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Results of the Test Program for Replacement of AK-225G Solvent for Cleaning NASA Propulsion Oxygen Systems

ABSTRACT: Since the 1990's, when the Class I Ozone Depleting Substance chlorofluorocarbon-113 was banned, NASA's propulsion test facilities at Marshall Space Flight Center and Stennis Space Center have relied upon the solvent Asahiklin AK-225 (hydrochlorofluorocarbon-225ca/cb or HCFC-225ca/cb) and, more recently AK-225G (the single isomer form, HCFC-225cb) to safely clean and verify the cleanliness of large scale propulsion oxygen systems. Effective January 1, 2015, the production, import, export, and new use of Class II Ozone Depleting Substances, including AK-225G, was prohibited in the United States by the Clean Air Act. In 2012 through 2014, NASA test labs at MSFC, SSC, and Johnson Space Center's White Sands Test Facility collaborated to seek out, test, and qualify a solvent replacement for AK-225G that is both an effective cleaner and safe for use with oxygen systems. This paper summarizes the tests performed, results, and lessons learned.

KEYWORDS: solvent, oxygen cleaning, ozone depletion, HCFC-225, cleanliness verification, nonvolatile residue

Introduction

Liquid and gaseous oxygen (LOX/GOX) systems used in bipropellant propulsion systems require a high level of cleanliness. Systems to be wetted by gaseous or liquid oxygen, and systems providing pressurization and/or purge (P&P) gases to these systems, must be clean, particularly of hydrocarbons and large ($> 800 \mu$) particles [1], to avoid the potential hazard of a reaction and subsequent fire or explosion. Solvents used to clean and verify the cleanliness of oxygen systems and supporting test hardware must be compatible with the materials of construction of these systems and effective at removing expected contaminants to the level required. When complete removal of residual cleaning solvent from the component or system cannot be verified with a high level of confidence, the solvent must also be minimally reactive with LOX/GOX at the system use conditions.

Historically, chlorofluorocarbon-113 (CFC-113) solvent was used for these applications. When CFC-113, a Class I ozone depleting substance (ODS), was banned in the 1990's, a Class II ODS, hydrochlorofluorocarbon-225³ (HCFC-225ca/cb, a dual isomer form, and later HCFC-

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³ Manufactured by Asahi Glass Company, Ltd., Tokyo, Japan; supplied in North America by AGC Chemicals Americas, Exton, PA, under the product name Asahiklin AK-225. AK-225 is a blend of two isomers, 3,3-Dichloro-1,1,1,2,2-pentafluoropropane (HCFC-225ca) (Chemical Abstract Service [CAS] Registry Number 422-56-0), and 1,3-Dichloro-1,1,2,2,3-pentafluoropropane (HCFC-225cb) (CAS Number 507-55-1). References to HCFC-225 in this paper refer to any form of this chemical, as the ca or cb isomer or a blend of both isomers. AK-225 refers specifically to the Asahiklin ca/cb isomer blend. Asahiklin AK-225G is $>99\%$ HCFC-225cb.

225cb, the single isomer form), was selected by the Space Shuttle Program and NASA propulsion test facilities to replace CFC-113 for final cleaning and verification of oxygen systems hardware where a suitable non-ODS replacement could not be found. Effective January 1, 2015, the production, import, export, and new use of HCFC-225 was banned in the United States.

An Inter-Center NASA test plan was developed for evaluating solvents to be used for cleaning of propulsion oxygen systems and associated hardware at Marshall Space Flight Center (MSFC) and Stennis Space Center (SSC), and for sampling of these systems for verification of cleanliness. In this test program, Asahiklin AK-225G (>99% HCFC-225cb) was used as the baseline solvent for comparison of cleaning efficiency and materials compatibility. The goal was to identify a single replacement solvent that meets or exceeds the performance of AK-225G in all rocket propulsion oxygen system applications at MSFC and SSC.

Background

MSFC in Huntsville, Alabama; MSFC's Michoud Assembly Facility (MAF) in New Orleans, Louisiana; and SSC on the Gulf Coast of Mississippi are the principal users of HCFC-225 at NASA. HCFC-225 is used at MSFC and SSC primarily for precision cleaning and verification of cleanliness of launch vehicle propulsion oxygen systems hardware, ground support equipment (GSE), and associated test systems. HCFC-225 was used at MAF for processing of Space Shuttle External Tank hardware until the retirement of the Space Shuttle in 2011. Future need is anticipated for a replacement for HCFC-225 for processing of oxygen system components for the next NASA launch vehicle, the Space Launch System. Two other NASA locations, Johnson Space Center's White Sands Test Facility (WSTF) and Goddard Space Flight Center have reported low levels of HCFC-225 usage primarily for laboratory operations.

During the time frame that this project was being formulated the Defense Logistics Agency (DLA) – Hazardous Minimization and Green Products Branch funded a project for MSFC to identify and test two candidate solvents for replacement of HCFC-225.⁴ The candidate solvents tested in the DLA study were Honeywell Solstice™ Performance Fluid (PF) and 3M L-14780 developmental solvent. These solvents were compared to two baseline solvents, AK-225G and DuPont™ Capstone® 4-I⁵. Capstone 4-I, a chemical intermediate composed primarily of perfluorobutyl iodide (PFBI), was provided by DuPont as a substitute for DuPont Ikon® Solvent P, a cleaning solvent that had been discontinued by DuPont.

The tests performed for the DLA study were based on those reported in AFRL-ML-WP-TR-2003-4040, *The Wipe Solvent Program*. [2] Oxygen compatibility tests for the DLA study were performed at the MSFC Materials Combustion Research Facility. The tests for the DLA study were complimentary and provided very useful input to the test program reported here. Data obtained during the performance of the DLA study was used to supplement and inform the down-selection process during the course of this project.

⁴ Military Interdepartmental Purchase Request SC04001200481, *Solvent Replacement for HCFC-225 for Cleaning Oxygen System Components*, final report delivered August 26, 2014.

⁵ Earlier trade names used by DuPont for their family of PFBI-based products were Zonyl® and Ikon®. Capstone® 4-I is marketed by DuPont only as a chemical intermediate.

Test Plan Development

The test plan was developed jointly by a team of Materials and Processes (M&P) engineers, laboratory personnel, and end users from MSFC, SSC, and WSTF, and reviewed by the NASA Engineering and Safety Center (NESC). The solvent selection criteria described in ASTM G 127, *Standard Guide for the Selection of Cleaning Agents for Oxygen Systems*, were referenced for the development of this test plan. [3] The materials and contaminants specified in this plan were selected by an engineering team from MSFC and SSC using ASTM G 127 and ASTM MNL36 *Safe Use of Oxygen and Oxygen Systems* as a guide; [4] considering historical and current propulsion system designs; and with input from users at MSFC and SSC propulsion test facilities and cleaning facilities regarding cleaning challenges with LOX/GOX systems encountered at their locations. Test reports from the 1990's and 2000's to qualify HCFC-225 as a replacement for CFC-113 were also reviewed to capture previous test methods where applicable. [5 – 9]⁶

The test procedures, metals and nonmetals to be tested for compatibility, and contaminants to be tested to compare cleaning effectiveness of the solvent candidates were detailed in the test plan. The test program was managed by the MSFC M&P Laboratory Environmental Effects Branch Contamination Control Team and, at the request of the Rocket Propulsion Test Program manager, monitored by NESC representatives from MSFC and SSC. Test responsibilities were assigned to laboratories at MSFC, SSC, and WSTF based on the availability of laboratory facilities and expertise.

Tests were scheduled to provide data to support Technical Interchange Meetings (TIM) to narrow the list of candidates. At these TIMs the engineering team reviewed the data gathered to date and selected the most promising candidates to proceed to the next phase of tests. Measurement of the nonvolatile residue (NVR) background of each cleaning solvent was performed prior to use, therefore this test was repeated several times during the course of the project when new lots of material were received from the supplier. The test sequence and down selection milestones were as follows:

Test Phase 1: NVR background of neat solvent and quick screen solvency tests.

First Down-Selection: Select three solvents to proceed to phase 2.

Test Phase 2: Metals compatibility, nonmetals compatibility, and initial oxygen compatibility tests.

Second Down-Selection: Select two solvents to proceed to phase 3.

Test Phase 3: NVR removal efficiency and oxygen compatibility tests.

Final Down-Selection: Select one solvent to proceed to phase 4. If considered a viable alternative, then the solvent not selected was to be retained as a backup should any insurmountable issues arise during the final test phase.

⁶ In addition to the published references, NASA Materials and Processes Technical Information System (MAPTIS) reports for material codes 01229 (AK-225) and 04619 (AK-225G) were also reviewed.

Test Phase 4: Component level cleaning tests (field cleaning) and assessment of implementation requirements.

Initial results from the ambient pressure LOX Mechanical Impact ignition tests performed at WSTF were found to be significantly different from those performed at MSFC for the DLA study following the same ASTM test method. Additional oxygen compatibility tests beyond the scope of the initial test plan were performed at MSFC and WSTF to support an independent assessment of the test variables and conditions that could affect the results when testing the reactivity of liquids in LOX and to provide final input for an oxygen compatibility analysis. Heat of Combustion (HOC) tests were also performed on the final two solvents and AK-225G to provide comparative data to support the oxygen compatibility analysis.

Selection of Solvent Candidates

During the initial phase of the DLA study, the critical performance parameters were identified and a thorough survey of the cleaning solvent industry was performed to identify promising candidates for cleaning oxygen systems. Solvent manufacturers and major blenders were contacted to identify new solvents that were in the developmental phase, but could be made available for test. A summary of this search was presented at the 2012 International Workshop on Environment and Alternative Energy; 4-7 Dec. 2012; Greenbelt, MD; United States. [10]

The solvent selection criteria fell into two primary categories: Safety, Health and Environmental (SHE) characteristics, and technical performance parameters. Should more than one solvent be found to perform acceptably, cost and availability could determine the final selection. Expected cost and availability were considered during the initial evaluation, but were secondary to the SHE and technical performance criteria.

Safety, Health and Environmental Characteristics

Solvents that were classified by the United States Environmental Protection Agency (EPA) as a Class I or Class II ODS or contained a constituent classified as a Hazardous Air Pollutant were not accepted as test candidates. In addition, chemicals classified as human carcinogens were not accepted for consideration.

Solvents that meet NASA criteria for oxygen compatibility are expected to be inherently safer for personnel from a flammability perspective. The toxicity of solvent candidates was evaluated based on Acceptable Exposure Limits (AEL), 8-hour Time Weighted Average or equivalent, as reported in the solvent Safety Data Sheet (SDS). Solvents with the highest AEL were the most preferred.

Solvents were preferred that were lower in Volatile Organic Compound (VOC) content or VOC exempt as determined by the EPA; and lower in 100-Year Global Warming Potential (GWP) as published in assessment reports by the Intergovernmental Panel on Climate Change. [11] Solvents were also preferred based on expected ease of recapture, re-purification, and re-use. Bio-based products as defined by the United States Department of Agriculture Bio-

Preferred Program were considered during the search for candidate solvents. No bio-based products met the screening criteria.

Technical Performance Criteria

Technical performance criteria included materials compatibility, cleaning effectiveness, and oxygen compatibility. Boiling point was also considered because solvents with high boiling points can be difficult to dry and solvents with boiling points lower than common ambient temperatures may require pressurized storage and may evaporate too quickly for practical use.

Materials compatibility was evaluated based on vendor information and published literature. Solvents must not be corrosive to metals used in the construction of oxygen system hardware and existing test systems. Aqueous cleaning products, known to be corrosive to many metals when inadequately rinsed and dried, were not considered as candidates to replace HCFC-225 due to the difficulty of cleaning large scale propulsion test systems in the field without corrosion risks.

HCFC-225 and other halogenated solvents are known to be incompatible with some nonmetals. Better compatibility with nonmetals used in oxygen systems is preferred but was not an initial selection criterion.

The solvent used to clean oxygen systems must be capable of removing contaminants that pose a safety hazard to oxygen systems. This includes particulate, and hydrocarbon-based hydraulic fluids, oils, and greases. [1] [3] [4] Silicone oils are also of concern, but are generally prohibited around NASA propulsion system hardware and thus were not included in the test program. Solvents are preferred that can also remove halogenated greases which are commonly used with oxygen systems due to their low reactivity. The solvent must be capable of removing NVR to below $1 \text{ mg}/0.1 \text{ m}^2$ ⁷, the pass/fail criterion for most NASA LOX/GOX propulsion system components, when cleaning is performed by ambient temperature flush. Due to the large scale of launch vehicle components and test systems, cleaning methods that rely on mechanical action (e.g. ultrasonic agitation) and/or high temperatures are not practical in many cases. Ambient temperature flush was identified by the test team as the most challenging cleaning process for solvent performance at MSFC and SSC.

Kauri-butanol (Kb) value as reported by the vendor, determined in accordance with ASTM D1133 *Standard Test Method for Kauri-Butanol Value of Hydrocarbon Solvents*, was used to estimate the cleaning power of the solvent. [12] A review of past test data showed that solvents with Kb < 20 performed poorly at removing the soils of concern for oxygen systems. When a Kb value was not available, cleaning performance was estimated based on vendor literature and other published data.

Wetting Index has been used to estimate the ability of a cleaning agent to enter small orifices and lift particulate from surfaces. Solvents with a higher wetting index are expected to perform better at removal of particulate. The Wetting Index is calculated as:

⁷ NASA specifications historically stated requirements for NVR in mg/ft². In later revisions, these units were changed to mg/0.1 m² with the footnote: "For the purpose of this specification 0.1 square meter = 1 square foot."

$$\text{Wetting Index} = (1000 \times \text{density}) / (\text{surface tension} \times \text{viscosity}) \quad (1)$$

Solvents were preferred that had both a high Kb value and a high wetting index.

Due to the potential consequences of a fire, solvents used for final cleaning and verification of NASA propulsion test systems must demonstrate very low reactivity in oxygen. Solvents that reported a flash point in air on the SDS were considered unacceptable. Solvents with no lower or upper explosion limit as reported on the SDS and solvents that demonstrated favorable performance on previous NASA oxygen compatibility tests were preferred as test candidates.

Many fluorinated solvents with low Kb value are blended with trans-1,2 dichloroethylene (tDCE), alcohols, or other solvents to boost cleaning power. Nonflammable fluorinated constituents suppress the flammability of tDCE and alcohols. Only true azeotropic blends were considered as candidates. Historical solvent flammability data in MAPTIS showed that all blends containing an alcohol failed the ambient LOX Mechanical Impact Ignition test. Therefore, solvents containing any alcohol were excluded. Also, MAPTIS data showed that solvent blends higher in tDCE content tended to fail this test. While insufficient data was available to establish a tDCE threshold, blends containing more than 50% tDCE were considered highly unlikely to be compatible with LOX/GOX and were eliminated from the candidate list.

Solvents Selected as Test Candidates

The solvents selected for this test program were:

- AGC Chemicals Americas (distributor for Asahi Glass Company, Japan): Asahiklin AE3000 (1,1,2,2-tetrafluoroethyl-2,2,2-trifluoroethyl ether [hydrofluoroether (HFE)-347pc-f2] [CAS 406-78-0]).
- AGC Chemicals Americas: Asahiklin AE3000AT (45% trans-1,2 dichloroethylene [CAS no. 156-60-5]/ 55% 1,1,2,2- tetrafluoroethyl-2,2,2-trifluoroethyl ether [HFE-347pc-f2] [CAS 406-78-0] azeotrope).
- E. I. du Pont de Nemours and Company: Capstone 4-I, chemical intermediate (perfluorobutyl iodide [CAS no. 423-39-2], with other constituents).
- 3M: L-14780 Developmental Solvent⁸ (22% trans-1,2 dichloroethylene [CAS no. 156-60-5]/ 78% methyl perfluoropropyl ether [HFE-347mcc3] [CAS no. 375-03-1] azeotrope).
- Solvay Fluorides, LLC: Solkane[®] 365 mfc (1,1,1,3,3 pentafluorobutane [CAS no. 406-58-6]).

⁸ 3M does not currently market this blend of solvents. "L-14780 Development Solvent" is the 3M designation for the azeotropic blend of two commercially available products, 3M Novec[™] 7000 (methyl perfluoropropyl ether) and trans-1,2 dichloroethylene. This product also contains stabilizer chemicals in concentrations below 1%.

- Solvay Fluorides LLC: Solvokane[®] (30% trans-dichloroethylene [CAS no. 156-60-5]/ 70% HFC-365 mfc 1,1,1,3,3 Pentafluorobutane [CAS no. 406-58-6] azeotrope).
- Honeywell: Solstice Performance Fluid (PF) (trans-1-chloro-3,3,3,-trifluoropropene [HCFO-1233zd(E)] [CAS no. 102687-65-0]).
- E. I. du Pont de Nemours and Company: Vertrel[®] MCA (38% trans-dichloroethylene [CAS no. 156-60-5]/ 62% 1,1,1,2,2,3,4,5,5,5-Decafluoropentane azeotrope [HFC-43-10mee] [CAS No. 138495-42-8]). This solvent contains a different stabilizer than some Vertrel MCA formulations previously tested by NASA.

Solvent samples were provided by the suppliers for this test program. AGC Chemicals Americas was unable to obtain test samples of AE3000 and AE3000AT from Japan in time to meet the schedule requirements. Vertrel MCA did not fully pass previous NASA oxygen compatibility tests [5], but was used for more than 10 years at NASA's Kennedy Space Center as the first step in a two-step cleaning process. This two-step process was identified as a fallback option for MSFC and SSC if no solvent was found to be acceptable as a replacement for HCFC-225 in a single step process.

For these tests, AK-225G solvent was supplied to the test labs at MSFC and SSC from the production facilities. This solvent was drawn from production stock and verified to meet the NVR requirements of ≤ 2 ppm. It may have been recycled and distilled prior to delivery for test.

Nonvolatile Residue Background in Neat Cleaning Solvents

NVR background in each solvent was tested at both MSFC and SSC on receipt of a new lot of material. The AK-225G that was supplied by MSFC and SSC production facilities and the Solstice PF were very low in NVR. The other solvents except Capstone 4-I were moderately low in NVR and varied between labs and between gravimetric and Fourier Transform Infrared (FTIR) test methods. This was noted for future investigation to determine whether these differences were due to lot-to-lot variability, solvent stability, NVR type, or other factors. The Capstone 4-I was the highest in NVR background and required filtering to remove a large quantity of particulate.

A calibrated FTIR method is used at MSFC and SSC propulsion test facilities for measurement of NVR in AK-225G for verification of solvent purity and for measurement of NVR in the solvent rinsed from a cleaned part to verify cleanliness. Ideally, the solvent rinse sample would be injected directly into the infrared (IR) liquid cell to measure NVR. CFC-113 was used this way. AK-225G, however, produces IR peaks in the same wavelength range as the hydrocarbon contaminants being measured. When measuring NVR in AK-225G, the residue is dried and then reconstituted in tetrachloroethylene for FTIR analysis. None of the solvent candidates were compatible with direct measurement in the IR cell. Furthermore, the L-14780 appeared in some tests to leave a trace constituent in the residue that may interfere with the FTIR analysis.

Quick Screen Solvency, Miscibility, and Odor Studies

As a quick way to evaluate the cleaning power of the candidate solvents, each candidate was challenged with a mixed batch of contaminants representative of cleaning challenges for propulsion oxygen system components. The contaminant mix was prepared by dissolving approximately 0.5 g of equal parts of the following contaminants in 100 ml of AK-225G:

- a. Mineral oil – pharmaceutical grade – mixed aliphatic
- b. MIL-PRF-83282 – ester based hydraulic fluid [13]
- c. Di-2-ethylhexylsebacate (gauge calibration oil), MONOPLEX[®] DOS (The C. P. Hall Co.)
- d. WD-40[®] (medium-heavy hydrocarbons) (WD-40 Company).
- e. Krytox[®] GPL103 (fluorocarbon lubricant for oxygen systems)

Aluminum weighing pans were doped with the contaminant mix, dried, weighed, and then cleaned by flushing with 100 ml of the test solvent in three steps, 30 ml, 30 ml, and then 40 ml. The solvent flush was collected in a clean beaker that was then dried and weighed to determine NVR removed. Each solvent was tested ten times and the results were averaged. The NVR cleaning efficiency (%) is reported as:

$$\text{Percent Cleaning Efficiency} = (\text{mg of NVR removed} / \text{mg of NVR applied}) \times 100 \quad (2)$$

The average cleaning efficiency results are shown in figure 1.

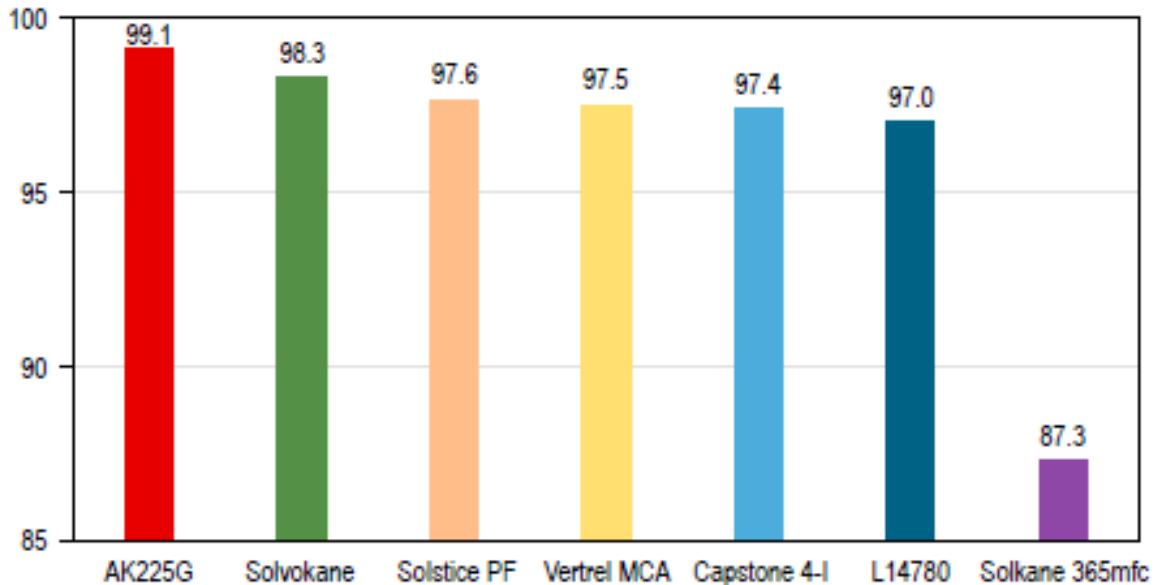


Figure 1 - Solvent Screening Test Results for Cleaning Efficiency

During the quick look screening tests at SSC and cleaning efficiency tests performed at MSFC for the DLA study, it was noted that some contaminants in high doses tended to separate from several candidate solvents. The miscibility of various contaminants in the candidate solvents was investigated further during the quick look screening tests. Solstice PF and AK-225G showed a significantly higher saturation capacity than the other test candidates for the contaminants tested: MIL-PRF-83282 hydraulic fluid, RP1 petroleum-based rocket propellant, and heavy weight petroleum-based gear oil.

During the screening tests, SSC laboratory chemists were asked to provide general user feedback on the candidate solvents. The solvents were scored on a scale of 1-10 on odor strength, 1 being most favorable. AK-225G, Solstice PF, and L-14780 scored 5 or below. Solkane 365 mfc scored a 9 and Capstone 4-I, Vertrel MCA, and Solvokane scored a 10. Only the odor of the Capstone 4-I was considered to be highly objectionable.

First Down-Selection

To narrow the test set to three candidates, data from NVR background tests and the quick screen solvency tests were reviewed. Initial observations were shared regarding solvent odor, contaminant saturation capacity, and evaporative characteristics. Data obtained to date from the parallel DLA study on Solstice PF, L-14780, and Capstone 4-I was also shared. MSFC reported that these solvents performed well in the cleaning tests and the oxygen compatibility tests, but that Capstone 4-I was highly contaminated with particulate, was corrosive to the metals tested, and appeared to be unstable, rapidly changing color during test activities.

Solstice PF, Solvokane, and L-14780 were selected for further testing. All three solvents performed well in the quick screening solvency test and demonstrated other favorable characteristics.

Solstice PF demonstrated good cleaning efficiency (>97%), and had the most favorable SHE profile. Based on MSFC oxygen compatibility test data from the DLA study, it was expected to pass oxygen compatibility tests to be performed at WSTF. It was also observed during initial handling of Solstice PF that despite the low boiling point of 19°C (66°F) the solvent did not rapidly boil away and could be used for cleaning processes similarly to AK-225G and other higher boiling point solvents.

L-14780 demonstrated good cleaning efficiency (>97%). Based on historical test data from WSTF and MSFC oxygen compatibility test data from the DLA study, it was expected to again pass the oxygen compatibility tests to be performed at WSTF. The environmental data on this material was not as favorable as Solstice PF, but was more favorable than other candidates that were higher in VOC content and GWP.

Solvokane demonstrated good cleaning efficiency (>98%). Although the Solkane 365 mfc component is relatively high in GWP, it was the least toxic constituent of the azeotropes evaluated. No data was available at this point on oxygen compatibility.

Metals Compatibility

The corrosiveness of Solstice PF, L-14780, and Solvokane on metals was evaluated under conditions of total immersion in accordance with ASTM F 483-09, *Standard Practice for Total Immersion Corrosion Test for Aircraft Maintenance Chemicals* and in solvent vapor for comparison. [14] AK-225G was also included in this test set for comparison.

Six test coupons of each metal were weighed and suspended, three immersed in boiling solvent and three in the vapor region above the solvent, within a high pressure borosilicate tube. See figures 2 and 3. One coupon of each metal was weighed and retained as a control. The solvent was maintained at the boiling point using a constant temperature water bath. Test coupons were exposed for 24 hours, removed for inspection and weighing, and returned to the solvent for an additional 144 hours.



Figure 2 - Metal coupons suspended in a High Pressure Rated Glass Tube



Figure 3 - Coupons immersed in solvent and suspended in the vapor zone.

Thirteen ferrous and non-ferrous alloys were tested:

- a. 304L stainless steel
- b. A-286 PH stainless steel
- c. 17-4 PH stainless steel
- d. 440C stainless steel
- e. 4140 low alloy carbon steel
- f. Tin-Bronze
- g. Brass – Admiralty brass CDA 443 (Copper Development Association)
- h. Cobalt-Chromium-Nickel alloy - Elgiloy[®] (Elgiloy[®] Specialty Metals)
- i. 2219-T6 aluminum
- j. 6061-T6 aluminum
- k. 2195-T8 aluminum-lithium (plate stock)
- l. Inconel[™] 718 nickel alloy (Specialty Metals Corp.)
- m. Monel[™] 400 nickel alloy (Specialty Metals Corp.)

No visual change was observed in any of the metal coupons immediately after exposure and no significant weight changes were observed. However, four weeks after completion of solvent exposure, discoloration consistent with corrosion product was observed on the three specimens of 4140 low alloy carbon steel that had been immersed in the L-14780. This test was

repeated later in the program and corrosion of 4140 carbon steel exposed to L-14780 was confirmed. The relationship between this observed corrosion and the individual components in L-14780 including the stabilizers has not been determined.

Nonmetals Compatibility

The compatibility of the solvent with nonmetals was evaluated under conditions of total immersion at the boil temperature of the solvent. This test method was similar to the ASTM F 483-09 test for metals compatibility, but exposure duration was much shorter. AK-225G solvent was included in the test matrix for comparison.

Three test specimens of each nonmetal, formed as O-rings or gaskets with a hole for hanging, were dried in a desiccator for 24 hours, weighed, measured for outer diameter in two directions, and immersed in boiling solvent within a high pressure borosilicate tube for 15 minutes. See figure 4. Elastomers were measured for hardness in accordance with ASTM D 2240 *Standard Test Method for Rubber Property – Durometer Hardness Type A* (Shore A durometer) at the point of maximum thickness prior to immersion. [15] One specimen of each material was weighed and retained as a control. The solvent was maintained at the boiling point using a constant temperature water bath. After immersion, the specimens were removed to the desiccator for 30 minutes and then weighed, measured, and inspected for evidence of deterioration. Specimens exhibiting a change in weight or linear swell of greater than 1% from the initial readings were returned to the desiccator for 24 hours and then re-measured. Specimens continuing to exhibit a change in weight or linear swell of greater than 1% from the initial readings were returned to the desiccator for an additional six days and then re-measured.



Figure 4 - O-rings suspended in solvent, ready for insertion into a heated water bath.

Nine nonmetals were tested:

Elastomers:

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- a. FKM poly(hexafluoropropylene-co-vinylidene fluoride) elastomer, FKM Compound V0747-75 (Parker Hannafin) [equivalent to Viton A (DuPont)]
 - b. FFKM poly(tetrafluoropropylene-co-perfluoromethylvinyl ether) elastomer, Kalrez[®] (DuPont)
 - c. NBR poly(acrylonitrile-co-butadiene) rubber (Buna N) – Mil-G-21569B Class I [16]
- Non-elastomers:
- d. PTFE Polytetrafluoroethylene – Algoflon[®] E2 (Solvay Solexis)
 - e. FEP fluorinated ethylene-propylene copolymer – FEP Teflon[®] (DuPont)
 - f. Polychlorotrifluoroethylene (PCTFE) – Kel-F[®] 81 (3M)
 - g. Aromatic polyimide – Vespel[®] SP21 (DuPont)
 - h. PEEK polyether ether ketone – Ketron[®] PEEK (Quadrant Engineering Plastic Products)
 - i. PTFE with silica fiber – Gylon[®] style 3502 for oxygen service (Garlock)

Weight gain and linear swell recorded for each nonmetal with each of the four solvents are shown in tables 1 through 4.⁹ The three candidate solvents performed equal to or better than AK-225G in these nonmetal compatibility tests. Current cleaning protocols at MSFC and SSC that require removal of incompatible nonmetallic components prior to cleaning with AK-225G will be acceptable when using any of the three alternate candidates.

Table 1 - AK-225G Test Results for Solvent Compatibility with Nonmetals

Material	% Weight Gain			% Linear Swell		
	Post Test	After 24 hours	After 7 days	Post Test	After 24 hours	After 7 days
FKM (V0747-75) ^a	12.6	7.8	5.5	5.0	3.0	2.7
	16.0	9.4	6.7	4.9	3.6	3.7
FFKM (Kalrez)	14.5	7.7	5.0	4.0	2.7	1.7
NBR (Buna-N)	14.7	6.6	3.0	1.4	-1.5	-2.1
PTFE Algoflon	0.2	-	-	1.1	0.4	-
FEP Teflon	0.5	-	-	-1.2	-0.5	-
PCTFE (Kel-F)	0.2	-	-	0.5	-	-
Vespel 21	0.0	-	-	-0.2	-	-
PEEK	0.0	-	-	0.9	-	-
Gylon	0.2	-	-	-	-	-

⁹ The process to cut Gylon from sheet results in an irregular outer edge. Diameter and linear swell was not measured for this material.

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- a. Test for FKM (VO747-75) compatibility with AK-225G was performed twice.

NASA AK-225G Solvent Replacement Test Program Results

Table 2 - Solstice PF Test Results for Solvent Compatibility with Nonmetals

Material	% Weight Gain			% Linear Swell		
	Post Test	After 24 hours	After 7 days	Post Test	After 24 hours	After 7 days
FKM (V0747-75)	12.4	6.2	3.6	4.6	2.3	0.4
FFKM (Kalrez)	4.2	2.1	1.3	1.1	-0.4	1.8
NBR (Buna-N)	5.6	1.6	0.0	0.6	-0.5	-0.1
PTFE Algoflon	0.2	-	-	0.5	-	-
FEP Teflon	0.3	-	-	-0.2	-	-
PCTFE (Kel-F)	0.1	-	-	0.7	-	-
Vespel 21	0.0	0.0	-	-0.4	0.1	-
PEEK	0.0	-	-	0.9	-	-
Gylon	0.0	-	-	-	-	-

Table 3 - L-14780 Test Results for Solvent Compatibility with Nonmetals

Material	% Weight Gain			% Linear Swell		
	Post Test	After 24 hours	After 7 days	Post Test	After 24 hours	After 7 days
FKM (V0747-75)	5.9	3.6	2.6	1.8	1.0	0.7
FFKM (Kalrez)	6.0	3.6	2.5	2.9	1.9	1.4
NBR (Buna-N)	6.3	1.7	-0.3	2.0	0.6	0.4
PTFE Algoflon	0.1	-	-	0.0	-	-
FEP Teflon	0.3	-	-	-0.2	-	-
PCTFE (Kel-F)	0.0	-	-	-0.1	-	-
Vespel 21	0.1	-	-	0.0	-	-
PEEK	0.1	-	-	0.2	-	-
Gylon	0.1	-	-	-	-	-

Table 4 - Solvokane Test Results for Solvent Compatibility with Nonmetals

Material	% Weight Gain			% Linear Swell		
	Post Test	After 24 hours	After 7 days	Post Test	After 24 hours	After 7 days
FKM (V0747-75)	17.8	9.3	6.0	8.3	4.8	3.0
FFKM (Kalrez)	1.7	1.1	0.7	0.4	2.4	-1.0
NBR (Buna-N)	12.9	4.0	0.7	2.9	0.6	-0.3
PTFE Algoflon	0.1	-	-	1.1	-	-
FEP Teflon	0.1	-	-	-0.4	-	-
PCTFE (Kel-F)	0.0	-	-	0.2	-	-
Vespel 21	0.3	-	-	-0.1	-	-
PEEK	0.1	-	-	-0.1	-	-
Gylon	0.0	-	-	-	-	-

Initial Oxygen Compatibility Tests

The test methods specified in the test plan for oxygen compatibility were selected to parallel tests performed in the past by NASA for replacement of ODS solvents for cleaning oxygen systems. Two tests were performed at WSTF to evaluate the ignition sensitivity in LOX/GOX. These tests had also been performed at MSFC for the DLA study.

Ambient LOX Mechanical Impact Test Method

Each solvent was tested in accordance with ASTM G86-98a (Reapproved 2011), *Standard Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Ambient Liquid Oxygen and Pressurized Liquid and Gaseous Oxygen Environments*, section 4.2, *Ambient LOX Impact Test*, at 98 J (72 ft-lb) impact energy. [17] This test is referred to as the LOX Mechanical Impact test. This test method is specified in NASA-STD-6001 *Flammability, Offgassing, and Compatibility Requirements and Test Procedures*, as Test 13A. [18]

Solvent was transferred to a small sample cup (referred to as the “grease cup”) to obtain a sample thickness (depth) of 1.27 ± 0.13 mm (0.050 ± 0.005 in.). The solvent in the grease cup was frozen on a chill bar (WSTF) or cold box (MSFC) using liquid nitrogen. The grease cup with solid solvent was placed into a one-piece sample cup and the cup was filled with LOX. This LOX-filled sample cup containing the grease cup with frozen solvent was then placed into the base of the pre-chilled impact tester, the striker pin was placed, and the impact was performed in accordance with ASTM G86-98a. The pass criteria, as in previous NASA tests, was no reactions in 20 impacts or not more than 1 reaction in 60 impacts. Specimens that did not pass at 98J (72 ft-lb) impact force were tested at progressively lower impact energies until no reactions were observed.

Autogenous Ignition Temperature (AIT) Test Method

Each solvent was tested in accordance with ASTM G72-09, *Standard Test Method for Autogenous Ignition Temperature of Liquids and Solids in a High Pressure Oxygen-Enriched Environment*, modified for testing of volatile liquids. [19] Tests were performed at 0.34 MPa (50 psia) and 13.8 MPa (2000 psia) based on previous solvent tests performed by NASA in a joint program with the U.S. Naval Sea Systems Command [5] and for the U.S. Air Force. [20]

ASTM G72-09 does not contain specific instructions for handling of volatile liquids to assure that sufficient material is present to obtain valid data after purging of the test chamber with oxygen. Previous AIT test reports for solvents from the 1990's did not specify how the sample was handled to prevent excessive loss. During AIT tests performed by MSFC for the DLA study, the solvent and sample tubes were chilled with an ice bath to reduce loss prior to test. For these tests of Solstice PF, the sample tubes were filled with 0.50 g of solvent rather than 0.20 +/- 0.03 g as specified in ASTM G72-09 to assure that sufficient solvent would be present in liquid form after purging of the test chamber with oxygen to achieve an oxygen concentration >99%.

During the performance of the initial AIT tests at WSTF, due to the failure of several of the solvent samples to react up to the temperature threshold limit (TL) of the test apparatus (427°C [800°F]), concerns were raised that the test results may not be valid. WSTF performed several additional tests to investigate the effects of greater fuel quantity (sample weight), greater pressures than 0.34 MPa (50 psia) for the low pressure test, sample temperature, and test chamber purging.

As a result of these investigations, NASA proposed changes to ASTM G72-09 to improve repeatability when testing volatile liquids. This proposal was submitted to ASTM committee G04 on Compatibility and Sensitivity of Materials in Oxygen-Enriched Atmospheres, the governing committee for this standard.

Oxygen Compatibility Tests Performed Prior to Second Down-Selection

Test results on Solstice PF, L-14780, and Solvokane for LOX Mechanical Impact performed by MSFC for the DLA study indicated that Solvokane was significantly more reactive than Solstice PF or L-14780. Data to date on materials compatibility and cleaning effectiveness were very similar for the three remaining solvent candidates therefore additional oxygen test data was required as a discriminator. The oxygen compatibility tests specified in the test plan to be performed at WSTF were expedited to provide additional information prior to the second down-selection milestone.

Oxygen Compatibility Tests performed at WSTF

The oxygen compatibility tests performed by WSTF prior to the second down-selection included LOX mechanical impact at ambient pressure and at elevated pressure with threshold determination; and AIT tests using the modified test parameters described above.

NASA AK-225G Solvent Replacement Test Program Results

None of the three solvents tested for LOX Mechanical Impact at WSTF, Solstice, L-14780, and Solvokane, passed the screening criteria at 98 J (72 ft-lb) impact energy. Each solvent was tested at lower impact energies to determine ignition threshold. In addition, WSTF tested each solvent for LOX Mechanical Impact at 98 J (72 ft-lb) impact energy in variable pressure accordance with ASTM G 86-98a (NASA-STD-6001 Test 13B). The results of the LOX Mechanical Impact tests performed at WSTF, and performed by MSFC for the DLA study, prior to the second down-selection are shown in table 5.

Table 5 - Comparison of LOX Mechanical Impact Ignition Test Results

Solvent	WSTF			MSFC	
	Ambient LOX Impact at 98 J (72 ft-lb)	LOX Impact Threshold for 0/20 Reactions ^a	Pressure Threshold for No Reactions at 98 J (72 ft-lb)	Ambient LOX Impact at 98 J (72 ft-lb)	LOX Impact Threshold ^b
Solstice PF	Fail	20 J (15 ft-lb)	52 MPa (7500 psi)	Pass - 0/20	98 J (72 ft-lb)
L-14780	Fail	54 J (40 ft-lb)	52 MPa (7500 psi)	Pass – 0/20	98 J (72 ft-lb)
Solvokane	Fail	< 14 J (10 ft-lb) ^c	< 3.5 MPa (500 psi) ^c	Fail – 2/6	74 J (54 ft-lb)

a. Energy Threshold Screening Method in accordance with ASTM G 86-98a.

b. Determined by the Bruceton sensitivity test method.

c. Lower limit of the test apparatus. Threshold could not be determined.

The significant discrepancy in reaction thresholds observed in the ambient LOX Mechanical Impact tests at WSTF and MSFC was unexpected. While it was apparent that Solvokane was the most reactive, this data did not provide confidence to distinguish between the reactivity of Solstice PF and L-14780. An Independent Assessment Team (IAT) sponsored by the NESC was formed to investigate the test variables and conditions that could affect the test results when testing the reactivity of liquids in LOX and to establish a modified test protocol for a subsequent set of tests that would provide a reliable reactivity ranking of the candidate solvents.

AIT test results obtained at MSFC for the DLA study were available for L-14780 and Solstice PF prior to the first down-selection. AIT results from MSFC and WSTF, when a reaction was observed below the TL of the apparatus, are compared in table 6 (shown in °F for comparison to historic test data). At WSTF, the sample size was 1000 mg. At MSFC, the sample size for Solstice PF was 500 mg; for L-14780 it was 200 to 230 mg.

Table 6 - Comparison of AIT Test Results at WSTF and MSFC

	Low Pressure ^a				13.8 MPa (2000 psia)			
	WSTF		MSFC		WSTF		MSFC	
	Avg °F	Std Dev	Avg °F	Std Dev	Avg °F	Std Dev	Avg °F	Std Dev
AK-225G	528 @ 0.48 MPa (70 psia)	5			446	-		
AK-225 ^b	TL	-			TL	-		
L-14780	454 @ 1.4 MPa (200 psia)	-	300 ^c	1	322	15	TL	-
Solstice PF	464	16	510	9	360	5	411	34
Solvokane	496	22	-	-	305	3	-	-

a. Low Pressure AIT test was performed at 0.34 MPa (50 psia) unless otherwise noted. At WSTF, if TL was observed the test was repeated at a higher pressure.

b. Historic data for AK-225. [5] [20]

c. Two of three tests showed unusual low energy reactions. The third test was a TL.

The AIT acceptance criteria stated in the test plan were based on criteria used in past solvent evaluations for use with oxygen systems. [20]

Category A - AIT > 400 °F – Acceptable for use in oxygen systems.

Category B - AIT 250 °F to 400 °F – May be used with caution in oxygen systems.

Category C - AIT < 250 °F – Not recommended for use in oxygen systems.

When a reaction was observed with the larger sample size, all three solvent candidates met the AIT criteria to be acceptable for use at lower pressures and acceptable for use with caution at higher pressures.

Historic AIT test data for AK-225 (the dual isomer form), assumed to have been performed with the standard sample size of approx. 200 mg, showed no reactions up to the TL of the test apparatus. When AK-225G was tested for this project, using a larger sample size, reactions were observed at 0.48 MPa (70 psia) and 13.8 MPa (2000 psia) indicating that AK-225G is more reactive than previously thought.

Second Down-Selection

At a February 2014 TIM at WSTF, the project team reconvened to narrow the test set to two candidates. The data reviewed at this TIM included the test results for quick screen solvency, metals compatibility, nonmetals compatibility, and the results for the oxygen compatibility tests completed to date. Test results for metals and nonmetals compatibility available to date from the DLA study were also reviewed. The DLA tests for materials compatibility had been performed by ambient immersion for longer time periods (21 days for metals, and 30 days for nonmetals) on a smaller set of materials. The DLA tests for nonmetals,

which included exposure up to 90 days, were not complete at the time of this TIM. Solvokane was not tested for metals compatibility in the DLA study. The DLA test results for materials compatibility were shown to correlate well with the test results shown in this report.

In addition to the test data obtained to date, the project team also reviewed the environmental and health data for the solvent candidates. This included status updates on VOC exemption and EPA Significant New Alternatives Policy approval for Solstice PF.

Solstice PF and L-14780 were selected to proceed to the next phase of testing for the following reasons:

1. All three solvents performed equal to or better than AK-225G for nonmetals compatibility. All of the metals compatibility tests were determined to be acceptable although corrosion of 4140 carbon steel after exposure to L-14780 was noted for further investigation. Low alloy carbon steel is unusual in the construction of oxygen systems, but is used in associated test apparatus.
2. Solstice PF was preferred based on environmental and health criteria. L-14780 was preferred over Solvokane based on environmental criteria, but Solvokane was preferred over L-14780 based on toxicity.
3. Solvokane was significantly more reactive in LOX than Solstice PF or L-14780. The AIT data did not reveal a clear distinction between the three solvents. It was noted that the two primary components in Solvokane, Solkane 365 mfc and tDCE, are stated on their Safety Data Sheets to be flammable in air. Based on the LOX Mechanical Impact test data and the component data, the project team concluded that use of Solvokane with oxygen systems was contraindicated. This was the deciding factor in the down-selection.

Nonvolatile Residue Removal Efficiency

The purpose of the nonvolatile residue removal efficiency test was to assess the ability of a solvent to remove specific contaminants by ambient temperature flush. This test method was designed to mimic the NVR verification sampling process used at MSFC and SSC for oxygen systems hardware. Stainless steel test panels were fabricated for this test based on the test panel used in ASTM E 1235-08 *Standard test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft*, [21] but with $\frac{1}{4}$ the surface area to yield a 152 mm x 152 mm (6 in x 6 in) flat, lipped surface. Individual contaminants were applied to the test panel to achieve a target contamination level of approximately 40 mg/0.1 m². The contaminant was air dried and then baked for two hours at 55 °C (130 °F). Under a fume hood, the contaminated panel was mounted on a stand and gently flushed first with 200 ml of the test solvent and then with 200 ml of AK-225G. Each solvent flush was captured in a clean beaker. The test configuration is shown in figure 5. The captured solvent was analyzed for NVR content by drying and gravimetric analysis. These tests were performed for Solstice PF, L-14780, and AK-225G for comparison. Each solvent/contaminant combination was tested three times. If the results did not agree within 10%, then the test was repeated. The contaminant removal efficiency for the test solvent was calculated as:

$$\frac{(m_T - m_{TB})}{(m_T - m_{TB}) + (m_{AK} - m_{AKB})} \times 100 = \% \text{ NVR removed}, \quad (3)$$

where:

- m_T = mass of contaminant removed by the test solvent
- m_{TB} = mass of the background NVR in the test solvent blank
- m_{AK} = mass of the contaminant removed by the second rinse with HCFC-225cb
- m_{AKB} = mass of the background NVR in the HCFC-225cb solvent blank



Figure 5 - Configuration to flush a test panel for the NVR removal efficiency test.

The NVR contaminants used to challenge the candidate solvents were:

- a. Mineral oil – (representative of RP-1 fuel, petroleum-based hydraulic oils, and most petroleum-based motor oils and tube bending oils).
- b. Petroleum-based machine tool hydraulic fluid - Mobil DTE™ 25 (Exxon Mobil Corp.).
- c. Synthetic hydraulic fluid, MIL-PRF-83282 - Brayco Micronic® 882 (Castrol Industrial North America).
- d. Di-2-ethylhexyl sebacate - MONOPLEX® DOS
- e. Fingerprint simulated by synthetic sebum, modified Spangler soil per ASTM D 4265-98 (R 2007), *Standard Guide for Evaluating Stain Removal Performance in Home Laundering*, section A2.16.2 [22] (Scientific Services S/D, Inc., Sparrow Bush , NY).
- f. Fluorocarbon grease - Krytox 240AC (DuPont™ Chemical Solutions).
- g. Heavy paraffinic grease (crane grease), U-101 Big Red Grease (Universal, Inc.)
- h. WD-40® aerosol

- i. Fluorocarbon grease - Christo-lube[®] MCG 111 (Lubrication Technology, Inc.)

The cleaning efficiencies of the candidate solvents were determined to be similar to the cleaning efficiencies of AK-225G.

Extended Oxygen Compatibility Tests and Assessment

Refinement of Liquid Oxygen Mechanical Impact Test for Volatile Liquids

In response to the discrepant results between the ambient LOX Mechanical Impact tests performed at MSFC and WSTF, tests were repeated at each site witnessed by representatives of the NESC IAT. A matrix was developed to identify the variables to be tested at each location. Engineers and technicians from each test facility traveled to the other facility with cleaned and packaged sample cups, disks, and striker pins to perform tests to explore these variables. The results were compared and the IAT recommended a set of modified test parameters to maximize repeatability and confidence in the data. Ambient LOX Mechanical Impact tests of Solstice PF and L-14780 were repeated at WSTF using the modified test parameters. A complete discussion of this investigation is captured in NASA technical publication NASA/TP-2015-218207 *Replacement of Hydrochlorofluorocarbon-225 Solvent for Cleaning and Verification Sampling of NASA Propulsion Oxygen Systems Hardware, Ground Support Equipment, and Associated Test Systems*. [23]

The modified test parameters developed as a result of the independent assessment do not deviate from the ASTM G 86-98a test protocols, but are refinements determined to be necessary for repeatability when testing volatile liquids such as cleaning solvents using a grease cup. The refined test parameters were:

- a. Acceptance criteria: Reactions occurring when the striker pin impacts the test material alone are counted towards the acceptance criteria. Reactions resulting from non-uniform impacts on the edge of the grease cup, which may expose fresh aluminum or create point or line contact, are unreliable indicators of the reactivity of the material and are disregarded.
- b. Use of rebound catcher: The rebound catcher, required for the high pressure mechanical impact test (NASA-STD-6001 Test 13B), is not required for Test 13A by ASTM G 86-98a. Use of the rebound catcher reduces uncontrolled variables such as a shift in position of the grease cup after the initial impact or partial vaporization of the test sample that may result in a reaction on a secondary impact after no reaction on the initial, controlled impact.
- c. Use the stainless steel insert disk under the grease cup within the sample cup: Use of the insert disk provides a more rigid impact surface and thus a more rigorous test. ASTM G 86-98a is unclear on when the insert disk is to be used.
- d. Solvent sample preparation: Filter the solvent to remove any background particulate prior to test.

- e. Humidity control: Prepare test samples in an area with relative humidity less than 60% for improved test sensitivity.
- f. Grease cup dimensions: Use the original ASTM G 86-89 [24] grease cup dimensions for consistency. Conversion of ASTM G 86 from inch-pound to metric units created a slight disparity in grease cup dimensions from older stock to newly manufactured stock.

LOX Mechanical Impact tests of Solstice PF and L-14780 were repeated at WSTF in September 2014 using the modified test parameters. In these tests, both solvents met the screening criteria at 98 J (72 ft-lb). Solstice reacted once in 61 impacts at 98 J (72 ft-lb). L-14780 showed no reactions in 70 impacts at 98 J (72 ft-lb).

Heat of Combustion Tests

To supplement the AIT and LOX Mechanical Impact data, Heat of Combustion (HOC) tests in accordance with ASTM D 4809, *Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method)* [25], were performed at WSTF for Solstice PF, L-14780, and AK-225G. HOC is a measure of the energy released per gram of material should an ignition occur. It is an indicator of the potential for the material, if ignited, to ignite an adjacent material within the oxygen system and thus propagate the kindling chain. The HOC for these solvents were similar to several nonmetals commonly used within oxygen systems.

Oxygen Compatibility Analysis of Solvents

WSTF performed an analysis of the oxygen compatibility of Solstice PF and L-14780 versus AK-225G using the approach described in ASTM G 63 *Standard Guide for Evaluating Nonmetallic Materials for Oxygen Service* for performing an Oxygen Compatibility Assessment (OCA). [26] An OCA as defined in ASTM G 63-99 requires definition of the system within which the material will be used, including temperature, pressure, and configuration. The oxygen system within which a cleaning solvent might be entrapped due to a process escape is undefined, as is the quantity of solvent that might be entrapped. To evaluate the potential risk of using a solvent with known reactive properties for cleaning an oxygen system, engineering judgment is required to compare the properties of the solvent with those of other solvents and nonmetals that have been successfully used with oxygen systems in the past. This is the basis of the oxygen compatibility analysis performed by WSTF for this project.¹⁰

Solstice PF and L-14780 were found to be flammable in enriched oxygen environments, and plausible ignition mechanisms have been demonstrated. These tests also showed AK-225G to be flammable in enriched oxygen environments. However, this does not preclude the safe use of these solvents in oxygen systems. A comparison of the HOC and AIT of Solstice PF and L-14780 versus AK-225G and common oxygen system materials showed these materials to be “in family”. Both solvent candidates were comparable to each other but poorer in oxygen compatibility compared to AK-225G.

¹⁰ WSTF # 15-46483, NASA White Sands test Facility Solvent Investigation Special Report, January 2015

The volatility of these two solvents provided a challenge in their evaluation; however, this characteristic will also aid in the safe use of the materials. Regardless of the ignition method, sufficient material is required for ignition, making for an unlikely scenario. In the event of ignition, testing has shown the energy released upon combustion is most likely insufficient to kindle soft goods.

Based on this oxygen compatibility analysis, Solstice PF and L-14780 were determined to be an acceptable flammability risk for cleaning and NVR verification of NASA propulsion oxygen systems and associated hardware. These solvents are safe for use with reasonable efforts to assure that the solvent is adequately removed prior to introduction of oxygen to the system. Because these solvents are somewhat more reactive than the historically used solvents, CFC-113 and HCFC-225, additional precautions, such as the use of hydrocarbon detectors to verify solvent removal, are recommended.

On-site Vendor Demonstrations

Honeywell Demonstrations

The boiling point of Honeywell Solstice PF is 19 °C (66 °F), below typical operating temperatures for cleanrooms and laboratories at MSFC and SSC, and below ambient temperatures in Huntsville, Alabama, and the Gulf Coast of Mississippi during a significant portion of the year. Propulsion test operations are performed year-around at both locations and component processing operations must frequently be performed without the benefit of air conditioning. Concerns were raised early in this project whether a solvent with a low boiling point would be impractical for use during the summer months in south Mississippi. To alleviate these concerns, representatives of Honeywell visited SSC to demonstrate Solstice PF at the SSC Component Processing Facility. This facility is an open air shop with large doors that uses localized filtration (such as clean tents) when required. The day of this demonstration was warm and the doors were open. The ambient temperature was 30°C (86°F) with 83% relative humidity.

Representatives of Honeywell also visited MSFC to discuss the potential use of Solstice PF in the vapor degreaser currently used with AK-225G. Honeywell brought a vapor degreaser demonstration unit to MSFC to show how their product could perform in equipment adapted for that Solstice PF. Representatives of Honeywell also demonstrated Solstice PF in field cleaning operations within MSFC propulsion test areas.

The feedback from the demonstrations of Solstice PF at SSC and MSFC was positive. Users at both facilities recommended proceeding with this solvent as a potential replacement candidate for AK-225G. Users at both facilities considered the requirements to transport and store Solstice PF in pressure vessels, discussed during these demonstrations, to be only a minor inconvenience that could even potentially save storage and disposal costs with a cylinder exchange program. Technicians at MSFC liked the convenience of dispensing solvent directly from a small pressure vessel with a hose and nozzle rather than transferring and dispensing it from the pressurized canister sprayers currently used with AK-225G.

Solstice PF Spray Cleaner, which contains Solstice 1234ze (trans-1,3,3,3-tetrafluoroprop-1-ene) as the propellant, was also demonstrated at MSFC. Unlike Solstice PF which has no Upper Explosion Limit (UEL) or Lower Explosion Limit (LEL) in air, the Solstice 1234ze propellant exhibits flame limits in air at temperatures in excess of 28°C (82.4°F). This propellant dissolved in Solstice PF could alter the flammability of the solvent in oxygen. Therefore, it was decided that Solstice PF Spray Cleaner should not be used for cleaning components for oxygen service without separate oxygen compatibility tests for this product. When needed, Solstice PF should be pressurized with gaseous nitrogen (GN2) or by heating the vessel in which it is stored with a heating blanket.

3M Demonstrations

The boiling point of L-14780 is 28-30°C (82-86°F), significantly lower than the boiling point of AK-225G and well within the range of ambient temperatures in Huntsville, Alabama, and the Gulf Coast of Mississippi during the summer months. Although this is higher than the boiling point of Solstice PF, L-14780 may also require modified procedures for shipping, storage, and use.

Representatives of 3M visited MSFC to discuss the potential use of L-14780 in the vapor degreaser currently used at MSFC, and to demonstrate the use of their product in manual cleaning applications to MSFC Test Laboratory engineers and technicians. They also answered questions from the users regarding solvent testing, distillation and handling.

During the 3M demonstration at MSFC, technicians in the Valve and Components Lab tried L-14780 with their NVR sampling and verification apparatus. During this trial, a residue was detected in the FTIR spectrometer that partially overlapped and interfered with the hydrocarbon peaks used to measure NVR contamination. This had been noted earlier when testing the solvent for background NVR. 3M later supplied samples of the stabilizers used in L-14780 for comparison to the residue detected in these tests.

The 3M representatives traveled to south Mississippi to demonstrate their product at the SSC Component Processing Facility the following day. A 3M chemist demonstrated use of L-14780 for cleaning of several components including complex tubing. Requirements for packaging, transportation, and shipping of L-14780 were discussed during these meetings. 3M informed NASA that L-14780 may be shipped and stored in four liter/one gallon bottles or smaller but larger volumes of L-14780 require stronger containers than the lined carbon steel pails and drums used for AK-225G. L-14780 may be shipped and stored in stainless steel 55 gallon drums or 5 gallon pressure vessels.

Users at both facilities recommended proceeding with L-14780 as a potential replacement candidate for AK-225G. However, interference by one of the solvent stabilizers with the hydrocarbon peaks measured during verification of NVR using FTIR would require MSFC and SSC labs to use a different quantitative method to verify NVR. It was also noted that use of a multi-component solvent would require more analytical monitoring for quality control during distillation and recycling than AK-225G, a single component solvent that does not require a stabilizer.

Final Down-Selection

A TIM was convened in October 2014 to select the final replacement candidate for AK-225G. All of the data obtained to date was summarized and reviewed. The oxygen compatibility test data was discussed in detail and the oxygen compatibility analysis was presented by WSTF. A table of decision parameters was presented and the committee rated the final solvent candidates on these parameters. The project team agreed that Solstice PF and L-14780 met the primary technical performance requirements, but with some questions remaining regarding L-14780.

The cleaning efficiency of both solvents was comparable to AK-225G for the contaminants of concern. Both solvents were capable of achieving the cleanliness requirements for MSFC and SSC propulsion oxygen systems hardware, GSE, and associated test systems.

Materials compatibility for both solvents was equal to or better than AK-225G, with the exception of L-14780 with low alloy carbon steel. Although used in test support apparatus, low alloy carbon steel is unusual in the construction of NASA propulsion oxygen systems, therefore the team concluded that this is not a barrier to use of L-14780 with systems that do not contain low alloy carbon steel. L-14780 should not be used to clean systems containing low alloy carbon steels without further investigation and precautions to prevent corrosion.

Solstice PF and L-14780 as tested were judged to be acceptable for use with NASA propulsion oxygen systems, with reasonable efforts to assure that the solvent is adequately removed prior to introduction of oxygen to the system.

Two concerns remained regarding the suitability of L-14780 for use in MSFC and SSC propulsion oxygen system applications:

1. A residue was detected in the test samples of L-14780 that interfered with the MSFC/SSC test method for verification of NVR by FTIR. In some samples tested gravimetrically, the residue exceeded the solvent residue limits required by MSFC and SSC specifications. If the residue was confirmed to be a stabilizer of L-14780 rather than a contaminant that could be removed by distillation without affecting the L-14780, then L-14780 as currently formulated may be unsuitable for use by MSFC and SSC. It was also recommended that the NVR residue attributed to the stabilizer should be tested for HOC to assure that this residue does not pose a greater flammability risk than other contaminants intended to be precluded by the 1 mg/0.1 m² NVR cleanliness limit for NASA propulsion oxygen systems.
2. The L-14780 formula tested is an azeotrope only at the boiling point of the solvent. The boiling point of the HFE component is 34°C (93°F), significantly lower than the boiling point of tDCE, 48°C (118°F). If an escape of L-14780 were left within an oxygen system component stored at a temperature below the boiling point, it is possible that the HFE fraction could evaporate preferentially leaving a solvent

enriched in tDCE. Whether this enriched blend would pass the NASA criteria for oxygen compatibility is unknown.

The technical performance concerns remaining for L-14780 could potentially be resolved with more testing and, if necessary, reformulation of the stabilizer.

Solstice PF was clearly preferred over L-14780 for its lower toxicity and favorable environmental characteristics. Solstice PF has a higher AEL than AK-225G or L-14780. Solstice PF is also one of a new class olefin-based halogenated chemicals that breaks down rapidly in the atmosphere. It fully meets all currently identified environmental requirements and goals including very low GWP. Neither solvent had an objectionable odor. Both solvents were expected to be accepted by the end users.

As a single component solvent that does not require stabilizers, Solstice PF was preferred over the four component L-14780 azeotropic blend with stabilizers for implementation and business considerations. A single component material will require less analytical effort to monitor its chemical composition for quality assurance. Also, the performance of L-14780 is dependent on chemical stabilizers to prevent chemical breakdown of the tDCE component. These implementation issues are minor when purchasing and dispensing a cleaning solvent from small containers for one time use, but become significant when the solvent is used in a vapor degreaser for an extended time period and when repeatedly recovered, distilled, and reused.

Honeywell Solstice PF was selected as the preferred solvent to replace AK-225G at MSFC and SSC. 3M L-14780 was identified as a potential backup should Solstice PF become unavailable in the future or should unforeseen obstacles to implementation arise during the final assessment phase. The final selection of Solstice PF was driven by SHE factors, by the remaining technical questions regarding use of L-14780, and for business considerations related to the cost and complexity of monitoring and maintaining a multiple-component material.

Component Level Cleaning and Implementation Assessments

Investigations were performed at MSFC and SSC to assess the use of the new solvent in place of AK-225G in actual launch vehicle and engine test operations. The purpose of these investigations was to confirm that the solvent was suitable for use in field operations and to identify any potential issues with user acceptance. Necessary modifications to equipment and procedures were noted during these activities to assess potential cost and schedule impacts of the transition from AK-225G to the new solvent. Personnel involved in these investigations were primarily the end users: engineers, test technicians, and laboratory personnel currently processing NASA propulsion oxygen system hardware.

End User Evaluations

Solstice PF was provided to the MSFC Propulsion Test office for direct comparison to AK-225G in actual field cleaning tasks. Solvent field cleaning trials performed at a test stand included flanges, B-nuts, tubing, piping and instrumentation. The technicians reported that they saw no difference in the cleaning performance or usage rate of Solstice PF versus AK-225G.

Solstice PF was noted to seem colder and evaporate faster than AK-225G, but the faster evaporation was seen as a plus. Some icing from moisture condensation was observed on the aluminum foil on which parts were placed for cleaning, but not on the parts themselves.

To verify the performance of the solvent in a shop operation, cleaning trials similar to those performed during the vendor demonstrations were repeated at SSC for Solstice PF and L-14780. Flex hose, rigid tubing, and fittings from the Component Processing Facility were contaminated, cleaned, and quantitatively analyzed to measure cleaning efficiency.

Laboratory Implementation Assessments

The MSFC M&P Contamination Lab performed three additional tests not described in the test plan. These tests were to:

- a. Confirm the effectiveness of Solstice PF to remove particulate.
- b. Confirm the capability of a real time hydrocarbon analyzer (“sniffer”) to detect the presence of residual Solstice PF entrapped within hardware.
- c. Evaluate the use of a Gas Chromatograph/Mass Spectrometer (GC/MS) for receiving inspection and determination of lot-to-lot consistency of Solstice PF.

The particulate removal test was designed to verify that rinsing with Solstice PF would meet the particulate requirements for propulsion system components. In accordance with MSFC-SPEC-164D, oxygen systems and associated hardware must be verified free of particulate > 800 μ in longest dimension, and silting of fine particulate is prohibited. [1]

Three clean stainless steel panels, one for each solvent to be tested, were heavily contaminated with Course Test Dust, ISO 12103-1, *Road Vehicles – Test Dust for Filter Evaluation – Part 1: Arizona Test Dust*, Type A4). [27] The test dust was brushed through a sieve onto panels wetted with distilled water. The panels were then air dried in a filtered flow bench. The contaminated panels were rinsed with AK-225G, Solstice PF, and L-14780 from a PTFE squeeze bottle. A visual inspection showed that the particulate was not removed. The solvent flush was repeated with a pressurized canister sprayer. This removed most, but not all, of the particulate. The remaining particulate was successfully removed with solvent moistened lint free wipers.

In a second particulate removal test, clean, dry stainless steel panels were lightly dusted with Course Test Dust using a sieve and brush. For each of the three solvents, a flush from the PTFE squeeze bottle achieved visual cleanliness. The particulate removed from the panels was visible in the solvent captured from the flush.

The conclusion of these particulate removal tests was that flushing of lightly contaminated surfaces is sufficient to meet the required particulate cleanliness levels, but heavily contaminated surfaces must be pre-cleaned prior to flushing or cleaned manually. This is the current standard practice.

Trials were performed to evaluate the drying properties of Solstice PF and to confirm that a real-time sensor could be used to detect the presence of Solstice PF within a component that was not adequately dried. The sensor used for this test was a hand held Yellow Jacket AccuProbe UV Leak Detector, Model 69336, with solid electrolyte sensor, set on “high sensitivity”. The sensor is used by holding a “sniffer” probe at an orifice of a tube or other component to detect trace vapor from solvent that was inadequately removed from the part.

For each test, solvent was poured into a test article, the test article was capped (for tubes), and rocked or rotated to wet all interior surfaces. The tubes were stainless steel of various lengths and OD. The solvent was then poured out, the article was dried (or not) and the sniffer was used to detect residual solvent. These tests showed that:

- a. The sniffer detected AK-225G, Solstice PF, and L-14780.
- b. AK-225G and Solstice PF were removed to below the detection limit of the sniffer by a low pressure purge.
- c. Ambient drying for two hours was insufficient to remove AK-225G from inside a pipe but was sufficient to remove Solstice PF.
- d. Solstice PF appeared to dry more rapidly than AK-225G, consistent with the lower boiling point.

Three samples of Solstice PF from three lots were submitted to the MSFC Chemistry Laboratory for analysis by GC/MS to determine the constituents of the solvent and compare these lots for consistency. The analysis showed that GC/MS could be used for receiving inspection and that all three lots contained >99% trans-1-chloro-3,3,3-trifluoropropene.

Investigation of L-14780 Residue

During the demonstrations of L-14780 at MSFC and SSC, residue from L-14780 was detected during NVR verification by the FTIR method. The SSC Gas and Materials Science laboratory performed an evaluation of solvent residue from L-14780. A 200 ml sample of the solvent evaporated to dryness yielded 5.7 mg of residue, visible as dried droplets in the evaporating pan. This residue was analyzed by FTIR and compared to mineral oil and to a sample provided by 3M of one of the proprietary stabilizers used in L-14780. The residue from L-14780 matched the reference spectra of the stabilizer.

To be suitable for use as an NVR verification solvent for NASA rocket engine test facilities, solvents are required to have NVR background residue < 2 ppm weight/weight or <1 mg NVR/200 ml. For use as a cleaning solvent for propulsion systems, the MSFC purity requirement is ≤ 10 mg NVR per 500 ml of solvent. [1] The quantity of stabilizer detected in L-14780 exceeded both of these requirements. The solvent purity requirement is permitted to be achieved by distillation in-house to remove impurities. The stabilizer, however, is not an impurity, but is an essential component to inhibit the deterioration of the solvent. Discussion with a 3M chemist confirmed that the quantity of the stabilizer required by the 3M solvent

specification exceeds these NASA purity requirements. This makes the current formulation of L-14780 unsuitable for use at MSFC or SSC.

Many different chemical formulas are used by solvent manufacturers to stabilize their products. It is possible that L-14780 could be reformulated by 3M with a different stabilizer formula to meet the MSFC and SSC requirements for NVR background residue. This would be considered a new product by NASA and require testing.

Conclusions and Lessons Learned

Solstice PF is acceptable and preferred as the replacement for HCFC-225 solvent for cleaning and NVR verification sampling of NASA propulsion oxygen systems hardware, GSE, and associated test systems. Solstice PF is recommended for cleaning and NVR sampling in applications at NASA propulsion test facilities where AK-225G is currently used.

3M L-14780 as formulated does not meet the solvent purity requirements for cleaning and NVR verification of NASA propulsion oxygen systems hardware due to the required concentration of the stabilizer. This issue could potentially be resolved by reformulation of the stabilizer and testing of the reformulated product.

Solstice PF and L-14780 were shown to be flammable in enriched oxygen environments and more reactive than AK-225G. However, it was concluded that these products could be safely used with propulsion oxygen systems when reasonable precautions are in place to assure that the solvent is adequately removed prior to introduction of oxygen to the system. Uncertainties remain regarding the stability of the HFE/tDCE ratio of L-14780 when stored at a temperature lower than the boiling point of the azeotrope. This could potentially affect the flammability risk of solvent inadvertently left within an oxygen system component. Also, the relative flammability of a stabilizer residue versus other NVR contaminants has not been assessed. Due to these uncertainties, L-14780 is judged to be a higher flammability risk than Solstice PF.

Volatile liquids are difficult to test for oxygen compatibility. Additional controls are required for repeatability than specified in the current ASTM test methods. Furthermore, all halogenated solvents are reactive in oxygen under some conditions. The ASTM G63 Oxygen Compatibility Assessment approach is useful to evaluate the risks of use.

Stabilizer additives, although less than 1% of the solvent, can affect NVR results and must be considered and controlled for oxygen system cleaning applications.

Limitations

No claim is made here regarding the safety or efficacy of Solstice PF or L-14780 with materials or contaminants other than those tested.

No claim is made here regarding the suitability of Solstice PF or L-14780 for use with breathing oxygen systems. Use of a halogenated cleaning solvent with breathing oxygen systems

requires an appropriate toxicology assessment by the using organization. Evaluation of these solvents for suitability with breathing oxygen systems was beyond the scope of this project.

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Trademarks

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