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Investigative Report

United States Air Force Wipe Solvent Testing

Lyndon B. Johnson Space Center
White Sands Test Facility
P. O. Box 20
Las Cruces, NM 88004
(505) 524-5011

WSTF-IR-0134

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United States Air Force Wipe Solvent Testing

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Prepared By: ______________________
Jan S. Goldberg
Honeywell Technology Solutions Inc.

Prepared By: ______________________
Rafael H. Delgado
Honeywell Technology Solutions Inc.

Prepared By: ______________________
Paul Biesinger
AlliedSignal Technical Services Corp. Team

Reviewed By: ______________________
Steven D. Hornung
Honeywell Technology Solutions Inc.

Approved By: ______________________
Harold D. Beeson
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1.0 Introduction

The Wright-Patterson Air Force Base (WPAFB), as part of the Air Force Material Command, requested that NASA Johnson Space Center (JSC) White Sands Test Facility (WSTF) conduct testing and analyses in support of the United States Air Force Wipe Solvent Development Project. The purpose of the wipe solvent project is to develop an alternative to be used by Air Force flight line and maintenance personnel for the wipe cleaning of oxygen equipment.

This report provides material compatibility, liquid oxygen (LOX) mechanical impact, autogenous ignition temperature (AIT), and gauge cleaning test data for some of the currently available solvents that may be used to replace CFC-113 and methyl chloroform. It provides data from previous WSTF test programs sponsored by the Naval Sea Systems Command, the Kennedy Space Center, and other NASA programs for the purpose of assisting WPAFB in identifying the best alternative solvents for validation testing.

The primary solvents include:

- AK-225 and AK-225S, manufactured by Asahi Glass America
- HFE-7100 (L-13532), HFE-7200 (L-13556), HFE-301 (L-13791), and HFE-71IPA, manufactured by 3M
- Vertrel XF, Vertrel MCA, and Vertrel X-P10, manufactured by DuPont
- Ikon Solvent P, manufactured by Ikon Corporation

Data from a wide variety of alternative solvents are presented in this report. Under some circumstances, these solvents may be considered potentially hazardous to either human health or the environment, or both. The use of these solvents may be regulated under national or local law in some countries, while it may not be controlled in others. It is important to consider regulations pertinent to maintenance operations when evaluating each alternative solvent.

2.0 Objective

The objective of this report is to summarize the performance data for previously tested solvents in the areas of material compatibility, LOX mechanical impact, AIT, and cleaning efficiency.

3.0 Approach

Material compatibility testing was performed in accordance with Boeing North American (BNA) Space Systems Division procedures as described in Wittman and Garrard (1995). LOX mechanical impact testing was performed according to ASTM G 86 (formerly ASTM D 2512). AIT testing was performed according to ASTM G 72. Cleaning efficiency tests were performed according to WSTF Job Instruction SVC-CSS-0032.[1]

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[1] In-house document. WSTF Job Instruction SVC-CSS-0032. *Final Cleaning and Cleanliness Verification of Hardware in the Class 100 Clean Room*. NASA White Sands Test Facility, Las Cruces, NM, November 24, 1998.

4.0 Experimental

4.1 Material Compatibility Tests

Material compatibility testing for all solvents was performed following a similar test protocol. All material compatibility testing except that for HFE 7100 was conducted by BNA Space Systems Division. The BNA results are reported under the following report numbers:

- LTR 6608-4083 (Wittman and Eichinger 1995)
- LTR 6559-4089 (Gaede, Apel, and Eichinger 1995a)
- LTR 6618-4090 Rev. 1 (Gaede, Apel, and Eichinger 1995b)
- MPR 6718-2000 (Meyer 1996)
- MPR 6933-2001 (Nguyen and Meyer 1997)
- LTR 7005-4076 (Wittman 1997)
- LTR 7195-4020 (Apel, Eichinger, and Syms 1997)
- LTR 6932-4034 (Czerwinski, Wittman, and Syms 1998)
- LTR 7408-4080 (Antaya, Wittman, and Syms 1999)

HFE 7100 testing was conducted at WSTF. Sections 4.1.1 and 4.1.2 summarize the experimental procedures followed for that testing. These procedures are detailed in WSTF-IR-97-0078 (Delgado et al. 1998).

4.1.1 Exposure Conditions

The exposures were carried out in glass jars sealed with polyseal caps. Only one sample was placed in each container with enough test solvent for the sample to be fully immersed, approximately 20 to 50 mL. The samples were exposed at ambient temperature for a period of 90 days. The samples were removed at 30 and 60 day intervals for visual inspection; the percent swell was determined for the elastomeric samples. The samples were then replaced in the same fluid.

Two of the sample materials, titanium in the stressed condition and ethylene propylene rubber (EPR), were also exposed at elevated temperature. This exposure was conducted in sealed glass Fischer-Porter sample vessels with stainless steel caps and either neoprene o-rings or Teflon PTFE seals. These samples were maintained at 71 ± 2 °C (160 ± 3 °F) in a water bath for 24 h.

The compatibility of CRES 17-4 with a 95/5 volume-to-volume MON-3 oxidizer/HFE 7100 mixture was evaluated with a CRES 17-4 coupon and a stressed c-ring in sealed Fisher-Porter sample vessels. The test samples were stored for 90 days at room temperature.

4.1.2 Sample Characterization

The materials were prepared for the exposure test as either coupons or stress samples; the latter were used to test for stress corrosion cracking. An additional sample was prepared as a control and was not exposed. The stress samples were prepared by placing a bolt through a machined c-ring and tightening to 95 percent of yield, which created a sample with transverse tensile stress. The outer diameter, measured between the two bolt holes, and edges of the c-ring were polished before assembly. The metallic coupon samples were polished on one face. All of the coupons were weighed, measured, and in the case of the nonmetals, the hardness was determined using a Shore A or Shore D durometer both before and after exposure. The percent swell of the elastomers, as a function of thickness only, was determined both immediately after removal from the test fluid and after being air-dried for one week. For those samples exhibiting any change as a result of exposure, their respective fluids were evaporated to dryness after test, and the nonvolatile residue (NVR) was analyzed by either infrared spectrophotometry or energy dispersive x-ray spectroscopy, or both. The MON-3 oxidizer/HFE 7100 mixtures were evaporated, and the NVR was analyzed using ion chromatography.

4.2 LOX Mechanical Impact Tests

LOX mechanical impact tests were performed according to ASTM G86 (formerly ASTM D 2512). Because of the evaporation rate of test solvents at room temperature, sample weight, thickness, and diameter could not be measured. For each test, a volume of approximately 0.3 mL of the test solvent was placed into sample cup approximately 1.783 cm (0.702 in.) in diameter. The test medium was 100 percent LOX at a temperature of -183 °C (-297 °F) and at WSTF ambient pressure, 85.5 kPa (12.4 psia).

According to this test procedure, a material is considered to have passed if the test produces no reactions in 20 impacts at any energy level or not more than one reaction in 60 impacts at 72 ft-lbf. The test typically consists of 20 impacts at any energy level except 72 ft-lbf, but will be extended at 72 ft-lbf to 60 if only one reaction is produced during the first 20 impacts. The commonly recommended energy levels are 72, 65, 60, 55, 50, 45, 40, 35, 30, 25, 20, 15, and 10 ft-lbf.

4.3 AIT Tests

AIT tests were performed according to ASTM G 72. In most cases, the material was tested three times in 100 percent oxygen at a pressure of approximately 345 kPa (50 psia) and five times in 100 percent oxygen at a pressure of approximately 13.8 MPa (2000 psia). Some solvents were tested at 7.2 MPa (1050 psia). The average sample weight for each test was 0.22 ± 0.01 g. The heating rate in the reaction vessel was 5 ± 1 °C (9 ± 2 °F) per minute for the entire heating range. The maximum temperature of the reaction vessel was set at 450 °C (842 °F). The sample weight, starting temperature, starting pressure, AIT, and pressure at ignition for each test were recorded.

The average AIT was calculated for each set of tests. With regard to AIT results, discussion between WSTF and WPAFB led to an agreement of the following acceptance criteria:

Category A	AIT <250 °F	Not recommended for use in oxygen systems
Category B	AIT 250 to 400 °F	Caution when used in oxygen systems
Category C	AIT >400 °F	Recommended for use in oxygen systems

4.4 Cleaning Efficiency Tests

The cleaning efficiency of the test solvents was determined by contaminating Bourdon gauges and simulated Bourdon gauges (16 in. long sections of 0.125 in. dia stainless steel tubing) with a five-component mixture of common contaminants, rinsing the test articles with 200 mL of the test solvents, followed by a final rinse with 100 mL of CFC-113. An NVR analysis was performed on the CFC-113 rinse, and the difference between the initial contaminant loading and the final CFC-113 rinse NVR was taken to be the amount removed by the test solvent. Cleaning efficiency was calculated as the percent of initial contaminant loading removed.

4.4.1 Test Article Precleaning and Contamination

Each test article was cleaned using standard WSTF cleaning procedures, and the cleanliness level was verified to meet Level 50A per JPG 5322.1, Rev. D (2000). The standard contaminant solution was prepared by adding 1 g each of Krytox®[1] 240AC, MIL-H-5606 and MIL-PRF-83282 hydraulic fluids, Spinesstic 22®[2], and sebacate to 100 mL of CFC-113; each 100 µL of the CFC-113 solution contained 5 mg of the contaminant mixture. The cleaned and verified article was weighed and contaminated with a known amount of standard solution, and the CFC-113 solvent carrier was allowed to evaporate. The amount of contaminant was verified by injecting the same amount of standard solution injected into the test article into a tared Petri dish and allowing the CFC-113 to evaporate. The Petri dish was then reweighed, and the contaminant weight was recorded.

4.4.2 Nonvolatile Residue Analysis

NVR was determined gravimetrically by evaporating 100 mL of test solvent to a volume of 10 mL. The concentrated fluid was transferred to a tared Petri dish and heated in an oven at 105 °C (221 °F) for 30 min. The remaining residue was weighed and recorded.

5.0 Results and Discussion

5.1 Material Compatibility Tests

Results for the material compatibility tests for HFE 7100, performed at WSTF, are reported in WSTF-IR-97-0078. Results for material compatibility tests for AK-225 and Vertrel MCA, performed by BNA, are reported in LTR 6673-4093. Material compatibility results for Ikon Solvent P are contained in a proprietary report; inquiries should be directed to Mr. Ed Snyder at (937) 255-9036.

All of the metals and stressed metals tested by BNA were generally compatible with the solvents tested. Of all the metals and stressed metals tested by WSTF, only tungsten carbide and Custom 455 c-ring showed some corrosion when exposed to HFE 7100.

The compatibility results for elastomers and polymers with HFE 7100 are shown in Table 1. Only Kalrez 1045 and Viton V0747 showed some swelling when exposed to HFE 7100. The compatibility results for elastomers and polymers with AK-225, Vertrel MCA, and CFC-113 are shown in Table 2. The results indicate that EPR E740, Neoprene C557, and Viton V0747 showed significant effects when exposed to Vertrel MCA. Viton V0747 also showed significant effects when exposed to CFC-113 and AK-225. All lubricants and coatings tested with these solvents showed general compatibility.

5.2 LOX Mechanical Impact and AIT Tests

AIT test results for solvents that passed LOX mechanical impact testing at 72 ft-lbf are summarized in Table 3. The recommendation (i.e, Category A, B, or C) is based solely on the criteria stated in Section 4.3 and does not consider toxicity, material compatibility, or cleaning ability. The solvents that did not pass LOX mechanical impact testing at 72 ft-lbf are listed in Table 4.

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[1] Krytox® is a registered trademark of E. I. DuPont de Nemours and Co.

[2] Spinesstic® is a registered trademark of Exxon Corp.

5.3 Cleaning Efficiency Tests

Results of the cleaning efficiency tests performed at WSTF are listed in Table 5, where they are hyperlinked to the appropriate WSTF data report. AK-225, HFE-71DE, and Vertrel MCA showed the highest cleaning efficiency, with 99, 98, and 98 percent, respectively. Those solvents with the lowest cleaning efficiency were tetrachloroethylene at 87 percent, ethanol at 85 percent, and Vertrel XF at 84 percent.

6.0 Conclusions

Based on results to date of testing conducted at WSTF or analyzed in this report, the following can be concluded.

- All the solvents tested for compatibility were in general compatible with those metals tested, and their compatibililty with nonmetals varied from solvent to solvent. The user should refer to the individual hyperlinked reports for appropriate solvent compatibility, particularly for nonmetals, before designing or changing process specifications.

- The LOX mechanical impact test, a pass/fail test, and the AIT test were useful for distinguishing the oxygen compatibility of the various solvents tested in this program. Those solvents that performed best in oxygen compatibility testing were AK-225, AK-225G, and HFE 7100.

- Several solvents showed better or nearly equivalent cleaning efficiency to CFC-113. Other solvents showed acceptable performance and may be chosen according to environmental or toxicological concerns.

Table 1

Results of Elastomer and Polymer Compatibility Testing with HFE 7100

Material	Results 30 Day		Results 60 Day		Results 90 Day		Hardness[b]	Posttest Description[c]
	Swell (%)[a]	Weight Change (%)[a]	Swell (%)[a]	Weight Change (%)[a]	Swell (%)[a]	Weight Change (%)[a]		
Teflon PTFE	5	1	2	2	2	2	57 (58)	No visual change
Vespel SP-21	0	-1	2	< 1	-1	1	84 (84)	No visual change
Kalrez 1045	21	51	21	50	20	50	60 (80)	Swelling, clear fluid
EPR E740-75	-1	1	1	2	-1	2	68 (69)	No visual change
EPR E740-75 (@ 50 °C for 24 h) [a]	< 1	1	N/A	N/A	N/A	N/A	68 (70)	No visual change
Rulon AR	-5	4	-5	4	-8	4	50 (57)	No visual change
Kynar Grade 460	-6	< 1	-6	< 1	-7	< 1	79 (76)	No visual change
Neoprene C557	-1	-1	1	-1	0	-1	63 (65)	No visual change
Viton V0747	12	27	12	28	11	27	64 (75)	Swelling, clear fluid
15% Glass Filled PTFE Teflon	5	5	3	5	5	5	51 (60)	No visual change
Butyl Rubber B318-70	1	< 1	1	< 1	< 1	1	61 (63)	No visual change
Kel-F 81	< 1	0	1	< 1	-1	< 1	77 (76)	No visual change

[a] Data collected immediately after removal from test fluid.

[b] Shore hardness (A or D) measured after 90-day immersion in test fluid; the number in parenthesis indicates the pretest hardness.

[c] Observations from comparison of control sample and exposed sample after removal from 90-day test fluid immersion.

Table 2

BNA Results of Materials Compatibility Testing With CFC-113, AK-225, and Vertrel MCA (90 d Exposure at 22 °C)

Sample Number	Material	Fluid	Swell[1] %	Swell, After 1 Week (%)	Shore[2]	Weight Change (%)[3]	Comments
1	Teflon PTFE	CFC-113	1	1	56(60)	1	No visual change in appearance
		AK-225	2	2	55(60)	2	
		Vertrel MCA	1	1	54(59)	2	
2	Vespel SP-21	CFC-113	<1	<1		<1	No visual degradation evident
		AK-225	<1	<1	N/A	<1	
		Vertrel MCA	4	2		7	
3	Kalrez 1045	CFC-113	15	2	70(78)	8	No visual degradation evident
		AK-225	19	4	67(77)	7	
		Vertrel MCA	14	3	70(77)	7	
4a	EPR E740	CFC-113	23	<1	77(73)	-4	Slightly sticky/tacky residue found on Vertrel MCA-exposed sample only; NVRs of 22, 10, and 3 mg respectively; EDS shows base C for all three NVRs
		AK-225	6	<1	76(74)	-2	
		Vertrel MCA	15	<1	76(75)	-1	
4b	EPR E740	CFC-113	22	<1	75(75)	-3	Slightly sticky/tacky residue found on Vertrel MCA-exposed sample only
		AK-225	5	<1	75(76)	-2	
		Vertrel MCA	22	<1	75(76)	<-1	
5	Rulon AR	CFC-113	1	<1	60(63)	1	No visual change in appearance
		AK-225	2	1	61(65)	1	
		Vertrel MCA	<1	<1	61(64)	1	
6	Kynar	CFC-113	<1	<1	81(80)	<1	No visual change in appearance
		AK-225	<1	<1	81(80)	<1	
		Vertrel MCA	<1	<1	79(80)	<1	
7	Neoprene C557	CFC-113	5	<1	75(73)	3	White bloom on Vertrel MCA-exposed sample; faint trace only on CFC-113-exposed sample
		AK-225	3	<1	76(73)	<1	
		Vertrel MCA	9	<1	75(73)	<1	Yellow NVR from all three samples, with white particulate in Vertrel MCA NVR only (38, 36 and 16 mg of residue, respectively); EDS for CFC-113: b. maj. C; 1. maj. O; 1.min.S; trace Cl, Ca, Si Vertrel MCA: (white particle) maj. Mg, F; h. min. Na,Cl;1.min. S, Ca, K
8	Viton V0747	CFC-113	8	5	73(77)	12	Very obviously swollen samples
		AK-225	23	4	73(77)	10	NVRs of 1, 10, 15 mg, respectively
		Vertrel MCA	29	4	71(77)	10	EDS: C, F for all three NVRs

Table 3

AIT Results for Solvents That Passed LOX Mechanical Impact Testing at 72ft-lbf[1]

Solvent	AIT	Category
CFC-113 (Trichlorotrifluoroethane)	@ 50 psia, no ignition @ 2000 psia, no ignition	C
Genetron 141b	@ 50 psia, no ignition @ 2000 psia, no ignition	C
Genesolv 2000/HCFC-141b (20 ppm NVR Blend)	@ 50 psia, no ignition @ 2000 psia, 514 °F	C
AK-225 AES	@ 1050 psia, 474 °F	C
AK-225	@ 50 psia, no ignition @ 2000 psia, no ignition	C
Tetrachloroethylene	@ 50 psia, 276 °F @ 2000 psia, 323 °F	B
Trichloroethylene	@ 50 psia, 226 °F @ 2000 psia, 171 °F	A
Vertrel SMT w/o Nitromethane	@ 1050 psia, 394 °F	B
HFE 7100	@ 50 psia, no ignition @ 2000 psia, no ignition	C
HFE 7200	@ 50 psia, no ignition @ 2000 psia, 503 °F	C
HFE 301	@ 50 psia, no ignition @ 2000 psia, no ignition	C
HFE 301 (77.6%) / Trans-1,2-DCE (22.4%) Azeotropic Mixture	@ 50 psia, no ignition @ 2000 psia, no ignition	C
Vertrel XF	@ 50 psia, no ignition @ 2000 psia, 467 °F	C

[1] The solvent hyperlink is to the appropriate LOX mechanical impact test data report; the AIT hyperlink is to the appropriate AIT test data report.

Table 4
Solvents that Failed LOX Mechanical Impact Testing

EnSolv Precision Vapor Degreasing and Cleaning Solvent
HFE-71DE
HFE-71IPA
Ikon Solvent P
OS-10 Volatile Methylsiloxane Fluid
PFC 265-163
Vertrel MCA
Vertrel SMT w/Nitromethane
Vertrel X-P10
Vertrel XF/Dichloroethylene 62/38 Blend

Table 5
Solvent Cleaning Efficiencies

Solvent	Cleaning Efficiency (%)
AK-225	99
HFE-71 DE	98
Vertrel MCA	98
CFC-113	97
HCFC 141B	97
AK–225S	96
HFE 301/HCFC 141B Blend	96
HFE 301 77.6% /Trans-1,2-DCE (22.4%) Azeotropic Mixture	96
HFE 301/L-11412 Blend	95
HFE 301/Krytox Alcohol Blend	92
HFE 7100	92
Trichloroethylene	92
HFE 301	90
HFE 301/FC-170-C Blend	88
Tetrachloroethylene	87
Ethanol	85
Vertrel XF	84

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