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RESEARCH ON INHIBITED N<sub>2</sub>O<sub>4</sub>

Second Quarterly Report

H. E. Dubb  
A. D. Lev

March 1967

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

TECHNICAL REPORT AFRPL-TR-67-85

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RESEARCH ON INHIBITED  $N_2O_4$

Second Quarterly Report

H. E. Dubb  
A. D. Lev

March 1967

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## 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 F04611-67-C-0008, with Ralph Fargnoli, 1st/Lt/USAF, RPCL, serving as Project Monitor.

The work described covers the period 1 December 1966 through 28 February 1967. The Responsible Scientist for this program is Dr. Hubert E. Dubb of Physical and Engineering Chemistry, which is managed by Dr. K. H. Mueller. The work was conducted by members of Physical Chemistry, supervised by Dr. A. E. Axworthy; Engineering Chemistry, supervised by Dr. W. Unterberg; Analytical Chemistry, supervised by Dr. V. H. Dayan; and the Materials and Processes section, managed by Mr. G. A. Fairbairn. The principal contributors to the program were Dr. Dubb, Mr. A. D. Lev, Mr. G. Brull, and Mr. J. Fisher.

This report has been assigned the Rocketdyne report No. R-6831-2.

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.

W. H. EBELKE, Colonel, USAF  
Chief, Propellant Division

## ABSTRACT

This program is concerned with extending the engineering evaluation of the new storable liquid oxidizer INTO which is NTO inhibited with 1 to 3 weight percent  $\text{FNO}_2$ .

Storability tests are being conducted on INTO at ambient temperature and at 70 C in aluminum, iron, and titanium tanks of 10- to 30-gallon volume.

Small-scale storability testing at 130 C for 24 hours has been completed. Small-scale storability tests (20 milliliters) in iron and titanium at ambient temperature and at 70 C are in their second month.

Titanium 6Al-4V specimens stressed to 90,000 psi and stored in INTO at ambient temperature and at 70 C will be observed for possible stress corrosion failures. These tests will start next quarter.

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## INTRODUCTION

The use of nitrogen tetroxide (NTO) 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 demonstrated under Contract AF04(611)-10809 (Ref. 1) that the addition of a fluorine oxidizer to NTO leads to a marked reduction of the nitric and nitrous acid content of the propellant with the concurrent production of HF. It has also been shown (Ref. 1) that if the fluorine oxidizer is  $\text{FNO}_2$ , the resulting oxidizer system is storable at 70 C in passivated aluminum, stainless-steel, and nickel containers.

The present program consists of an extended engineering evaluation of inhibited nitrogen tetroxide (INTO), which is NTO containing 1 to 3 weight percent  $\text{FNO}_2$ . INTO is being evaluated with respect to its storability at ambient temperature and at 70 C in large tanks of 10- to 30-gallon capacity (Task I), and at ambient temperature, 70 C, and 130 C in small bombs of 20-milliliter capacity (Task III). It is also being evaluated at ambient temperature and at 70 C with respect to its effect upon the stress corrosion problem encountered when NTO is stored under pressure in titanium 6Al-4V tanks (Task II).

## TASK I: LARGE-TANK STORAGE

### SUMMARY

The objectives of this task are to investigate the storability of inhibited NTC (INTO) in tanks of approximately 10-gallon capacity and to investigate the formation of  $\text{FNO}_2$  (the inhibiting agent) in the tanks in situ. To date, the two 18-gallon, 347 stainless-steel tanks have been loaded with wet NTO (0.22 weight percent water equivalent) and then inhibited with  $\text{FNO}_2$ . A 4.4 mole percent  $\text{FNO}_2$  concentration has been detected in one of the tanks. The other has yet to be sampled. The 10 remaining tanks (aluminum, steel, and titanium) are at the test facility and will be loaded shortly.

### EXPERIMENTAL

The two 347 stainless-steel tanks have been loaded employing the experimental procedures outlined previously (Ref. 2). The two tanks, No. 1-A and 2-T, were filled with 168 and 162 pounds of wet NTO, respectively. Fluorine was bubbled through the tanks at an average rate of 0.07 lb/hr. This slow rate ensures the formation of  $\text{FNO}_2$ , whereas rapid fluorine addition might result in the formation of some  $\text{FNO}$ . The reaction leading to  $\text{FNO}_2$  formation ( $\text{N}_2\text{O}_4 + \text{F}_2 \rightarrow 2\text{FNO}_2$ ) was well controlled in the loading operation with no pressure or temperature surges being detected.

Fluorine was injected into the tanks at pressures up to 100 psi instead of the anticipated 50 psi. This 100-psi loading pressure was necessary because of higher than expected tank ullage pressures. Tables 1 and 2 show data from the logbook for the loading of tank 1-A, and illustrate the ullage pressure history. For the  $\text{FNO}_2$  concentrations in question, the vapor pressure of the INTO should range between 15 and 25 psig (Ref. 1). As Table 1 shows, ullage pressures have been as high as 70 psig. Two reasons have been postulated for this high pressure: (1) an  $\text{FNO}_2$ -nitric

TABLE 1

## TANK 1-A DATA SHEET

Date, 1967	F <sub>2</sub> Loading Volume Pressure, psig	Tank Ullage Pressure, psig	Remarks
2-7	50	7.5	Tank ullage vented
	35	25	
	49	10	
	35.5	28.5	
2-8	58	5	Tank ullage vented
	44.5	40	
	65	15	
	55	50	
	78	21.5	
	65	60	
	85	30	
	73.5	65	
2-9	80	27.5	Ullage dropped 28 psig overnight (vented to 27.5)
	73.5	50	
2-13	72	0	Tank ullage vented
	50	45	
2-14	79	11	Ullage dropped 18 psig overnight (vented to 11)
	60	54	
2-15	84	14	Ullage dropped 15 psig overnight (vented to 14)
	61	57.5	
2-16	84	20	Ullage dropped 12.5 psig overnight (vented to 20) Tank ullage vented
	67	63	
	84	20	
	70	66	
2-17	90	18	Ullage dropped 15 psig overnight (vented to 18)
	72	70	
2-20	95	19	Ullage dropped 20 psig over week-end (vented to 19)
	70	67	
2-21	--	61	Ullage vented and tank stored
	--	36	

TABLE 2

## TANK 1-A FLUORINE MASS BALANCE

	<u>Weight,</u> <u>pounds</u>
Wet NTO Loaded	168
Total H <sub>2</sub> O Equivalent in NTO	0.37
F <sub>2</sub> Required to React H <sub>2</sub> O	0.78
FNO <sub>2</sub> Concentration (4.4 mole percent)	
F <sub>2</sub> as FNO <sub>2</sub>	1.52
F <sub>2</sub> Reacted	0.78 + 1.52 = 2.30
F <sub>2</sub> Added	2.65
F <sub>2</sub> Balance (vented or passivated out)	0.35

and nitrous acid reaction resulting in gaseous oxygen evolution and (2) a slow rate of dissolution of  $\text{FNO}_2$  into the bulk liquid.

The mass balance in Table 2 shows that the formation of  $\text{FNO}_2$  is nearly quantitative. Only one-seventh of the fluorine added contributes to venting losses and tank passivation. Additional passivation will probably take place during storage. The remarks in Table 1 show that the tank ullage pressure decreases 12.5 to 28 ps overnight. Temperature drop overnight could account for no more than 5 psi of this decrease. It was noted during the loading of tank 2-T that the higher the ullage pressure, the greater the pressure drop overnight. It appears that equilibrium of  $\text{FNO}_2$  between the vapor phase and the solution is being established slowly, thus the second postulate of slow dissolution of  $\text{FNO}_2$  is favored.

#### CONCLUSIONS AND FUTURE EFFORT

The tank loading operation will be expedited by system and procedure modifications. An attempt will be made to increase the dissolution rate by perforating all tank diaphragms. This will decrease  $\text{F}_2$  bubble size, which will increase the  $\text{F}_2$ -NTO interface area. This increased interface area should enhance the rates of both the  $\text{FNO}_2$  formation and dissolution.

Rocketdyne experience thus far has indicated that there is little hazard involved in the  $\text{FNO}_2$  formation reaction. The system will be modified so that fluorine can be bubbled through all eight of the 10-gallon tanks simultaneously. NTO will be transferred to the tanks individually, but the tanks will then be set up behind the blastproof wall and loaded exactly as described in Ref. 2. The only differences will be that the  $\text{F}_2$  fill line will be manifolded to the eight tank loading valves and the vent line will be manifolded to the eight tank vent valves. The 30-gallon titanium tanks will be loaded individually. The tank sampling procedure will not be modified. All of the remaining tanks will be loaded during the next quarter.

## TASK II: TITANIUM STRESS CORROSION

### SUMMARY

The titanium stress corrosion frames and test specimens have been machined, cleaned, and prepared for testing. The determination of the elastic modulus of the material used to make the test specimens has been completed. Three control samples have been loaded into stress frames at 90,000 psi and are presently being stored in a desiccator. The titanium 6Al-4V tanks in which the tests are to be conducted are scheduled for delivery on 15 April 1967. Testing will commence as soon as the tanks are received and cleaned.

### EXPERIMENTAL

The construction of the stressing frames and 0.125-inch-diameter tensile specimens for the stress corrosion tests has been completed. Design drawings for these components were given in the first quarterly report of this program (Ref. 2).

The value of the elastic modulus ( $\epsilon$ ) was obtained from the stress ( $\sigma$ ) - strain ( $E$ ) curves of control specimens ( $\sigma = \epsilon E$ ). The stress on the test specimens is established by determining the imposed strain. The same extensometer used for measuring the elastic modulus is being used for stressing the test specimens. This technique eliminates errors caused by variances in the calibration of extensometers and recording equipment. All materials used in the stressing frames as well as the test specimens were solution treated and aged (1750 F, 1 hour, water quenched; and 1000 F, 8 hours, air cooled) to a minimum ultimate tensile strength of 160,000 psi and yield strength of 150,000 psi. Three control specimens have been loaded into stress frames at 90,000 psi in this manner and are now being stored in a desiccator. Pressurization tanks in which the stress corrosion specimens will be stored in contact with INT0 are on order, and delivery is scheduled for 15 April 1967.

A schematic of the tanks was published previously (Ref. 2). The only change from the schematic has been to decrease the cylindrical diameter of the tank from 9 to 8-1/2 inches and to increase tank wall thickness from 0.040 to 0.060 inch. There will still be sufficient space to load the test specimens in the smaller diameter tanks, and the increased wall thickness will provide a greater safety factor. The tanks are being fabricated by tungsten/inert gas welding of Ti-6Al-4V in the solution-treated condition. After welding, they will be aged 6 to 8 hours at 1000 F. The aging will serve the dual purpose of strengthening the parent metal and of stress relieving the weldments. The tanks will be thoroughly cleaned after aging to remove blue oxide interference film from their inner surfaces.

#### CONCLUSIONS AND FUTURE EFFORT

The specimens will be stored in INTO at ambient temperature and at 70 C for periods of 60 days. They will be examined by X-ray radiography twice each week for the first 4 weeks and once weekly thereafter. The rate of failure of the samples in INTO will be compared to the rate of failure of similar specimens exposed to NTO where such data are available in the literature. If failures occur, metallographic, fractographic, electron probe, and chemical analysis of the fractured specimens will be performed in an attempt to determine the mechanism of the failures. Storage of the stressed Ti-6Al-4V specimens in INTO will begin as soon as the pressurization tanks are received.



### TASK III: SMALL-BOMB STORABILITY

#### SUMMARY

Small-bomb storability tests are being conducted with INTO prepared from dry NTO (< 0.10 weight percent H<sub>2</sub>O) and of INTO prepared from wet NTO (> 0.10 weight percent H<sub>2</sub>O). The bombs are fabricated of three materials: (1) titanium 6Al-4V, (2) type 250 maraging steel, and (3) type A 286 steel welded with Hastelloy-W rod. They have an internal volume of 20 milliliters. Storage tests for 24 hours at 130 C have been completed. FNO<sub>2</sub> was found in the INTO at the completion of the tests after the bombs had been sufficiently passivated. The titanium bombs required considerably more passivation than the steel bombs. Less FNO<sub>2</sub> was detected in them even after extensive passivation. Twelve-month tests are now in progress at ambient temperature and at 70 C.

#### Experimental

The bombs were loaded according to the procedure described in the first quarterly progress report of this program (Ref. 2). Analysis for FNO<sub>2</sub> was carried out by expanding a sample of the liquid phase of the INTO in the bombs to 200 mm Hg pressure in an infrared cell. The concentration of FNO<sub>2</sub> was then calculated from the absorbance at 12.18 microns (NF stretch; Ref. 1). All bombs were passivated for 96 hours at 70 C with a 30 mole percent solution of FNO<sub>2</sub> in NTO prior to the start of the tests. The results of the 130 C tests are summarized in Table 3.

It may be seen from Table 3 that the FNO<sub>2</sub> contents of the bombs made of 250 maraging steel and of A286 steel welded with Hastelloy W were reduced only approximately 25 to 30 percent during the 24-hour test. However, the FNO<sub>2</sub> was completely consumed in the titanium 6Al-4V bombs during the first 24 hours at 130 C. After the addition of more FNO<sub>2</sub> (equivalent to more extensive passivation), the bombs lost 50 to 75 percent of their FNO<sub>2</sub> in 24 hours at 130 C.

TABLE 3

## RESULTS OF 24-HOUR STORAGE TESTS AT 130 C

Bomb Material	Total Water Content, w/o	FNO <sub>2</sub> Content (m/o), hours			
		0	24	48	76
250 Maraging Steel	0.08*	20.0*	14.0		
	0.2**	12.5**	10.0		
A 286 Steel Welded With Hastello <sup>W</sup> W	0.08	20.0	14.0		
	0.2	12.5	9.4		
Titanium 6Al-4V	0.08	20.0	0(15.6)***	7.5	3.8
	0.2	12.5	0(8.8)***	3.0(8.3)***	1.9

\*Initial water content of the NTO was 0.08 w/o; F<sub>2</sub> was added until FNO<sub>2</sub> reached 20.0 m/o.

\*\*An additional 0.12 w/o water was then added to part of the INTO resulting in an initial FNO<sub>2</sub> content of 12.5 m/o for the "wet" NTO samples.

\*\*\*FNO<sub>2</sub> was added to bring FNO<sub>2</sub> content to that shown in parentheses.

The 12-month ambient temperature and 70 C storage tests were started. The bombs were loaded with the same solutions of INTO as in the 130 C tests (Table 3). The bombs will be sampled periodically during the remaining 10 months of these tests.

#### CONCLUSIONS AND FUTURE EFFORT

The 130 C tests indicate that titanium 6Al-4V either requires considerably more passivation with INTO than the steel alloys or that INTO is not storable in titanium for extended periods of time. The results of the 12-month ambient temperature and 70 C storability tests should clarify the situation.

## SUMMARY

The tanks for the Task I large-tank storage tests are all in house and loading has commenced. The two 347 stainless-steel tanks have been loaded. A manifold system has been constructed which will allow the simultaneous filling of 8 of the remaining 10 tanks.

All of the equipment for the Task II titanium stress corrosion study has been constructed with the exception of the tanks in which the stressed specimens are to be stored. The tanks are scheduled to be delivered by 15 April 1967. The elastic modulus of the titanium stock which is to be tested has been determined.

The Task II 24-hour, 130 C, small-scale storage tests have been completed. The 12-month ambient temperature and 70 C tests are now in progress. The 130 C tests indicated that titanium 6Al-4V requires considerably more passivation to INTO than do 250 maraging steel and A286 steel welded with Hastelloy W.

#### REFERENCES

1. AFRPL-TR-66-320, Final Report, Inhibited N<sub>2</sub>O<sub>4</sub>, Contract No. AF04(611)-10809, Rocketdyne, a Division of North American Aviation; Inc., Canoga Park, California, January 1967.
2. AFRPL-TR-66-347, First Quarterly Report, Research on Inhibited N<sub>2</sub>O<sub>4</sub>, Contract No. F04611-67-C-0008, Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, January 1967.

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