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AFFPL-TR-66-168

INHIBITED N_2O_4
Third Quarterly Progress Report

Composition Research Unit
Chemistry Section
Research Division

Rocketdyne
A Division of North American Aviation, Inc.
Canoga Park, California

July 1966

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Air Force Rocket Propulsion Laboratory
Research and Technology Division
Edwards Air Force Base, California
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AFRPL-TR-66-166

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Air Force Rocket Propulsion Laboratory
Research and Technology Division
Edwards Air Force Base, California
Air Force Systems Command
United States Air Force

FOREWORD

The research reported herein was supported by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Edwards Air Force Base, California, Air Force Systems Command, United States Air Force, under Contract AF04(611)10809, Project Number 3148, BPSN 623148, Program Structure Number 750 G, with Ralph Fagnoli, 2nd/Lt/USAF, RPCL, serving as Project Monitor. This quarterly technical progress report was prepared under Rocketdyne G.O. 8728 in compliance with Part I, Paragraph B, and Line Item 6 of DD Form 1423, and IL-STD-847 (USAF).

The work described covers the period 1 April 1966 through 30 June 1966. The Responsible Scientist for this program is Dr. Hubert E. Dubb of the Analytical Chemistry Group, headed by Dr. B. L. Tuffly. The work was carried out by members of the Composition Research Unit supervised by Dr. V. H. Dayan, the Synthetic Chemistry Group headed by Dr. E. A. Lawton, and the Propellant Engineering Group, headed by Dr. J. J. Kalvinskas.

The following personnel were the principal contributors to the report: J. Gerhauser, A. D. Lev, H. H. Rogers, R. Rushworth, D. F. Sheehan, and R. D. Wilson.

This report has been assigned the Rocketdyne report No. R-6354-3

Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

GEORGE F. BABITS, Lt. Colonel, USAF
Chief, Propellant Division

ABSTRACT

This program is concerned with evaluating a new storable liquid oxidizer INTO, which is nitrogen tetroxide (NTO) inhibited with FNO_2 .

Corrosion tests of steel and aluminum alloys are being conducted in wet NTO, dry NTO, and dry NTO + HF. The tests are being conducted at ambient temperature for 30 days and for 20 months, and at 70 C for 30 days. The results of the 30-day ambient temperature test is reported herein. A definite passivation layer was noted both visually and by weight change on the aluminum samples exposed to INTO made from dry NTO.

Storability tests of INTO in stainless-steel, aluminum, and nickel containers at 70 C have now been in progress for 4 months with no apparent change in the composition of the propellants.

INTO has been prepared by bubbling F_2 through liquid-propellant-grade NTO at ambient temperature. The reaction proceeded smoothly and no difficulties are anticipated in scaling up the operation.

The conductivities of INTO prepared from wet and dry NTO have been measured. They were found to be 3.4 and 4.0×10^{-11} $\text{ohm}^{-1} \text{cm}^{-1}$. It is not anticipated that INTO will present more galvanic corrosion problems than does NTO.

Measurements of the vapor pressures and freezing points of INTO solutions have been initiated.

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INTRODUCTION

The use of nitrogen tetroxide (NTO), the most widely used storable liquid oxidizer in the United States, has been continually hampered by corrosion problems. Dry NTO is not a highly corrosive liquid when in contact with most common metals of construction, but moist NTO is extremely corrosive because of the formation of nitric and nitrous acids by the reaction of NTO with water.

It has previously been shown (Ref. 1 and 2) that the addition of a fluorine oxidizer to NTO leads to a reduction of the nitric and nitrous acid content of the propellant with the concurrent production of HF. It has also been shown (Ref. 2) that if the fluorine oxidizer is FNO_2 , the resulting oxidizer system is storable at 70 C in passivated aluminum, stainless-steel, and nickel containers.

The present program consists of an engineering evaluation of INTO, which is NTO containing 1 to 3 weight percent FNO_2 . INTO is being evaluated with respect to corrosion properties at ambient temperature and at 70 C, long-term (20 month) storability at the same temperatures, conductivity as it may relate to galvanic corrosion, vapor pressure and solubility of FNO_2 in NTO, and freezing points of INTO solutions. In addition, analytical chemical methods have been developed for the analysis of INTO and of NTO (Ref. 1 and 2). The results of the program to date are presented in the following sections.

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CORROSIVITY TESTS

SUMMARY

Corrosivity tests are being conducted on six ferrous alloys and five aluminum alloys (Table 1) in five different propellant compositions. To date the 30-day ambient tests have been completed and the 20-month ambient tests are in progress for the following compositions:

1. Dry NTO
2. Wet NTO
3. Dry NTO + FNO_2 (INTO)
4. Dry NTO + HF

The 30-day and 20-month tests with wet NTO + FNO_2 have been delayed because of difficulties with the vacuum line used for loading. Problems are currently being resolved, and loading should commence shortly. The 30-day high temperature test bombs are currently being assembled. Preliminary results of the study have been promising. NTO with FNO_2 apparently forms a white passivation layer on aluminum alloys. Control tests show that this layer is not caused by HF in solution. The 30-day, high-temperature and 20-month ambient-temperature tests will have to be completed before reliable conclusions can be drawn.

PROCEDURE

Evaluation of Tests

All corrosion tests are evaluated in the same manner to ensure a common basis of comparison. An overall observation of the specimens, including the taking of color photographs, is made immediately upon removal from the test bombs. The alloys which have been obviously affected by exposure to the oxidizer are noted. Weights of each specimen are then taken to determine the absolute and percent weight change. The specimens are then

TABLE 1

ALLOYS BEING SUBJECTED TO CORROSIVITY TESTING

Iron Alloys	Aluminum Alloys
304 Stainless Steel	6061-T6
316 Stainless Steel	7075-T73
321 Stainless Steel	Tens 50
440 C Stainless Steel	2014-T6
AM 350 SCT	2024
1018 Carbon Steel	Welded samples
Welded Samples	

photomicrographed at 15X magnification. Observations on the surface condition of the specimens are made by a comparison of the photomicrograph of the exposed specimen to a photomicrograph of a control specimen. Photomicrographs of two ferrous and two aluminum alloys with controls are presented in Fig. A-1 through A-9 of Appendix A. The unexposed specimens are presented photographically in Fig. A-10 and A-11.

Selected representative samples are then cross-sectioned, polished, and photomicrographed at high magnification to inspect for intergranular corrosion. No high magnification photomicrographs have yet been taken, because all tests at a given temperature must be concluded before the most representative samples can be selected.

Unloading of Test Bombs

Unloading procedures were set up to preserve the condition of the specimens as they were upon removal from the test bombs. The initial unloading procedure was used for the wet and dry NT0 tests with both aluminum and ferrous alloys. After dumping the liquid from the test bombs, the coupon strings were rapidly placed in a glove bag (portable plastic bag with attached gloves) through which a dry gaseous nitrogen purge was run. When the strings of coupons no longer showed signs of degassing NT0,

they were removed from the glove bag and given three rinses: methanol, 50-percent methanol/50-percent water, and water. The purpose of the rinses was to remove any residual nitric acid which would continue to corrode the specimens. The specimens were then removed from the strings, blotted dry, photographed, and weighed.

A second procedure was developed for the removal of the aluminum specimens from the dry INTO test bomb. Modifications were made to preserve the anticipated passivation layer. After dumping the liquid, a dry gaseous nitrogen purge was run through the bomb. The bomb with the specimen stringer inside was placed in the glove bag where the stringer was then removed. The specimens had a thin white passivation layer. One specimen was removed and weighed. It appeared to be losing weight. After approximately 1.5 hours exposure to the atmosphere, the layer disappeared. After 24 hours in the glove bag, the remaining samples showed significant passivation layer loss. It was apparent that a more inert atmosphere than that available in the glove bag was required for preserving the passivation layers.

All tests subsequent to the aluminum alloys in dry INTO were opened in a dry box through which a moisture free gaseous nitrogen purge is run. This will be the procedure in the future. The dry box is approximately 4 by 3 by 3 feet with an antechamber which enables hardware to be placed into and removed from the box while maintaining an inert atmosphere. A Mettler balance was placed in the box enabling the weighings to be made in an inert atmosphere. Except for the dumping of the liquid in the bombs and the photographing of the specimens, all work is performed in the dry box. An attempt will be made to preserve the passivation layers by sealing the specimens in Saran Wrap packets upon removal from the dry box. This unloading procedure has proved satisfactory with the dry NTO + HF test bombs and the ferrous specimens in the dry INTO test bomb.

TEST RESULTS

Wet and Dry NTO Tests

The ferrous and aluminum specimens exposed to wet and dry NTO were, in general, only very slightly corroded if affected at all. The only two alloys which were consistently affected by exposure were 1018 carbon steel and 440C stainless steel. Both were slightly discolored upon removal from solution (Fig. A-12 and A-13). The 1018 carbon steel showed increased rusting after the three rinses. Visual observation revealed that none of the aluminum alloys appeared affected (Fig. A-14 and A-15). A yellow powder was observed on the aluminum and ferrous alloys in both solutions. Samples of this powder were taken and were analyzed by emission spectrographic analysis. Results of these analyses are presented in Table 2. Because iron is a major constituent even in the aluminum test sample, it is believed that the source of the iron is not from the test bombs but from the container used for storing the NTO before loading. Several of the other elements are stainless-steel constituents. These could come from the test bombs or, more likely (because they are present in the aluminum test sample), from the pretest storage container.

Each specimen was weighed before and after testing. The weight changes presented in Tables A-1 through A-4 are averages based on four samples of each alloy (one welded and one nonwelded in the vapor and liquid phases). The only exceptions to this averaging of four samples are for the 2024 and 7075 aluminum alloys (one nonwelded sample in each phase) or where otherwise noted in the comments. Most of the weight changes in Tables A-1 and A-2 are not really significant relative to the probable errors incurred during handling and reweighing the specimens after the tests. The weight changes are valid to approximately ± 0.0002 gram. This represents approximately ± 0.0009 and ± 0.023 weight percent for the ferrous and aluminum alloys respectively. Of the 22 averages in Tables 1 and 2, 17 are within this range. There are 8 weight increases and 11 weight decreases which again show a random scatter in the data.

TABLE 2

EMISSION SPECTROGRAPHIC ANALYSES OF NTO TEST RESIDUES

Test	Major Constituents	Minor Constituents	Minor-Trace Constituents	Trace Constituents
Dry NTO, 30-Day Ambient, Ferrous Alloys	Fe	—	Cr, Al, Si, Mg, Ni, Mo, Cu, Ti, Na	Zr
Wet NTO, 30-Day Ambient, Ferrous Alloys	Fe, Cr	Ni, Cu	—	Mn, Ti, Mg, Zr, Al, Si
Wet NTO, 30-Day Ambient, Aluminum Alloys	Fe	Cr, Ni, Cu	Si	Mn, Mg, Zr, Al

Comments on the surface condition of the specimens made from comparison of photomicrographs of control and exposed specimens are presented in Tables A-5 through A-8. Samples of these photomicrographs are shown in Fig. A-1 through A-9. Because the interpretation of the photomicrographs is inherently subjective in nature, observations were made by two people independently, and the comments made are a compilation of these observations. Although observations were made on photomicrographs of two specimens in each phase, in Tables A-5 through A-8 the observations have been presented as vapor- and liquid-phase comments, because in almost all cases the welded and nonwelded samples exhibited the same effect in a given phase. Most of the comments in Tables A-5 and A-6 express minimal surface attack or no effect at all. These comments substantiate the insignificant corrosion (if any) reflected by the weight changes discussed above.

The corrosive effect of wet and dry NTO on aluminum and ferrous alloys is not strong enough to show up during a 30-day ambient test. It is anticipated that the effect will appear during the 30-day, high-temperature (70 C) tests and especially the 20-month ambient tests. The corrosivity of NTO should appear with certain alloys in the form of significant weight changes in a given direction. Photomicrographs should show more distinct surface attack.

Dry INTO Tests

The results of the dry INTO test with aluminum alloys are most promising in that an obvious white passivation layer was formed on all specimens in both phases. The ferrous specimens in dry INTO showed no such layer. The only observable effect with the ferrous specimens was a discoloring of 1018 carbon steel and 440 C stainless steel alloys in both phases (Fig. A-16). Although the results of the aluminum test are promising, the test will have to be repeated for two reasons: (1) infrared analysis showed that there was no FNO_2 present in the bomb when it was opened (it was later determined that this was caused by a leak in the vapor end of the bomb), (2) the passivation layers reacted while in the glove bag yielding no photomicrographs and questionable weight changes. When the test is repeated, the bomb will be opened in the dry box enabling weights to be taken in an inert atmosphere. The specimens will be enclosed in Saran Wrap packets when removed from the dry box, and photomicrographs will be taken through the protective packets. Samples of the passivation layer will be analyzed for both cations and anions.

All test bombs with dry INTO have shown a decrease in FNO_2 concentration in the liquid phase with time. The 30-day ferrous alloys test had an initial FNO_2 concentration (in the liquid phase) of 4.4 mole percent. This had dropped off to 1.6 percent after 3-1/2 weeks. The 30-day aluminum alloys test initially had a 6.6-percent FNO_2 concentration. This had dropped off to 0 after 4 weeks (because of a leak in the bomb). The 20-month aluminum alloys test started with 5.3 percent FNO_2 . After 3-1/2 weeks, the concentration was 0.9 percent, and after 4 weeks it had dropped off to 0. FNO_2 was recently added to this 20-month bomb to bring the concentration back up to 3.8 percent. The 20-month ferrous alloys test has shown a decrease from 5.3 to 1.3 percent over a 4-week period. The decrease in FNO_2 concentration is caused by the passivation of the samples and test bombs. It is anticipated during the 20-month tests that the FNO_2 concentration will be constant after complete passivation of the bomb and samples has occurred. This passivation phenomenon is consistent with the results obtained during the storability tests (Ref. 2).

Table A-3 presents the average weight changes for the dry INT0 tests. Although the aluminum weights were taken after some passivation layer loss had occurred (about 5 hours after unloading), it can be seen that the weight increases are still much greater than those of any other test completed. The weight changes of the ferrous specimens are not nearly as great as the aluminum specimens, but they are all positive and are all greater than the probable weighing error. These increases are probably caused by slight passivation layers as substantiated by the comments on the photomicrographs presented in Table A-7.

Dry NT0 + HF Tests

The ferrous and aluminum alloys exposed to dry NT0 + HF exhibited slight weight increases probably caused by the formation of very thin surface layers. The three alloys which showed obvious surface and weight changes are TENS-50 aluminum, 1018 carbon steel, and 440 C stainless steel Fig. A-17 and A-18 and Table A-4. The aluminum specimens did not exhibit an apparent passivation layer as did the specimens in dry INT0. Except for 304 C stainless steel, the ferrous specimens showed greater weight increases in NT0 + HF than in dry INT0. The comments on the aluminum specimens (Table A-8) when compared to the weight changes (Table A-4) seem to imply that the layers formed on the aluminum specimens are either very thin or transparent. It would seem that more affirmative observations of surface layers should go with the given weight increases.

CONCLUSIONS

The most significant result to date is that dry INT0 forms a relatively heavy passivation layer on aluminum alloys. The results of the dry NT0 + HF test support the fact that this layer is caused by FNO_2 in solution and not HF. None of the ferrous alloys exhibited obvious

passivation layers. Of the ferrous alloys tested, 1018 carbon steel and 440 C stainless steel were consistently most affected by exposure to the propellant compositions. Other tests must be completed before reliable conclusions on the corrosion inhibition of NTO by FNO_2 can be drawn.

STORABILITY TESTS

The storability of DNT0 at 70 C is being investigated in aluminum, stainless steel, and nickel bombs. The tests have been in progress since 1 March 1966. No change in FNO_2 content was detected, on 29 April 1966 or on 20 May 1966. Sampling is now being performed on a bimonthly basis. Analysis is by calibrated infrared spectrophotometry (Ref. 2).

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FIELD PREPARATION OF FNO_2

One of the most promising aspects of using FNO_2 as an additive in NTO has been the possibility of forming FNO_2 in the field by the reaction of F_2 with NTO. During the past quarter this possibility has been investigated on a bench scale with complete success. No major difficulties are anticipated in further scaling up the operation.

EXPERIMENTAL

A 1-liter passivated Hoke cylinder was loaded with 1120 grams (750 C) of liquid N_2O_4 . Provisions were made for the withdrawal of liquid samples and of samples of the gas above the liquid as well as measurements of pressure (Matheson SS gage 23538-1) and temperature (Tera-Tron thermocouple U-T2) changes (Fig. 1). The gaseous fluorine was stored in a 500-cc Hoke cylinder reservoir at 50 to 60 psig and was bubbled through the liquid N_2O_4 in small increments by means of the pressure differential. The details of the addition are summarized in Table 3.

Throughout the whole experiment the reaction vessel was agitated intermittently to insure thorough mixing and reaction. The only warming observed was when the F_2 was passed through rapidly during steps No. V, VI, and VIII. An overall 5 C temperature rise, corresponding to the change in ambient temperature during the experiment, was noted. The heating was only observed at the top of the reaction vessel above the liquid phase. The thermocouple did not show any temperature rise in the liquid phase. Infrared sampling indicated that some FNO was also formed, towards the end of the experiment. A total of approximately 17 grams of F_2 was passed through or into the liquid N_2O_4 . At the end of the addition infrared analysis of the liquid phase by expansion of a portion of the liquid into an infrared cell indicated the presence 2.9 mole percent FNO_2 .

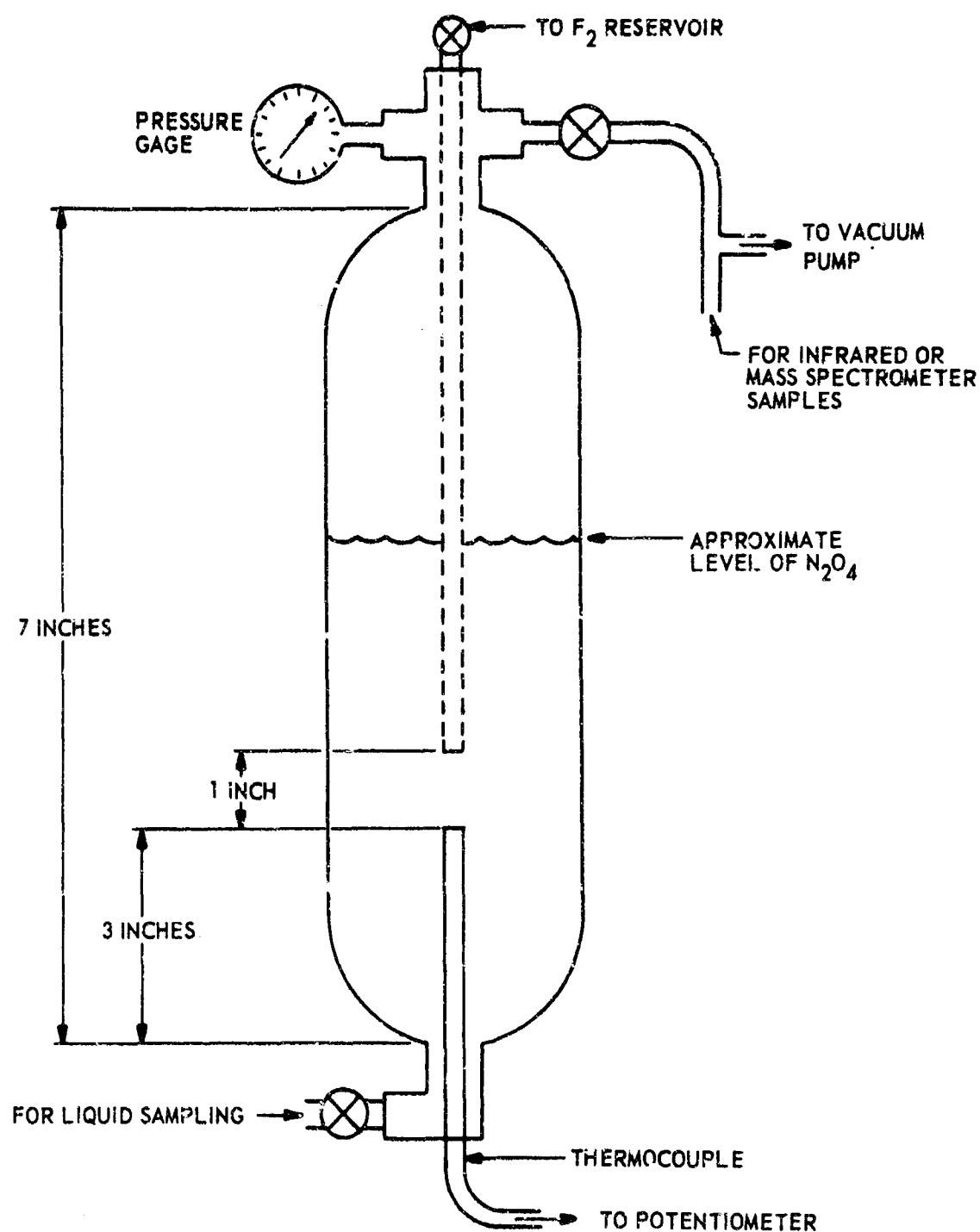


Figure 1. Field Preparation of INTO

TABLE 3

SUMMARY OF FLUORINE ADDITION DATA

Step Number	Number of Increments	Amount Added, cc	Time, hours	Gas-Phase Pressure, psig	Liquid Temperature Changes	Gas phase Products of N_2O_4
I	18	1200	3	20	None	None
II	11	1250	22	45	None	None
III	6	1100	1	45	None	$FN O_2$
IV	9	1800	24	56	None	$FN O_2$
V	*	1250	—	—	None; warm in gas-phase area	$FN O_2$
VI	*	650	—	—	None	
VII	1	750	—	—	None	
VIII	**	2100	—	—	None; warming in gas-phase area	$FN O_2$, $FN O$
Total		10,100				

*The fluorine reservoir was pressurized, then opened to the N_2O_4 reactor and the F_2 was sucked through the liquid by removal of the gases above the liquid.

**The above process was accomplished twice.

CONCLUSIONS

The experiment showed that approximately 8.5 of the 17 grams of fluorine added were used either to completely passivate the system and to react with a./ moisture in the N_2O_4 itself or were vented during the addition. The procedure adapted near the end of the experiment showed that a fairly rapid addition of fluorine to nitrogen tetroxide could be made without excessive heat resulting. Occasional venting may be desired to remove small amounts of oxygen formed from the reaction of fluorine or $FN O_2$ with any water present in the NTO .

GALVANIC CORROSION

SUMMARY

System compatibility is dependent on factors beyond the simple corrosive effects of the propellant on the individual materials of construction. One of the most important interactions is galvanic corrosion between dissimilar metals exposed to the propellant. The electrical conductivities of dry NTO (< 0.01 weight percent H_2O), wet NTO (> 0.10 weight percent H_2O), and of dry NTO + HF (0.3 weight percent) have been previously reported (Ref. 1 and 2). The conductivities of INTO made from dry and wet NTO are reported in the following paragraph. While there is no exact correlation between conductivity and galvanic corrosion, the data obtained here will help determine the necessity of later direct studies, because more conductive solutions have a larger tendency to exhibit this type of corrosion phenomenon.

EXPERIMENTAL

The conductivities of INTO made from wet (> 0.1 weight percent H_2O) and dry (< 0.1 weight percent H_2O) NTO have been measured. They were found to be 3.4 and $4.0 \times 10^{-11} \text{ ohm}^{-1} \text{ cm}^{-1}$, respectively.

CONCLUSIONS

Because of the low conductivities measured for the INTO solutions, it is not anticipated that galvanic corrosion will be more of a problem with INTO than it is with NTO.

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VAPOR PRESSURE AND FREEZING POINT

SUMMARY

Work has been initiated to determine the vapor pressures and freezing points of INTO solutions containing varying quantities of FNO_2 and to determine the vapor pressure of pure FNO_2 . The apparatus for this work has been previously described (Ref. 1). No final data are yet available because of problems encountered in obtaining complete passivation. The apparatus has been repassivated with FNO_2 at 30 psia and no further difficulties are anticipated.

EXPERIMENTAL

The technique of operation is as follows:

1. Load the bomb with an INTO solution
2. Measure the FNO_2 concentration in the liquid phase
3. Allow 3 hours for equilibration at each operating temperature
4. Measure the vapor pressure at each temperature
5. Cool the solution at a constant rate
6. Graphically record the measured internal temperature of the bomb against the rate of cooling
7. Determine the freezing point by observing the point of the discontinuous change in slope of the cooling curve
8. Redetermine the FNO_2 concentration

In attempts to carry out this procedure performed to date it has been found that the final and initial concentration measurements have not agreed because of the bombs undergoing additional passivation at elevated temperatures.

CONCLUSIONS

The vapor pressure and freezing point apparatus has worked as expected. No data are yet available because of the passivation problem described previously. A repassivation of the apparatus with 30 psia of FNO_2 is expected to solve this problem.

FUTURE EFFORT

The remainder of the corrosivity tests will be initiated. The 20-month storability tests will be continued. The corrosive effects of flowing INTO on valves will be studied. Attempts will be made to determine the efficacy of fluorine addition to wet NTO in reclaiming the out-of-specification propellant. The vapor pressure and freezing point studies will be completed.

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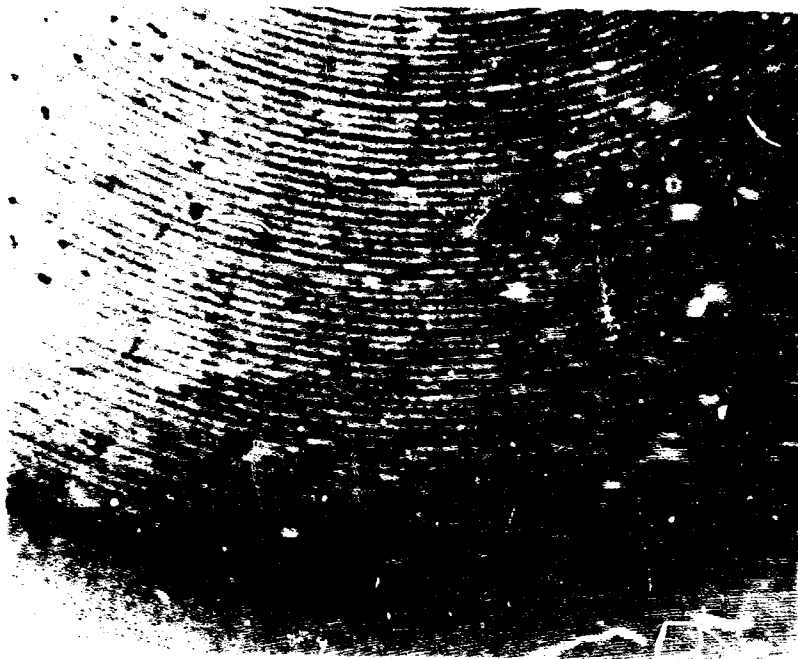
REFERENCES

1. R-6354-1, Inhibited N_2O_4 , First Quarterly Progress Report, Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, October 1965, CONFIDENTIAL.
2. R-6354-2, Inhibited N_2O_4 , Second Quarterly Progress Report, Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, April 1966, CONFIDENTIAL

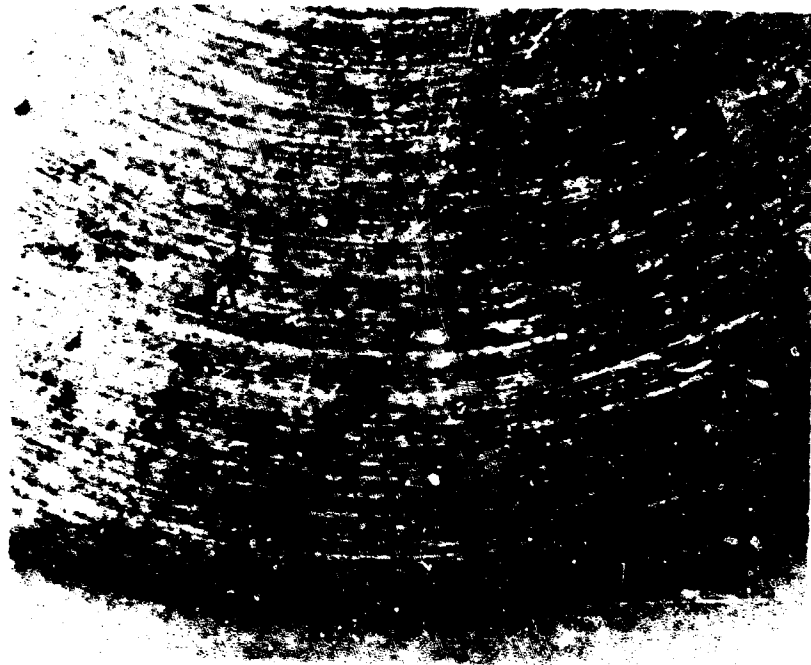
APPENDIX A

SUMMARY OF AMBIENT CORROSIVITY TESTS

This Appendix has been compiled to present a convenient summary of the data for the 30-day ambient corrosivity tests. Samples of photomicrographs of four selected alloys are presented in Fig. A-1 through A-4. Figures A-10 through A-18 show the samples as they were upon removal from the test bombs. The weight changes of the specimens are summarized in Tables A-1 through A-4. Comments on the surface condition of the exposed samples are presented in Tables A-5 through A-8.

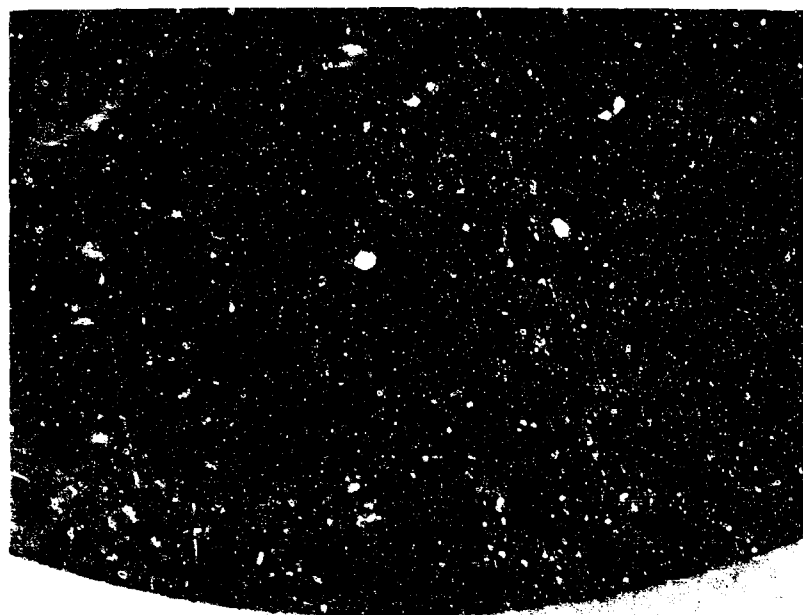


a. 440 Stainless Steel

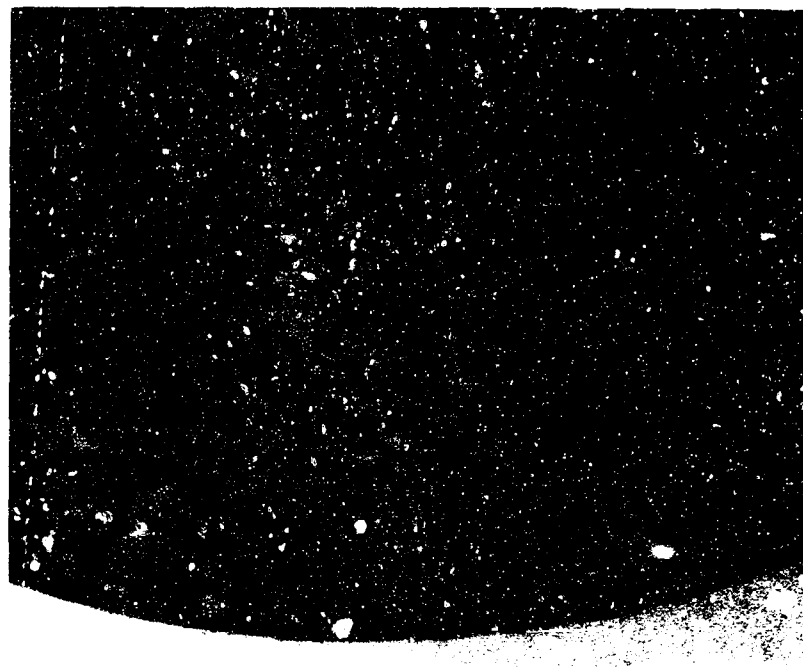


b. 1018 Carbon Steel

Figure A-1. Ferrous Control Specimens

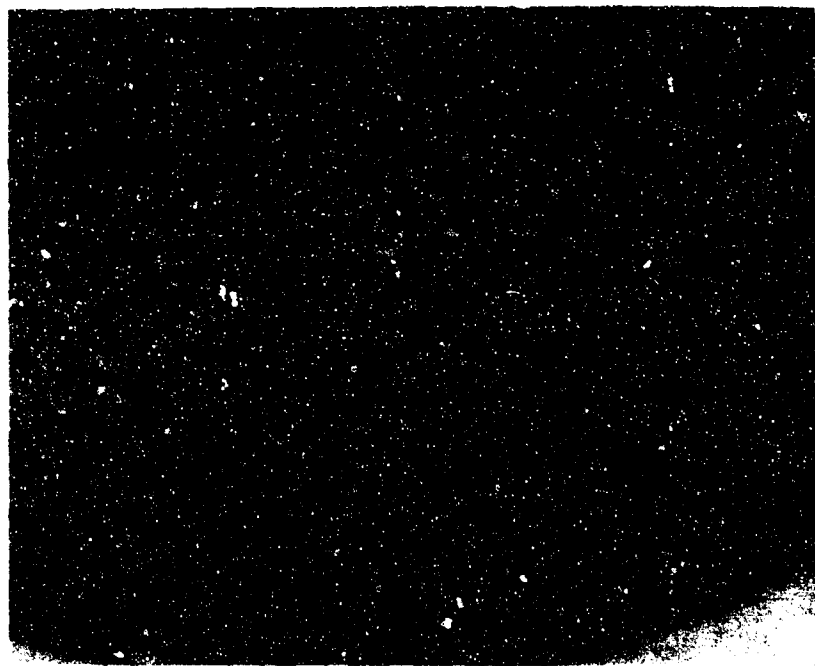


a. 440 Stainless Steel
(Vapor Phase)



b. 1018 Carbon Steel
(Liquid Phase)

Figure A-2. Ferrrous Specimens Exposed to Dry N₂O

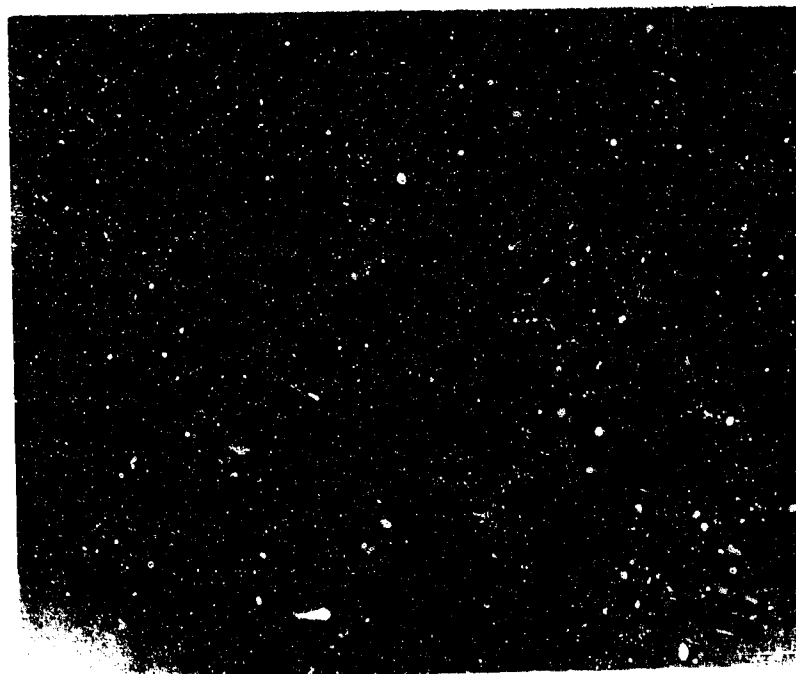


a. 440 Stainless Steel
(Liquid Phase)

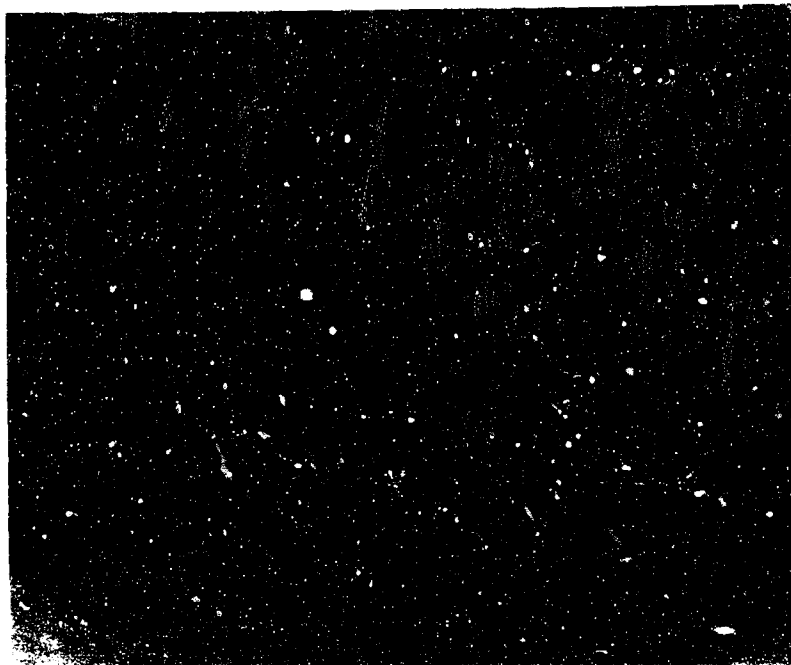


b. 1018 Carbon Steel
(Vapor Phase)

Figure A-3. Ferrous Specimens Exposed to Wet NTO



a. 440 Stainless Steel
(Vapor Phase)



b. 1018 Carbon Steel
(Liquid Phase)

Figure A-4. Ferrous Specimens Exposed to Dry NF_3 + FNO_2



a. 440 Stainless Steel
(Liquid Phase)

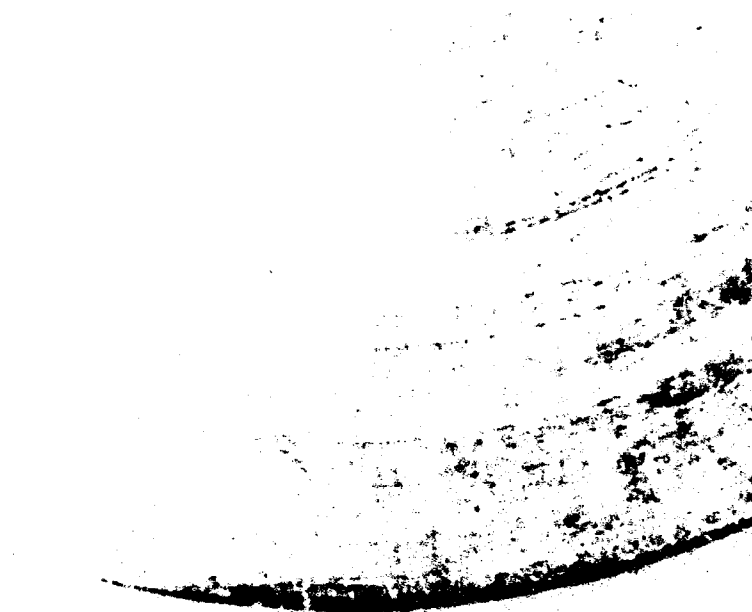


b. 1018 Carbon Steel
(Vapor Phase)

Figure A-5. Ferrous Specimens Exposed to Dry NTO + HF



b. TENS-50 Aluminum



a. 6061 Aluminum

Figure A-6. Aluminum Control Specimens



a. 6061 Aluminum
(Liquid Phase)

b. TENS-50 Aluminum
(Liquid Phase)

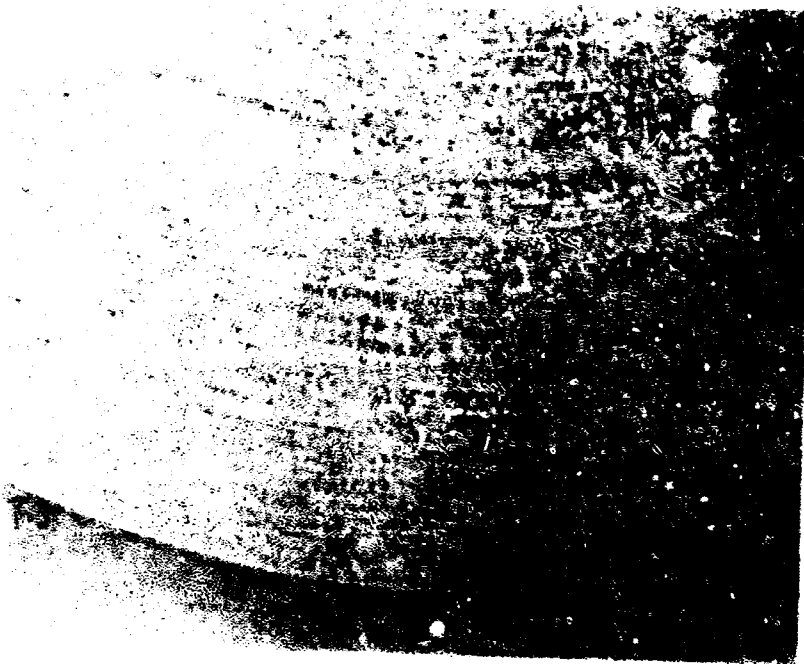
Figure A-7. Aluminum Specimens Exposed to Dry NTO



a. 6061 Aluminum
(Vapor Phase)

b. TENS-50 Aluminum
(Vapor Phase)

Figure A-8. Aluminum Specimens Exposed to Wet NTG



a. 6061 Aluminum
(Vapor Phase)



b. TENS-50 Aluminum
(Liquid Phase)

Figure A-9. Aluminum Specimens Exposed to Dry NiO + HF

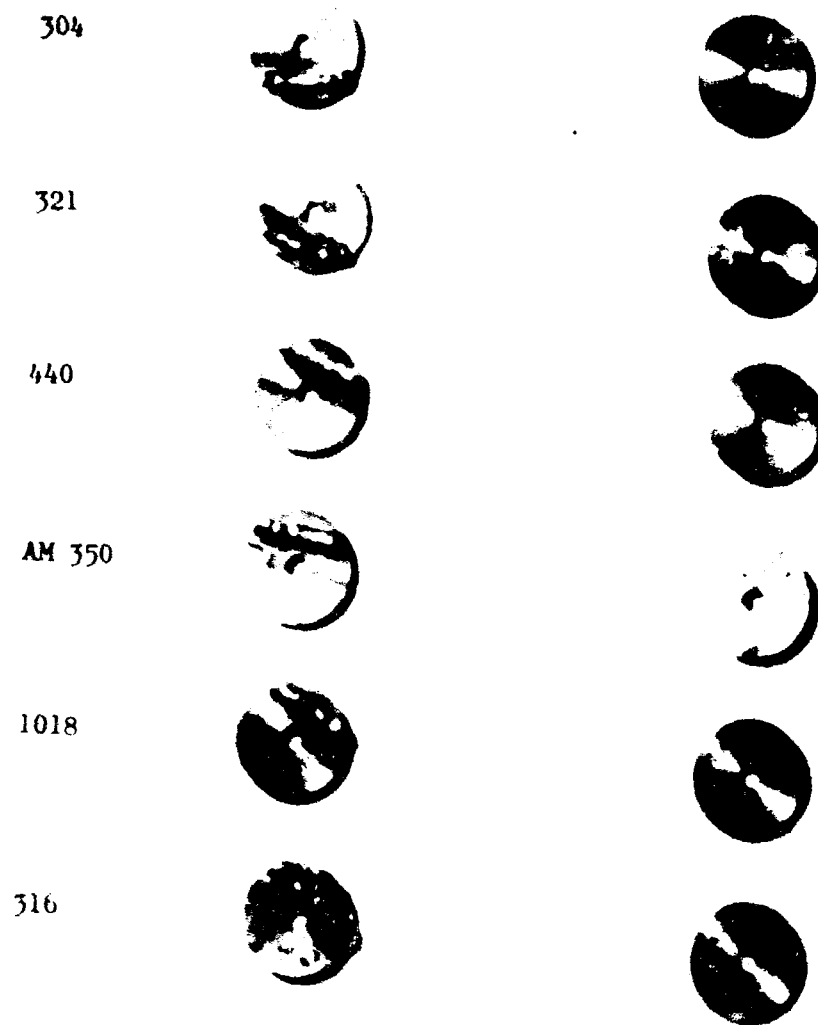


Figure A-10. Ferrrous Control Specimens

6061

7075 (Not Weldable)

TENS-50

2014

2024 (Not Weldable)

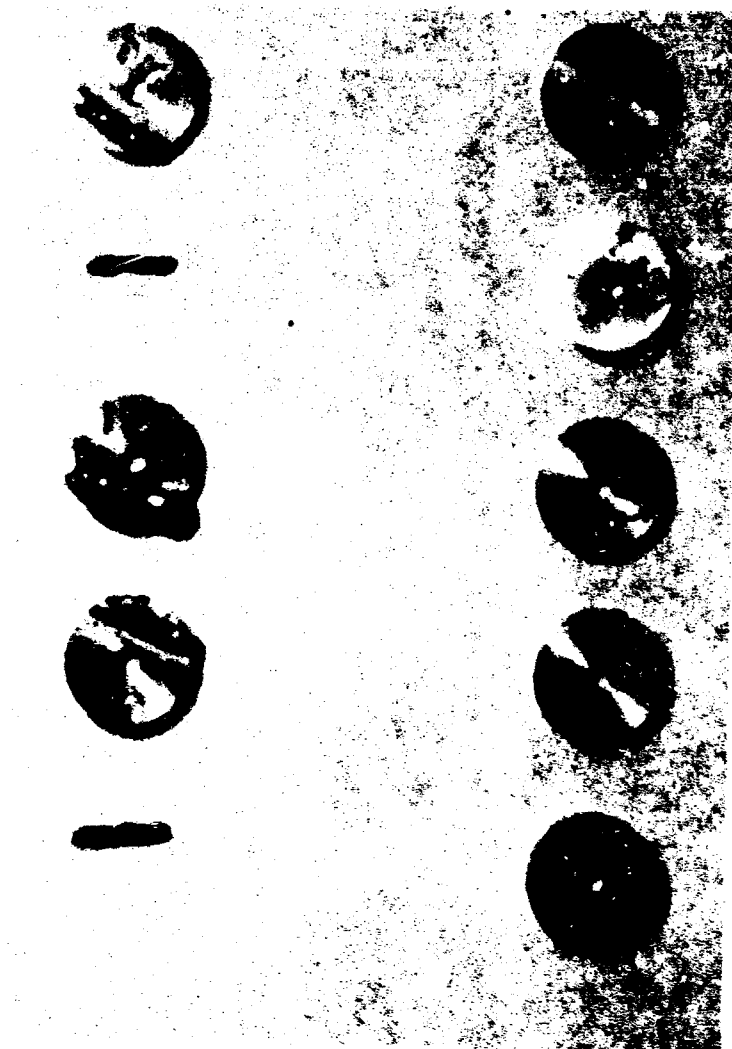


Figure A-11. Aluminum Control Specimens

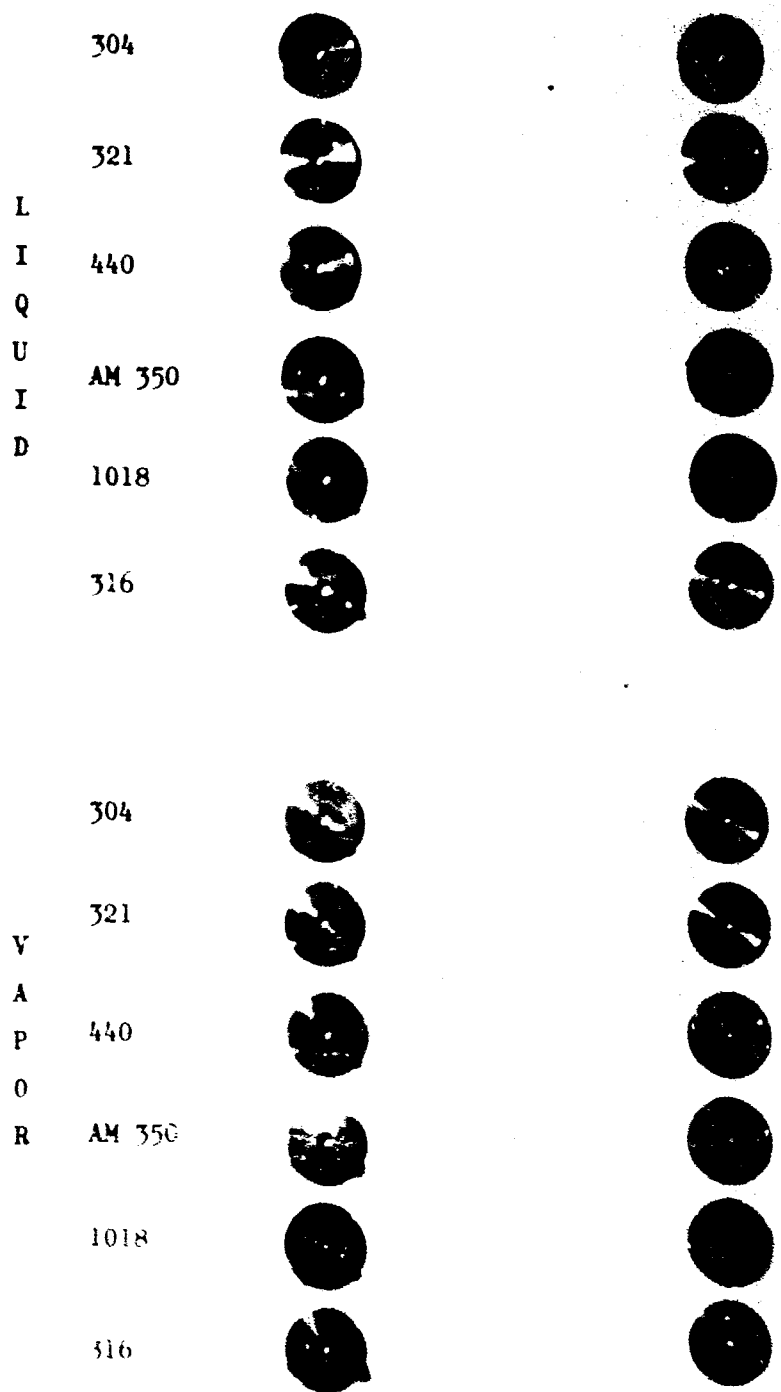


Figure A-12. Ferrous Specimens Exposed to Dry N₂O for 30 Days at Ambient Temperature

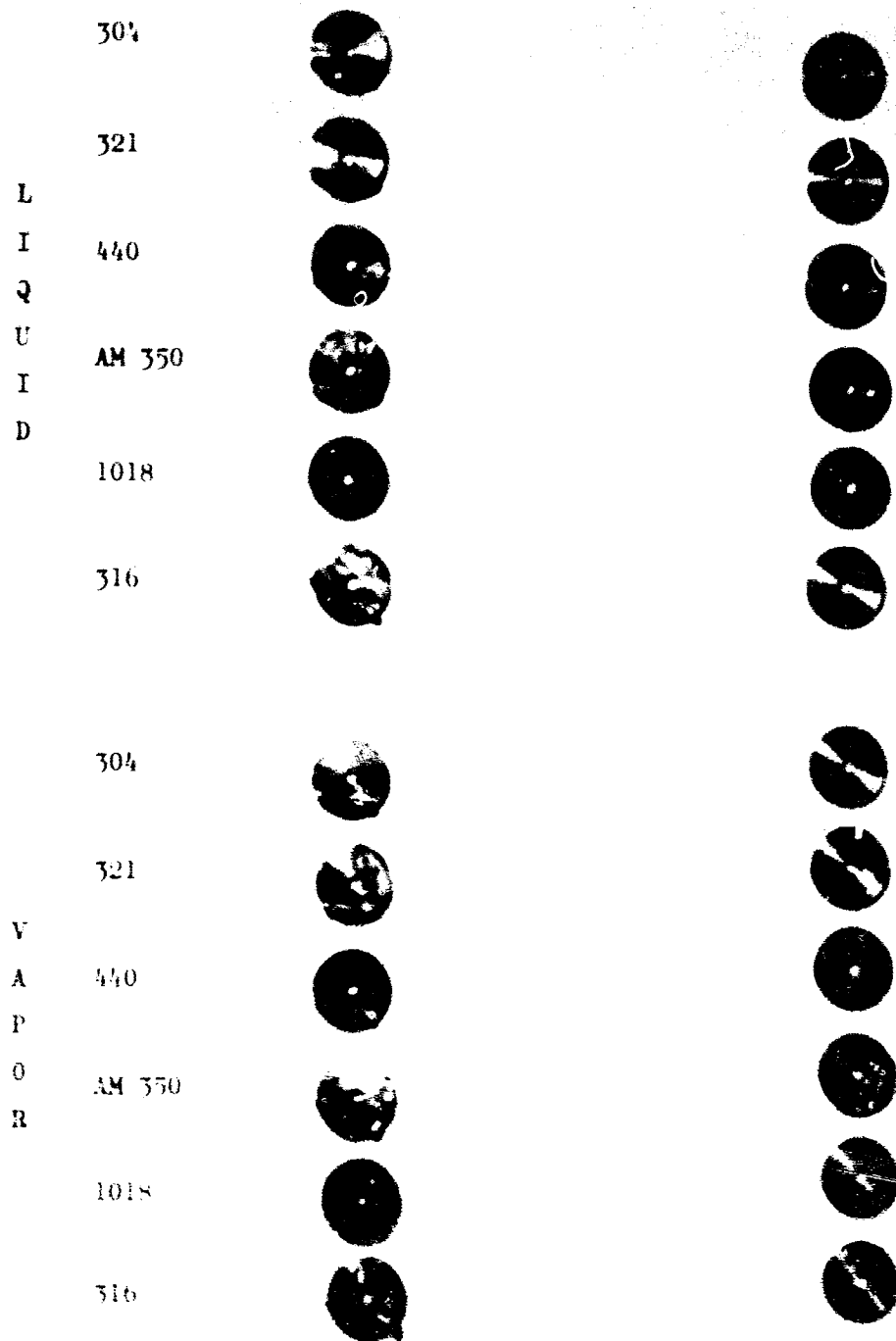


Figure A-13. Ferrrous Specimens Exposed to Wet NiO for 30 Days at Ambient Temperature

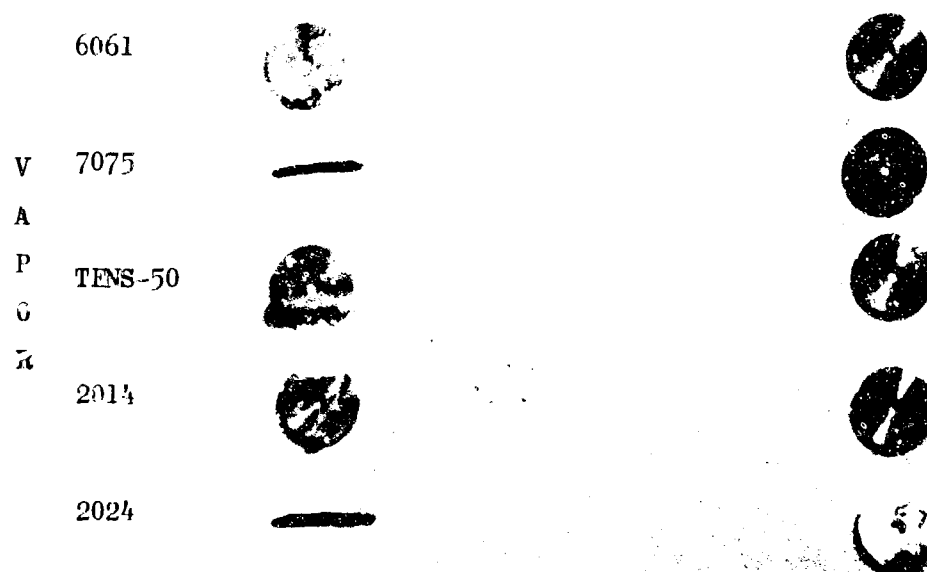
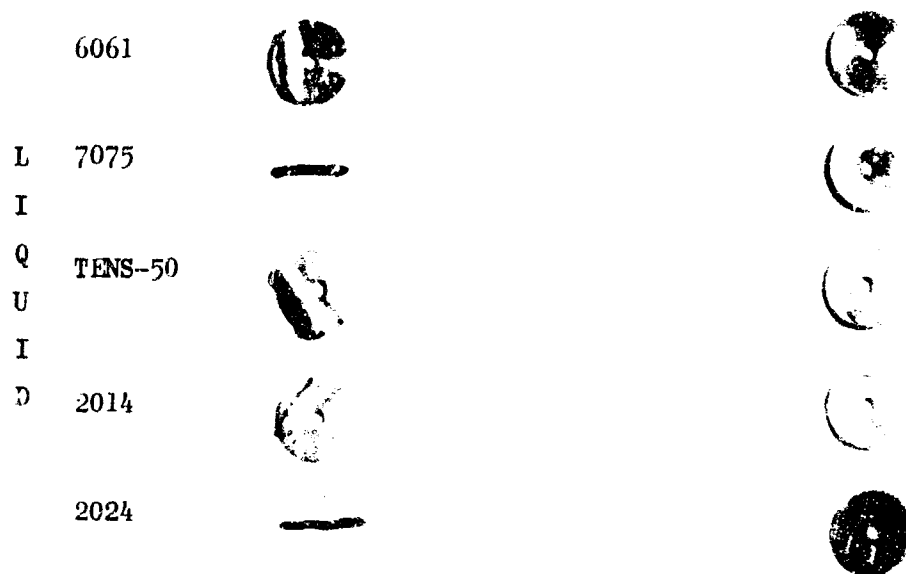


Figure A-14. Aluminum Specimens Exposed to Dry NTC for 30 Days at Ambient Temperature

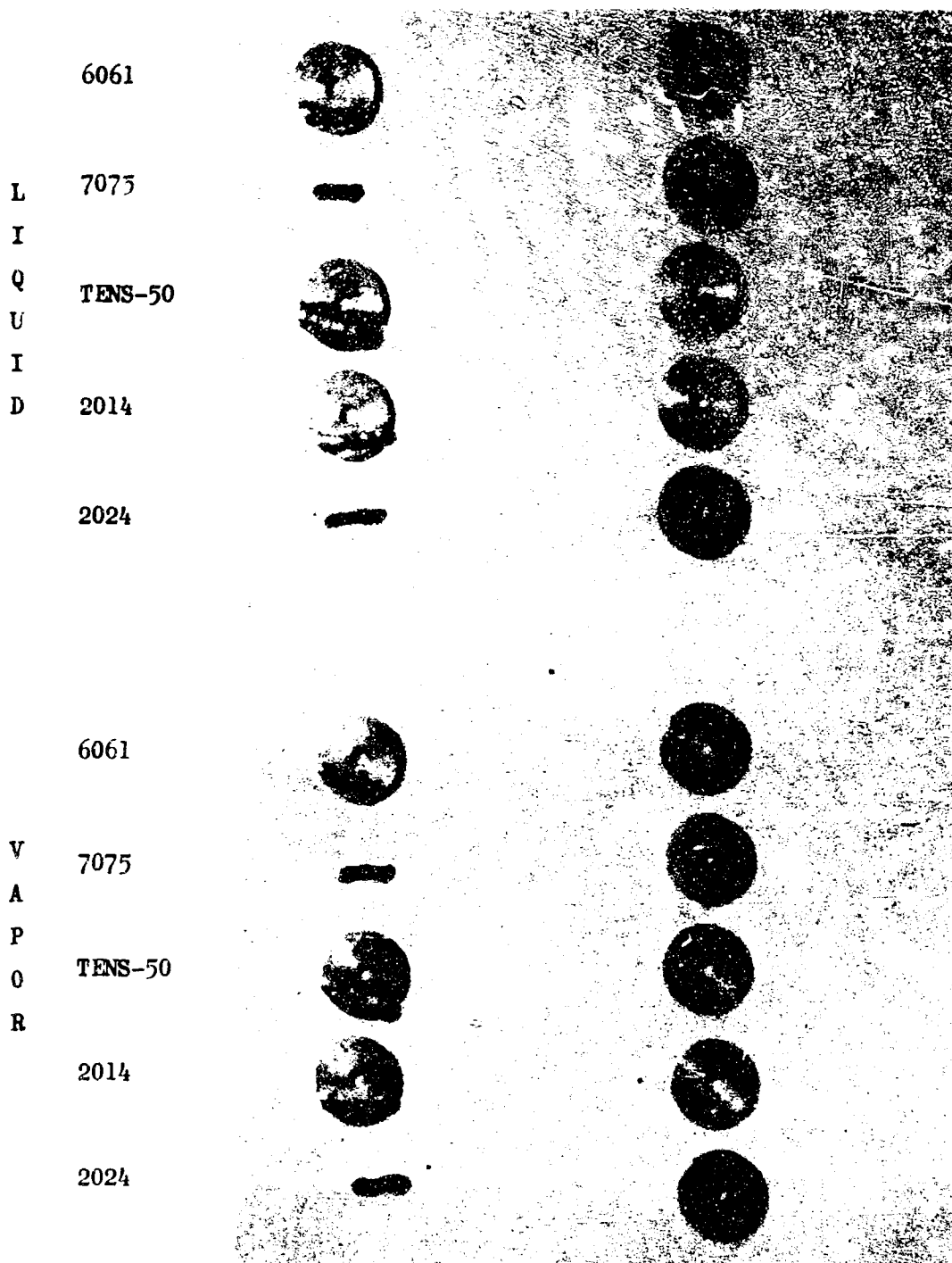
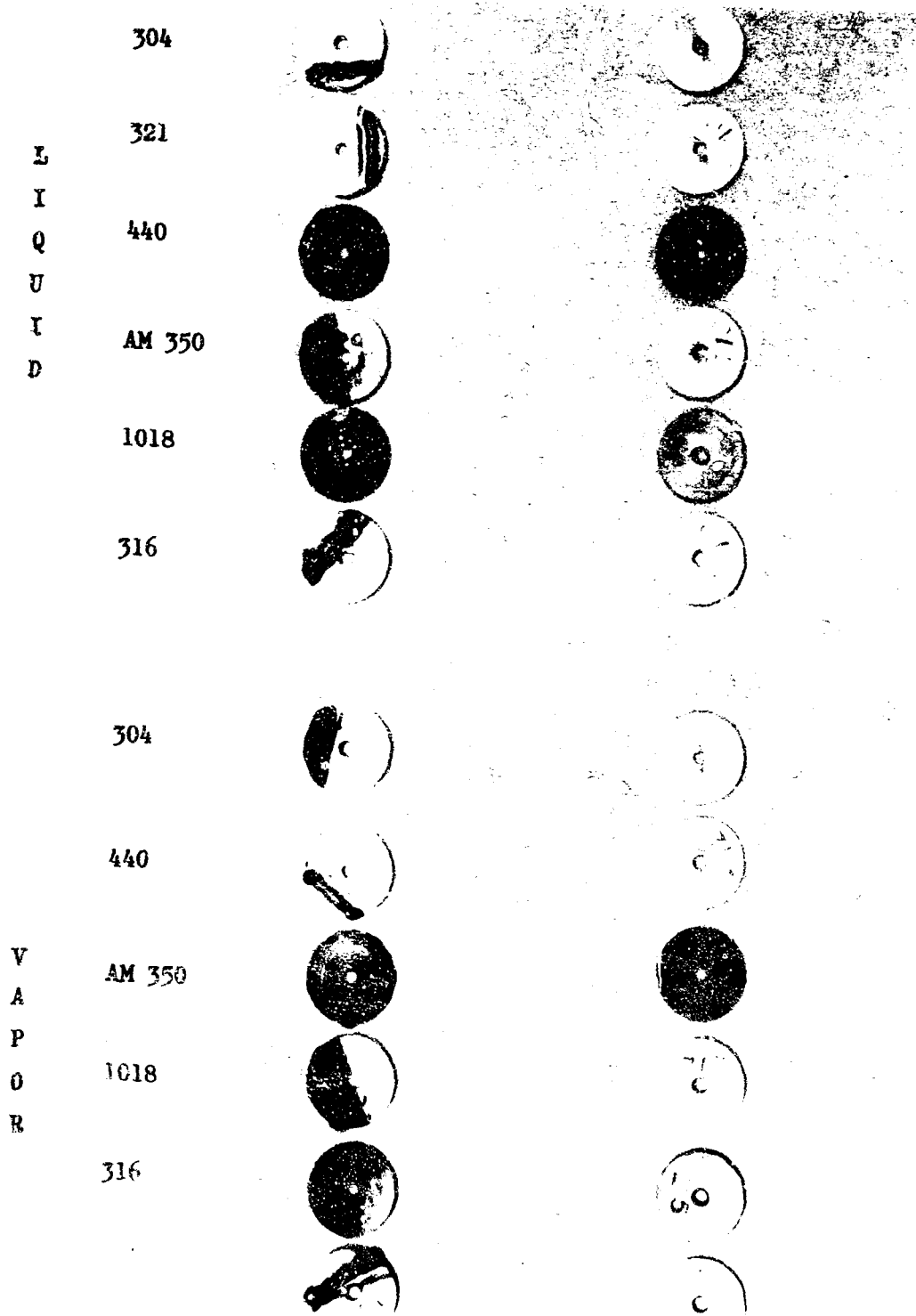


Figure A-15. Aluminum Specimens Exposed to Wet NT0 for 30 Days at Ambient Temperature



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Figure A-16. Ferrous Specimens Exposed to Dry NO + FNO_2 for 30 Days at Ambient Temperature

L
I
Q
U
I
D

304



321



440



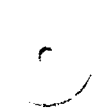
AM 350



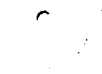
1018



316



304



321



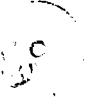
440



AM 350



1018



316



5AG26-6/1/66-S1*

Figure A-17. Ferrous Specimens Exposed to Dry N₂O + HF for 30 Days at Ambient Temperature

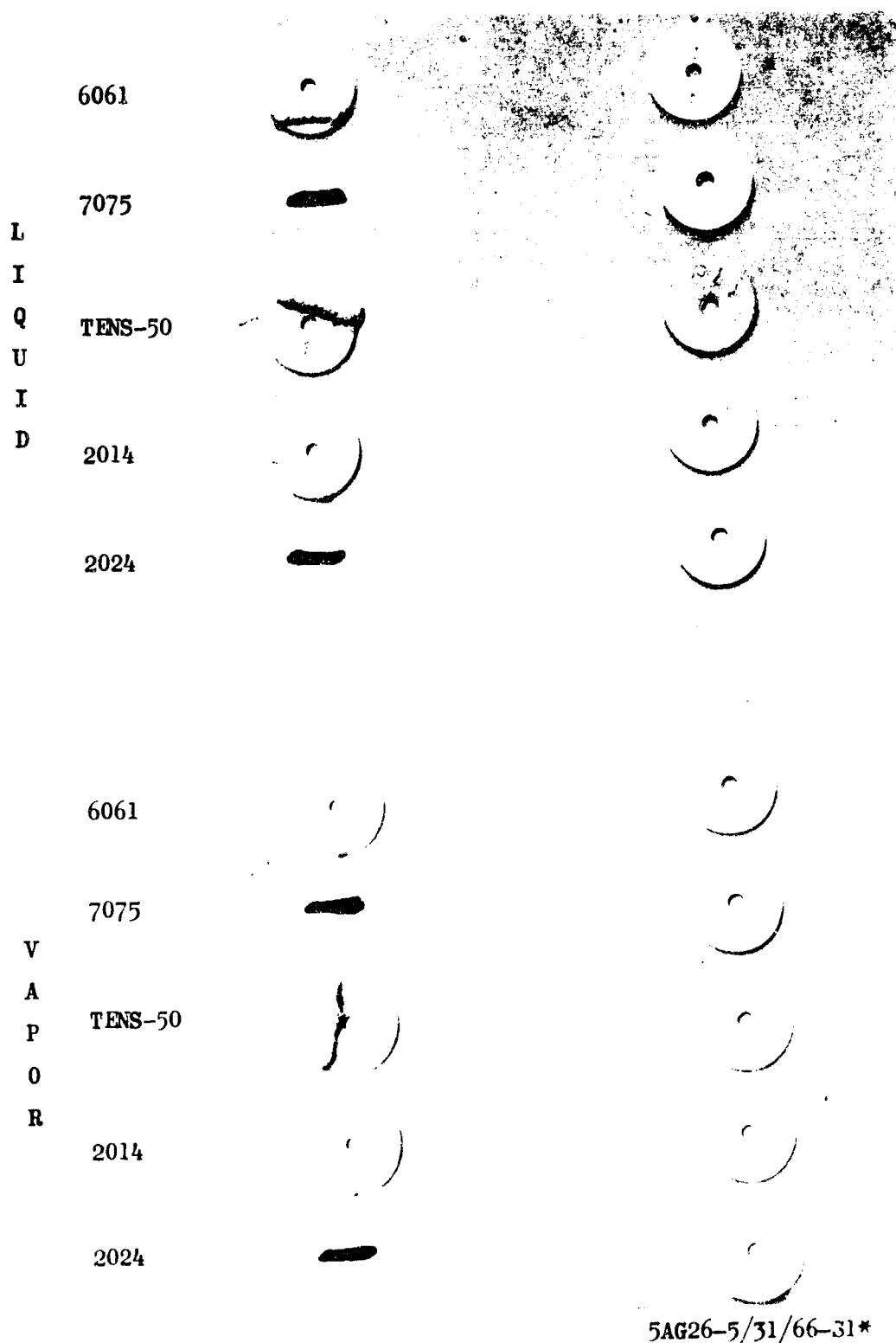


Figure A-18. Aluminum Specimens Exposed to Dry NTO + HF for 30 Days at Ambient Temperature

TABLE A-1

WEIGHT CHANGES OF SPECIMENS EXPOSED TO DRY N₂O
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Average Weight Change, grams	Average Percent Weight Change	Comments
Ferrous Specimens			
304 L	+0.0001	+0.006	Weight changes were approximately equal for all specimens.
316	+0.0002	-0.010	Welded specimens exhibited smaller weight decreases than nonwelded specimens.
321	+0.0001	+0.006	Liquid- and Vapor-phase welded specimens exhibited no weight changes.
AM 350	0	0	The liquid-phase nonwelded specimen showed an abnormal weight change and was discarded; vapor-phase nonwelded and liquid-phase welded specimen showed no weight change.
440	-0.0002	-0.009	The liquid-phase welded specimens showed no weight change. The vapor-phase welded specimen showed the smallest weight change.
1018	+0.0004	+0.011	The liquid-phase nonwelded specimen showed the smallest weight change.
Aluminum Specimens			
TENS-50	-0.0002	-0.025	None
2014	+0.0001	+0.024	The vapor-phase welded specimen showed a large negative weight gain whereas all others were positive. It was discarded in averaging.

TABLE A-1
(Concluded)

Alloy	Average Weight Change, grams	Average Percent Weight Change	Comments
2024	-0.0004	-0.055	The vapor-phase specimen showed three times the weight loss of liquid-phase specimen.
6061	-0.0005	-0.067	The vapor-phase welded specimen showed much larger weight decrease than others.
7075	+0.0004	+0.037	The vapor-phase specimen was discarded because it showed a large negative weight loss probably caused by dirt in a machining groove noted before the test.

TABLE A-2

WEIGHT CHANGES OF SPECIMENS EXPOSED TO WET NTO
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Average Weight Change, grams	Average Percent Weight Change	Comments
Ferrous Specimens			
304 L	+0.0002	+0.006	Weight changes were approximately equal for all specimens.
316	-0.0002	-0.010	Weight changes were approximately equal for all specimens.
321	-0.0002	-0.010	Weight changes were approximately equal for all specimens.
AM 350	-0.0001	-0.003	The liquid-phase welded specimen showed no weight change. The vapor phase-specimens showed a weight decrease. The liquid-phase nonwelded specimen showed a weight gain.
440	-0.006	-0.027	The vapor-phase weight losses were approximately four times those of the liquid phase.
1018	-0.0001	-0.006	The vapor-phase welded specimen was the only specimen to show a weight gain. The weight loss of the vapor-phase nonwelded specimen was four times that of liquid-phase specimens.
Aluminum Specimens			
TENS-50	+0.0001	+0.019	The vapor-phase welded specimen showed no weight change. The vapor-phase nonwelded specimen showed greater change than the others.

TABLE A-2
(Concluded)

Alloy	Average Weight Change, grams	Average Percent Weight Change	Comments
2014	0	0	Nonwelded specimens exhibited a slight weight increase. The vapor-phase welded specimen showed no weight change.
2024	-0.0002	-0.030	The vapor-phase specimen showed significant weight loss. The liquid-phase specimen showed no change.
6061	0	0	All samples except the liquid-phase welded specimen showed no weight change.
7075	+0.0002	+0.014	The liquid-phase specimen showed weight gain. The vapor-phase specimen showed a weight loss.

TABLE A-3

WEIGHT CHANGES OF SPECIMENS EXPOSED TO DRY $\text{N}_2\text{O} + \text{FeO}_2$
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Average Weight Change, grams	Average Percent Weight Change	Comments
Ferrous Specimens			
304 L	+0.0011	+0.046	The weight gain of the liquid-phase welded specimen was slightly less than the others.
316	+0.0005	+0.025	The liquid-phase welded gains were approximately 1.5 times those of the vapor phase.
321	+0.0006	+0.030	The vapor-phase nonwelded specimen exhibited approximately twice the weight gain of the other specimens.
AM 350	+0.0018	+0.061	The liquid-phase nonwelded specimen showed a smaller weight gain than other specimens.
440	+0.0021	+0.092	None
1018	+0.0020	+0.080	The welded specimens showed weight gains those of the nonwelded specimens.
Aluminum Specimens			
TENS-50	+0.0128	+1.726	None
2014	+0.0075	+1.068	Nonwelded specimens exhibited a slightly greater weight gain than welded specimens.
2024	+0.0056	+0.696	There was approximately the same weight gain for all specimens.
6061	+0.0243	+3.57	No weight was recorded for the liquid-phase welded sample; it was exposed to air and the passivation layer reacted.
7075	+0.0096	+0.892	None

TABLE A-4

WEIGHT CHANGES OF SPECIMENS EXPOSED TO DRY N₂O - HF
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Average Weight Change, grams	Average Percent Weight Change	Comments
Ferrous Specimens			
304 L	+0.0006	+0.029	Vapor-phase weight increases were greater than liquid-phase weight increases.
316	+0.0006	+0.025	Vapor-phase weight gains were twice those of liquid phase.
321	+0.0010	+0.044	Nonwelded specimens showed approximately twice the weight gain of welded specimens.
AM 350	+0.0023	+0.080	All specimens showed approximately the same weight increase. Welded specimens showed a slightly greater weight gain.
440	+0.0048	+0.216	All specimens showed approximately the same weight change.
1018	+0.0044	+0.191	The weight gain of the liquid-phase welded specimen was approximately one-third of the others.
Aluminum Specimens			
TENS-50	+0.0010	+0.125	All weight changes were approximately equal.
2014	+0.0009	+0.121	The vapor-phase welded specimen had a slightly greater weight gain than the others.
2024	+0.0004	+0.044	The liquid-phase specimen showed a significant weight gain. The vapor-phase specimen showed a slight weight.
6061	+0.0005	+0.070	Liquid-Phase specimens showed a slight weight change. Vapor-phase specimens showed a significant weight gain.
7075	+0.0012	+0.116	Both specimens showed approximately the same weight increase.

TABLE A-5

SURFACE CONDITION OF SPECIMENS EXPOSED TO DRY N₂O
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Phase	Comments
Ferrous Specimens		
304 L	Vapor	No effect
	Liquid	Crazing and very slight corrosion of machining grooves
316	Vapor	No effect
	Liquid	Very slight corrosion of machining grooves
321	Vapor	No effect
	Liquid	Very slight crazing and very slight corrosion of machining grooves
AM 350	Vapor	Slight surface attack
	Liquid	Very slight surface attack
440	Vapor	Pitting and apparent corrosion
	Liquid	Slight pitting and apparent machining groove corrosion
1018	Vapor	Bad pitting and scale formation
	Liquid	Bad pitting and scale formation
Aluminum Specimens		
7075-T6	Vapor	Very slight attack of machining groove
	Liquid	Very slight attack of machining grooves
2024	Vapor	No effect
	Liquid	Very slight surface attack
2025	Vapor	No effect
	Liquid	No effect
7050	Vapor	No effect
	Liquid	Slight pitting and corrosion
7073	Vapor	No effect
	Liquid	No effect

TABLE A-6

SURFACE CONDITION OF SPECIMENS EXPOSED TO WET NTO
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Phase	Comments
Ferrous Specimens		
304 L	Vapor	No effect
	Liquid	No effect
316	Vapor	No effect
	Liquid	No effect
321	Vapor	Very slight corrosion
	Liquid	Very slight corrosion and crazing
AM 350	Vapor	Very slight corrosion
	Liquid	Very slight corrosion
440	Vapor	Slight corrosion
	Liquid	Slight corrosion and spotting
1C18	Vapor	Slight corrosion of machining grooves
	Liquid	Slight corrosion of machining grooves
Aluminum Specimens		
TENS-50	Vapor	Pitting and slight corrosion of weld surface
	Liquid	Slight pitting and surface corrosion
2014	Vapor	Slight corrosion
	Liquid	Very slight corrosion
2024	Vapor	Very slight corrosion on edge
	Liquid	Very slight corrosion
6061	Vapor	Slight corrosion and slight pitting
	Liquid	Very slight corrosion and slight pitting or spotting
7075	Vapor	Pitting and slight corrosion
	Liquid	Very slight pitting and slight corrosion

TABLE A-7

SURFACE CONDITION OF SPECIMENS EXPOSED TO DRY $\text{N}_2\text{O} + \text{FNO}_2$
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Phase	Comments
Ferrous Specimens		
304 L	Vapor	Slight surface layer
	Liquid	Very slight surface layer
316	Vapor	No effect
	Liquid	Slight surface layer and spotting
321	Vapor	Slight surface layer and spotting
	Liquid	No effect
AM 350	Vapor	Very slight surface layer
	Liquid	Slight surface layer and spotting
440	Vapor	Surface layer and spotting
	Liquid	Apparent surface layer
1018	Vapor	Surface layer and spotting
	Liquid	Surface layer and spotting. Apparent residue on welded sample

NOTE: For aluminum specimens photographs were not taken because passivation layers reacted with moist atmosphere and disappeared. The test will be rerun.

TABLE A-8

SURFACE CONDITION OF SPECIMENS EXPOSED TO DRY NTO + HF
FOR 30 DAYS AT AMBIENT TEMPERATURE

Alloy	Phase	Comments
Ferrous Specimens		
304 L	Vapor	Slight surface layer
	Liquid	Spotted surface layer
316	Vapor	No effect
	Liquid	Spotting on slight surface layer
321	Vapor	Slight surface layer
	Liquid	Spotting on slight surface layer
AM 350	Vapor	Very slight spotting on slight surface layer
	Liquid	Very slight spotting on surface layer
440	Vapor	Spotted surface layer
	Liquid	Spotted surface layer; dense layer on nonwelded specimen
1018	Vapor	Apparent spotting on surface layer
	Liquid	Spotted surface layer
Aluminum Specimens		
TENS-50	Vapor	Spotted passivation layer
	Liquid	Spotted passivation layer
2014	Vapor	No effect
	Liquid	No effect
2024	Vapor	No effect
	Liquid	No effect
6061	Vapor	Apparent surface crazing
	Liquid	Slight passivation layer
7075	Vapor	Slight passivation layer
	Liquid	Passivation layer

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1 ORIGINATING ACTIVITY (Corporate author) Rocketdyne, a Division of North American Aviation, Inc., 6633 Canoga Avenue, Canoga Park, California		2a REPORT SECURITY CLASSIFICATION UNCLASSIFIED
		2b GROUP
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5 AUTHOR(S) (Last name, first name, initial) Dubb, H.; Gerhauser, J.; Lev, A. D.; Rogers, H. H.; Rushworth, R.; Sheehan, D. F.		
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8b PROJECT NO. 3148	9b OTHER REPORT NO(S) (Any other numbers that may be assigned this report) AFRPL-TB-66-166	
10 AVAILABILITY/LIMITATION NOTICES		
11 SUPPLEMENTARY NOTES	12 SPONSORING MILITARY ACTIVITY Air Force Rocket Propulsion Laboratory Research and Technology Division Edwards, California	
13 ABSTRACT This program is concerned with evaluating a new storable liquid oxidizer INTO, which is nitrogen tetroxide (NTO) inhibited with FNO_2. Corrosion tests of steel and aluminum alloys are being conducted in wet NTO, dry NTO, and dry NTO + HF. The tests are being conducted at ambient temperature for 30 days and for 20 months, and at 70 C for 30 days. The results of the 30-day ambient temperature test is reported herein. A definite passivation layer was noted both visually and by weight change on the aluminum samples exposed to INTO made from dry NTO. Storability tests of INTO in stainless-steel, aluminum, and nickel containers at 70 C have now been in progress for 4 months with no apparent change in the composition of the propellants. INTO has been prepared by bubbling F_2 through liquid-propellant-grade NTO at ambient temperature. The reaction proceeded smoothly and no difficulties are anticipated in scaling up the operation. The conductivities of INTO prepared from wet and dry NTO have been measured. They were found to be 3.4 and $4.0 \times 10^{-11} \text{ ohm}^{-1} \text{ cm}^{-1}$. It is not anticipated that INTO will present more galvanic corrosion problems than does NTO. Measurements of the vapor pressures and freezing points of INTO solutions have been initiated. (U)		

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14 KEY WORDS	LINK A		LINK B		LINK C	
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Nitrogen Tetroxide						
Fluorine						
Corrosion						
Storability						
Infrared Spectroscopy						

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