucosate in preference to inorganic chromates was based on its superiority in overcoming pitting corrosion at very low concentrations. This advantage disappears at the higher inhibitor loading possible with a recirculating system. Potassium dichromate has the advantages of being less costly, less likely to be degraded in use, and commercial experience is available in the treatment technology of waste effluents containing potassium dichromate.

It is recommended that the sanitary sewage system be utilized where possible to dispose of chromate wastes. Where the sanitary sewer water is free of chromates, chromate tainted fresh water rinse can be blended with the sanitary sewer water to dilute the chromate to acceptable levels. However, if the chromate maximum in the effluent is exceeded, this will not be possible. Each case will have to be considered individually. Where chromate removal plants are installed, consideration should be given to utilizing the sanitary sewage settling basins to collect the precipitate, thus saving the cost of separate sludge handling and disposal.

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DISCUSSION

Chemical Considerations

Reference (b) is a report of an investigation of corrosion inhibitors for a fresh water aircraft rinse. Therein, it was concluded that the most effective inhibitors for water rinses were those that contained hexavalent chromium. Quachrom glucosate and potassium dichromate provide nearly equivalent corrosion protection at concentrations above 0.02% and slightly better protection than other hexavalent chromiums tested. At concentrations below 0.02% quachrom glucosate was the only material that provided adequate protection. The inorganic chromates were unable to stop localized pitting at this low level of application. For a one-shot rinse, economic considerations limited the inhibitor concentration to not more than 0.05%, and quachrom glucosate was recommended as the inhibitor which would provide protection with a greater margin of safety. For a recirculating system, however, the inhibitor concentration can be many times greater. Therefore, equivalent protection can be anticipated for both quachrom glucosate and potassium dichromate inhibitors in a recirculating rinse. Potassium dichromate is the inhibitor recommended for the recirculating system. It is more readily available. It is an inorganic chemical, and not as subject to decomposition. It has some cost advantage and waste treatment processes for potassium dichromate have been established.

Laboratory evaluations were made to establish the relationship between inhibitor concentration and dissolved minerals for adequate corrosion protection. The requirements of the rinse are that it prevent pitting or staining of 1010 steel and magnesium, and that it greatly reduce the galvanic corrosion between 1010 steel and magnesium. An outline of the test procedure is given in Appendix C. The water quality at the Naval Air Stations varies considerably in both kind and quantity of dissolved salts. An inhibitor's effectiveness is dependent upon both concentration and upon the presence of interfering ions in the water. In the case of the above metals, tests indicate that only the chloride ion significantly alters the corrosiveness of water. To achieve a realistic estimate of inhibitor performance, a test water was formulated for use in evaluating the inhibitors at high salt loadings. Appendix A describes the selection of the test water. This test water contained:

255 ppm HCO₃
72 ppm Cl⁻

10 ppm Mg⁺⁺

100 ppm Na+

25 ppm Ca++

15 ppm S04"

De-ionized water was used for the make-up. To gather data on the concentration effect of the dissolved ions the test water was diluted with de-ionized water in the ratios of 0:1, 1:1, 3:1, 1:3 and 1:0. Figure 1 illustrates the minimum concentration of potassium dichromate inhibitor necessary to achieve adequate protection as the level of dissolved ions in the water is varied. This minimum concentration is as low as .002% in de-ionized water to 0.3% for the test water. A summary of the data is contained in Appendix E. It should be remembered that these are minimum concentrations and that a higher concentration is desirable if complete protection is to be assured. It should also be noted that the flat portion of the curve at 0.3% inhibitor concentration is a temporary plateau. At higher contaminant loadings, particularly if the contaminant is C1°, the required inhibitor increases sharply. However, in most localities 0.1% inhibitor in the recirculating system will be adequate.

Since the concept of a recirculating chromate rinse is to reuse the chromate again and again, it was necessary to evaluate the effects of contaminants. Fortunately the list of probable contaminants is small. Also, the term rinse is somewhat of a mismomer in that just enough inhibited water should be used to coat the aircraft but not enough to accomplish any washing or rinsing action.

It is important that the aircraft always be rinsed with fresh water to remove dissolvable contaminants, particularly salt, whose chloride ion, as noted above, is most likely to degrade the chromate rinse. It will therefore be necessary to maintain a close check on any chloride build-up in the chromate rinse, particularly where evaporation losses tend to concentrate the soluble salts in the system.

Another source of contamination is the leakage of small amounts of lubricating oil, hydraulic oil and fuel from the aircraft that enter the rinse. These materials are practically insoluble in water and will be separated from the rinse by flotation and filtering before reuse (see Figure 3). The small amounts of these materials that might dissolve or become entrained in the rinse by emulsification have no significant effect as indicated by the tests summarized in Appendix D. It is not anticipated that materials of this type will degrade the inhibitor rinse.

Gaseous emissions from the aircraft engines, as well as industrial impurities in the air, will contact exposed chromate rinse. Of the possible gases which might be expected, sulfur dioxide (SO₂) is the most likely to react with the chromate. Sulfur dioxide reacts with hexavalent chromium to reduce it to trivalent chromium, an ineffective corrosion inhibitor. Fortunately there are two favorable factors. Contact between the inhibitor rinse and exhaust gases is expected to be minimal, and the sulfur content of present jet fuels is very low. Thus, sulfur dioxide emissions should have only a negligible effect.

In the same area of concern is the potential degradation and reduction of the chromate inhibitor by other factors. Trivalent chromium was added to

test solutions of potassium dichromate to evaluate its effect on the inhibitors' performance. It did not significantly affect the inhibiting action of the chromate. Therefore, the only concern is that the level of active inhibitor could be lowered to a point where protection is reduced.

When the aircraft is sprayed with the inhibited rinse only a little will actually adhere to the surface. The liquid that adheres will be distributed in two ways. There will be a thin layer of film spread across the surface, particularly on exposed metal. However, the majority of the liquid will form droplets which dry and adhere to form noticeable spotting, particularly on white painted surfaces. Spotting will be increased on deteriorated painted surfaces. This problem is not one of corrosion prevention, but rather one of aesthetics. Spotting can be reduced through the addition of a surfactant to the chromate rinse. The surfactant or wetting agent will promote uniform wetting of the surfaces by reduction of surface tension; low surface tension allows the film to dry without forming droplets and the resulting spotting. However, many surfactants tend to be slightly corrosive when used alone, and tests should be conducted to evaluate any surfactant before it is introduced into the system.

Design Criteria

Fresh Water rinse: As a first step, the aircraft should be taxied through a fresh water rinse. This rinse is intended primarily to remove all traces of salt picked up during the mission. The rinse will also remove any chromate residue from a previous chromate rinse. Based upon a rinse of 1,000 gallons and an allowable chromate content of .05 ppm in the effluent, only .5 grams of potassium dichromate is permitted to wash off the aircraft. The tests reported in Appendix B indicate that as much as 4 grams of potassium dichromate would be dissolved in the fresh water rinse even when the concentrated rinse is limited to 0.1% potassium dichromate. This value is eight times greater than the maximum allowable for the effluent. Thus it is apparent that the fresh water rinse must have chromate removal treatment or be diluted before being dumped.

Two basic types of water rinse facilities have been established at the Naval Air Stations. (a) The deluge type, such as that at Patuxent River which develops a wall of water spray 100 ft. wide by 35 ft. deep through which the aircraft is taxied, and (b) the selective wash system such as that at NAS, Moffet Field in which a series of washing nozzles are timed to rinse sections of the aircraft individually as it passes each station. The latter is intended primarily to conserve water in a water scarce area. No effort has been made to compare the effectiveness of the two types of systems, though both are reported to be satisfactory.

Recirculating Chromate Rinse

SPRAY SYSTEM: The chromate inhibited rinse will be applied after the fresh water rinse. The chromate runoff will be collected, filtered and then reused. The system should be designed to use a minimal volume of the chromated water.

The spray should be divided into separate groupings aimed at the lower wing surfaces, lower fuselage, upper wing surfaces, upper fuselage, and tail surfaces. The spray nozzle should provide a flat non-atomized spray pattern that impacts the aircraft with a maximum of force. The system should use 100 to 200 gpm. The rinse would be scattered less if instant actuation of the flow can be achieved, and if an air blow-off system can be installed to remove excess rinse, although the cost of the latter may be prohibitive. The basic design considerations of this spray system are.

- 1. To impact the aircraft with a hard hitting spray that can drive into openings, cracks or crevices.
 - 2. To eliminate any non-working spray that might be blown away.
- 3. To prevent chromate inhibitor from escaping from the spray area or having excess inhibitor carried away by the aircraft.

EFFLUENT COLLECTION: The collection system for the chromate rinse should be as simple as possible to minimize costs and maintenance. Basically the system should consist of a large waterproof apron to catch the run-off and channel it to a collection pipe. The collection pipe must be fitted with appropriate valving and instrumentation to discriminate between the chromated run-off of the recirculating rinse and rain water. The recirculating chromate rinse will be sent to a separator and holding tank, whereas the rain run-off should be collected in a run-off water collection tank. During a rain storm it should be necessary to collect only the initial chromate containing run-off. The later run-off can be safely emptied into the storm sewer. The collection tank will serve two purposes: (a) prevent contaminated rain water from being washed away and (b) provide high quality make-up water provided the rain itself is not polluted. The edge of the apron should be provided with a jet blast screen to prevent pick-up and scatter of the chromate solution by back wash from the engines.

Treatment

The heart of the treatment system will be a large holding tank or contaminant separation tank as illustrated in Figure 2. Run-off from the spray is emptied into the tank where particles of dirt are allowed to settle and droplets of oil will float to the top. Skimmers will remove the dirt and oil. When reused, the solution is pumped through a final or polishing filter which removes fine solid matter and absorbs emulsified oil contaminants. Periodically the contents of the holding tank should be sampled to establish the condition of the solution. Provisions will be made to adjust the pH and replenish the chromate via a chemical addition station. A separate testing station will also be needed to monitor the condition of the run-off water, chromate rinse and water supply.

Solution Maintenance

Solution maintenance is expected to be relatively simple, consisting only of make-up of lost solution and possibly an occasional adjustment of the pH. No data has been generated to indicate what to expect in terms of probable solution adjustments or usable life, however, it is anticipated that drag-out

losses will not be greater than 1% of the rinse. Depending on the proximity of the clear water rinse to the inhibited rinse station, water input resulting from drag-out of the clear water rinse may approximate the chromate solution drag-out losses. In this case solution maintenance will be mostly a matter of make-up of the chromate drag-out losses. The useable life of the chromate solution appears to be indefinite, dependant only upon the contaminant build-up.

Water Purification

Where the primary water supply contains many dissolved contaminants, particularly if there is a high chloride content, a purifier is needed to remove these contaminants before the water is used to make up the chromate rinse solution. Selection of type and size of the water purifier will depend upon the quality of the water supply. Where the quality of the primary water supply is good, purification of make-up water is not necessary. Either reverse osmosis or ion-exchange may be used for the purification.

Chromate Disposal

A determination of when to replace the chromate solution cannot be gauged without field experience. Evaluation of chromate content and contaminant levels will be the criteria for gauging correct disposal times. However, unanticipated factors are likely to enter the picture and a maximum life should be placed on the solution to avert complications. The choice of a final disposal method ultimately depends upon solution life. If the life is sufficiently long and a treatment plant is not needed for the fresh water effluent, then perhaps the spent solution can be most economically handled by trucking it to a disposal plant. Where a treatment plant must be built the chemical destruction method should be used. There are two commercially accepted processes; a metabisulfite process and a sulfur dioxide process. The SO₂ process is more economical and probably more convenient for use at a Naval air station.

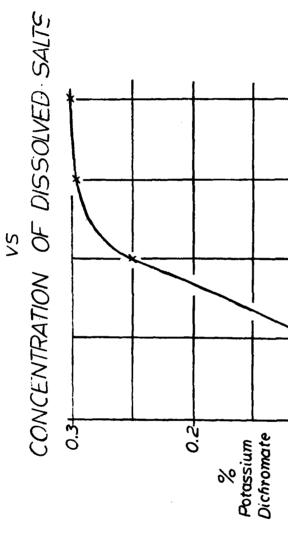
Chromate Blow-Off

It is an important consideration to prevent excess chromate from being carried away from the rinse site. Excess chromate solution that dries on the surface of the aircraft will be washed off by rain or in the clear water rinse. Although the quantity of chromate involved is small, it may cause objectionable staining of aprons, and pollution of rain run-off. Therefore, an air blow-off system should be considered as a means of removing excess chromate from aircraft as it exits the chromate rinsing station. Some operational experience is desirable, before the added cost of this system can be justified.

REFERENCES

- (a) NAEC Project Order P.O. 1-8027

 Naval Corrosion Preventive Maintenance Capabilities.
- (b) Report No. NADC-MA-7005 of 24 July 1969 —
 Investigation of Corrosion Inhibitors for Aircraft
 Fresh Water Rinse System.



Deionized
Water

Ratio of Deionized to Test Water

Figure

0.1-

Figure 2

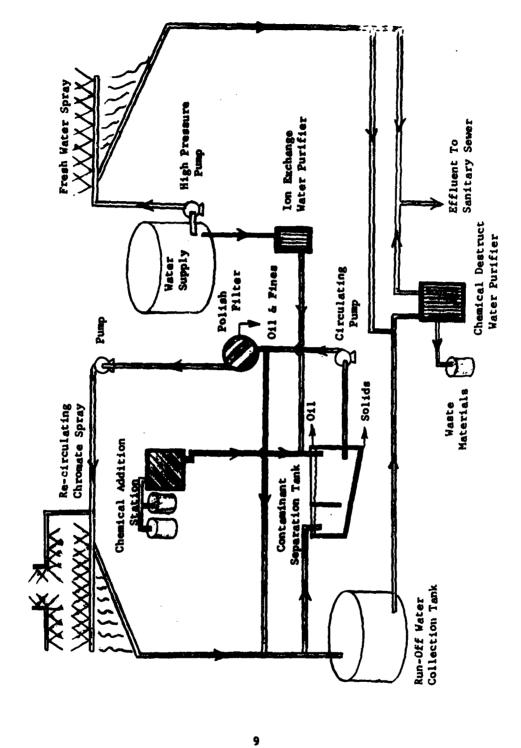


figure 3

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APPENDIX A

TEST WATER

For test purposes a test water was formulated which contained various amounts of the dissolved ions normally found in most water supply systems. The concentrations were selected after an examination of the composition of the waters used at five naval air stations. The concentration of chloride ion was deliberately made high (about 5 times higher than the average) since it is the ion which most strongly counteracts the effect of a chromate inhibitor. The sodium ion is also necessarily high because sodium chloride was added to get the desired chloride concentration. Intermediate values were selected for the other ions.

The following is a partial analysis of water from five naval air stations compared with the composition of the test water:

ION	CONCENTRATION IN PARTS PER MILLION						
	Patuxent River	Moffet Field	New River	Jacksonville	Cherry Point	TEST WATER	
HCO ₃	200	50	200	300	275	255	
∞_3	nil	nil	nil	2.5	-		
C1	3	25	10	< 20	15	72	
Mg	10	nil	10	15	20	10	
Na	45	9	-	15	-	100	
Ca	13	•	84	54	180	25	
so ₄	7	25	-	112	25	15	

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APPENDIX B

ESTIMATION OF CHROMATE DRAG-OUT

In order to determine the amount of hexavalent chromium which would be rinsed off an aircraft which had previously gone through the chromate rinse system, eight test panels, measuring 3 by 6 by 0.020 inches, which had been fabricated from aluminum alloy conforming to QQ-A-250/5, were utilized. Five of the test panels were painted with the various coatings commonly used on aircraft, and three were unpainted, in order to simulate the type surfaces encountered on aircraft.

The test panels were dipped into a 1.0% aqueous solution of potassium dichromate, withdrawn, and allowed to air dry. The dried panels were dipped into a container of 500 ml of deionized water where the residue of potassium dichromate remaining on the panels was washed off. This procedure was repeated five times, utilizing the same water to wash off the chromate residue.

Two series of tests were run. In the first set, the bottom edge of the panels was blotted to remove adhering droplets of water. This procedure was eliminated in the second set.

A series of 15 potassium dichromate solutions were accurately made whose concentration varied from .05 ppm to 100 ppm of potassium dichromate. These were then used as color standards for determining the concentration of potassium dichromate.

The average test results obtained were:

•	1st Run	2nd Run
Aluminum Alloy Panels	12 ppm	16 ppm
Painted Panels	13 ppm	33 ppm

Since the volume of wash water (500 ml), the concentration of potassium dichromate, and the surface area of the panels is known, the volume of the 1% potassium dichromate solution which adhered to the panels can now be calculated. Then knowing that the approximate surface area of a P3C aircraft is 10,000 square feet, the estimated volume of chromate rinse water which will adhere to it can also be calculated.

(.1322)

EIF

100 ml of 17 K2Cr207 1 gram of K2Cr207 (1 gallon) 3782 ml 10,000 ft for P3C aircraft Wo. of ft for test panels (500 grams of) 1,000,000 grams of wash water No. of grams of K2Cr207 (No. of mi of carryover) ı 1% K,Cr₂0, Carryover on test panels Volume in gallons of carryover on Volume in m l of P3C afreraft

letting X - PPM of $K_2 Gr_2^{0}$, and Y - ft² of test panels and combining the two equations gives:

(X) (500) (100) (10,000) (1) (1,000,000) (1) (Y) (3782) ı Volume in gallons of carryover on P3C aircraft The estimated volume of carryover for a P3C aircraft based on each of the four teats is as follows: Aluminum alloy panels (1st run) = 0.5 gallons

Aluminum alloy panels (2nd run) - 0.7 gallons (2nd run) - 0.9 gallons (lst run) - 0.4 gailons Painted panels Painted panels

The maximum amount of hexavalent chromate permitted is 0.05 ppm. This is equivalent to 0.141 ppm of potassium dichromate.

Based on an estimate that the carryover from a P3C aircraft will be approximately one gallon (since the highest estimated volume of carryover was 0.9 gallons), and that 1,000 gallons of fresh water will be used to rinse the aircraft, the concentration of the potassium dichromate in the used wash water will be:

5 ppm if 0.5% $K_2Cr_2O_7$ is used in the concentrated rinse 1 ppm if 0.1% $K_2Cr_2O_7$ is used in the concentrated rinse 0.5 ppm if 0.05% $K_2Cr_2O_7$ is used in the concentrated rinse

These values are all above the 0.141 ppm maximum allowable limit.

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APPENDIX C

TEST TO DETERMINE CHROMATE PROTECTION

The degree of protection provided by addition of a chromate inhibitor was determined by:

- (1) the reduction in galvanic current produced by it
- (2) the effect on a magnesium specimen
- (3) the effect on a 1020 steel specimen

The reduction in galvanic current was determined by measuring the current produced by a galvanic cell consisting of one electrode of 1020 steel and the other of magnesium with a microammeter. The electrodes measured 3" x 1/2" x 1/16" and were freshly abraded and cleaned. They were located parallel to each other, one inch apart, and immersed two inches in the test liquid. The reduction in galvanic current was determined by comparing the conductivity of a test water sample before and after the addition of an inhibitor.

The test electrodes were immersed in the test solution for 1/2 hour, afterwhich they were examined for visible corrosion.

The lowest concentration of an inhibitor for a given test water at which no corrosion occurs on either the steel or magnesium electrodes was considered the minimum concentration of potassium dichromate necessary to achieve adequate protection.

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APPENDIX D

EFFECTS OF CONTAMINANTS IN THE TEST WATER ON INHIBITING CAPACITY OF 0.3% POTASSIUM DICHEOMATE

Contaminant	Galvanic Current	Effect on Magnesium	Effect on Steel
JP-5 fuel	No change	No corrosion	No corrosion
Hydraulic Oil	No change	No corrosion	No corrosion
1010 011	Slight Reduction*	No corrosion	No corrosion
1100 011	Slight Reduction*	No corrosion	No corresion
CrCl ₃ (Trivalent Chromium) saturated solution	Slight Increase	No corrosion	No corrosion
H ₂ SO ₃ (0.05%)	Very Slight Increase	'No corrosion	No corrosion
C1 (NaC1) ** 0.05%	>100% increase	Scattered Pitting	Scattered Pitting (less than for the magnesium)

NOTES:

- * The actual value depended on the amount of oil coating the panel
- ** The chloride is in addition to the chloride in the test water

APPENDIX E

INHIBITOR NEEDED TO ASSURE ADEQUATE PROTECTION

% Inhibitor Ratio of Test Water to I				ionized Water		
(K ₂ Cr ₂ O ₇)	0:1	1:3	1:1	3:1	1:0	
1.0	N. C.	n. C.	N. C.	N. C.	N. C.	
0.5	N. C.	N. C.	M. C.	N. C.	N. C.	
0.3	N. C.	N. C.	N. C.	N. C	N. C.	
0.25	N. C.	N. C.	N. C.	Mg - 2 pits Steel OK	Mg - 2 pits Steel - 2 spots	
0.20	N. C.	n. C.	Mg - OK Steel - 2 spots		Mg - 2 pits Steel - 3 spots	
0.10	N. C.	N. C.	Mg - 2 pits Steel - 2 spots		Mg - 7 pits Steel 2 spots	
0.05	N. C.	Mg - 2 pits Steel - OK	·			
0.01	N. C.					
0.002	N. C.				***	
0.001	Staining on Mg interfac					

NOTES:

N. C. = No Corrosion

Visual observations made at 7% magnification

TABLE OF EQUIVALENTS FOR TEST WATER

		% K,Cr,07 needed for		
Ratio		<u> </u>	Salt (NaCl)	Adequate Protection
0:1	100%	72 ppm	119 ppm	0.3%
1:3	75%	54 ppm	89 ppm	0.3%
1:1	50%	36 ppm	59 ppm	0.25%
3:1	25%	18 ppm	30 ppm	0.10%
1:0	0%	0 ppm	O ppm	0.002%
			K-1	