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(Unclassified Title)

ADDENDUM TO FINAL REPORT, PREPARATION AND CHARACTERIZATION OF A NEW HIGH-ENERGY OXIDIZER

M. T. Constantine
F. D. Raniere
V. Bedwell

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

TECHNICAL REPORT AFRPL-TR-66-184

September 1966

Group 4
Downgraded at 3-Year Intervals
Declassified After 12 Years

THIS MATERIAL CONTAINS INFORMATION AFFECTING THE NATIONAL DEFENSE OF THE UNITED STATES WITHIN THE MEANING OF THE ESPIONAGE LAWS, TITLE 18 U.S.C., SECTIONS 793 AND 794, THE TRANSMISSION OR REVELATION OF WHICH IN ANY MANNER TO AN UNAUTHORIZED PERSON IS PROHIBITED BY LAW.

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Prepared Under Contract AF04(611)-9563
For
Air Force Rocket Propulsion Laboratory
Research and Technology Division
Edwards, California 93523
Air Force Systems Command
United States Air Force

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Prepared Under Contract AF04(611)-9563
For
Air Force Rocket Propulsion Laboratory
Research and Technology Division
Edwards, California 93523
Air Force Systems Command
United States Air Force

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FOREWORD

This report is an addendum to the final report (Ref. 1) issued under Contract AF04(611)-9563 and is submitted under Rocketdyne G.O. 8553 in compliance with the first and second modifications reflected in Supplemental Agreement No. 5 to the contract. The research reported herein represents additional effort conducted over a period of 15 November 1964 through 15 November 1965 under a contract extension. This research was sponsored by the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, Edwards, California, with Mr. G. Allen Beale acting as Air Force Project Engineer.

The effort was conducted in the Chemical Research Section of the Rocketdyne Research Department, with Dr. J. Silverman serving as Program Manage[illegible] and Mr. M. T. Constantine serving as Responsible Project Scientist.

This report has been assigned the Rocketdyne identification number R-6445.

This technical report has been reviewed and is approved.

CURTIS E. LUNDBLAD
Deputy Chief, Propellant Division

CONFIDENTIAL ABSTRACT

The results of long-term, ambient-temperature materials compatibility and storability studies with chlorine pentafluoride (ClF_5) are reported as an extension of efforts conducted under Contract AF04(611)-9563. Duplicate sets of 34 different materials of construction, exposed to the liquid and vapor phases of ClF_5 for a period of 19 months, exhibited corrosion rates similar to those demonstrated previously in 30-day materials compatibility studies under both ambient-temperature and 160 F conditions. Periodic analyses of ClF_5 samples stored at ambient temperatures in 321 stainless steel, 6061 aluminum, Monel 400, and oxygen-free copper for a period of 13 months indicated an absence of propellant decomposition and/or reaction.

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INTRODUCTION AND SUMMARY

Under Contract AF04(611)-9563, Rocketdyne initiated an exploratory development program on the engineering characterization of ClF_5 for the purpose of advancing the state-of-the-art of this storable high-energy oxidizer. During the first year of the contract, 15 November 1963 to 14 November 1964, the program consisted of two phases. Phase I was designed to produce at least 80 pounds of ClF_5 while Phase II comprised the experimental and analytical investigation of the physical and engineering properties of ClF_5, including density, vapor pressure, critical properties, surface tension, compressibility, specific heat, heat of vaporization, viscosity, thermal conductivity, dielectric constant, electrical conductivity, thermal stability, detonation sensitivity, materials compatibility, and storability. This initial 12-month effort was completed and a final report (Ref. 1) was issued in April 1965.

A 12-month extension of the contract was granted to permit continuation of experimental characterization of selected physical and engineering properties of ClF_5. Primarily, this effort was directed at long-term materials compatibility and storability studies; however, additional viscosity and electrical conductivity studies were planned. These areas of study, as well as other efforts conducted within the scope of this program since 15 November 1964, are described in terms of experimental techniques and results.

During this 12-month extension of the contract, Rocketdyne supplied 1 pound of 99.2+ weight percent purity ClF_5 to the Battelle Memorial Institute as a continuation of Phase I of the original effort.

During Phase II, duplicate sets of 34 different materials of system construction were exposed to the liquid and vapor phases of ClF_5 for a period of 19 months at temperatures of 30 to 100 F. The effects of this closed-system exposure, evaluated on the basis of visual inspection and weight gain/loss analysis, indicated that all the materials were compatible under the test conditions.

Samples of ClF_5 were stored in 321 stainless steel, 6061 aluminum, Monel 400, and oxygen-free copper containers (initial ullages of ~20 to 30 percent) for 13 months at ambient temperatures. In addition to weekly monitoring of container pressures and ambient temperatures, small liquid and vapor samples were removed from each container and chemically analyzed monthly. No significant changes were noted in pressure or composition during the entire storage period.

The viscosity of liquid ClF_5 from -38 C (-36.4 F) to 20 C (68 F) was completed in time for inclusion in Ref. 1. Contemplated extension of electrical conductivity measurements was deleted from the planned efforts.

All ClF_5 engineering property data generated during the active period of this program as well as pertinent data derived from other programs, have been collected and will be assembled for publication in a Chlorine Pentafluoride Engineering Properties Handbook under Contract F04-611-67-C-0006.

TECHNICAL PROGRAM

PHASE I: CHLORINE PENTAFLUORIDE PRODUCTION

OBJECTIVE

The objective under Phase I of the original 12-month program was the production of a minimum quantity of 80 pounds of 98+ percent ClF_5 and utilization of this material to satisfy Phase II propellant requirements as well as provide limited quantities for shipment to other users as directed by the Air Force.

RESULTS

The original Phase I objectives were satisfied and the details were reported in Ref. 1. However, during the 12-month extension of the contract, an additional shipment of 1 pound of ClF_5 was made on 19 March 1965 to the Battelle Memorial Institute at the request of the Air Force. The purity of the ClF_5, as determined by chemical analysis prior to shipment, was 99.2+ weight percent. This shipment was the sixth such shipment and brought the total quantity of ClF_5 shipped under this contract to approximately 14.5 pounds.

PHASE II: ENGINEERING PROPERTY CHARACTERIZATION OF ClF_5

OBJECTIVE

The primary objective of the 12-month extension to the program was to continue the Phase II chlorine pentafluoride engineering property determinations. The latter included the following:

1. Long-term ambient temperature materials compatibility
2. Long-term ambient temperature storability
3. Completion of viscosity measurements
4. Extension of electrical conductivity measurements

LONG-TERM MATERIALS COMPATIBILITY

Experimental Technique

As reported previously (Ref. 1), 34 different materials were selected for static compatibility screening studies with both liquid and vapor ClF_5. Selection was based on materials normally utilized in the aerospace industry. An attempt was made to select at least one representative from each class of materials which would be of interest. Simulation of actual propellant storage conditions was considered foremost in the investigation.

All sample materials (Table 1) were machined into test disks (approximately 11/16-inch diameter and 1/32-inch thick with a center hole measuring 0.11 inch). An identification stamp was imprinted, and each sample was degreased with trichloroethylene and acetone. The metal samples were then subjected to heat treatments compatible with typical end-use conditions (Table 2). The final heat treatment or condition of all materials tested prior to immersion in ClF_5 is given in Table 1. After heat treatment,

TABLE I

MATERIALS COMPATIBILITY SAMPLES

Material	Heat Treatment or Condition
Group 1	
1100 Al	0
2024 Al	T3
2024 Al	T351
2219 Al	T351
6061 Al	T651
6066 Al	T6
7075 Al	T6
7079 Al	T651
5457 Al	0
X7002 Al	0
5096 Al	0
Group 2	
Inconel-X	Tensile, 150,000 psi
304 Stainless Steel	Full hard
316 Stainless Steel	Full hard
347 Stainless Steel	Full hard
Rene' 41	1950 F, 1 hour, air cooled; 1400 F, 16 hours, air cooled
Hastelloy-C	Solution heat treated
Nickel 200	Annealed
Nickel 211	Annealed
Kel-F 81	
Group 3	
410 Stainless Steel	Tensile, 180,000 psi
PH 15-7 Mo	RH1050
AM 350	SCT 850
AM 355	SCT 1000
FEP Teflon	
TFE Teflon	
Group 4	
Monel 400	Annealed
Monel 402	Annealed
Monel K-500	Aged
Monel 501	Aged
Monel R-405	Annealed
Monel 505	Annealed
Monel 507	Annealed
ETP Copper	Hard

TABLE 2

SAMPLE HEAT TREATMENT

Material	Vendor Heat No.	Sample Harness			Final Heat-Treat Condition
		After Machining	After Annealing	After Aging	
Monel 400	M0882RSP	Rockwell B92	Rockwell B54		Annealed
Monel 402	M8092BL	Rockwell B82	Rockwell B48		Annealed
Monel K-500	M8507K	Rockwell B102		Rockwell B100	Aged
Monel 501	M8556KR	Rockwell B79		Rockwell B100	Aged
Monel R-405	M7292B	Rockwell B95	Rockwell B59		Annealed
Nickel 200	N7493A	Rockwell B90	Rockwell B40		Annealed
Nickel 211	N7779D	Rockwell B78	Rockwell B60		Annealed
Monel 505	S3439	Rockwell B95	Rockwell B97		Annealed
Inconel-X 750	3654X	Rockwell B78		Rockwell C33	Aged
Monel 507	RH3041	Rockwell B80	Rockwell B83		Annealed
FEP Teflon		Shore D65			
TFE Teflon		Shore D65			
Hastelley-C	IC 81	Rockwell B83			Solution heat treated
ETP Copper		Rockwell H104			Hard
Kel-F 81		Shore D80			
AM 355	HT24383-9	Rockwell A70		Rockwell C44	SCT 1000
AM 350	HT69756	Rockwell B78		Rockwell C38	SCT 850

each of the metal samples was pickled and/or resurfaced with No. 240 emery paper according to Table 3 (Ref. 2). Finally, the weight and dimensions of each sample were recorded.

The specimens were divided into four groups as indicated in Table I. Each group was loaded into a separate bomb with specimens of each material in both the liquid and vapor phases. This separation of materials into groups was intended to eliminate erroneous results caused by possible interaction of dissolved components between sample groups. A set of four bombs, each containing an individual group of specimens, comprised one system for study.

Each container bomb assembly was constructed from a 10-inch-long, 1-inch-diameter, stainless-steel tube with a stainless-steel bellows-type valve at the top and an AN plug in the bottom. The tube was lined with Kel-F sheet to prevent galvanic corrosion between the samples and the wall of the bomb.

The groups of specimens were suspended on a Teflon-coated wire with alternate Teflon spacers. In addition to the spacers, each specimen was isolated from other specimens, below and above, by alternating Kel-F disks. After assembly, the specimens had a spacing of 0.625 inch. The configuration of specimen, spacer, and Kel-F disk is shown in Fig. 1. This method of suspending specimens prevented scale and other corrosion products from dropping on the sample directly beneath. When loaded, sample bombs were suspended vertically, which ensured a horizontal orientation for the specimens and Kel-F disks.

Four complete and identical systems were loaded with high-purity uncontaminated (closed-system loading) ClF_5 and prepared for materials compatibility screening under the following conditions:

1. One system in a 30-day ambient-temperature test
2. One system in a 30-day high-temperature test (160 F)
3. Two systems in a long-term ambient-temperature test

DESC

Material	Appearance After Heat Treatment
Monel 400	Heavy scale, some coppering
Monel 402	Heavy scale, rusty appearance
Monel K-500	No appreciable scale
Monel 501	No appreciable scale
Monel R-405	Heavy scale, some coppering
Nickel 200	No appreciable scale
Nickel 211	Heavy scale, no coppering
Monel 505	Heavy scale, no coppering, less discoloration than Nickel 211
Inconel-X 750	Not badly scaled, dark blue color
Monel 507	Heavy scale, no coppering
AM 355	Heavy scale, rusty appearance
AM 350	Heavy scale, rusty appearance
ETP Copper	No heat treatment
Hastelloy C	No heat treatment
Kel-F 81	No heat treatment
Teflon	No heat treatment

[a]Formula 5

Water, cc	1000
Hydrochloric Acid (20 degrees Be'), cc	500
Cupric Chloride, grams	50
Temperature, F	180
Time, minutes	20 to 40

Rinse in hot water prior to next Formula immersion

[b]Formula 6

Water, cc	1000
Sulfuric Acid (66 degrees Be'), cc	100
Sodium Dichromate, grams	132
Temperature, F	70 to 100
Time, minutes	5 to 16

Rinse in cold water and neutralize in 1 to 2 v/o ammonia

[c]Formula 7

Water, cc
Sodium Hydroxide, grams
Potassium Permanganate, g
Temperature, F
Time, hours

Rinse and pickel in Form

[d]Form

Water, cc
Nitric Acid (42 degrees
Hydrofluoric Acid (50 deg
Temperature, F, maximum
Time, minutes

Add 7 to 10 grams alumin
solution.

Caution: Samples are sub
attack in this solution.
a minimum.

TABLE 3

CALING TREATMENT

	Descaling Treatment
	Formula 5[a] (30 minutes), followed by Formula 6[b] (10 minutes)
	Formula 5 (30 minutes), followed by Formula 6 (10 minutes)
	Formula 5 (30 minutes), followed by Formula 6 (10 minutes)
	Formula 5 (1 hour), followed by Formula 6 (20 minutes)
	Formula 5 (1 hour), followed by Formula 6 (20 minutes)
	Formula 5 (1 hour), followed by Formula 6 (10 minutes)
	Formula 5 (1 hour), followed by Formula 6 (10 minutes)
	Formula 5 (1 hour), followed by Formula 6 (20 minutes)
	Formulas 7[c], 10[d], and 3[e], respectively; no results; finally sanded with emery
	Formula 5 (1 hour), followed by Formula 6 (20 minutes)
	24.7 w/o HNO_3 at ambient temperature
	24.7 w/o HNO_3 at ambient temperature
	--
	--
	--
	--

	1000
	266
grams	67
	212
	1 to 2

mula No. 10

mula 10

	1000
Be'), cc	296
egrees Be'), cc	50
	125
	5 to 60

num or iron per 1000 cc of

ubject to intergranular
. Keep immersion time to

[e]Formula 3

Water, cc	1000
Sulfuric Acid (60 degrees Be'), cc	1500
Nitric Acid (42 degrees Be'), cc	2250

Allow to cool and add:

Sodium Chloride, grams	30
Temperature, F	70 to 100
Time, seconds	5 to 20

Rinse in water and neutralize in dilute ammonia

2

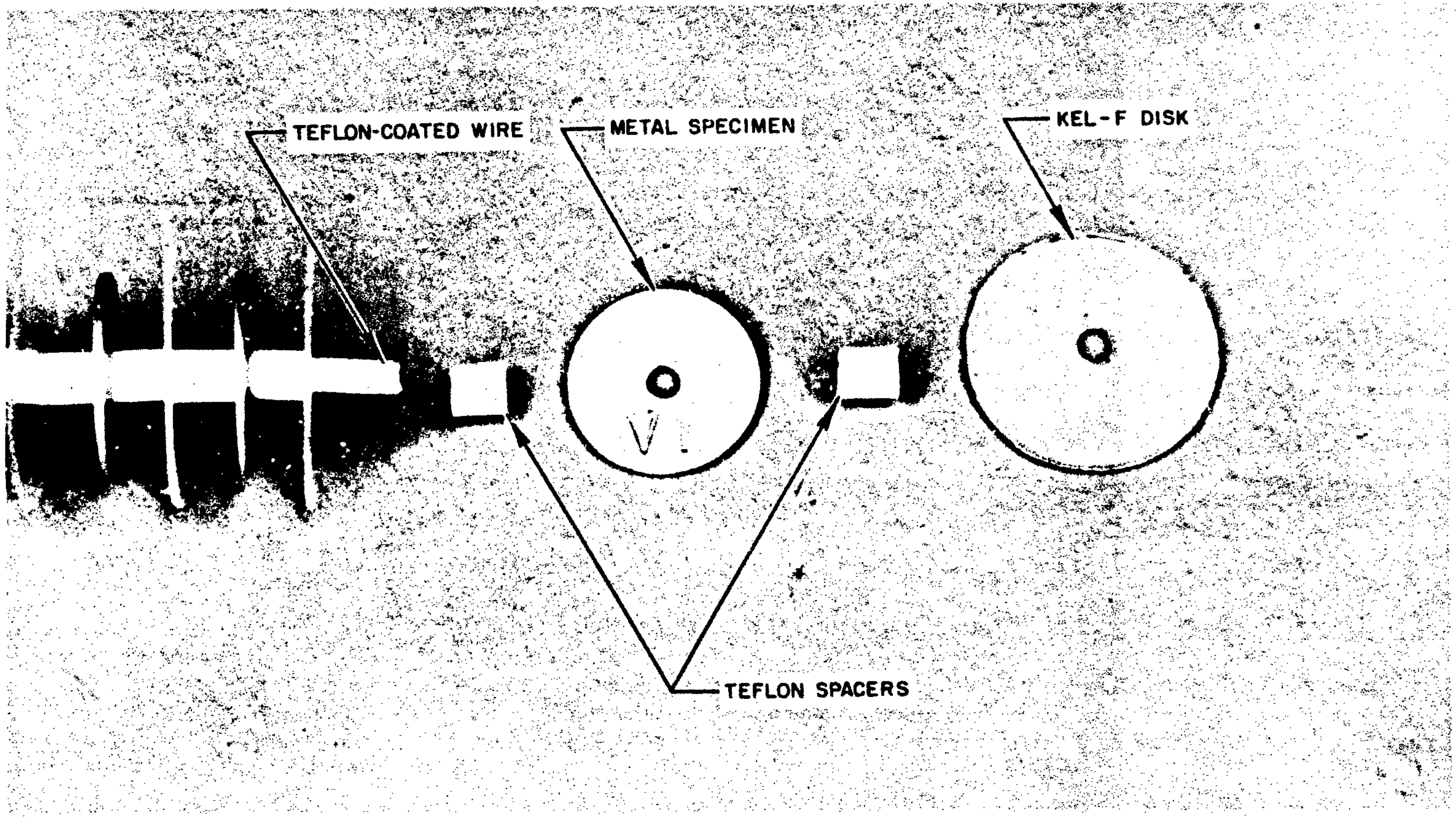

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Figure 1. Suspension of Sample Disks

In the closed-system loading of ClF_5, each bomb was first passivated with ClF_5 vapors at 25 psig and loaded with liquid so that half of the samples were exposed to the liquid and an identical half to the vapors. The closed-system transfer of ClF_5 into the bombs, from a calibrated supply system, was used to prevent air and moisture contamination of ClF_5.

In addition to these tests, a selected group of samples was studied in a preliminary 30-day compatibility test with moisture and air contaminated ClF_5 at a controlled temperature of 86 ±2 F. Sample bombs for each material specimen were fabricated from Teflon and loaded with ClF_5 in a direct open-air transfer. This test was used to evaluate the effect of handling ClF_5 under contaminated system conditions. The ClF_5 was obviously contaminated with air and moisture that had condensed on the prechilled bombs.

This test program, with the exception of the long-term ambient-temperature tests, was concluded during the initial 12 months of the contract and reported in Ref. 1. Long-term ambient-temperature tests were initiated on two complete and duplicate systems during this period (April 1964) and continued through the 12-month extension of the contract.

Results and Discussion

Long-term materials compatibility screening tests with ClF_5 were concluded in November 1965 after 19 months (580 days) of closed-system storage at prevailing ambient temperatures ranging from 30 to 100 F. The compatibility bombs were opened in a controlled atmosphere box under a dry gaseous nitrogen atmosphere. After removal from the ClF_5 liquid and vapor, the material specimens were dried by dry nitrogen gas over a period of ~24 hours. The specimens were transferred under a dry inert atmosphere to a weighing dish. Weights and surface conditions of the materials were noted and recorded.

Results of the long-term materials compatibility tests are summarized in Table 4. The group number assigned to each material identifies the container in which it was tested and relates materials tested together.

Weight changes in the materials are given in three different units, milligrams per square decimeter, milligrams per square decimeter per day (mdd), and mils per year, representing various means of comparing corrosion rate data. In the nomenclature used in material weight gain/loss analysis, a positive sign represents a weight loss; otherwise a weight gain is indicated. The visual appearance of the materials is the condition of the surface before extended exposure of the specimens to air (and moisture).

These data are compared to results from previous 30-day, ambient-temperature and 160 F tests (Ref. 1) in Table 5. The comparison is based on weight change per exposed surface area (mg/sq dm) without a time factor. This method was selected because it was apparent that corrosion (weight loss) and passivation (weight gain) rates based on the long-term tests were very small in comparison with those based on short-term tests. The significance of this is discussed in the following paragraphs.

No metallographic studies were conducted on the long-term specimens, to compare with those conducted previously (Ref. 1) because of cost limitations. However, all specimens have been retained and can be tested if future funding is available.

Aluminum Alloys. All aluminum alloys tested demonstrated weight losses in both the liquid-exposed and vapor-exposed specimens. The magnitude of the weight losses, which was similar for each alloy tested, was well within the corrosion resistance rating level (<2.0 mils/year) established as excellent. It was noted that materials tested in container 1B generally exhibited slightly greater weight changes then those tested in container 1A.

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TABLE 4

MATERIALS COMPATIBILITY RESULTS* FOR 580-DAY AMBIENT-TEMPERATURE EXPOSURES TO ClF_5

Material	Group**	Material Weight Change						Visual Appearance	
		Liquid-Exposed Samples			Vapor-Exposed Samples				
		mg/sq dm	mg/sq dm-day	mils/year	mg/sq dm	mg/sq dm-day	mils/year	Liquid-Exposed Samples	Vapor-Exposed Samples
Aluminum Alloys									
1100 Al	1A	+7.33	+0.013	+0.007	0	0	0	No visible change	No visible change
	1B	+12.92	+0.022	-0.012	+16.54	+0.028	+0.015		
2024 Al (T3)	1A	-1.90	+0.003	-0.002	-7.6	+0.013	-0.007	No visible change	No visible change
	1B	+15.30	+0.026	+0.014	-17.21	+0.030	+0.016		
2024 Al (T351)	1A	+7.63	+0.013	+0.007	0	0	0	No visible change	No visible change
	1B	+19.16	+0.033	+0.018	+17.27	+0.030	-0.016		
2219 Al (T351)	1A	+7.50	+0.013	+0.007	+24.30	+0.042	+0.022	No visible change	No visible change
	1B	+16.64	+0.029	+0.015	+18.59	+0.032	+0.017		
5086 Al	1A	-5.58	+0.010	-0.005	+7.42	+0.013	+0.007	No visible change	No visible change
	1B	+16.57	+0.029	+0.016	+16.73	+0.029	+0.016		
5457 Al	1A	-9.49	-0.016	+0.009	+9.49	+0.016	+0.009	No visible change	No visible change
	1B	+13.28	+0.023	+0.012	+15.21	+0.026	+0.014		
6061 Al (T651)	1A	-7.39	+0.013	+0.007	+7.39	+0.013	+0.007	No visible change	No visible change
	1B	+16.64	+0.029	+0.022	+14.70	+0.025	+0.013		
6066 Al (T6)	1A	+1.87	+0.003	+0.002	+11.15	+0.019	+0.010	No visible change	No visible change
	1B	+18.66	+0.032	+0.017	+14.87	+0.026	+0.014		
X7002 Al	1A	+9.49	+0.016	+0.008	-7.53	+0.013	+0.011	No visible change	No visible change
	1B	+9.43	-0.016	+0.008	+15.18	+0.026	+0.014		
7075 Al (T6)	1A	+9.51	+0.016	+0.009	+11.49	+0.020	+0.011	No visible change	No visible change
	1B	+15.21	+0.026	+0.014	+11.21	+0.019	+0.010		
7079 Al (T651)	1A	-7.38	+0.013	+0.007	+3.71	+0.006	+0.003	No visible change	No visible change
	1B	+16.73	+0.029	+0.015	+16.69	+0.029	+0.015		
Austenitic Stainless Steels									
304 SS	2A	0	0	0	+1.88	+0.003	+0.0005	No visible change	No visible change
	2B	5.66	0.010	0.002	3.77	0.007	0.001		
316 SS	2A	5.66	0.010	0.002	5.66	0.007	0.001	No visible change	No visible change
	2B	1.90	0.003	0.0005	+89.02(?)	+0.153(?)	+0.028(?)		
347 SS	2A	0	0	0	+1.89	+0.003	+0.0005	No visible change	No visible change
	2B	5.71	0.010	0.002	1.89	0.003	0.0005		
Nonaustenitic Stainless Steels									
PH 15-7 Mo	3	1.98	0.003	0.0005	0	0	0	Discolored, dull	Discolored, dull
	3	5.62	0.010	0.002	0	0	0		
AM 350	3	5.77	0.010	0.002	1.95	0.003	0.0005	Discolored, dull, slight surface attack	Discolored, dull, slight surface attack
	3	0	0	0	+1.93	+0.003	+0.0005		
AM 355	3	25.69	0.044	0.008	27.72	0.048	0.009	Discolored, dull, slight surface attack	Discolored, dull, slight surface attack
	3	23.72	0.041	0.008	15.81	0.027	0.005		
410 SS	3	62.74	0.108	0.019	70.08	0.121	0.022	Discolored, film on surface	Discolored, film on surface
	3	56.82	0.098	0.018	62.50	0.108	0.019		

*A positive sign indicates a weight loss; otherwise a weight gain was observed

**Group represents test container

(?)Indicates a probable weight measurement error

TABLE 4

(Concluded)

Material	Group**	Material Weight Change						Visual Appearance	
		Liquid-Exposed Samples			Vapor-Exposed Samples				
		mg/sq dm	mg/sq dm-day	mils/year	mg/sq dm	mg/sq dm-day	mils/year	Liquid-Exposed Samples	Vapor-Exposed Samples
Nickel and Nickel-Base Alloys									
Nickel 200	2A	0	0	0	+1.87	+0.003	-0.0004	Slight dullness	Slight dullness
	2B	1.87	0.003	0.0004	0	0	0		
Nickel 211	2A	1.90	0.003	0.0004	+1.90	+0.003	+0.0004	Discolored, dull	Discolored, dull
	2B	3.79	0.007	0.001	-1.98	-0.003	-0.0004		
Inconel X-750	2A	+1.96	+0.003	+0.0005	+1.98	+0.003	-0.0005	Discolored, dull	Discolored, dull
	2B	3.96	0.007	0.001	3.94	0.007	0.001		
Rene 41	2A	3.74	0.006	0.001	5.66	0.010	0.002	Slight discoloration	Slight discoloration
	2B	3.78	0.007	0.001	3.75	0.006	0.001		
Hastelloy-C	2A	0	0	0	-5.83	+0.010	+0.002	No visible change	No visible change
	2B	3.89	0.007	0.001	+91.62(?)	+0.158(?)	+0.025(?)		
Copper and Monel Alloys									
Monel 400	4A	5.83	0.010	0.002	11.63	0.020	0.003	Discolored, dull, dark spots	Discolored, dull, dark spots
	4B	5.78	0.010	0.002	3.91	0.007	0.001		
Monel 402	4A	11.65	0.020	0.003	1.91	0.003	0.0004	Discolored, dull	Discolored, dull
	4B	7.71	0.013	0.002	7.59	0.013	0.002		
Monel R-405	4A	37.66	0.065	0.010	32.69	0.056	0.009	Discolored, dull, dark spots	Discolored, dull, dark spots
	4B	7.65	0.013	0.002	7.66	0.013	0.002		
Monel K-500	4A	+1.93	+0.003	+0.0005	7.72	0.013	0.002	Discolored, dull	Discolored, dull
	4B	1.93	0.003	0.0005	+1.93	+0.003	+0.0005		
Monel 501	4A	23.12	0.040	0.007	27.03	0.047	0.008	Discolored, dull, dark spots	Discolored, dull, dark spots
	4B	19.23	0.033	0.006	17.27	0.030	0.005		
Monel 505	4A	9.86	0.017	0.003	14.06	0.024	0.004	Discolored, dull, pitted	Discolored, dull, pitted
	4B	2.01	0.003	0.0005	2.01	0.003	0.0005		
Monel 507	4A	20.12	0.035	0.006	30.00	0.052	0.009	Pitted, dull, discolored	Pitted, dull, discolored
	4B	12.02	0.021	0.004	16.10	0.028	0.005		
ETP Copper	4A	13.51	0.023	0.004	21.44	0.037	0.006	Dull	Dull
	4B	3.88	0.007	0.001	3.88	0.007	0.001		
Nonmetallics									
FEP Teflon	3	346.3	0.597	0.399	347.5	0.599	0.400	Black discoloration around perimeter	Black discoloration around perimeter
TFE Teflon	3	197.3	0.340	0.228	170.2	0.293	0.196	Black discoloration around perimeter	Black discoloration around perimeter
Kel-F 81	2A	728.17	1.255	0.851	491.19	0.847	0.574	No visible change	No visible change
	2B	745.02	1.285	0.871	711.50	1.227	0.832		

*A positive sign indicates a weight loss; otherwise a weight gain was observed

**Group represents test container

(?)Indicates a probable weight measurement error

TABLE 5

COMPARISON OF MATERIALS COMPATIBILITY RESULTS* FOR EXPOSURE TO ClF_5 UNDER VARIOUS CONDITIONS

Material	Material Weight Change, mg/sq dm					
	Liquid-Exposed Samples			Vapor-Exposed Samples		
	30-Day, ambient	30-Day, 160 F	500-Day**, ambient	30-Day, ambient	30-Day, 160 F	500-Day,** ambient
Aluminum Alloys						
1100 Al	1.85	15.69	+7.33 +12.92	1.85	19.60	0 +16.94
2024 Al (T3)	+5.74	9.81	+1.90 +15.30	+5.73	11.76	+7.6 +17.21
2024 Al (T351)	+9.52	7.83	+7.63 +19.16	+3.82	+9.66	0 +17.27
2219 Al (T351)	+5.54	7.83	+7.50 +16.64	+5.56	11.76	+24.30 +18.59
5086 Al	0	11.76	+5.58 +16.57	0	3.93	+7.42 +16.73
5457 Al	+3.81	9.81	+9.49 +13.28	+5.69	13.71	+9.49 +15.21
6061 Al (T651)	+9.24	11.76	+7.39 +16.64	+1.86	13.71	+7.39 +14.70
6066 Al (T6)	+7.46	15.69	+1.87 +18.66	+1.86	13.72	+11.15 +14.87
X7002 Al	3.80	7.83	+9.49 +9.43	3.79	9.81	+7.53 +15.18
7075 Al (T6)	3.79	7.83	+9.51 +15.21	5.67	5.88	+11.49 +11.21
7079 Al (T651)	1.87	11.76	+7.38 +16.73	5.36	13.71	+3.71 +16.69
Austenitic Stainless Steels						
304 SS	22.43	7.83	0 5.66	15.01	7.83	+1.88 3.77
316 SS	24.57	9.81	5.66 1.90	47.17	5.88	5.66 +89.02(?)
347 SS	22.77	11.76	0 5.71	26.46	13.71	+1.89 1.89
Nonaustenitic Stainless Steels						
PH 15-7 Mo	39.47	15.69	1.98 5.62	19.15	11.76	0 0
AM 350	7.71	11.76	5.77 0	+7.66	17.70	1.93 +1.93
AM 355	17.61	23.55	25.69 23.72	11.72	37.20	27.72 15.81
410 SS	20.64	60.90	62.74 56.82	9.48	41.10	70.08 62.50
Nickel and Nickel-Base Alloys						
Nickel 200	22.47	+9.81	0 1.87	24.57	+7.83	+1.87 0
Nickel 211	32.26	1.95	1.90 3.79	20.75	0	+1.90 +1.98
Inconel X-750	37.40	0	+1.96 3.96	37.62	0	+1.98 3.94
Rene 41	30.42	11.76	3.74 3.78	23.81	13.71	5.66 3.75
Hastelloy-C	21.53	1.95	0 3.89	27.29	0	+5.83 +91.62(?)

*A positive sign indicates weight loss; otherwise weight gain was observed

**Duplicate samples were tested under same test conditions resulting in two data points

(?)Indicates a probable weight measurement error

TABLE 5
(Concluded)

Material	Material Weight Change, mg sq dm					
	Liquid-Exposed Samples			Vapor-Exposed Samples		
	30-Day, ambient	30-Day, 160 F	380-Day**, ambient	30-Day, ambient	30-Day, 160 F	380-Day**, ambient
Copper and Monel Alloys						
Monel 400	40.62	3.93	5.83 5.78	31.10	5.88	11.63 3.91
Monel 402	30.30	+3.93	11.63 7.71	25.19	+1.93	1.91 7.59
Monel R-405	35.02	+9.81	37.66 7.65	40.23	5.88	37.66 7.66
Monel K-500	30.89	3.93	+1.93 1.93	30.89	3.93	7.72 +1.93
Monel 501	61.54	23.55	23.12 19.23	37.47	3.88	27.03 17.27
Monel 503	29.94	+48.90	9.86 2.01	33.79	0	14.06 2.01
Monel 507	45.73	57.00	20.12 12.02	42.17	57.10	30.00 16.10
ETP Copper	33.20	31.50	13.51 3.88	27.24	11.76	21.44 3.88
Nonmetallics						
FEP Teflon	—	111.9	346.4		105.90	347.5
TFE Teflon	—	51.0	197.3		48.90	170.2
Kel-F 81	—	—	728.17 745.02			491.19 711.50

*A positive sign indicates weight loss; otherwise weight gain was observed
**Duplicate samples were tested under same test conditions resulting in two data points
(?)Indicates a probable weight measurement error

Visual examination of the aluminum specimens indicated very little or no change in the surface of any specimen. No corrosion pitting was obvious and the surfaces possessed the same bright appearance of the pretest condition. Some dulling of this shiny condition was noted after the specimens had been exposed to air for several days.

The results exhibited by the aluminum alloys during the long-term tests were slightly different from those exhibited previously during 30-day, ambient-temperature and 160 F tests as shown in Table 5. During the shorter term tests, four of the ambient test samples and all of the 160 F test samples showed weight gains (indicative of passivation) instead of weight losses. Comparison of those that exhibited weight losses shows that the magnitude of the weight change was generally similar.

Austenitic Stainless Steels. The 300-series stainless steels demonstrated (Table 4) very small weight gains in the liquid-exposed samples and some conflicting results for the vapor-exposed samples. (The value shown for the 316 stainless steel specimen exposed to ClF_5 vapors in container 2B appears to be a discrepancy and probably represents an error in pretest weight measurement.) This small change in weight is further supported by visual observations in which no change was noted in surface appearance.

Table 5 shows that the magnitudes of weight gains during the long-term tests were comparable to the 160 F tests and slightly less than those of 30-day ambient tests.

Nonaustenitic Stainless Steels. Of the nonaustenitic stainless steels tested, the PH 15-7 Mo and AM 350 samples showed slight weight gains, with larger weight gains experienced with the AM 355 and 410 stainless-steel samples. Surfaces of all specimens of this group were discolored and dull, with slight surface attack noticeable at spots on the AM 350 and AM 355 specimens. Although there appeared to be a thin film or coating on all of the specimens, this film was quite obvious on the 410 stainless-steel specimen.

The material weight gains per unit area were similar for each of these specimens over the various test conditions (Table 5), except for the PH 15-7 Mo specimen which exhibited less change during the long-term tests than in the shorter tests.

Nickel and Nickel Base Alloys. Long-term exposure of the nickel and nickel base alloys to liquid ClF_5 resulted in small weight gains; however, generally, the vapor-exposed samples experienced small weight losses (a probable weight measurement error exists in the high value for Hastelloy-C specimen). The nickel 211 and Inconel X-750 specimens were discolored and dull with some evidence of surface reaction. Surfaces of the Rene 41 and nickel 200 specimens had only very slight evidence of film formation (i.e., slight dulling and discoloration of the surface finish), while all Hastelloy-C specimens showed no evidence of surface reaction.

In general, comparison of results of the various test conditions for the nickel and nickel base alloys indicated some similarities between the 160 F and long-term ambient tests, but very little agreement with the 30-day ambient tests. It should be noted that very similar conclusions were in evidence in the Table 5 comparison of the austenitic stainless steels. These two types of materials shared the same test container during each of the test periods.

Copper and Monel Alloys. The copper and Monel alloys, subjected to long-term exposure to ClF_5, experienced weight gains of relatively slight-to-moderate magnitudes. Although all of the Monel surfaces were discolored and dull, evidence of attack on the two cast Monels, 505 and 507, was demonstrated in the form of surface pitting in the area of the identification indentation. The K-500 and 402 specimens showed no evidence of local attack, while the 400, R-405, and 501 specimens had small areas of dark grey spots indicative of some sort of localized attack. No attack was visible on the surfaces of the ETP copper specimens with only a dulling of the surface finish. This dullness was increased with continued exposure to air.

Similar results (Table 5) were experienced with previous exposure of the copper and Monel alloys to ClF_5 during 30-day, ambient-temperature and 160 F tests, but the magnitude of the weight gains were generally larger during the 30-day ambient tests. In addition, some weight losses had been measured during the 160 F testing of this group.

Nonmetallics. Long-term testing of the nonmetallics, FEP Teflon, TFE Teflon, and Kel-F 81, resulted in the expected large weight gains experienced in previous testing. All of the specimens absorbed small amounts of ClF_5 with magnitudes between materials running in the order of Kel-F 81 > FEP Teflon > TFE Teflon. No visible attack was noticeable on the Kel-F 81 specimen; however, both Teflon specimens had black discoloration around the perimeter surface.

General Summary. In summarizing the results of the long-term materials compatibility tests as well as their comparison with previous short-term tests, it was obvious that none of the materials tested showed evidence of severe attack by either the liquid or vapor phases of ClF_5 under the conditions tested. Although there was a wide range of variation in the magnitude of weight changes and visual appearances between the various samples, the resistance of all materials tested (to attack by ClF_5) would be placed in the excellent category. The magnitude of weight changes for all materials under all conditions was very small based on accepted corrosion standards (i.e., excellent ratings are given to materials with < 2 mils/year change).

Of the metallic materials, only the aluminum alloys showed definite weight losses, indicative of corrosion. (However, there was visible evidence of corrosion in other groups, i.e., Monels and nonaustenitic stainless steel, although overall sample weight gains were noted.) Weight gains, indicative of a passivation film formation, were noted and observed on most of the other materials. However, visual observations indicated that the passivation film was not "fluffy" and did adhere to the metal. In addition, very little evidence of additional reaction appeared following lengthy exposure of the posttest specimens to air (and moisture). The Group 2 materials, particularly the austenitic stainless steels, nickel 200, Rene 41, and Hastelloy-C, showed very little evidence of any surface reaction.

Some discrepancies were noted in comparison of results between the various test conditions; but, for the most part, there was agreement. In one particular case (30-day ambient tests of Group 2 materials), the discrepancies may have been caused by very slight moisture contamination or slight change in the ClF_5 composition loaded into a particular container. This conclusion was reached because of the uniform discrepancy created with all specimens in that container.

Because of the agreement in the magnitude of weight changes per unit area (and large differences based on rate) between results from various conditions, it is concluded that most of the reaction of the ClF_5 (either passivation or corrosion) with the metallic samples occurs on initial contact. After that point, provided there are no additional changes created by external conditions (such as propellant composition change, dynamic movement or jarring loose of the passive film), very little reaction is expected.

In static applications, the Teflon and Kel-F plastics appear to be acceptable; however, these materials do absorb ClF_5 liquid and vapor.

LONG-TERM STORABILITY

Experimental Technique

Long-term ambient-temperature ClF_5 storability tests were conducted in four large-capacity bombs, 6.5 inches long, 1.5 inches in ID, and having a 1/8-inch wall thickness. A typical bomb is shown in Fig. 2. The four bombs and their top flanges were machined from 321 stainless steel, 6061 aluminum, Monel 400 (cold), and oxygen-free copper stock, respectively. The flanges were sealed with Teflon O-rings. Stainless-steel, bourdon-tube type pressure gages were used on each bomb to monitor vapor pressure fluctuations. Valves (300-series Hoke), used in conjunction with a sampling tee (as shown in Fig. 2) and the pressure gage, were constructed of the same material as the bomb except for the copper bomb, which utilized Monel valves. The positioning of the three-valve arrangement on each bomb allows for easy sampling of both liquid and vapor.

Each bomb was cleaned, dried, and passivated prior to closed-system transfer of approximately 300 grams (167 cc) of high-purity ClF_5 into the bombs. The initial ullages were kept small to maintain exposure of a large metal surface to the liquid and to readily show any significant pressure rises indicative of propellant breakdown. After loading, the bombs were transferred to an outdoor storage observation area and stored at prevailing ambient temperatures. The initial chemical analyses of the storability samples were conducted from 9 to 13 November 1964; thereafter, chemical analyses of liquid and vapor phase samples from the bombs were conducted at approximately monthly intervals. In addition, the ambient temperature and the internal pressures of the containers were measured and recorded weekly.

The transfer of a representative liquid sample (~4 cc) from a storability bomb was accomplished by inverting the bomb and connecting it to a vacuum line. After passivation of the transfer system and sampling tee with ClF_5

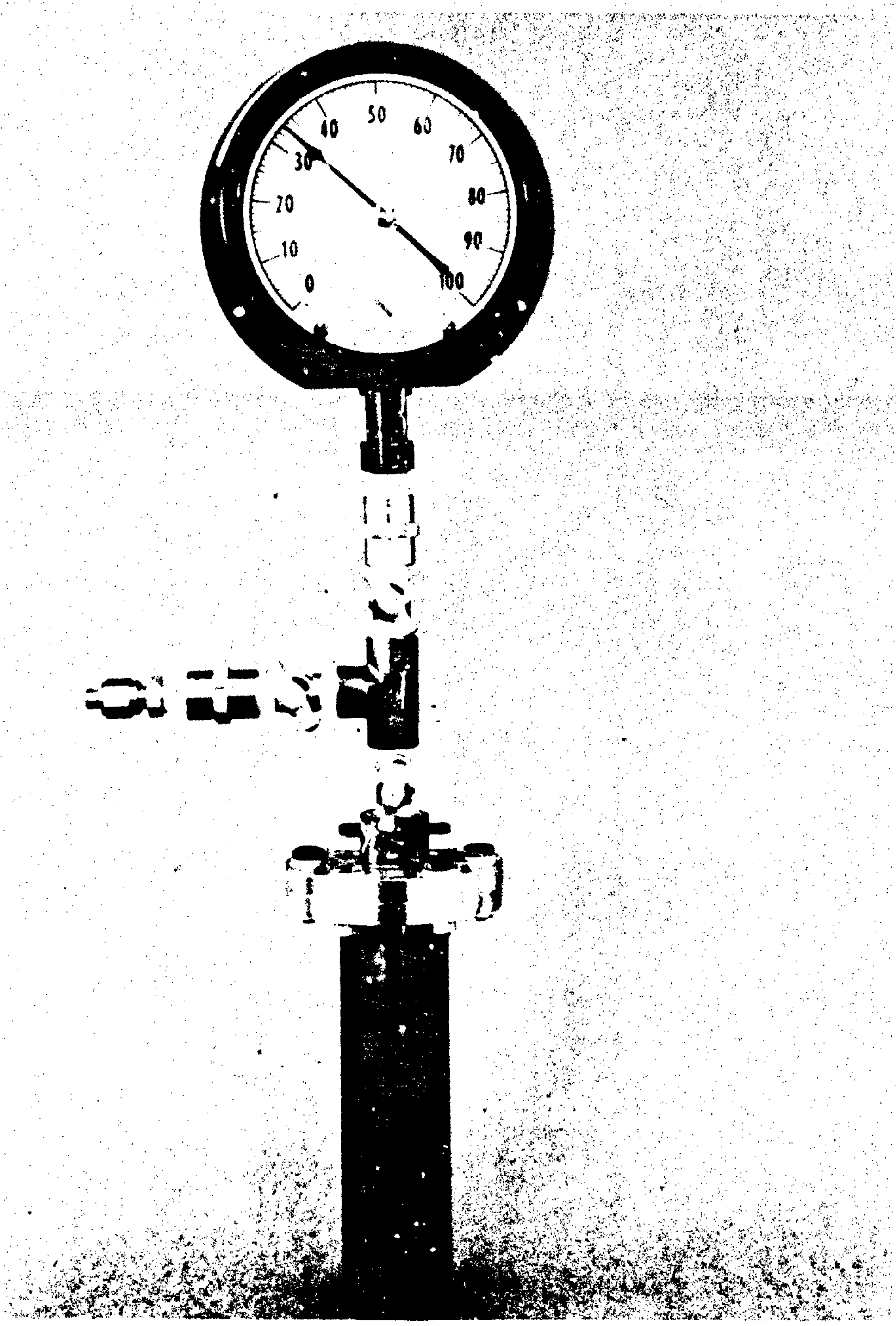

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Figure 2. Long-Term Storability Bomb

and subsequent evacuation, the tee outlet valve was closed. The bomb outlet valve was opened to fill the tee with liquid ClF_5 and then closed. The tee outlet valve was opened and the liquid sample expanded on a vacuum line into a 1.7-liter stainless-steel gas sampler. This sampler was connected to the inlet manifold of a gas chromatograph and infrared cell for a simultaneous analysis of all constituents by a combination of gas chromatography and infrared spectrophotometry. Vapor phase samples were similarly transferred and analyzed with the bomb in a normal upright position.

Weekly monitoring of the container pressures was established as a crude check of potential decomposition or reaction of the storability samples, rather than an accurate pressure-temperature measurement. This was accomplished by opening the bomb outlet valve and gage inlet valve, and recording the value from the gage; these two valves were then closed. This procedure of closing the valves between measurements was used to offer more protection against the accidental loss of the storability samples. Temperatures were recorded from a laboratory thermometer, which was placed in the immediate vicinity of the storability bombs.

Results and Discussion

The eleventh and final analyses of the storability samples under this program were conducted from 7 to 13 December 1965 to complete storage periods of approximately 13 months. During this time, prevailing ambient temperatures at the storage site ranged from ~30 to 100 F.

Results of periodic chemical analyses of samples from ClF_5 stored in 321 stainless steel, 6061 aluminum, Monel 400, and copper are presented in Tables 6 through 9, respectively. Each table shows the dates and results of the liquid (given in weight percent) and vapor (given in volume or mole percent) phase analyses, the accumulated storage periods,

TABLE 6

RESULTS OF LONG-TERM STORAGE OF ClF_5 IN 321 STAINLESS STEEL[1]

Date	Storage[2] Period, months	Liquid Phase Chemical Analysis, weight percent						Liquid Volume,[3] cc
		ClF_5	ClF_3	ClF	$F_2 + CF_4$[4]	HF	Cl_2	
11-12-64	0	99.1	< 0.4	< 0.04	< 0.03	0.4	-	167.2
12-15-64	1.1	99.2	< 0.4	< 0.02	< 0.01	0.3	-	162.1
1-27-65	2.5	99.3	0.6	< 0.02	< 0.01	NA[6]	-	156.9
3-3-65	3.6	99.2	0.7	< 0.02	< 0.01	NA[6]	-	154.1
4-6-65	4.8	99.0	0.6	< 0.02	< 0.01	0.25	-	149.7
5-6-65	5.7	99.2	0.7	< 0.02	< 0.01	NA[6]	-	147.3
6-9-65	6.9	98.9	0.7	< 0.02	< 0.01	0.3	-	144.9
7-14-65	8.0	99.1	0.6	< 0.02	< 0.01	0.2	-	139.3
8-6-65	8.8	98.8	0.8	< 0.02	< 0.01	0.4	-	135.6
9-13-65	10.0	98.9	0.7	< 0.02	< 0.01	0.4	-	132.4
12-7-65	12.8	98.8	0.6	< 0.02	< 0.01	0.5	-	127.3

Date	Storage[2] Period, months	Vapor Phase Chemical Analysis, volume percent							Ullage,[3] volume percent
		ClF_5	ClF_3	ClF	Cl_2	$F_2 + CF_4$[4]	ClO_2	SF_6	
11-9-64	0	98.5	< 0.5	0.2	< 0.1	0.6[7]	-	< 0.1	19.0
12-15-64	1.2	98.1	< 0.5	0.4	< 0.1	0.8[7]	-	< 0.1	21.3
1-21-65	2.4	98.3	< 0.5	0.4	0.1	0.6[7]	-	< 0.1	24.0
2-24-65	3.5	98.7	< 0.5	0.2	< 0.1	0.4	-	< 0.1	25.3
3-29-65	4.6	98.8	< 0.5	0.2	< 0.1	0.3	-	< 0.1	27.5
4-27-65	5.5	98.7	< 0.5	0.2	< 0.1	0.4	-	< 0.1	28.7
6-2-65	6.8	98.8	0.7	0.1	0.1	0.2	-	< 0.1	29.8
7-1-65	7.7	98.9	< 0.5	0.2	< 0.1	0.2	-	< 0.1	32.4
8-2-65	8.7	98.9	< 0.5	0.2	0.1	0.2	-	< 0.1	34.3
9-2-65	9.8	98.9	< 0.5	0.2	0.2	0.1	-	< 0.1	35.9
12-7-65	12.9	99.0	< 0.5	0.2	0.1	0.1	-	< 0.1	38.4

NOTE: Explanatory notes relating to this table are presented on page 28.

TABLE 7

RESULTS OF LONG-TERM STORAGE OF ClF_5 IN 6061 ALUMINUM[8]

Date	Storage[2] Period, months	Liquid Phase Chemical Analysis, weight percent						Liquid Volume,[3] cc
		ClF_5	ClF_3	ClF	$F_2 + CF_4$[4]	HF	Cl_2	
11-13-64	0	99.2	< 0.4	< 0.03	< 0.02	0.25	-	164.5
12-29-64	1.5	99.2	< 0.4	< 0.02	< 0.01	0.3	-	159.6
1-28-65	2.5	99.5	< 0.4	< 0.02	< 0.01	NA[6]	-	154.4
3-12-65	4.0	99.2	0.7	< 0.02	< 0.01	NA[6]	-	151.0
4-5-65	4.7	98.6	0.8	< 0.02	< 0.01	0.5	-	146.6
5-12-64	6.0	98.9	0.7	< 0.02	< 0.01	0.3	-	143.6
6-8-65	6.8	98.8	0.8	< 0.02	< 0.01	0.35	-	138.1
7-13-65	8.0	99.0	0.6	< 0.02	< 0.01	0.3	-	133.4
8-6-65	8.8	98.8	0.8	< 0.02	< 0.01	0.4	-	128.2
9-3-65	9.7	98.8	0.6	< 0.02	< 0.01	0.5	-	124.1
12-9-65	12.9	98.7	0.6	< 0.02	< 0.01	0.6[7]	-	120.1

Date	Storage[2] Period, months	Vapor Phase Chemical Analysis, volume percent							Ullage,[5] volume percent
		ClF_5	ClF_3	ClF	Cl_2	$F_2 + CF_4$[4]	ClO_2	SF_6	
11-9-64	0	98.6	< 0.5	0.1	< 0.1	0.6	-	< 0.1	26.8
12-22-64	1.4	98.6	< 0.5	0.3	-	0.5	-	< 0.1	31.0
1-21-65	2.4	98.2	< 0.5	0.6	< 0.1	0.5	-	< 0.1	33.1
2-24-65	3.5	98.6	< 0.4	0.4	< 0.1	0.4	-	< 0.1	34.8
4-1-65	4.6	98.7	< 0.5	0.2	0.2	0.3	-	< 0.1	36.6
4-27-65	5.5	98.5	< 0.5	0.3	0.1	0.5	-	< 0.1	37.9
6-1-65	6.7	98.4	< 0.5	0.4	0.1	0.5	-	< 0.1	40.3
7-7-65	7.9	97.8	0.7	0.5	0.3	0.6	-	< 0.1	42.3
8-3-65	8.8	97.4	0.7	0.7	0.2	0.9[7]	-	< 0.1	44.6
9-3-65	9.8	97.7	0.6	0.6	0.2	0.7[7]	-	< 0.1	46.4
12-9-65	13.0	98.3	0.5	0.3	0.1	0.3	-	< 0.1	48.2

NOTE: Explanatory notes relating to this table are presented on page 28.

TABLE 8

RESULTS OF LONG-TERM STORAGE OF ClF_5 IN MONEL 400[9]

Date	Storage[2] Period, months	Liquid Phase Chemical Analysis, weight percent						Liquid Volume,[3] cc
		ClF_5	ClF_3	ClF	$F_2 + CF_4$[4]	HF	Cl_2	
11-13-64	0	99.0	< 0.4	< 0.04	0.3	0.25	-	160
12-[illegible]-64	1.1	98.7	< 0.4	< 0.05	< 0.05	0.3	0.5[7]	155.5
1-2[illegible]-65	2.5	99.2	0.6	< 0.02	< 0.01	NA[6]	-	150.2
3-[illegible]-65	[illegible]	[illegible]	0.8	< 0.02	< 0.01	NA[6]	-	147.2
4-[illegible]-65	[illegible]	[illegible]	[illegible]	< 0.02	< 0.01	0.25	< 0.05	143.0
5-13-65	[illegible]	98.9	0.8	< 0.02	< 0.01	0.2	-	139.9
6-11-65	[illegible]	98.8	0.8	< 0.02	< 0.01	0.3	-	134.8
7-15-65	8.1	98.8	0.7	< 0.02	< 0.01	0.4	-	129.3
8-9-65	8.9	98.8	0.7	< 0.02	< 0.01	0.4	-	125.8
9-15-65	[illegible]	98.9	0.7	< 0.02	< 0.01	0.3	-	122.2
12-13-65	[illegible]	98.7	0.6	< 0.02	< 0.01	0.6	-	117.6

Date	Storage[2] Period, months	Vapor Phase Chemical Analysis, volume percent						Ullage,[3] volume percent
		ClF_5	ClF_3	ClF	Cl_2	$F_2 + CF_4$[4]	ClO_2	SF_6	
11-10-64[10]	0	79	0.5	2.0	1.0	17.3	-	< 0.1	26.6
11-11-64	0	93	0.5	0.4	-	3.9	-	< 0.1	26.6
12-15-64	1.2	97+	0.7	0.2	-	1.7	-	< 0.1	28.6
1-22-65	2.4	95.6	< 0.5	0.5	< 0.1	3.2	-	< 0.1	31.0
2-25-65	3.5	95.8	< 0.5	0.2	-	3.4	-	< 0.1	32.4
3-31-65	4.7	95.6	< 0.5	0.7	< 0.1	3.0	-	< 0.1	34.4
4-30-65	5.7	95.5	< 0.5	0.1	0.5[7]	2.8	0.5[7]	< 0.1	35.8
6-2-65	6.8	96.1	< 0.5	0.8	0.3	2.2	-	< 0.1	38.1
7-6-65	7.9	97	0.5	0.5	0.2	1.7	-	< 0.1	40.7
8-4-65	8.8	93.7	< 0.5	1.4	0.3	3.8	-	< 0.1	42.2
9-7-65	9.9	94.8	< 0.5	1.0	0.2	3.4	-	< 0.1	43.3
12-13-65	13.1	95.5	< 0.5	0.7	0.2	3.0	-	< 0.1	46.0

NOTE: Explanatory notes relating to this table are presented on page 28.

TABLE 9

RESULTS OF LONG-TERM STORAGE OF ClF_5 IN COPPER[1/]

Date	Storage[2/] Period, months	Liquid Phase Chemical Analysis, weight percent						Liquid Volume,[3/] cc
		ClF_5	ClF_3	ClF	$F_2 + CF_4$[4/]	HF	Cl_2	
11-12-64	0	98.7	< 0.4	< 0.01	< 0.02	0.8[7/]	-	168
12-8-64	0.9	99.1	0.5	< 0.02	< 0.06	0.3	-	163.2
1-26-65	2.5	99.3	0.6	< 0.02	< 0.01	NA[6/]	-	159.6
3-2-65	3.6	99.4	0.5	< 0.02	< 0.01	NA[6/]	-	156.6
4-7-65	4.8	98.9	0.7	< 0.02	< 0.01	0.3	-	152.8
5-5-65	5.7	99.2	0.7	< 0.02	< 0.01	NA[6/]	-	149.9
6-3-65	6.7	98.8	0.8	< 0.05	< 0.03	0.3	-	147.4
7-12-65	8.0	98.8	0.8	< 0.05	< 0.03	0.25	-	143.0
8-5-65	8.8	98.8	0.7	< 0.05	< 0.03	0.35	-	138.0
9-10-65	9.9	99.0	0.6	< 0.05	< 0.03	0.3	< 0.03	134.0
12-9-65	12.9	98.9	0.6	< 0.05	< 0.03	0.4	-	129.5

Date	Storage[2/] Period months	Vapor Phase Chemical Analysis, volume percent							Ullage,[5/] volume percent
		ClF_5	ClF_3	ClF	Cl_2	$F_2 + CF_4$[4/]	ClO_2	SF_6	
11-9-64	0	98	< 0.5	0.6	0.3	0.6[7/]	-	< 0.1	21.9
12-7-64	0.9	97.8	< 0.5	0.6	0.4	0.6[7/]	-	< 0.1	24.1
1-20-65	2.4	98.6	< 0.5	0.4	0.2	0.2	0.8[7/]	< 0.1	25.7
2-23-65	3.5	98.2	< 0.5	<0.1	< 0.1	0.2	-	< 0.1	27.1
3-30-65	4.6	97.8	0.7[7/]	0.2	0.7[7/]	0.5[7/]	-	< 0.1	28.9
4-26-65	5.5	98.7	< 0.5	0.2	0.1	0.4	-	< 0.1	30.3
5-26-65	6.5	98.9	< 0.5	0.2	< 0.1	0.3	-	< 0.1	31.4
6-30-65	7.7	98.8	< 0.5	0.2	0.2	0.3	-	< 0.1	33.5
8-2-65	8.7	98.4	0.6	0.2	0.4	0.3	-	< 0.1	35.8
9-3-65	9.8	98.5	0.5	0.2	0.4	0.3	-	< 0.1	37.7
12-9-65	13.0	98.6	< 0.5	0.3	0.3	0.3	-	< 0.1	39.8

NOTE: Explanatory notes relating to this table are presented on page 28.

EXPLANATORY NOTES

The following notes are applicable to Tables 6 through 9.

1. Initial container volume of 206.5 cc; loaded with 301 grams ClF_5
2. Storage period is elapsed storage time since initial analysis
3. Represents liquid volume calculated before indicated sample is removed
4. Also could include N_2, O_2, Ar, etc; infrared analysis indicates values > 0.3 v/o are CF_4
5. Represents ullage calculated at 22 C (71.6 F) before indicated sample is removed
6. Not analyzed because of inoperative instrument
7. Values questionable; probably contamination of analytical sample during transfer of sample from bomb to analytical apparatus
8. Initial container volume of 231.5 cc; loaded with 296 grams ClF_5
9. Initial container volume of 218 cc; loaded with 288 grams ClF_5
10. A reaction occurred during initial pressure measurements; all subsequent vapor phase analysis results affected
11. Initial container volume of 215 cc; loaded with 302 grams ClF_5

and the preanalysis liquid volumes or ullages. The liquid volumes and ullages at each analysis were calculated from the weight of ClF_5 remaining in the bombs from the previous analyses and based on a propellant density of 1.8 corresponding to the established density at a propellant temperature of ~22 C (71.6 F).

Results reflected in the liquid phase analyses indicate there were no significant changes in the composition of the storability samples or any apparent differences in effects between the four storage materials. The fractional changes in quantities of the different components are within the inherent experimental error of the analytical techniques and do not represent any trend toward reduction or buildup. In some instances, there are questionable values noted; these have been attributed to minute contamination of the transfer system with moisture, etc. during removal of an assay sample from the bomb. The hook-up and use of a system to transfer a small amount of the storability sample to the analytical apparatus, without contamination of the assay sample, represented the most difficult part of the test.

Similar results are shown in the analyses of vapor phase compositions. The vapor-phase analysis was used primarily to observe any significant increase of fluorine in the system; however, such an increase was not evident in any of the containers. The initial high analysis of $F_2 + CF_4$ in the vapor phase of the Monel storability system was caused by a reaction that occurred in the pressure [illegible] during initial container-pressure measurements. The effects of the reaction on the vapor composition in the Monel bomb is clearly demonstrated during subsequent analyses. The intermittent appearance of unusual results, such as ClO_2 content, was again caused by sampling problems.

Results of weekly measurements of container pressures also indicated the absence of propellant breakdown and/or reaction. Although the pressure measurement results were erratic, significant gross pressure buildups were not noted. Approximate differences between the recorded pressures and the vapor pressure of the ClF_5 at the observed ambient temperature ranged from -7 to 20 psi with no significant differences observed between containers during a particular set of measurements. The difference between the container pressure and the vapor pressure at the recorded temperature was attributed to the following factors:

1. The temperature of the outside thermometer did not accurately reflect the temperature of the ClF_5.
2. No attempt was made to achieve a liquid/vapor equilibrium.
3. After the first few measurements, the indicator needle on the gages began to stick; it was necessary to jar the needle loose. The accuracy of the gages after that point was questionable.

On the basis of these results, there was no evident decomposition, breakdown, and/or gross reaction of ClF_5 when stored under uncontaminated conditions in 321 stainless steel, 6061 aluminum, Monel 400, and oxygen-free copper containers over a 13-month period at temperatures of ~30 to 100 F. These results verify the original conclusions reached in Ref. 1.

Future Effort

The ClF_5 storability tests will be continued over the next several years under a Rocketdyne-sponsored effort. Chemical analyses of liquid and vapor samples will be conducted at yearly intervals and the results will be reported to the appropriate government and industrial laboratories.

VISCOSITY

During the initial 12-month period of the contract, experimental liquid ClF_5 viscosity measurements were conducted with a modified (Zhukev) Ostwald Viscometer over a temperature range of 5 C (41 F) to 20 C (68 F).

Continuation of these measurements over a wider temperature range was planned in the extension of the contract. The additional measurements, which extended the temperature range to -38 C (-36.4 F) and improved the accuracy of the original measurements, were completed during the thirteenth month of the program. Because of this early completion date, it was possible to include all of the data in Ref. 1.

ELECTRICAL CONDUCTIVITY

The specific conductivity and dielectric constant of the ClF_5 liquid and vapor phases were measured during the original 12-month effort. Additional effort on a chemical analysis technique for determining HF content in ClF_5 through use of electrical conductivity or dielectric constant measurements was contemplated during the 12-month extension. However, this work was dropped because of a lack of remaining funding.

ENGINEERING PROPERTIES HANDBOOK

All ClF_5 engineering property data generated in this program, as well as pertinent data derived from other programs, have been collected and will be assembled in a Chlorine Pentafluoride Engineering Properties Handbook under Contract F04-611-67-C-0006.

REFERENCES

1. AFRPL-TR-65-51, Final Report, Preparation and Characterization of a New High Energy Oxidizer, Rocketdyne, a Division of North American Aviation, Inc., Canoga Park, California, April 1965, CONFIDENTIAL.
2. Technical Bulletin T-20, Heating and Pickling Huntington Alloys, The International Nickel Company, Inc., Huntington, West Virginia.

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APPENDIX A

AFRPL-TR-65-51 ERRATA SHEET

1. On page 13, the vapor pressure at 20 C (68 F) should read 3.31 atmospheres (48.7 psia).

2. On page 13, the compressibility (isothermal) at 20 C (68 F) should read 3.04×10^{-4} at m^{-1} (2.07×10^{-5} psi^{-1}).

3. On page 42, line 10 should read. . . found to be 4.066×10^{-8} cm, and β_1 is 2.07×10^{-5} psi^{-1} at 20 C.

4. On page 82, under Group 1, Table 12, 5096 Al should read 5086 Al.

5. On page 88, Table 14, the liquid-exposed samples, ambient-temperature rate of Inconel X750 should be 1.247 instead of 1.9.

   The vapor-exposed samples, ambient temperature rate for Monel K-500 should read 1.03 instead of 1.94; for Inconel X750 it should read 1.25; for 7075 aluminum it should read 0.189; and for 316 stainless steel it should read 1.572.

6. On page 101, the last line should read. . . initiated on 3 November 1964.

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11 SUPPLEMENTARY NOTES

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13 ABSTRACT

The results of long-term, ambient-temperature materials compatibility and storability studies with chlorine pentafluoride (ClF_5) are reported as an extension of efforts conducted under Contract AF04(611)-9563. Duplicate sets of 34 different materials of construction, exposed to the liquid and vapor phases of ClF_5 for a period of 19 months, exhibited corrosion rates similar to those demonstrated previously in 30-day materials compatibility studies under both ambient-temperature and 160 F conditions. Periodic analyses of ClF_5 samples stored at ambient temperatures in 321 stainless steel, 6061 aluminum, Monel 400, and oxygen-free copper for a period of 13 months indicated an absence of propellant decomposition and/or reaction. Other work pertinent to the scope of the program, including the preparation of a Chlorine Pentafluoride Engineering Properties Handbook, is described. (C)

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14 KEY WORDS	LINK A		LINK B		LINK C	
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Compatibility						
Storability						
Corrosion Rates						
Decomposition						

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