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

H.D. Burns, M.A. Mitchell, and J.H. McMillian
Marshall Space Flight Center, Huntsville, Alabama

B.R. Farner
Stennis Space Center, Mississippi

S.A. Harper and S.F. Peralta
Johnson Space Center-White Sands Test Facility, New Mexico

N.M. Lowrey
Jacobs Technology, Inc./Jacobs ESSSA Group, Huntsville, Alabama

H.R. Ross
A2 Research, Stennis Space Center, Mississippi

A. Juarez
Jacobs Technology, Inc./MEI Technologies, Johnson Space Center-White Sands Test Facility, New Mexico

April 2015

The NASA STI Program…in Profile

Since its founding, NASA has been dedicated to the advancement of aeronautics and space science. The NASA Scientific and Technical Information (STI) Program Office plays a key part in helping NASA maintain this important role.

The NASA STI Program Office is operated by Langley Research Center, the lead center for NASA's scientific and technical information. The NASA STI Program Office provides access to the NASA STI Database, the largest collection of aeronautical and space science STI in the world. The Program Office is also NASA's institutional mechanism for disseminating the results of its research and development activities. These results are published by NASA in the NASA STI Report Series, which includes the following report types:

- TECHNICAL PUBLICATION. Reports of completed research or a major significant phase of research that present the results of NASA programs and include extensive data or theoretical analysis. Includes compilations of significant scientific and technical data and information deemed to be of continuing reference value. NASA's counterpart of peer-reviewed formal professional papers but has less stringent limitations on manuscript length and extent of graphic presentations.

- TECHNICAL MEMORANDUM. Scientific and technical findings that are preliminary or of specialized interest, e.g., quick release reports, working papers, and bibliographies that contain minimal annotation. Does not contain extensive analysis.

- CONTRACTOR REPORT. Scientific and technical findings by NASA-sponsored contractors and grantees.

- CONFERENCE PUBLICATION. Collected papers from scientific and technical conferences, symposia, seminars, or other meetings sponsored or cosponsored by NASA.

- SPECIAL PUBLICATION. Scientific, technical, or historical information from NASA programs, projects, and mission, often concerned with subjects having substantial public interest.

- TECHNICAL TRANSLATION. English-language translations of foreign scientific and technical material pertinent to NASA's mission.

Specialized services that complement the STI Program Office's diverse offerings include creating custom thesauri, building customized databases, organizing and publishing research results…even providing videos.

For more information about the NASA STI Program Office, see the following:

- Access the NASA STI program home page at <http://www.sti.nasa.gov>

- E-mail your question via the Internet to <help@sti.nasa.gov>

- Phone the NASA STI Help Desk at 757–864–9658

- Write to:
  NASA STI Information Desk
  Mail Stop 148
  NASA Langley Research Center
  Hampton, VA 23681–2199, USA

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

H.D. Burns, M.A. Mitchell, and J.H. McMillian
Marshall Space Flight Center, Huntsville, Alabama

B.R. Farner
Stennis Space Center, Mississippi

S.A. Harper and S.F. Peralta
Johnson Space Center-White Sands Test Facility, New Mexico

N.M. Lowrey
Jacobs Technology, Inc./Jacobs ESSSA Group, Huntsville, Alabama

H.R. Ross
A2 Research, Stennis Space Center, Mississippi

A. Juarez
Jacobs Technology, Inc./MEI Technologies, Johnson Space Center-White Sands
Test Facility, New Mexico

National Aeronautics and
Space Administration

Marshall Space Flight Center • Huntsville, Alabama 35812

April 2015

Acknowledgments

In addition to the authors, many engineers and technicians at Marshall Space Flight Center (MSFC), Stennis Space Center (SSC), Johnson Space Center-White Sands Test Facility (JSC-WSTF), and Langley Research Center (LaRC) participated in the development and execution of this test program. The authors wish to acknowledge the following for significant contribution to this project:

- MSFC: Curtis Bahr (Jacobs Technology/Jacobs ESSSA Group), Eddie Davis, Timothy T. Gautney, Gary Glass (METTS), Gail Gordon, Bobby Graves, Mark Griffin (METTS), Judson M. Hudson (METTS), Richard J. Joye (METTS), Buford Moore (METTS), James E. Morgan (ERC Inc. / Jacobs ESSSA Group), Barry Morris, Jennifer Nye (METTS), Sandra D. Pearson, Scott Rohe, James L. Smith, Brannon Standridge, Stefanie L. Wallburg (METTS / SERC), and Roger Welker (Jacobs Technology / Jacobs ESSSA Group)

- SSC: Randall R. Canady, Thomas R. Galloway, Dale Green (Jacobs Technology, Inc.), Erick Guttierrez (A2 Research), Marry Kerschbaum (A2 Research), Kenneth J. McCormick, Darrin Spansel (Lockheed Martin), and Taylor Davie (A2 Research)

- JSC-WSTF: Steve Bailey (Jacobs Technology), John Bouvet (Jacobs Technology), Horacio Perez (Jacobs Technology), Christina Pina-Arpin, and Joel M. Stoltzfus (Jacobs Technology)

- LaRC: Brian J. Jensen, Michael D. Smiles, and Steven J. Gentz

The authors gratefully acknowledge the contribution of test solvent and technical support by the following solvent suppliers:

Honeywell: Dr. Ryan Hulse, Jon T. Herdlein, Gary M. Knopek, Diana Mercier, and Jeff Beard
3M: Thomas J. Brodbeck, James W. Simmons, Karl Manske, and David Hesselroth
DuPont Vertrel® Specialty Products: Robert A. Lee
DuPont Chemicals and Fluoroproducts: Ray Davenport and Rick L. Raessler
Solvay Fluorides LLC: Kenneth Neugebauer and Lisa Norton
AGC Chemicals Americas: Richard W. Lamp

TRADEMARKS

Available from:

NASA STI Information Desk
Mail Stop 148
NASA Langley Research Center
Hampton, VA 23681–2199, USA
757–864–9658

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

TABLE OF CONTENTS (Continued)

LIST OF FIGURES

LIST OF FIGURES (Continued)

LIST OF TABLES

LIST OF ACRONYMS AND SYMBOLS

AEL	Acceptable Exposure Limit
AFRL	Air Force Research Laboratory
AIT	autogenous ignition temperature
ASTM	American Society for Testing and Materials
BP	boiling point
CAS	Chemical Abstracts Service
CFC	chlorofluorocarbon
CFR	Code of Federal Regulations
DLA	Defense Logistics Agency
DoD	Department of Defense
EPA	Environmental Protection Agency
FEP	fluorinated ethylene-propylene
FTIR	Fourier transform infrared
GC/MS	gas chromatograph/mass spectrometer
GN_2	gaseous nitrogen
GOX	gaseous oxygen
GSE	ground support equipment
GWP	Global Warming Potential
HCFC	hydrochlorofluorocarbon
HFE	hydrofluoroether

LIST OF ACRONYMS AND SYMBOLS (Continued)

HOC	heat of combustion
IAT	Independent Assessment Team
IR	infrared
JSC-WSTF	Johnson Space Center-White Sands Test Facility
Kb	Kauri-butanol
LOX	liquid oxygen
MAF	Michoud Assembly Facility
MAPTIS	Materials and Processes Technical Information System
M&P	Materials and Processes
MSFC	Marshall Space Flight Center
NESC	NASA Engineering and Safety Center
NVR	nonvolatile residue
OCA	Oxygen Compatibility Assessment
OD	outer diameter
ODS	ozone-depleting substance
PCTFE	polychlorotrifluoroethylene
PEEK	polyether ether ketone
PF	Performance Fluid
PFBI	perfluorobutyl iodide
P&P	pressurization and/or purge
PTFE	polytetrafluoroethylene

LIST OF ACRONYMS AND SYMBOLS (Continued)

SDS	Safety Data Sheet
SHE	safety, health, and environmental
SNAP	Significant New Alternatives Policy
SSC	Stennis Space Center
SSP	Space Shuttle program
tDCE	trans-1,2 dicholoroethylene
TIM	Technical Interchange meeting
TL	threshold limit
TP	Technical Publication
USAF	United States Air Force
VOC	volatile organic compound

Return to Menu

TECHNICAL PUBLICATION

REPLACEMENT OF HYDROCHLOROFLUOROCARBON-225 SOLVENT FOR CLEANING AND VERIFICATION SAMPLING OF NASA PROPULSION OXYGEN SYSTEMS HARDWARE, GROUND SUPPORT EQUIPMENT, AND ASSOCIATED TEST SYSTEMS

1. INTRODUCTION

Liquid oxygen (LOX) and gaseous oxygen (GOX) systems used in bipropellant propulsion systems require a high level of cleanliness. Systems to be wetted by GOX or LOX, and systems providing pressurization and/or purge (P&P) gases to these systems, must be clean, particularly of hydrocarbons and large (>800 μ) 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, chlorofluorcarbon-113 (CFC-113, Chemical Abstracts Service (CAS) No. 76-13-1) solvent was used for these applications. When CFC-113, a class I ozone-depleting substance (ODS) was banned in the 1990's, hydrochlorofluorocarbon-225[*] (HCFC-225ca/cb (a dual isomer form), CAS No. 422-56-0 and CAS No. 507-55-1), a class II ODS, was selected by the Space Shuttle program (SSP) to replace CFC-113 for final cleaning and verification of oxygen systems hardware where a suitable non-ODS replacement could not be found[2] (see footnote †). Beginning in 1999, the SSP moved from HCFC-225ca/cb to the single isomer HCFC-225cb[‡] (CAS No. 507-55-1), a less toxic product.[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) (CAS No. 422-56-0), and 1,3-dichloro-1,1,2,2,3-pentafluoropropane (HCFC-225cb) (CAS No. 507-55-1). References to HCFC-225 in this Technical Publication (TP) 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.

[†] HCFC-225 was authorized for use on the Space Shuttle External Tank by Change Order 60 to SE-S-0073 Revision F (ref. 2), dated April 15, 1996. HCFC-225 was later authorized for use on the Space Shuttle Orbiter by Change Order 63, dated February 10, 1997.

[‡] Manufactured by Asahi Glass Company, Ltd., Tokyo, Japan; supplied in North America by AGC Chemicals Americas, Exton, PA, under the product name Asahiklin AK-225G. AK-225G contains only the less toxic isomer (>99%) 1,3-dichloro-1,1,2,2,3-pentafluoropropane (HCFC-225cb) (CAS No. 507-55-1). AK-225 and AK-225G were approved for use on the SSP for cleaning and verification of propulsion oxygen systems. Purchase of AK-225 by NASA was discontinued when the less toxic form, AK-225G, was determined to be an acceptable alternative.

At that time, NASA Marshall Space Flight Center (MSFC) implemented HCFC-225cb for cleaning propulsion test hardware. In 2002, NASA Stennis Space Center (SSC) adopted HCFC-225cb to perform final rinsing and verification of nonvolatile residue (NVR) cleanliness for oxygen test system hardware that could be cleaned by water-based processes. When referenced in this TP, HCFC-225 refers to both HCFC-225ca/cb and HCFC-225cb.

An inter-Center NASA test plan (app. A) was developed for evaluating solvents to be used for cleaning of propulsion oxygen systems and associated P&P systems, ground support equipment (GSE), and test systems at MSFC and SSC, and for sampling of these systems for verification of cleanliness. In this test program, Asahiklin AK-225G (app. B.1) (>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.

Return to Menu

2. BACKGROUND AND TEST PLAN DEVELOPMENT

2.1 NASA Use of Hydrochlorofluorocarbon-225

Prior to initiation of this project, the NASA Principal Center for Regulatory Risk Analysis and Communication group polled the NASA Centers to identify users of HCFC-225 (as HCFC-225cb or as older stock of HCFC-225ca/cb) within the 2009–2011 timeframe, as well as users of stockpiled CFC-113. Three NASA locations—MSFC, MSFC-Michoud Assembly Facility (MAF), and SSC reported a high level of usage of HCFC-225 during this 3-year period, ranging from 3,600 to 18,000$^+$ kg (8,000 to 40,000$^+$ lb). MAF is a manufacturing facility in New Orleans, LA, managed by MSFC. HCFC-225 was used at these facilities primarily for precision cleaning and verification of cleanliness of launch vehicle propulsion oxygen systems hardware, GSE, and associated test systems. HCFC-225 was used at MAF for processing Space Shuttle external tank hardware until the retirement of the SSP in 2011. Future need is anticipated for a replacement for HCFC-225 at MAF for processing oxygen system components for the next NASA launch vehicle, the Space Launch System.

Two other NASA locations, Goddard Space Flight Center and Johnson Space Center-White Sands Test Facility (JSC-WSTF) reported low levels of HCFC-225 usage, less than 23 kg (50 lb) in this time period, primarily for laboratory operations.

2.2 Collaboration With the Department of Defense on Hydrochlorofluorocarbon-225 Replacement

During the timeframe that this project was being formulated, via communications with the U.S. Department of Defense (DoD)/NASA Joint Service Solvent Substitution Working Group, it was determined that NASA and DoD users shared a common interest in replacement of HCFC-225 for cleaning oxygen systems. The Defense Logistics Agency (DLA), Hazardous Minimization and Green Products Branch, funded a 2-year project for MSFC to identify and test two candidate solvents for replacement of HCFC-225.[§]

The candidate solvents tested in the DLA study were Honeywell Solstice™ Performance Fluid (PF) (app. B.2) and 3M L-14780 Developmental Material (app. B.3). These solvents were compared to two baseline solvents, AK-225G and DuPont™ Capstone® 4-I[¶] (app. B.4). 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 PFBI cleaning solvent approved by the U.S.

[§] Military Interdepartmental Purchase Request SC04001200481, Solvent Replacement for HCFC-225 for Cleaning Oxygen System Components, Final Report, August 26, 2014.

[¶] 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. DuPont does not currently offer a product based on PFBI as a cleaning solvent.

Return to Menu

Air Force (USAF) as a replacement for HCFC-225 for hand-wipe cleaning of components for aviator's breathing oxygen systems where HCFC-225 is prohibited or unavailable. Ikon Solvent P cleaning solvent was 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, the test program used to qualify Ikon Solvent P for USAF applications.[4] The test methods used in the DLA study for NVR background, materials compatibility, and cleaning effectiveness were different than those used for this project and a smaller set of materials and contaminants were tested. 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.

The schedule for the DLA study overlapped the schedule for this test program, with an earlier required completion date. Data obtained during the performance of the DLA study were used to supplement and inform the down-selection process during the course of this project.

2.3 Test Plan Development

2.3.1 Test Plan

The test plan (app. A) was developed jointly by a team of Materials and Processes (M&P) engineers, laboratory personnel, and end users from MSFC, SSC, and JSC-WSTF, and reviewed by the NASA Engineering and Safety Center (NESC). The solvent selection criteria described in ASTM G127, Standard Guide for the Selection of Cleaning Agents for Oxygen Systems,[5] were referenced for the development of this test plan.The materials and contaminants specified in this plan were selected by an engineering team from MSFC and SSC using ASTM G127 and ASTM MNL36, Safe Use of Oxygen and Oxygen Systems,[6] as a guide; 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.[7–11] [**]

2.3.2 Assignment of Test Responsibilities

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, SSC, and Langley Research Center. Test responsibilities were assigned to laboratories at MSFC, SSC, and JSC-WSTF based on the availability of laboratory facilities and expertise as follows:

- NVR background in neat cleaning solvents by gravimetric and Fourier transform infrared (FTIR) methods: SSC and MSFC.

[**] NASA Materials and Processes Technical Information System (MAPTIS) reports for material codes 01229 (AK-225) and 04619 (AK-225G)).

- Metals compatibility: SSC.
- Nonmetals compatibility: MSFC.
- Quick screen solvency: SSC.
- NVR removal efficiency: MSFC.
- Oxygen compatibility: JSC-WSTF.
- Component level cleaning tests (field cleaning): MSFC and SSC.

2.3.3 Test Sequence and Down-Selection Milestones

Tests were scheduled to provide data to support Technical Interchange meetings (TIMs) to narrow the list of candidates prior to the expenditure of funds on costly and time-consuming tests. 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 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 tests, 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 retained as a backup if any insurmountable issues arose during the final test phase.
- Test phase 4: Component level cleaning tests (field cleaning) and assessment of implementation requirements.

2.3.4 Additional Oxygen Compatibility Tests and Assessment

Initial results from the LOX mechanical impact tests performed at JSC-WSTF were found to be significantly different from those performed at MSFC for the DLA study following the same ASTM test method. As a result, an Independent Assessment Team (IAT) sponsored by the NESC was formed to investigate the test variables and conditions that could affect the 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. Additional oxygen compatibility tests beyond the scope of the initial test plan were performed at MSFC and JSC-WSTF to support this independent assessment and provide final input for an oxygen compatibility analysis. Final LOX mechanical impact threshold ignition tests were performed at JSC-WSTF using the detailed test parameters recommended by the IAT. Heat of Combustion tests in accordance with ASTM D4809, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method),[12] were also performed on the final two solvents and AK-225G to provide comparative data to support the final oxygen compatibility analysis.

3. SELECTION OF SOLVENT CANDIDATES

3.1 Identification of Promising 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. This survey included a review of publications from the efforts to replace CFC-113 and qualify HCFC-225 in the 1990's and 2000's, analysis of all cleaning solvent data recorded in the MAPTIS Material Selection Database, an extensive internet search, and evaluation of alternatives to ODS solvents approved under the U.S. Environmental Protection Agency (EPA) Significant New Alternatives Policy (SNAP). 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.[13]

3.2 Solvent Selection Criteria

The solvent selection criteria fell into two general categories: safety, health, and environmental (SHE) characteristics and technical performance parameters. Subordinate to the SHE criteria and the technical performance requirements, business issues influencing cost and availability were also considered.

3.2.1 Safety, Health, and Environmental Characteristics

3.2.1.1 Unacceptable Safety, Health, and Environmental Characteristics. Solvents that were classified by the EPA as a class I or class II ODS or contained a constituent classified as a Hazardous Air Pollutant[14] were not accepted as test candidates. In addition, chemicals classified as human carcinogens were not accepted for consideration.

3.2.1.2 Safety and Industrial Health. 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 (AELs), 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 with an AEL lower than 200 ppm were anticipated to require facility modifications or additional personal protective equipment for safe use.

3.2.1.3 Environmental. Solvents were preferred that were lower in volatile organic compound (VOC) content or VOC exempt as listed in the U.S. Federal Register[15] and published EPA determinations, and lower in 100-Year Global Warming Potential (GWP) as published in assessment reports by the Intergovernmental Panel on Climate Change.[16] Solvents were also preferred based on expected ease of recapture, repurification, and reuse.

3.2.1.4 Bio-Preferred Cleaning Solvents. Although spacecraft systems and launch support equipment are exempt from the requirements for federal procurement of bio-based cleaning agents,[17] bio-based products as defined by the U.S. Department of Agriculture Bio-Preferred program were considered during the search for candidate solvents. No bio-based products met the screening criteria for test candidates.

3.2.2 Technical Performance Criteria

Technical performance criteria included materials compatibility, cleaning effectiveness, and oxygen compatibility. Boiling point (BP) was also considered because solvents with high BPs (above 65 °C (150 °F)) can be difficult to dry and solvents with BPs lower than common ambient temperatures may require pressurized storage and may evaporate too quickly for practical use.

3.2.2.1 Materials Compatibility. Materials compatibility was evaluated based on vendor information and published literature:

- Metals corrosion: 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.

- Compatibility with nonmetals: HCFC-225 and other halogenated solvents are known to be incompatible with some nonmetals. Current practice at MSFC and SSC is to remove most seals and other nonmetallic materials from oxygen system components prior to cleaning with halogenated solvents. Better compatibility with nonmetals used in oxygen systems is preferred but was not an initial selection criterion.

3.2.2.2 Cleaning Effectiveness. 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 (refs. 1, 5 (sec. 6), and 6 (chapter 9)). 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 $\text{mg}/0.1\ \text{m}^2$, (ref. 1 and footnote [††]) 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.

[††] NASA specifications historically stated requirements for NVR in mg/ft^2. In later revisions, these units were changed to $\text{mg}/0.1\ \text{m}^2$ with the footnote: "For the purpose of this specification, 0.1 square meter = 1 square foot" (ref. 18).

The 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,[19] was used to estimate the cleaning power of the solvent. High Kb values indicate relatively strong solvency. While Kb value is a limited measure of solvent performance, 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.

The 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 follows:

Wetting index = (1,000 × density) / (surface tension × viscosity).

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

3.2.2.3 Oxygen Compatibility.

3.2.2.3.1 Flammability Data. Flammable cleaning agents inadvertently remaining within an oxygen system pose a significant fire hazard. When a flammable solvent is used with an oxygen system component, extreme care must be taken to assure that the component is thoroughly dried and all solvent is removed. Precautions such as bake-out and vacuum drying are often impractical, and inspection of large test systems to assure complete solvent removal may be difficult. 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.

3.2.2.3.2 Flammability of Solvent Blends. Many fluorinated solvents with low Kb values 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. To assure that the performance properties will remain stable over time, only true azeotropic blends were considered as candidates. Historical solvent flammability data in MAPTIS showed that all blends containing alcohol (e.g., methanol, ethanol, isopropanol, etc.) 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 LOX mechanical impact. While insufficient data were 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.

Return to Menu

3.2.3 Business Considerations

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.

3.3 Solvents Selected as Test Candidates

The following solvents were selected for this test program:

- AGC Chemicals Americas (distributor for Asahi Glass Company, Japan): Asahiklin AE3000 (new product) (1,1,2,2-tetrafluoroethyl-2,2,2-trifluoroethyl ether (hydrofluoroether (HFE)-347pc-f2) (CAS No. 406-78-0)). Reference appendix B.5.

- AGC Chemicals Americas (distributor for Asahi Glass Company, Japan): Asahiklin AE3000AT (new product) (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 No. 406-78-0) azeotrope). Reference appendix B.6.

- E. I. DuPont de Nemours and Company: Capstone 4-I, chemical intermediate (perfluorobutyl iodide (CAS No. 423-39-2), with other constituents). Reference appendix B.4.

- 3M: L-14780 Developmental Material[‡‡] (22% trans-1,2 dichloroethylene (CAS No. 156-60-5)/ 78% methyl perfluoropropyl ether (HFE-347mcc3) (CAS No. 375-03-1) azeotrope). Reference appendix B.3.

- Solvay Fluorides, LLC: Solkane® 365 mfc (new product) (1,1,1,3,3 pentafluorobutane (CAS No. 406-58-6)). Reference appendix B.7.

- Solvay Fluorides LLC: Solvokane® (new product) (30% trans-dichloroethylene (CAS No. 156-60-5)/70% HFC-365 mfc 1,1,1,3,3 pentafluorobutane (CAS No. 406-58-6) azeotrope). Reference appendix B.8.

- Honeywell: Solstice PF (new product) (trans-1-chloro-3,3,3,-trifluoropropene (HCFO-1233zd(E)) (CAS No. 102687-65-0)). Reference appendix B.2.

- 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)). Reference appendix B.9. This solvent contains a different stabilizer than some Vertrel MCA formulations previously tested by NASA.

[‡‡] 3M™ does not currently market this blend of solvents. L-14780 Developmental Material 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%.

Return to Table List

The selection criteria for these solvents are shown in table 1.

Table 1. Solvent candidates selected for test.

Single Component	Kb ≥ 20	Expected to Pass LOX Test	BP > 38 °C (100 °F)	AEL-8hr ≥ 200	Safe With Metals	VOC Exempt	100-Year GWP(1)
AGC AE3000	No	Yes	Yes	No	Yes	Yes(2)	Mid
DuPont Capstone 4-I	Yes(3)	Yes(4)	Yes	Yes	(5)	Unknown	Unknown
Solvay Solkane 365 mfc	No	Unknown	Yes	Yes	Yes	Yes	Mid
Honeywell Solstice PF	Yes	Yes	No	Yes	Yes	Yes(2)	Low
Azeotrope							
AGC AE3000AT	Yes	Unknown	Yes	No	Yes	No(6)	Mid
3M L-14780	Yes(3)	Yes(4)	No	Yes	Yes	No(6)	Mid
Solvay Solvokane	Yes	Unknown	No	Yes	Yes	No(6)	Mid
DuPont Vertrel MCA	Yes	Unknown	Yes	Yes	Yes	No(6)	High

Notes:
(1) For any component in the solvent: High is >1,000; mid is 10–1,000; low is <10.
(2) When solvent candidate selection was made, the request for VOC exemption for this solvent was in progress. EPA approval of VOC exemption has since been received.
(3) No Kb data available, but previous industry test data showed good cleaning performance.
(4) Historical LOX mechanical impact test data showed this material to be LOX compatible.
(5) A Boeing internal test report in support of the SSP indicated that this solvent might not be compatible with aluminum.
(6) This solvent blend contains trans-1,2 dichloroethylene which is not VOC exempt.

3.3.2 Solvents Tested

The suppliers provided solvent samples 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. The SHE characteristics and expected performance of AE3000 and AE3000AT were less favorable than the other test candidates so this is not considered a significant omission. L-14780 and Vertrel MCA had been previously tested as replacements for CFC-113. L-14780 had performed well in previous NASA oxygen compatibility tests (test report 97-31610, JSC-WSTF). Vertrel MCA did not fully pass previous NASA oxygen compatibility tests[7] (MAPTIS reports for material code 04091, Vertrel MCA), but was used for 10^+ years at NASA's Kennedy Space Center as the first step in a two-step cleaning process. Vertrel MCA was used to clean the hardware, followed by a rinse with an oxygen-compatible solvent with lower cleaning efficiency. 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. NASA had not previously tested any of the other solvent candidates for cleaning propulsion oxygen systems.

3.3.3 Baseline Solvent

Asahiklin AK-225G was used as the baseline solvent for this test program. At MSFC and SSC, AK-225G solvent was supplied to the test labs 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.

Return to Menu

4. NONVOLATILE RESIDUE BACKGROUND IN NEAT CLEANING SOLVENTS

4.1 Nonvolatile Residue Background Test Results

NVR residue reports from SSC and MSFC are shown in appendix C. 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 FTIR test methods. These were considered acceptable for further testing, but would require purification to meet the requirements for use as an NVR verification solvent. The differences in NVR results between labs and between test methods were 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. During these tests it was necessary to filter a large quantity of particulate from the Capstone 4-I. It was also noted that the Capstone 4-I had a very strong, objectionable odor.

4.2 Compatibility With Fourier Transform Infrared Analysis of Nonvolatile Residue

A calibrated FTIR method (app. A, section 3.1.3) is used at MSFC and SSC propulsion test facilities for measurement of NVR in AK-225G for initial 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 the 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, in some tests, the L-14780 appeared to leave a trace constituent in the residue that may interfere with the FTIR analysis. This residue was suspected to be a stabilizer component of the L-14780, an issue identified for further investigation.

5. QUICK SCREEN SOLVENCY

5.1 Quick Screen Solvency Test Results

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. This is described in detail, and the results shown, in appendix D. The contaminant mix was prepared by dissolving 0.5240 gram (g) of equal parts of the following contaminants in 100 mL of AK-225G:

- 0.1023 g of mineral oil—pharmaceutical grade—mixed aliphatic.
- 0.1018 g of MIL-PRF-83282D[20]—ester-based hydraulic fluid.
- 0.1025 g of Di-2-ethylhexylsebacate (gauge calibration oil), MONOPLEX® DOS.
- 0.1112 g of WD-40® (medium-heavy hydrocarbons).
- 0.1062 g of 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 10 times and the results were averaged. The NVR cleaning efficiency (%) is reported as:

Percent cleaning efficiency = (mg of NVR removed / mg of NVR applied) × 100.

The average cleaning efficiency results are shown in figure 1.

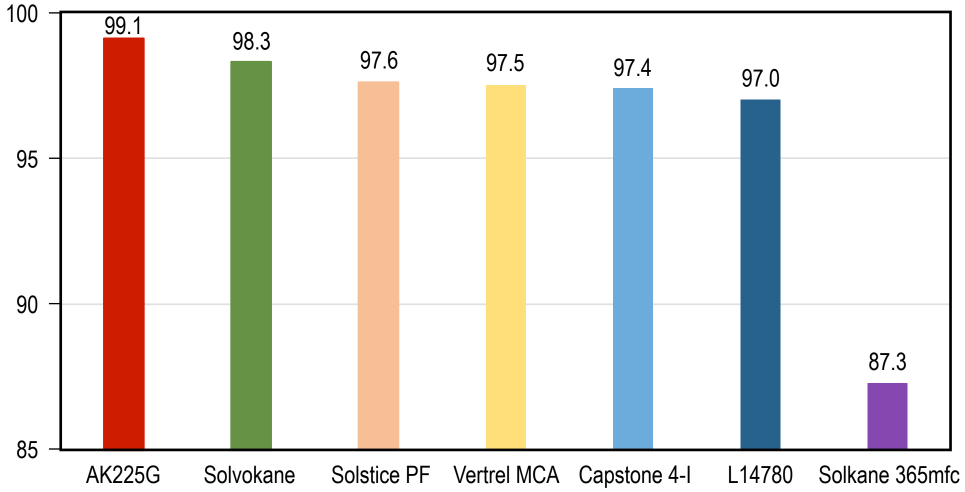


Figure 1. Solvent screening test results for cleaning efficiency—average percent NVR removed for each solvent.

Return to Menu

5.2 Observations on Miscibility and Saturation

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 substance solubility (e.g., hydraulic fluid) in a solvent can be measured as the saturation concentration or by the substance miscibility with the solvent. The miscibility of various contaminants in the candidate solvents was investigated further during the quick look screening tests. These miscibility tests are also shown in appendix D. It was noted that Solstice PF and AK-225G showed a significantly higher saturation capacity than the other test candidates for the contaminants tested. The contaminants tested were:

- MIL-PRF-83282D[20] hydraulic fluid.
- RP1 petroleum-based rocket propellant.
- Heavy-weight (85–140 W), petroleum-based gear oil.

5.3 Observations on Odor

During the quick look screening tests, the SSC laboratory chemists were asked to provide general user feedback on the candidate solvents. The solvents were scored on a scale of 1 to 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.

6. FIRST DOWN-SELECTION

6.1 Data Reviewed for the First Down-Selection

At the September 2013 TIM at SSC, the initial down-selection was performed to narrow the test set to three candidates. Data from NVR background tests and the quick screen solvency test were reviewed and 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 were also shared. These data included cleaning effectiveness tests, metals corrosion tests at ambient temperature, and initial LOX mechanical impact and autogenous ignition temperature (AIT) tests. MSFC reported that these three 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.

6.2 Solvents Selected for Further Testing

Solstice PF, Solvokane, and L-14780 were selected for further testing. All three solvents performed well in the quick screen solvency test and demonstrated other favorable characteristics. The cleaning performance of Solkane 365 mfc was significantly lower than the other candidates and it was determined that this solvent would not be acceptable for removing hydrocarbon contaminants to the low levels required for oxygen systems. It was also decided at this point that sufficient data were already available on Vertrel MCA to support future consideration as a fallback option should the three solvents selected for further testing fail to meet performance requirements.

6.2.1 Factors Supporting Continued Evaluation of Solstice Performance Fluid

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 JSC-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.

6.2.2 Factors Supporting Continued Evaluation of L-14780

L-14780 demonstrated good cleaning efficiency (>97%). Based on historical test data from JSC-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 JSC-WSTF. The environmental data on this material were not as favorable as Solstice PF, but were more favorable than other candidates that were higher in VOC content and GWP.

6.2.3 Factors Supporting Continued Evaluation of Solvokane

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 were available at this point on oxygen compatibility.

Return to Menu

7. METALS COMPATIBILITY

7.1 Metals Compatibility Tests

The corrosiveness of Solstice PF, L-14780, and Solvokane on metals was evaluated under conditions of total immersion in accordance with ASTM F483-09, Standard Practice for Total Immersion Corrosion Test for Aircraft Maintenance Chemicals,[21] and in solvent vapor for comparison. 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. One coupon of each metal was weighed and retained as a control. See figures 2 and 3. 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. Thirteen ferrous and nonferrous alloys were tested:

(1) 304L stainless steel.
(2) A-286 PH stainless steel.
(3) 17-4 PH stainless steel.
(4) 440C stainless steel.
(5) 4140 low alloy carbon steel.
(6) Tin-bronze.
(7) Brass—Admiralty brass CDA 443 (Copper Development Association).
(8) Cobalt-chromium-nickel alloy—Elgiloy® (Elgiloy Specialty Metals).
(9) 2219-T6 aluminum.
(10) 6061-T6 aluminum.
(11) 2195-T8 aluminum-lithium (plate stock).
(12) Inconel™ 718 nickel alloy (Specialty Metals Corp.).
(13) Monel™ 400 nickel alloy (Specialty Metals Corp.).

7.2 Metals Compatibility Test Results

Test details, tables of the test results, and images of the metal coupons after exposure are shown in appendix E. No visual change was observed in any of the metal coupons immediately after exposure and no significant weight changes were observed. However, 4 weeks after completion of solvent exposure, discoloration consistent with corrosion 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.

List of Figures

Figure 2. Metal coupons suspended in a high-pressure-rated glass tube.

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

Return to Menu List of Figures

8. NONMETALS COMPATIBILITY

8.1 Nonmetals Compatibility Tests

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 F483-09 test[21] for metals compatibility, but exposure duration was much shorter. Exposure to the baseline AK-225G solvent was included in the test matrix for comparison. AK-225G is not compatible with several nonmetallic materials used in oxygen systems and no halogenated cleaning solvent is expected to be compatible with all nonmetals.

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 (OD) 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 D2240, Standard Test Method for Rubber Property—Durometer Hardness Type A (Shore A durometer) at the point of maximum thickness prior to immersion.[22] 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 >1% from the initial readings were returned to the desiccator for 24 hours and then remeasured. Specimens continuing to exhibit a change in weight or linear swell of >1% from the initial readings were returned to the desiccator for an additional 6 days (7 days after immersion, total) and then remeasured.

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

Return to Menu

Nine nonmetals—six nonelastomers and three elastomers—were tested:

- Nonelastomers:

  (1) PTFE—Algoflon® E2 (Solvay Solexis).
  (2) Fluorinated ethylene-propylene (FEP) copolymer—FEP Teflon® (DuPont).
  (3) Polychlorotrifluoroethylene (PCTFE)—Kel-F® 81 (3M).
  (4) Aromatic polyimide—Vespel® SP21 (DuPont).
  (5) Polyether ether ketone (PEEK)—Ketron® PEEK (Quadrant Engineering Plastic Products).
  (6) PTFE with silica fiber—Gylon® style 3502 for oxygen service (Garlock).

- Elastomers:

  (1) FKM poly(hexafluoropropylene-co-vinylidene fluoride) elastomer, FKM compound V0747-75 (Parker Hannafin) (equivalent to Viton A (DuPont)).
  (2) FFKM poly(tetrafluoropropylene-co-perfluoromethylvinyl ether) elastomer, Kalrez® (DuPont).
  (3) NBR poly(acrylonitrile-co-butadiene) rubber (Buna N)—Mil-G-21569B class I.[23]

8.2 Nonmetals CompatibilityTest Results

Test results for AK-225G, Solstice PF, L-14780, and Solvokane with the listed nonmetals are shown in appendix F. Weight gain and linear swell recorded for each nonmetal with each of the four solvents are shown in tables 2–5, using the following colors for visual comparison of the relative compatibility of these materials:

- Red = Post test gain >10% or gain after 7 days >5% or weight loss >5%.
- Yellow = Post test gain >5% or gain after 7 days >3% or weight loss >2%.
- Green = Post test gain <5% and gain after 7 days <3% and weight loss <2%.

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. Although a red, yellow, or green cell is an indication of the degree of incompatibility, the decision whether or not to expose each of these nonmetals to a particular solvent should be determined by the user of that nonmetal.

Return to Table List

Table 2. 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) (Note 1)	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	–	–	(Note 2)	–	–

Note 1: Test for FKM (VO747-75) compatibility with AK-225G was performed twice.
Note 2: Linear swell measurements for Gylon not valid. The process to cut Gylon from sheet results in an irregular outer edge.

Table 3. 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	–	–	(Note 1)	–	–

Note 1: Linear swell measurements for Gylon not valid. The process to cut Gylon from sheet results in an irregular outer edge.

Return to Table List

Table 4. 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	–	–	(Note 1)	–	–

Note 1: Linear swell measurements for Gylon not valid. The process to cut Gylon from sheet results in an irregular outer edge.

Table 5. 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	–	–	(Note 1)	–	–

Note 1: Linear swell measurements for Gylon not valid. The process to cut Gylon from sheet results in an irregular outer edge.

Return to Menu

9. INITIAL OXYGEN COMPATIBILITY TESTS

9.1 Oxygen Compatibility Test Methods

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 to evaluate the ignition sensitivity in LOX/GOX. These tests had been performed at MSFC for the DLA study; those test reports are shown in appendix G.1.

9.1.1 Ambient Liquid Oxygen 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,[24] at 98 J (72 ft-lb) impact energy. 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.[25]

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 inch). The solvent in the grease cup was frozen on a chill bar (JSC-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 prechilled impact tester, the striker pin was placed, and the impact was performed in accordance with ASTM G86-98a.[24] 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.

9.1.2 Autogenous Ignition Temperature 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.[26] Tests were performed at 0.34 MPa (50 psia) and 13.8 MPa (2,000 psia) based on previous solvent tests performed by NASA in a joint program with the U.S. Naval Sea Systems Command[7] and for the USAF.[27]

9.1.2.1 Sample Handling for Volatile Liquids. ASTM G72-09 does not contain specific instructions for handling volatile liquids to assure that sufficient material is present to obtain valid data after purging the test chamber with oxygen.[26] 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 development of the test plan, JSC-WSTF recommended elimination of the purging step rather than using a larger sample size.

During the performance of the initial AIT tests at JSC-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 ºC (800 ºF)), concerns were raised that the test results may not be valid. JSC-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. The results of this investigation are described in appendix G.2.

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. The following changes were recommended :

(1) Solid or nonvolatile liquid sample weight should be 0.20±0.03 g.

(2) For volatile liquids such as cleaning solvents, a larger sample weight up to 1.00±0.10 g may be required to obtain a valid AIT result. It is good practice to prechill volatile liquids with boiling points near or below room temperature using an ice bath to prevent excessive loss of solvent prior to test. It is recommended a final weight be taken immediately before test to verify quantity present.

(3) Note: A lab may choose to incrementally approach the sample size of 1.00±0.10 g, evaluating pressure spikes and system safety limits as sample size increments are increased.

(4) Note: A nonignition at maximum temperature when testing at lower pressures (<6.9 MPa (<1,000 psia)) may indicate an insufficient oxidizer to fuel ratio. When testing at lower pressures, if obtaining a nonignition at maximum temperature, it is recommended that testing be performed at higher pressures until an AIT is obtained. If suspected, testing at the standard 10.3 MPa (1,500 psia) or higher and increased sample mass (suggested 1.00±0.10 g) is recommended to confirm an unreactive material.

9.2 Oxygen Compatibility Tests Performed Prior to the 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 (app. G.1.) AIT tests for Solvokane were not completed at MSFC prior to the TIM for the second down-selection. Data to date on materials compatibility and cleaning effectiveness were very similar for the three remaining solvent candidates, therefore additional oxygen test data were required as a discriminator. The oxygen compatibility tests specified in the test plan to be performed at JSC-WSTF were expedited to provide additional information prior to the second down-selection milestone.

Return to Menu Return to Table List

9.3 Oxygen Compatibility Tests Performed at Johnson Space Center-White Sands Test Facility

The results of the oxygen compatibility tests performed by JSC-WSTF prior to the second down-selection are shown in appendix G.2.

9.3.1 Initial Liquid Oxygen Mechanical Impact Ignition Test Results

None of the three solvents—Solstice, L-14780, or Solvokane—tested for LOX mechanical impact at JSC-WSTF, 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, JSC-WSTF tested each solvent for LOX mechanical impact at 98 J (72 ft-lb) impact energy in variable pressure accordance with ASTM G86-98a[24] (NASA-STD-6001 Test 13B).[25] The results of the LOX mechanical impact tests performed at JSC-WSTF, and performed by MSFC for the DLA study, prior to the second down-selection are shown in table 6.

Table 6. Comparison of LOX mechanical impact ignition test results.

	JSC-WSTF			MSFC (DLA Study)	
Solvent	**Test 13A Ambient LOX Impact at 98 J (72 ft-lb)**	**Test 13A LOX Impact Threshold for 0/20 Reactions(1)**	**Test 13B Pressure Threshold for No Reactions at 98 J (72 ft-lb)**	**Test 13A Ambient LOX Impact at 98 J (72 ft-lb)**	**Test 13A LOX Impact Threshold(2)**
Solstice PF	Fail	20 J (15 ft-lb)	52 MPa (7,500 psi)	Pass – 0/20	98 J (72 ft-lb)
L-14780	Fail	54 J (40 ft-lb)	52 MPa (7,500 psi)	Pass – 0/20	98 J (72 ft-lb)
Solvokane	Fail	<14 J (10 ft-lb)(3)	<3.5 MPa (500 psi)(3)	Fail – 2/6	74 J (54.6 ft-lb)

Notes:

(1) Energy Threshold Screening Method in accordance with ASTM G86-98a.[24]
(2) Determined by the Bruceton sensitivity test method.
(3) 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 JSC-WSTF and MSFC was unexpected. While it was apparent that Solvokane was the most reactive, these data did not provide confidence to distinguish between the reactivity of Solstice PF and L-14780. An 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. See section 12 and appendix I for additional detail.

9.3.2 Autogenous Ignition Temperature Test Results

The results of the AIT tests performed by JSC-WSTF are shown in appendix G.2 (shown in °F for comparison to historic test data). AIT test results obtained at MSFC for the DLA study were available for Solstice PF and L-14780 prior to the first down-selection (app. G.1). AIT results, when a reaction was observed below the TL of the apparatus, are compared in table 7. At JSC-WSTF, the sample size was 1,000 mg. At MSFC, the sample size for Solstice PF was 500 mg; for L-14780 the sample size was 200 to 230 mg.

Return to Table List

Table 7. Comparison of AIT test results at JSC-WSTF and MSFC.

	Low Pressure (Note 1)				13.8 MPa (2,000 psia)			
	JSC-WSTF		MSFC (DLA study)		JSC-WSTF		MSFC (DLA Study)	
Solvent	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 (Note 2)	TL	–	–	–	TL	–	–	–
L-14780	454 @ 1.4 MPa (200 psia)	–	300 (Note 3)	1	322	15	TL	–
Solstice PF	464	16	510	9	360	5	411	34
Solvokane	496	22	–	–	305	3	–	–

Notes:
(1) Low pressure AIT test was performed at 0.34 MPa (50 psia) unless otherwise noted. At JSC-WSTF, if TL was observed, the test was repeated at a higher pressure.
(2) Historic data for AK-225.[7,27]
(3) 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:[27]

- Category A—AIT >400 °F—Acceptable for use in oxygen systems.
- Category B—AIT 250 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 ≈200 mg, showed no reactions up to the TL of the test apparatus.[7,27] A repeat of this test for AK-225G with a larger sample size showed that, although no reaction was observed at 0.34 MPa (50 psia) to the TL, reactions were observed at 0.48 MPa (70 psia) and 13.8 MPa (2,000 psia), indicating that AK-225G is more reactive than previously thought.

Return to Menu

10. SECOND DOWN-SELECTION

10.1 Data Reviewed for the Second Down-Selection

At the February 2014 TIM at JSC-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 TP.

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 SNAP approval for Solstice PF.

10.2 Solvents Selected for Further Testing

The objective for the February 2014 TIM was to down-select the solvent candidates from three to two. 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) Oxygen compatibility: 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. Also discussed during this TIM was the flammability and reactivity of the two primary components in Solvokane: Solkane 365 mfc and tDCE. Both of these components are stated on their SDS to be flammable in air. Based on the LOX mechanical impact test data and the SDS data, the project team concluded that use of Solvokane with oxygen systems was contraindicated. This was the deciding factor in the down-selection.

Return to Menu List of Figures

11. NONVOLATILE RESIDUE REMOVAL EFFICIENCY

11.1 Nonvolatile Residue Removal Efficiency Test Method

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 E1235-08, Standard Test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft,[28] but with one-fourth the surface area to yield a 152 mm × 152 mm (6 in × 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 m^2. The contaminant was air dried and then baked for 2 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.

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

These tests were performed for Solstice PF, L-14780, and AK-225G. AK-225G was included 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:

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

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 AK-225G.
m_{AKB} = mass of the background NVR in the AK-225G solvent blank.

The NVR contaminants used to challenge the candidate solvents were:

- Mineral oil—CAS 8042-47-5 (representative of RP-1 fuel, petroleum-based hydraulic oils, and most petroleum-based motor oils and tube bending oils).
- Petroleum-based machine tool hydraulic fluid, ISO grade 46—Mobil DTE25™ (Exxon Mobil Corp.).
- Synthetic hydraulic fluid, MIL-PRF-83282,[20] fire resistant, synthetic hydrocarbon base—Brayco Micronic® 882 (Castrol Industrial North America).
- Di-2-ethylhexyl sebacate (gauge calibration oil)—MONOPLEX DOS (The C. P. Hall Co.).
- Fingerprint simulated by synthetic sebum, modified Spangler soil per ASTM D4265-98 (R 2007), Standard Guide for Evaluating Stain Removal Performance in Home Laundering, section A2.16.2 (Scientific Services S/D, Inc., Sparrow Bush , NY).[29]
- Fluorocarbon grease—Krytox 240AC (DuPont Chemical Solutions).
- Heavy paraffinic grease (crane grease), U-101 Big Red Grease (Universal, Inc.).
- WD-40 aerosol (medium-heavy aliphatic hydrocarbons) (WD-40 Company).
- CHRISTO-LUBE® MCG 111 (Lubrication Technology, Inc.) fluorocarbon grease was added to the test set after the release of the test plan when it was determined that CHRISTO-LUBE was commonly used as a substitute for Krytox 240AC at MSFC and SSC.

11.2 Nonvolatile Residue Removal Efficiency Test Results

Test results for AK-225G, Solstice PF, and L-14780 with the above contaminants are shown in appendix H. Charts comparing the removal efficiency of the candidate solvents versus AK-225G for each solvent are shown in figures 6 through 14. Cleaning efficiencies over 100% are due to the tolerance of the analytical balance and subtraction of solvent NVR background. The cleaning efficiencies of the candidate solvents were determined to be similar to the cleaning efficiencies of AK-225G and therefore suitable for cleaning NASA propulsion oxygen systems.

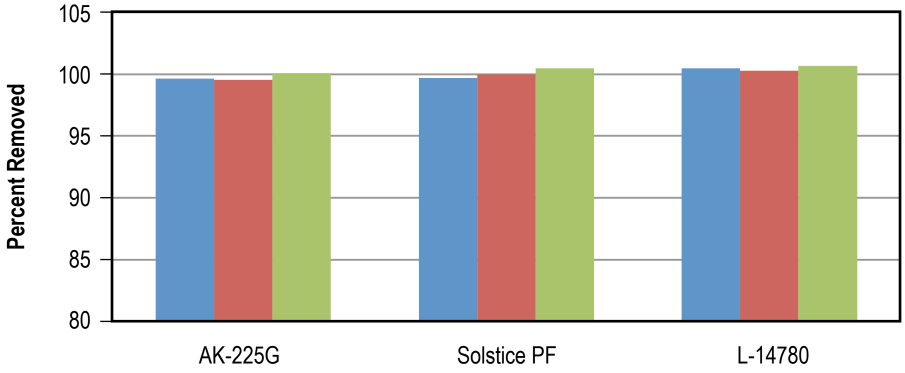


Figure 6. Mineral oil removal efficiency—percent removed by the test solvent of total removed. First rinse with test solvent, second rinse with AK-225G.

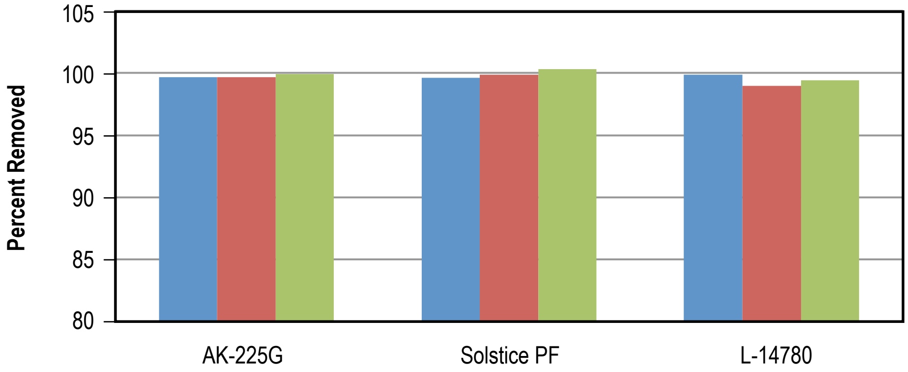
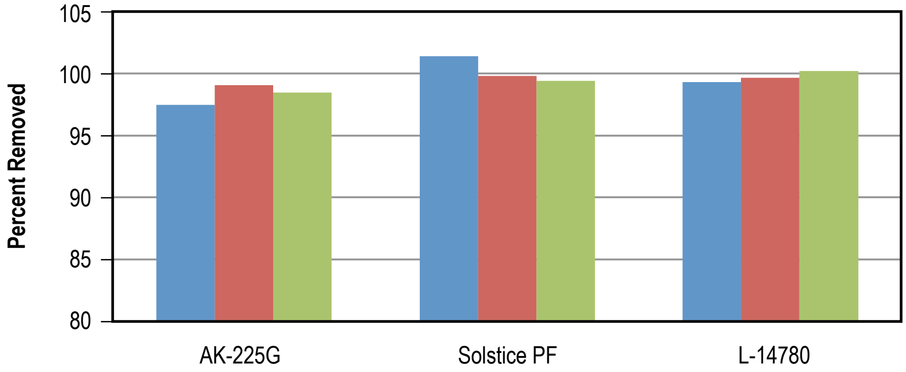


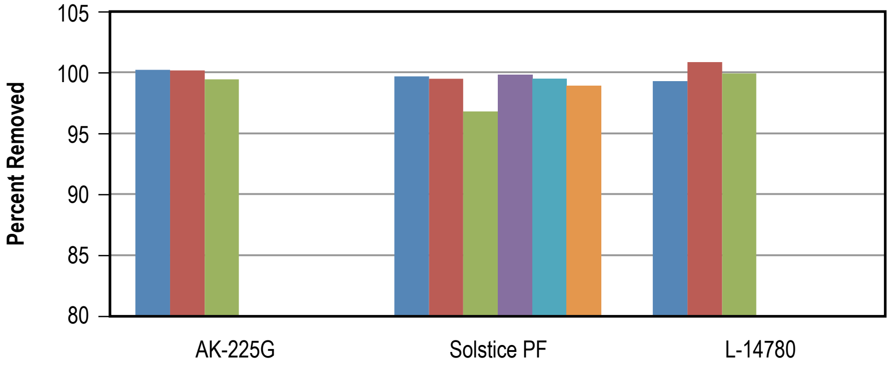
Figure 7. Mobil DTE25 hydraulic oil removal efficiency—percent removed by the test solvent of total removed. First rinse with test solvent, second rinse with AK-225G.



Note: Data from run 2. Test was repeated due to inconsistency of total contaminant recovered per plate due to spilled solvent during the tests of L-14780 and inadequate percentage of target containment mass collected.

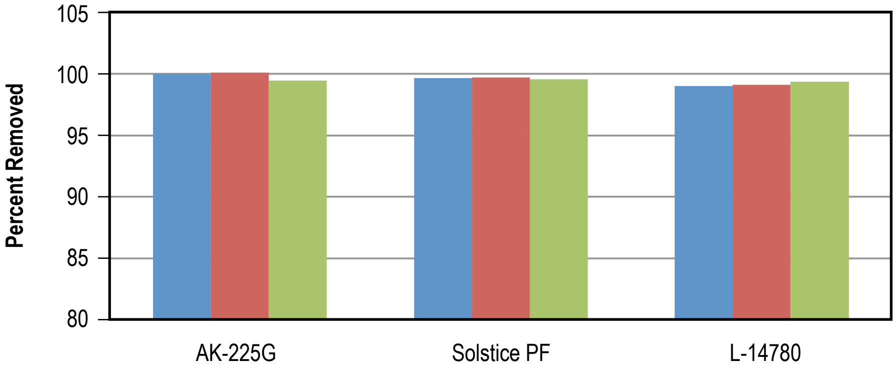
Figure 8. MIL-PRF-83282[20] hydraulic fluid removal efficiency—percent removed by the test solvent of total removed (data from run 2). First rinse with test solvent, second rinse with AK-225G.

List of Figures



Note: Data from runs 2 and 3. Test was repeated due to inconsistency of total contaminant recovered per plate in run 1. Three tests were added for Solstice PF (run 3) to confirm performance.

Figure 9. Di-2-ethylhexyl sebacate removal efficiency—percent removed by the test solvent of total removed (data from runs 2 and 3). First rinse with test solvent, second rinse with AK-225G.



Note: Data from run 2. Test was repeated due to inconsistency of total contaminant recovered per plate in run 1.

Figure 10. Synthetic sebum removal efficiency—percent removed by the test solvent of total removed (data from run 2). First rinse with test solvent, second rinse with AK-225G.

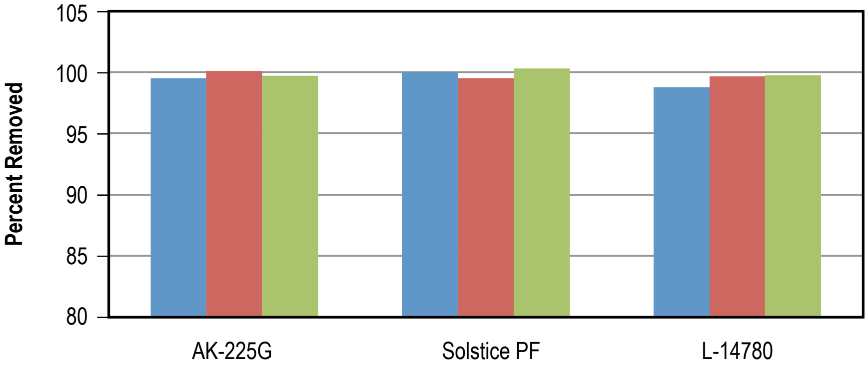


Figure 11. Krytox grease removal efficiency—percent removed by the test solvent of total removed. First rinse with test solvent, second rinse with AK-225G.

List of Figures

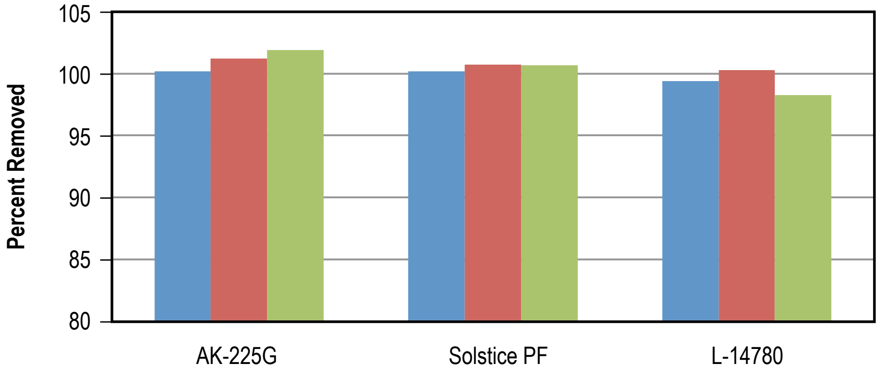
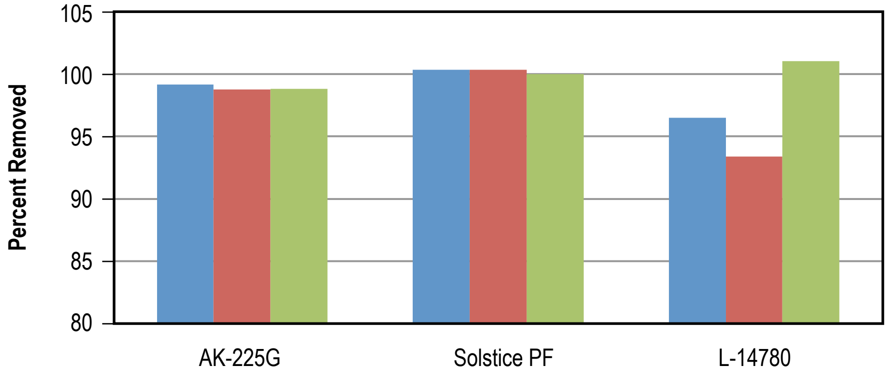


Figure 12. Crane grease removal efficiency—percent removed by the test solvent of total removed. First rinse with test solvent, second rinse with AK-225G.



Note: WD-40 contains a high percentage of volatile components that evaporate during initial drying. Total contaminant recovered was <30%. This creates a more variable result.

Figure 13. WD-40 grease removal efficiency—percent removed by the test solvent of total removed. First rinse with test solvent, second rinse with AK-225G.

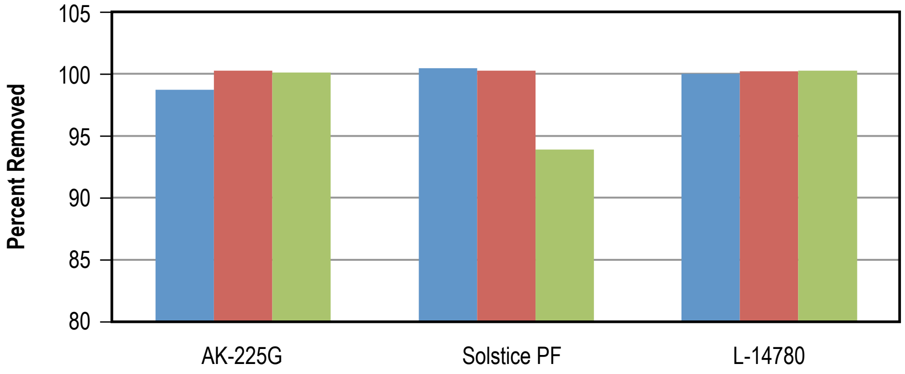


Figure 14. Christo-Lube grease removal efficiency—percent removed by the test solvent of total removed. First rinse with test solvent, second rinse with AK-225G.

Return to Menu

Return to Table List

12. EXTENDED OXYGEN COMPATIBILITY TESTS AND ASSESSMENT

12.1 Refinement of Liquid Oxygen Mechanical Impact Test for Volatile Liquids

In response to the discrepant results between the ambient LOX mechanical impact ignition tests performed at MSFC and SSC, tests were repeated at each site witnessed by representatives of the NESC IAT. A table was developed to identify the variables be tested at each location. (See table 8.) The IAT assessment, results, and recommendations are shown in appendix I.1. Appendix I.2 contains oxygen compatibility test data generated at MSFC for this assessment. Ambient LOX mechanical impact tests of Solstice PF and L-14780 were repeated at JSC-WSTF using the modified test parameters. Discussion of the test parameter analyses and final results are shown in appendix I.3.

Table 8. Matrix of variables tested for the independent assessment.

LOX Impact Threshold Testing	MSFC	JSC-WSTF
Insert disks	No inserts	Add Inserts
MSFC technician prepare samples		MSFC and JSC-WSTF cleaning
JSC-WSTF technician prepare samples	JSC-WSTF and MSFC cleaning	
11/16-inch sample cup		
Cleaning	JSC-WSTF cleaning	MSFC cleaning
Common solvent filtration	Same solvent container and filters	
Test with solvent from the same container	Standard MSFC method	Standard JSC-WSTF method
Humidity	Low	High
Rebound catcher	Use*	Use

*Test not performed due to shortage of specimen cups.

12.1.1 Modified Test Parameters

The modified test parameters developed as a result of the independent assessment do not deviate from the ASTM G86-98a test protocols,[24] but are refinements determined to be necessary for repeatability when testing volatile liquids such as cleaning solvents using a grease cup. These refinements will be recommended to ASTM committee G04 for potential incorporation into the test method. The refined test parameters were as follows:

(1) Acceptance criteria: Reactions occurring when the striker pin impacts the test material alone are counted towards the acceptance criteria. Reactions resulting from nonuniform 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.

Return to Menu

(2) Use of rebound catcher: The rebound catcher, required for the high-pressure mechanical impact test (NASA-STD-6001, Test 13B),[25] is not required for test 13A by ASTM G86-98a.[24] 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.

(3) 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 G86-98a[24] is unclear on when the insert disk is to be used.

(4) Solvent sample preparation: Filter the solvent to remove any background particulate prior to test.

(5) Humidity control: Prepare test samples in an area with relative humidity <60% for improved test sensitivity.

(6) Grease cup dimensions: Use the original ASTM G86-89[30] grease cup dimensions for consistency. Conversion of ASTM G86 from inch-pound to metric units created a slight disparity in grease cup dimensions from older stock to newly manufactured stock.

Due to the difficulty of placing and retaining the grease cup in the center of the sample cup that is then filled with LOX, the IAT recommended the development of a one-piece grease cup/sample cup for future tests. This would reduce the number of false indications due to the striker pin impacting the edge of the grease cup.

12.1.2 Liquid Oxygen Mechanical Impact Test Results Using the Refined Test Parameters

LOX mechanical impact tests of Solstice PF and L-14780 were repeated at JSC-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) (app. I.3). These results were consistent with the original test results seen at MSFC, therefore, the tests were not repeated with the modified test parameters at MSFC.

12.2 Heat of Combustion Tests

To supplement the AIT and LOX mechanical impact data, heat of combustion (HOC) tests were performed at JSC-WSTF for Solstice PF, L-14780, and AK-225G. HOC tests were performed in accordance with ASTM D4809, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method).[12] 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. HOC test results for these solvents, compared to some nonmetals commonly used within oxygen systems, are shown in table 9.

Return to Menu

Return to Table List

Table 9. Heat of combustion for solvents versus common nonmetals used in oxygen service.[30]

Comparison Materials		Heat of Combustion (cal/g) at 3.45 MPa (500 psia) JSC-WSTF Test Data 2014 Guidance per ASTM G63-99, Section 7.6.6: <2,500 cal/g Preferred 2,500 to 10,000 cal/g Use With Care >10,000 cal/g Poor
Common nonmetals used successfully in oxygen service	FluoroGreen E600	2,400*
	PTFE	1,700*
	PCTFE	2,557*
Honeywell Solstice PF		2,448
3M L-14780		1,925
Asahi AK-225G		1,153
Isopropyl alcohol (a flammable solvent)		7,165*

* Reference appendix I.3.

12.3 Oxygen Compatibility Analysis of Solvents

JSC-WSTF performed an analysis of the oxygen compatibility of Solstice PF and L-14780 versus AK-225G using the approach described in ASTM G63-99, Standard Guide for Evaluating Nonmetallic Materials for Oxygen Service[31] for performing an Oxygen Compatibility Assessment (OCA). An OCA as defined in ASTM G63-99 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 JSC-WSTF for this study and shown in appendix I.3.

Based on this oxygen compatibility analysis, Solstice PF and L-14780 have been 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/HCFC-225cb, additional precautions, such as the use of hydrocarbon detectors to verify solvent removal, are recommended.

Return to Menu

13. ON-SITE VENDOR DEMONSTRATIONS

13.1 Purpose of the On-Site Demonstrations

Both of the solvents selected as the final candidates to replace AK-225G have boiling points below 32 °C (90 °F) and therefore are expected to require some modifications to current procedures to prevent excessive loss. The suppliers of these solvents were invited to visit MSFC and SSC to demonstrate the use of their product to the end users and to answer questions. Honeywell and 3M accepted this invitation to provide a demonstration at both facilities. M&P engineers from MSFC witnessed these demonstrations at both facilities.

13.2 Honeywell Demonstrations

The boiling point of Honeywell Solstice PF is 19 °C (66 °F), below the typical operating temperatures of 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. Solstice PF must be shipped and stored in pressure vessels due to the low boiling point. The primary purpose of the Honeywell demonstrations was to alleviate concerns that the solvent would boil away too quickly for practical use, or require costly special handling methods.

13.2.1 Honeywell Demonstration at Stennis Space Center

Concerns were raised early in this project whether a solvent with a boiling point of 19 °C (66 °F) would be impractical for use during the summer months in south Mississippi. To alleviate these concerns, representatives of Honeywell visited SSC on September 18–19, 2013, to demonstrate Solstice PF to SSC engineers and technicians. A Honeywell chemist (fig. 15) demonstrated the product 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. The report for this demonstration at SSC is shown in appendix J.1.

13.2.2 Honeywell Demonstration at Marshall Space Flight Center

On November 4–6, 2013, representatives of Honeywell visited MSFC to discuss the potential use of Solstice PF in the vapor degreaser currently used with AK-225G in the MSFC Test Laboratory's Valve and Components Lab. The boiling point of Solstice PF is much lower than AK-225G; therefore, there was concern that Solstice PF would be ineffective as a vapor degreasing solvent. As a minimum, it was expected that major modifications to, or replacement of, this vapor degreaser would be required. To alleviate these concerns, Honeywell brought a vapor degreaser demonstration unit to MSFC to show how their product could perform in equipment adapted for that purpose.

Figure 15. A Honeywell chemist demonstrating Solstice PF with an SSC Component Processing Facility technician.

During this visit, representatives of Honeywell also demonstrated Solstice PF to MSFC engineers and technicians in field cleaning operations within MSFC propulsion test areas. Appendix J.2 contains a summary of these demonstrations.

13.2.3 Feedback From the Honeywell Demonstrations

The feedback from the demonstrations of Solstice PF at SSC and MSFC was positive. Users at both facilities recommended proceeding with Solstice PF as a potential replacement candidate for AK-225G. The requirements to transport and store Solstice PF in pressure vessels were discussed during the demonstrations at SSC and MSFC. Users at both facilities considered these requirements 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.

13.2.4 Solstice Performance Fluid Spray Cleaner Product

It was noted during the demonstration at MSFC that the Solstice PF Spray Cleaner product (app. B.10), which contains Solstice 1234ze as the propellant, is a different product that must be considered separately from the single component Solstice PF liquid. Unlike Solstice PF (1233zd(E), trans-1-chloro-3,3,3-trifluoropropene) which has no upper explosion limit or lower explosion limit in air (app. B.2), Solstice 1234ze (trans-1,3,3,3-tetrafluoroprop-1-ene) exhibits flame limits in air at temperatures in excess of 28 °C (82.4 °F) (app. B.11). Solstice 1234ze propellant dissolved in Solstice PF could alter the flammability of Solstice PF in oxygen. Therefore, Solstice PF spray cleaner should not be used as a substitute for Solstice PF for cleaning components for oxygen service without separate oxygen compatibility tests for this product. When needed, Solstice PF should be pressurized with gaseous nitrogen (GN_2). Solstice PF may also be pressurized by heating the vessel in which it is stored with a heating blanket.

13.3 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 from 3M were invited to demonstrate their product at MSFC and SSC to obtain user feedback prior to the final down-selection.

13.3.1 3M Demonstration at Marshall Space Flight Center

On August 20, 2014, representatives of 3M, including a solvent chemist, visited MSFC to discuss the potential use of L-14780 in the vapor degreaser currently used in the Valve and Components Lab, 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.

13.3.1.1 Nonvolatile Residue Verification With L-14780. 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 residual NVR contamination. This was the same concern noted in section 4.2. The 3M chemist stated that this residue was probably one of the stabilizers. 3M agreed to supply samples of the stabilizers used in L-14780 for comparison to the residue detected in these tests.

13.3.2 3M Demonstration at Stennis Space Center

Immediately after the demonstration at MSFC, the 3M representatives traveled to south Mississippi to demonstrate their product at SSC the following day. On August 21, 2014, the 3M representatives demonstrated their product at the SSC Component Processing Facility. A 3M chemist demonstrated use of L-14780 for cleaning of several components including complex tubing. (See fig. 16.)

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 4-liter (1-gallon) bottles or smaller but, due to its boiling point and vapor pressure, 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. For large quantity users, a container exchange program could be arranged.

Figure 16. A 3M chemist demonstrating L-14780 with an SSC Component Processing Facility technician.

13.3.3 Feedback From the 3M Demonstrations

The feedback from the demonstrations of L-14780 at SSC and MSFC was mostly positive. Users at both facilities recommended proceeding with L-14780 as a potential replacement candidate for AK-225G. It was noted, however, that one of the solvent stabilizers apparently interfered with the hydrocarbon peaks measured during verification of NVR using FTIR. This would require MSFC and SSC labs to use a different quantitative method to verify NVR. It was also noted that use of a four-component solvent (L-14780 is an azeotrope of methyl perfluoropropyl ether and tDCE plus two stabilizer chemicals) would require more analytical monitoring for quality control during distillation and recycling than AK-225G, a one component solvent that does not require a stabilizer.

Return to Menu Return to Table List

14. FINAL DOWN-SELECTION

14.1 Data Evaluated for the Final Down-Selection

A TIM was convened at JSC-WSTF on October 21–22, 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 were discussed in detail and the oxygen compatibility analysis was presented by JSC-WSTF. A table of decision parameters was presented and the committee rated the final solvent candidates on these parameters. The results of this assessment are shown in table 10. An X indicates the preferred solvent for that parameter.

Table 10. Decision point parameters for the selection of a replacement for AK-225G.

	Honeywell Solstice PF	3M L-14780	No Preference	Notes
SHE				
Environmental	X			Based on GWP and VOC comparison
Health and safety	X			Based on Acceptable Exposure Limit comparison
Technical/performance				
Metals compatibility			X	L-14780 corrosion on carbon steel after exposure and storage noted, but not considered a concern for selection
Nonmetal compatibility			X	
Cleaning effectiveness			X	
NVR verification	X			L-14780 complicates NVR analysis with the FTIR method. Correction for interference peak is required. Residue detected in some tests
Oxygen compatibility			X	L-14780—Analysis on the FTIR residue should be performed. Vendor commitment on stabilizer consistency required
Implementation				
Hands on	X*			Operator preference
Solvent cost	X			Based on vendor feedback, not firm quotes
Reclamation			X	
Facility mods			X	Both require some facility mods to vapor degreaser and distillers for different boiling point, heat of vaporization, etc.
Equipment needs		X		Solstice PF need for pressure vessels
Vendor readiness	X			Solstice PF now manufactured in Louisiana
Solvent maintenance cost	X			L-14780 requires four component monitoring / possible adjustments
Disposal cost	X			tDCE in L-14780 can go acidic requiring hazardous disposal

*Slight preference.

Return to Menu

14.2 Final Selection

14.2.1 Acceptability for Technical Performance

The project team agreed that Solstice PF and L-14780 met the primary technical performance requirements, but with some questions remaining regarding L-14780:

(1) 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.

(2) Materials compatibility for both solvents was equal to or better than AK-225G, with the exception of L-14780 with 4140 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 the use of L-14780 with systems that do not contain low alloy carbon steel. L-14780 should not be used to clean other systems containing low alloy carbon steels without further investigation and precautions to prevent corrosion.

(3) 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 the introduction of oxygen to the system. Although both solvents were shown to be more reactive in oxygen than CFC-113 or HCFC-225/HCFC-225cb, their flammability characteristics were ‘in family’ with other nonmetals that have been used safely within oxygen systems. Furthermore, the lower boiling points of these solvents contribute to faster drying and a reduction in the probability of solvent entrapment within an oxygen system component after cleaning.

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

(a) 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 m^2 NVR cleanliness limit for NASA propulsion oxygen systems.

(b) It was noted that 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. However, the greater certainty of performance of a single component material made Solstice PF the preferred candidate.

14.2.2 Safety, Health, and Environmental Factors

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.

14.2.3 Business Factors

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.

14.2.4 Selection of Honeywell Solstice Performance Fluid for Implementation

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 remaining technical performance concerns noted in 14.2.1(4) for L-14780 would need to be resolved. As noted in section 3.3.2, DuPont Vertrel MCA, used in a two-step cleaning process, was also identified as a backup. 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.

Return to Menu

15. COMPONENT LEVEL CLEANING AND IMPLEMENTATION ASSESSMENTS

15.1 Objectives of the 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 also 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. The objectives of the final component level cleaning and implementation assessments were to confirm the suitability of the replacement solvent in the intended final applications and to identify required modifications to equipment and procedures necessary to transition to the new solvent.

15.2 Marshall Space Flight Center 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 trials were performed at test stand 116. Items cleaned at test stand 116 included flanges, B-nuts, tubing, piping, and instrumentation. The Propulsion Test technicians reported that they saw no difference in the cleaning performance or usage rate of Solstice PF versus AK-225G. Solstice PF was reported 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.

Due to the age of the vapor degreasing system in the Valve and Component Lab now used with AK-225G, a request had already been submitted to the capital plan for a replacement. Discussions were held with a vapor degreaser supplier to identify the requirements for a system using Solstice PF and verify that a system could be supplied within the current cost estimate. No other new equipment or modifications were identified as necessary.

15.3 Stennis Space Cemter End User Evaluations

To verify the performance of the solvent in a shop operation, the cleaning trials similar to those performed during the vendor demonstrations were repeated 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. The results of these trials are shown in appendix K.

SSC identified a capital requirement for a new distillation system designed to purify and contain Solstice PF. Purchase of a second distillation system will support transition from AK-225G to Solstice PF without disruption of operations.

15.4 Laboratory Implementation Assessments

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

(1) Confirm the effectiveness of Solstice PF to remove particulate.

(2) Confirm the capability of a real time hydrocarbon analyzer ('sniffer') to detect the presence of residual Solstice PF entrapped within hardware.

(3) Evaluate the use of a gas chromatograph/mass spectrometer (GC/MS) for receiving inspection and determination of lot-to-lot consistency of Solstice PF.

15.4.1 Particulate Removal

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, Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, Specification for,[1] oxygen systems and associated hardware must be verified free of particulate >800 µ in longest dimension, and silting of fine particulate is prohibited. For fuel system hardware, the particulate size limit is 400 µ.

15.4.1.1 Particulate Removal Test 1. Three clean stainless steel panels, one for each solvent to be tested, were heavily contaminated with Course Test Dust (reference ISO 12103-1, Road Vehicles – Test Dust for Filter Evaluation—Part 1: Arizona Test Dust), type A4.[32] The test dust was brushed through a No. 60 sieve, 250-µ (0.01-in) pore size, 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 with white task lighting showed that the particulate was not removed. The solvent flush was repeated with a Sure Shot® sprayer (Milwaukee Sprayer Mfg. Co., Inc.) pressurized to 70 psi. This removed most, but not all, of the particulate. The remaining particulate was successfully removed with solvent-moistened, lint-free wipers.

15.4.1.2 Particulate Removal Test 2. Clean, dry stainless steel panels were lightly dusted with Course Test Dust using the No. 60 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 when inspected with white light.

15.4.1.3 Particulate Removal Conclusions. 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 precleaned prior to flushing or cleaned manually. This is the current standard procedure.

15.4.2 Drying and Verification of Removal of Residual Solvent

The objective of this trial was 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 handheld Yellow Jacket® AccuProbe UV™ Leak Detector, Model 69336, with solid electrolyte sensor (fig. 17). 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. The system was set on 'high sensitivity.'

Figure 17. Hydrocarbon leak detector.

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. The test scenarios and results are shown in table 11. L-14780 was not included in tests 3–8. These tests showed that:

- The sniffer detected AK-225G, Solstice PF, and L-14780.
- AK-225G and Solstice PF were removed to below the detection limit of the sniffer by low pressure purge for a sufficient length of time.
- Ambient drying for 2 hours was insufficient to remove AK-225G from inside a pipe but was sufficient to remove Solstice PF.
- Solstice PF appeared to dry more rapidly than AK-225G, consistent with the lower boiling point.

Return to Menu   Return to Table List

Table 11. Drying tests and test results.

Test No.	Test Article	Drying Method	Meter Reading		
			AK-225G	Solstice PF	L-14780
1	10-mL glass beaker	Evaporated to dryness on a filtered flow bench	0	0	0
2	30.5 cm (12 in) – 0.1 cm (3/8 in) OD tube, wetted with 7 mL solvent.	None. Tube sampled on a flow bench for 20 min	1–5	1–4	1–2
3	3 m (10 ft) – 1.9 cm (3/4 in) OD tube—straight	Purge with 34 kPa (5 psi) high purity air for 15 min	1	0	
4	3 m (10 ft) – 0.6 (1/4 in) OD tube—straight	Purge with 34 kPa (5 psi) high purity air for 15 min	0	0	
5	3 m (10 ft) – 0.6 (1/4 in) OD tube—curved	Purge with 34 kPa (5 psi) high purity air for 15 min	0	0	
6	1.2 m (4 ft) – 1.3 cm (1/2 in) OD pipe	2-hr ambient dry, no purge	1–4	0	
7	79 cm (31 in) – 0.1 cm (3/8 in) OD tube	15-min ambient dry, no purge	7	4	
8	79 cm (31 in) – 0.1 cm (3/8 in) OD tube	Purge with 34 kPa (5 psi) air for 5 min	2	4	

15.4.3 Receiving Inspection and Determination of Lot-to-Lot Consistency

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. This was a first step toward determining a receiving inspection test for the solvent.

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 (Marshall Space Flight Center Internal Report, EM10 (015-0001), dated January 7, 2015).

15.5 Follow-Up 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 spectrometer. The FTIR results were 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 exceeds both of these requirements. The solvent purity requirement may 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.

Solvent manufacturers use many different chemical formulas to stabilize their products. It is possible that the azeotrope of methyl perfluoropropyl ether and tDCE 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 as specified in appendix A.

16. CONCLUSIONS AND RECOMMENDATIONS

16.1 Conclusions of This Test Project

16.1.1 Honeywell Solstice Performance Fluid

Honeywell 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.

16.1.2 3M L-14780 Developmental Material

3M L-14780 as formulated does not meet the solvent purity requirements for cleaning and NVR verification of NASA propulsion oxygen systems hardware, GSE, and associated test systems, 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.

16.1.3 Flammability of Solstice Performance Fluid and L-14780 in Enriched Oxygen Environments

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 risk than Solstice PF.

16.1.4 Materials or Contaminants Other Than Those Tested

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

16.1.5 Breathing Oxygen Systems

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

Return to Menu

16.2 Recommendations

16.2.1 Implementation of Solstice Performance Fluid

MSFC and SSC user organizations should proceed to implement Solstice PF as the replacement for AK-225G on a schedule to avoid disruption of propulsion test operations, but prior to the depletion of stockpiled AK-225G. The following future work was identified during the test program to be addressed during the implementation phase:

- Purchase of a vapor degreaser system for the MSFC Test Laboratory Valve and Component Lab tailored for Solstice PF, to be used in parallel with the AK-225G system during the transition.

- Purchase of a distillation system for the SSC Component Processing Facility tailored for Solstice PF, to be used in parallel with the AK-225G system during the transition.

- Development of a NASA procurement specification for Solstice PF, to include cleanliness requirements and to prohibit use of pressurizing gases other than GN_2.

- Revision of MSFC and SSC procedures and work instructions to add Solstice PF as an approved solvent. This includes determination of required changes to process support equipment and processing parameters to implement Solstice PF in place of AK-225G in these procedures.

- Establishment of a procurement contract with Honeywell or a designated distributer to supply Solstice PF in bulk to MSFC and SSC, with arrangements for a container exchange program to minimize cost.

- Verification that Solstice PF is supplied to SSC and MSFC sufficiently pure to be used as a cleaner for propulsion oxygen systems. This includes visits to the Honeywell manufacturing facility for Solstice PF by NASA personnel to validate the Honeywell quality assurance processes, and the development and implementation of receiving inspection tests at NASA to verify quality.

16.2.2 3M L-14780 as a Backup Replacement Solvent

The 3M azeotrope of methyl perfluoropropyl ether and trans-1,2 dichloroethylene should be considered a backup replacement solvent for Solstice PF if the product can be formulated with a stabilizer that meets the SSC NVR requirement of <2 ppm. This reformulated product would require testing accordance with the test plan in appendix A and to resolve open questions regarding storage of an azeotropic product at nonazeotropic temperatures.

16.2.3 Testing of Solstice Performance Fluid Spray Cleaner

Solstice PF spray cleaner, which contains the propellant Solstice 1234ze, should be tested for oxygen compatibility to assure that this product form does not present a hazard when used with oxygen systems. Oxygen compatibility testing should include the following:

- Ambient LOX mechanical impact in accordance with ASTM G86,[24] using the modified test parameters specified in section 12.1.1.

- AIT in accordance with ASTM G72 [26] with changes as specified in section 9.1.2.1.

- Heat of Combustion in accordance with ASTM D4809.[12]

- Analysis of oxygen compatibility relative to Solstice PF as described in section 12.3.

Return to Menu

REFERENCES

1. MSFC-SPEC-164D, Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, Specification for, NASA Marshall Space Flight Center, Huntsville, AL, January 30, 2014.

2. JSC SE-S-0073F, Space Shuttle Specification: Fluid Procurement and Use Control, NASA Johnson Space Center, Houston, TX, May 20, 1994.

3. JSC SE-S-0073G, Space Shuttle Specification: Fluid Procurement and Use Control, NASA Johnson Space Center, Houston, TX, May 27, 1999.

4. Roberts, M.B.; Gschwender, L.J.; and Snyder, Jr., C.E.: AFRL-ML-WP-TR-2003-4040, The Wipe Solvent Program, Air Force Research Laboratory, Bedford, MA, February 2003.

5. ASTM G127 - 95(2008), Standard Guide for the Selection of Cleaning Agents for Oxygen Systems, ASTM International, West Conshohocken, PA, 2008, <http://www.astm.org>.

6. ASTM MNL36-2nd, Safe Use of Oxygen and Oxygen Systems: Handbook for Design, Operation, and Maintenance: 2nd Edition, H.D. Beeson, S.R. Smith, and W.F. Stewart, Eds., ASTM International, West Conshohocken, PA, 2007, <http://www.astm.org>.

7. Beeson, H.; Beisinger, P.; Delgado, R.; and Antin, N.: "Evaluation of Solvent Alternatives for Cleaning of Oxygen Systems," NASA/CP—1999–209258, *Proceedings of the Third Aerospace Environmental Technology Conference*, A.F. Whitaker, D.R. Cross, S.V. Caruso, and M. Clark-Ingram, Eds., NASA Marshall Space Flight Center, Huntsville, AL, pp. 373–377, April 1999.

8. Melton, D.: "Critical Surface Cleaning and Verification Alternatives," NASA/CP—2001–210417, *Proceedings of the 4th Conference on Aerospace Materials, Processes, and Environmental Technology*, D.E. Griffin and D. Cross Stanley, Eds., NASA Marshall Space Flight Center, Huntsville, AL, pp. 899–902, September 2000.

9. Melendez, O.; Trizzino, M.; and Fedderson, B.: "Evaluation of AK-225®, Vertrel® MCA, and HFE A 7100 as Alternative Solvents for Precision Cleaning and Verification Technology," in *Proceedings of the Second Aerospace Environmental Technology Conference*, A.F. Whitaker, M. Clark-Ingram, and S.L. Hessler, Eds., NASA Marshall Space Flight Center, Huntsville, AL, August 6–8, 1996, NASA Conference Publication 3349, pp. 319–327, March 1997.

10. Ross, H.R.: "The Use of HFC (CFC Free) Processes at the NASA Stennis Space Center," in *Proceedings of the Second Aerospace Environmental Technology Conference*, A.F. Whitaker, M. Clark-Ingram, and S.L. Hessler, Eds., NASA Marshall Space Flight Center, Huntsville, AL, August 6–8, 1996, NASA Conference Publication 3349, pp. 299–309, March 1997.

11. Wittman, C.L.; and Eichinger, E.C.: “The Search for a Replacement for CFC 113 in the Precision Cleaning and Verification of Shuttle Hardware,” in *Proceedings of the Second Aerospace Environmental Technology Conference*, A.F. Whitaker, M. Clark-Ingram, and S.L. Hessler, Eds., NASA Marshall Space Flight Center, Huntsville, AL, August 6–8, 1996, NASA Conference Publication 3349, pp. 311–318, March 1997.

12. ASTM D4809-13, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method), ASTM International, West Conshohocken, PA, 2013, <http://www.astm.org>.

13. Mitchell, M.; and Lowrey, N.: “The Search for Nonflammable Solvent Alternatives for Cleaning Aerospace Oxygen Systems,” <http://ntrs.nasa.gov/archive/nasa/casi.ntrs.nasa.gov/20130003208.pdf>, Conference Presentation, 2012 International Workshop on Environment and Alternative Energy, Greenbelt, MD, December 4–7, 2012.

14. National Emission Standards for Hazardous Air Pollutants for Source Categories, Code of Federal Regulations, Title 40, Vol. 9, Part 63, Subpart F, Table 2, <http://www.gpo.gov/fdsys/pkg/CFR-2011-title40-vol9/xml/CFR-2011-title40-vol9-part63.xml#seqnum63.56>, July 1, 2011.

15. Federal Register, 40 CFR, Vol. 1, Subchapter C, Part 51.100(s), <http://www.ecfr.gov/cgi-bin/retrieveECFR?gp=1&SID=8a0f4130892dbc70a23fd84d74dc96c0&ty=HTML&h=L&r=PART&n=pt40.2.51#se40.2.51_1100>, July 1, 1999.

16. Myhre, G.; Shindell, D.; Breon, F.-M.; et al.: “Anthropogenic and Natural Radiative Forcing,” in *Climate Change 2013*: *The Physical Science Basis. Contribution of Working Group I to the Fifth Assessment Report of the Intergovernmental Panel on Climate Change*, T.F. Stocker, D. Qin, G.-K. Plattner, et al., Eds., Cambridge University Press, Cambridge, United Kingdom and New York, NY, USA, 2013.

17. Federal Register, Vol. 77, No. 75, 48 CFR Parts 1, 2, 11, 23, and 52, Federal Acquisition Regulation; Biobased Procurements (final rule), section II.A.5, <http://www.gpo.gov/fdsys/pkg/FR-2012-04-18/html/2012-9291.htm>, April 18, 2012.

18. MSFC-SPEC-164B, Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, Specification for, NASA Marshall Space Flight Center, Huntsville, AL, November 19, 1994.

19. ASTM D1133-13, Standard Test Method for Kauri-Butanol Value of Hydrocarbon Solvents, ASTM International, West Conshohocken, PA, 2013, <http://www.astm.org>.

20. MIL-PRF-83282D, Amendment 1, Performance Specification: Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, NATO Code Number H-537, December 10, 1997.

21. ASTM F483-09 (Reapproved 2014), Standard Practice for Total Immersion Corrosion Test for Aircraft Maintenance Chemicals, ASTM International, West Conshohocken, PA, 2014, <http://www.astm.org>.

22. ASTM D2240-05 (Reapproved 2010), Standard Test Method for Rubber Property—Durometer Hardness, ASTM International, West Conshohocken, PA, 2010, <http://www.astm.org>.

23. MIL-G-21569B(SHIPS), Military Specification, Gaskets, Cylinder Liner Seal, Synthetic Rubber, Naval Sea Systems Command (Ship Systems), February 5, 1965; validated May 9, 1989.

24. 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, ASTM International, West Conshohocken, PA, 2011, <http://www.astm.org>.

25. NASA-STD-6001B, Flammability, Offgassing, and Compatibility Requirements and Test Procedures, National Aeronautics and Space Administration, Washington, DC, August 26, 2011.

26. ASTM G72/G72M-09, Standard Test Method for Autogenous Ignition Temperature of Liquids and Solids in a High-Pressure Oxygen-Enriched Environment, ASTM International, West Conshohocken, PA, 2009, <http://www.astm.org>.

27. WSTF-IR-0134, United States Air Force Wipe Solvent Testing, Investigative Report, Lyndon B. Johnson Space Center, White Sands Test Facility, Las Cruces, NM, November 13, 2000.

28. ASTM E1235-08, Standard Test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft, ASTM International, West Conshohocken, PA, 2008, <http://www.astm.org>.

29. ASTM D4265-98 (Reapproved 2007), Standard Guide for Evaluating Stain Removal Performance in Home Laundering, ASTM International, West Conshohocken, PA, 2007, <http://www.astm.org>.

30. ASTM G86-89 (Reapproved 1995), Standard Test Method for Determining Ignition Sensitivity of Materials to Mechanical Impact in Pressurized Oxygen Environments, ASTM International, West Conshohocken, PA, 1995, <www.astm.org>.

31. ASTM G63-99 (Reapproved 2007), Standard Guide for Evaluating Nonmetallic Materials for Oxygen Service, ASTM International, West Conshohocken, PA, 2007, <http://www.astm.org>.

32. ISO 12103-1, Road Vehicles—Test Dust for Filter Evaluation—Part 1: Arizona Test Dust, First edition, December 15, 1997.

Return to Menu Pg 2 Pg 4 Pg 46 Pg 48

APPENDIX A—TEST PLAN FOR SOLVENT REPLACEMENT FOR CLEANING AND VERIFICATION SAMPLING OF MSFC/SSC PROPULSION OXYGEN SYSTEMS HARDWARE, GROUND SUPPORT EQUIPMENT, AND ASSOCIATED TEST SYSTEMS, REVISION A, JULY 26, 2013

The test plan for solvent replacement for cleaning and verification sampling is given in appendix A.

Test Plan for Solvent Replacement for Cleaning and Verification Sampling of MSFC/SSC Propulsion Oxygen Systems Hardware, Ground Support Equipment, and Associated Test Systems

MSFC/SSC joint test plan

Contacts:

MSFC: Mark Mitchell (EM50)
M&P Environmental Effects Branch
(256) 544-5860

SSC: Bruce Farner (RA20)
(228) 688-2967

WSTF: Susana Harper (RF111) (For LOX/GOX flammability and reactivity testing)
(575) 524-5418

INTRODUCTION

Liquid and gaseous oxygen (LOX/GOX) systems used in bipropellant propulsion systems require a high level of cleanliness to support engine performance requirements and prevent mishaps. Systems to be wetted by gaseous or liquid oxygen, and systems providing pressurization or purge (P&P) gases to these systems, must be clean, particularly of hydrocarbons and large (> 800 μ) particles, 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. This test plan establishes test methods for evaluating solvents to be used for cleaning of propulsion oxygen systems and associated P&P systems, ground support equipment, and test systems at Marshall Space Flight Center (MSFC) and Stennis Space Center (SSC), and for sampling of these systems for verification of cleanliness.

Historically, chlorofluorcarbon-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, hydrochlorofluorocarbon-225[1] (HCFC-225), a Class II ODS, was selected by MSFC and SSC to replace CFC-113 for final cleaning and verification where a suitable non-ODS replacement could not be found. MSFC and SSC later converted from HCFC-225 to the single isomer HCFC-225cb[2], a less toxic product. In this test plan, HCFC-225cb is used as the baseline solvent for comparison of cleaning efficiency and materials compatibility. The goal is to identify a single replacement solvent that meets or exceeds the performance of HCFC-225cb in all user applications. Given the physical limitations of non-ODS solvents, this may not be fully achievable and selective compromises may be required.

---

[1] 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). This solvent is sometimes referred to as HCFC-225ca/cb.

[2] Manufactured by Asahi Glass Company, Ltd., Tokyo, Japan; supplied in North America by AGC Chemicals Americas, Exton, PA, under the product name Asahiklin AK-225G. AK-225G contains only the less toxic isomer (>99%) 1,3-Dichloro-1,1,2,2,3-pentafluoropropane (HCFC-225cb) (CAS Number 507-55-1). This solvent has been referred to in some publications as HCFC-225G.

1. Scope

1.1. This plan describes test methods for evaluating the suitability of solvents for cleaning of LOX/GOX and supporting system components and for sampling of these components to verify removal of nonvolatile residues (NVR).

1.2. Test procedures are specified for:

a. Measuring and analyzing the background nonvolatile residue (NVR) content in as-received (and purified, if required) solvent.

Rationale: Solvent used to clean and verify LOX/GOX systems must have a low background NVR. The limit for background NVR varies by application. The highest limit permitted for cleaning of MSFC propulsion systems is 4 mg/200mL (MSFC-SPEC-164C). Rinse solvent for NVR verification at SSC is limited to a background NVR of 1 mg/200mL. This test will determine whether the as-delivered test solvent must be purified prior to testing for cleaning efficiency and prior to production use.

b. Evaluating the compatibility of cleaning solvents with materials used to construct liquid and gaseous oxygen (LOX/GOX) systems.

Rationale: Solvents must not corrode the metals of the hardware being cleaned. To minimize processing costs, solvents that are compatible with commonly used nonmetals (soft goods) are preferred. However, aggressive NVR cleaning solvents such as HCFC-225cb are not compatible with some soft goods such as Viton. Testing of solvents with nonmetals is performed for comparison with other solvents and to identify materials that will require special handling, such as removal and replacement, when the solvent is used.

c. Measuring the effectiveness of the cleaning solvent at removing contaminants commonly requiring cleaning from LOX/GOX systems.

Rationale: The solvent must be capable of removing contaminants that pose a safety hazard to oxygen systems. The solvent must be capable of removing contaminants to below 1 $mg/0.1m^2$*, the pass/fail criterion for most LOX/GOX system components. No solvent is 100% effective at cleaning all types of contaminants; therefore a baseline solvent (in this case HCFC-225cb) is tested in parallel for comparison purposes.*

d. Evaluating the reactivity of the solvent in LOX/GOX.

Rationale: For safe use in LOX/GOX systems where complete removal of the solvent cannot be assured, the solvent must be non-reactive with LOX/GOX at the expected temperature and pressure conditions of the system. Historically, due to the variety of LOX/GOX systems that may be cleaned at MSFC and SSC and the extreme consequences of a fire in one of these systems, very conservative test methods and acceptance criteria have been used. CFC-113, HCFC-225ca/cb and HCFC-225cb all passed the highest acceptance criteria for the test methods in this plan.

1.3. These cleaning solvent criteria are discussed in ASTM G 127 *Standard Guide for the Selection of Cleaning Agents for Oxygen Systems.* ASTM G 127 lists materials and test methods to be considered when selecting cleaning agents. The materials and contaminants specified in this plan were selected using ASTM G 127 and ASTM MNL36 *Safe Use of Oxygen and Oxygen Systems* as a guide; considering historical and current propulsion system designs; and with input from users at NASA's Marshall Space

Flight Center and its Michoud Assembly Facility and at Stennis Space Center regarding cleaning challenges with LOX/GOX systems encountered at these locations. Future designs, system operating conditions, and other production and test facilities may require different materials and encounter different contaminants than those specified here. Additional testing may be required to assess the suitability of the solvent for new projects or applications.

1.4. The test methods specified in this plan for flammability and reactivity of solvents in LOX/GOX were selected to replicate tests that were performed in the past to evaluate HCFC-225ca/cb and HCFC-225cb for both NASA and the United States Air Force. This approach was selected to maximize the data available for direct comparison to HCFC-225cb performance. The HCFC-225ca isomer is much more toxic than the HCFC-225cb isomer, therefore HCFC-225ca/cb is no longer used at NASA. Testing for oxygen compatibility at higher pressures may be required for future systems and is beyond the scope of this plan.

1.5. The cleaning performance of a particular solvent depends upon the cleaning method used including details such as time, temperature, and mechanical action applied during cleaning. Cleaning performance also depends on the materials, configuration, and condition of the item being cleaned, including surface texture, surface finish, complexity of the part, and type of contaminants present. Final evaluation of the solvent is performed by end user testing on full-scale hardware items using current HCFC-225cb cleaning and NVR verification processes in use at MSFC and SSC.

Note: Evaluation of replacement solvents for applications that do not currently use HCFC-225cb, or for HCFC-225cb applications at other facilities that are substantially different from propulsion system applications, is beyond the scope of this plan.

1.6. In addition to these performance requirements, use of the solvent must meet applicable Safety, Health, and Environmental (SHE) requirements for the intended use. Evaluation of solvents for personnel safety precautions, life cycle cost, and environmental impact is the responsibility of the end user organization. These attributes must be assessed by the user for the expected end use application to determine required changes to equipment and operating procedures to protect the health of workers and to comply with federal, state, and local regulations.

1.7. During the performance of these test methods, minor adjustments to the test protocols described here may be required to accommodate solvents and contaminants that vary in properties. Adjustments to the test protocols will be agreed upon by the test project stakeholders and documented in the test report.

2. Reference Documents

2.1. NASA

KSC-SPEC-P-0022 *Solvent, Cleaning, 1,3-Dichloro-1,1,2,2,3-Pentafluoropropane, HCFC-225G, Specification for*

MSFC-SPEC-164C *Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems, Specification for*

RPTSTD-8070-0001 *Surface Cleanliness Standard of Fluid Systems for Rocket Engine Test Facilities of the NASA Rocket Propulsion Test Program*

2.2. Military

Mil-H-5606 *Hydraulic Fluid, Petroleum Base, Aircraft, Missile, and Ordnance*

MIL-PRF-83282 *Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Metric, NATO Code Number H-537*

2.3. General Services Administration

A-A-59743 *Cleaning Compound, Dichloropentafluoropropane (HCFC-225)*

2.4. Industry Voluntary Consensus Standards

ASTM D 1430 *Standard Classification System for Polychlorotrifluoroethylene (PCTFE)*

ASTM D 2109 *Standard Test Methods for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures*

ASTM D 2240 *Standard Test Method for Rubber Property – Durometer Hardness*

ASTM D 2512-95(2008) *Standard Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques)*

ASTM D4265-98(R 2007) *Standard Guide for Evaluating Stain Removal Performance in Home Laundering*

ASTM E 1235-08 *Standard test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft*

ASTM F 483-09 *Standard Practice for Total Immersion Corrosion Test for Aircraft Maintenance Chemicals*

ASTM G 86-98a (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*

ASTM G122 *Standard Test Method for Evaluating the Effectiveness of Cleaning Agents*

ASTM G127 *Standard Guide for the Selection of Cleaning Agents for Oxygen Systems*

ASTM MNL36 *Safe Use of Oxygen and Oxygen Systems*

ISO 12103-1 *Road vehicles — Test dust for filter evaluation — Part 1: Arizona test dust*

2.5 Technical Publications

SMC-TR-95-28 *Nonvolatile Residue Solvent Replacement*, Arnold, G. S. and Uht, J. C., The Aerospace Corporation, El Segundo, CA, March 1, 1995.

3. Test Procedures

3.1. Test Method for Nonvolatile Residue Background in Neat Cleaning Solvents

This test method is equivalent to ASTM D 2109, Standard Test Methods for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures Method C, using a solvent sample of 200 mL instead of 1000 mL.

3.1.1. Sampling Procedure

3.1.1.1. Gently agitate the as-received container of solvent prior to sampling to counteract any settling or stratification of constituents in the solvent being sampled. This may be performed by swirling the solvent in the container or rolling the container sideways prior to opening.

Return to Pg 11

3.1.1.2. On completion of agitation, draw 200 mL of solvent using a glass pipette and transfer to a glass container, or deliver directly to the filtration system.

Note 1. All pipettes, glassware, and evaporating dishes used in this procedure must be precleaned to remove background particulate and NVR to below 0.1 mg/0.1 m^2. Use of verified cleaning procedures for the dishware is adequate for acceptance of the cleanliness of these items.

3.1.1.3. Measure the pH of the solvent with litmus paper and record on a solvent data sheet.

3.1.2. Gravimetric NVR analysis procedure

3.1.2.1. Filter the solvent sample through a 10 micron pore size or less non-gridded filter membrane, compatible with the fluids being tested. Collect the filtered solvent into a cleaned container.

3.1.2.2. Separate the filtered 200 mL sample into a 180-190 mL portion to evaporate and reserve the 10-20 mL portion for rinsing of the evaporation vessel. Cover the container containing the remaining 20 mL with a watch glass or Teflon lined lid.

3.1.2.3. Evaporate a 180 mL of the solvent sample to a 10–20 mL volume using a steam bath or vacuum rotary evaporator or a thermostatically controlled hot plate.

3.1.2.4. Transfer the 10-20 mL evaporated sample to a clean 50 mL constant weight (within 0.1 mg), tared glass weighing container (see note 2), which was previously weighed to the nearest 0.1 mg. The cleaned weighing container shall be handled only with cleaned tongs or forceps. Rinse the sample container (evaporation flask) twice with approximately half of the remaining 20 mL of filtered fluid and transfer the rinse fluid to the weighing container. All of the remaining 20 mL of filtered fluid shall be used.

3.1.2.5. A calibrated analytical balance shall be used for all weight measurements.

Note 2. An aluminum weighing pan may be used if the compatibility of the solvent with aluminum has first been verified.

3.1.2.6. Continue evaporation by placing the weighing dish inside a constant-temperature oven at a temperature of 105 ± 5 ^{o}C. Allow the weighing dish to remain inside the oven until the fluid has just evaporated to dryness.

3.1.2.7. Remove the weighing dish from the oven and place in a desiccator to cool for 30 minutes. Remove the cool weighing dish and weigh to the nearest 0.1 mg.

3.1.2.8. Subtract the weight of the tared weighing dish to obtain the weight of the residue in mg. Record the weight as mg/200 mL.

3.1.2.9. Calculate the NVR in ppm by weight as follows:

$$\text{NVR ppm by weight} = \frac{(A)(1000)}{\rho}$$

Where:

A = residue in mg/200mL

ρ = density of solvent in kg/L at 25 ^{o}C

3.1.3. Infrared analysis of the nonvolatile residue

3.1.3.1. This procedure involves dissolving the residue obtained from the solvent in section 2 in tetrachloroethylene and analyzing the solution with an infrared spectrometer. This is a semi-quantitative procedure in that some residues may not exhibit infrared peaks within the detection range of the instrument. Comparison of the gravimetric result in mg/200 mL to the calculated result derived by this method may indicate that some of the residue has not been detected by the spectrometer and may warrant further investigation.

3.1.3.2. Equipment

a. Fourier Transform Infrared (FTIR) Spectrometer

b. Quartz cell, 20mm pathlength (10 mm optional)

c. Pharmaceutical grade mineral oil

d. Tetrachloroethylene, spectrometric grade

e. Temperature controlled oven (optional)

3.1.3.3. Calibration

a. Before using this method, the FTIR spectrometer shall be initially calibrated.

b. Follow manufacturer's instructions for internal optical alignment if required, and optimal energy through put. Instrument parameters (number of scans, wave number resolution, gain ranging radius, etc.) should be adjusted as needed to optimize results.

c. Using tetrachloroethylene in a 20 mm quartz cell, perform a background (single beam) scan daily or before use between 3200 cm^{-1} and 2600 cm^{-1}. The background spectrum will be used to develop absorbance spectra for the calibration standards in (d, e and f below).

d. For a 50 ml sample aliquot, calibrate the FTIR spectrometer by preparing mineral oil standards in tetrachlorethylene.

e. A minimum of five calibration points shall be obtained.

f. The final calibration concentrations shall range from 0.02 mg/ml to an upper limit of 0.36 mg/ml. The 0.02mg/ml mineral oil standard in tetrachloroethylene represents 0.5 mg of NVR in a 200ml sample brought down to dryness and reconstituted with 7 ml of terachloroethylene.

g. Progressing from the lowest calibration standard to the highest standard, scan the various hydrocarbon blends in the 3200 cm^{-1} and 2600 cm^{-1} range using a 20 mm quartz cell. If a representative aliquot or entire of the sample volume is analyzed, prepare equivalent calibration standards based on the total amount of solvent used (e.g., 50 ml of a 200 ml sample with an NVR of 1 mg/200 ml = 0.25 mgs. of NVR ml 0.25 mgs. reconstituted with 7 ml of tetrachloroethylene is 0.036 mg/ml. Therefore, an equivalent NVR (hydrocarbon) standard of 0.036 mg/ml would represent 1 mg/200 ml from a 50 ml aliquot.The quartz cell should be flushed twice with tetrachloroethylene and a minimum of two times using a standard of the next highest concentration level when changing standards.

h. Use the spectra from Sections D, E and F to compute the least squares regression calibration coefficients. The correlation coefficient for the linear regression or quadratic model should be 0.990 or better. Either peak intensity or area can be modeled. The calibration curve cannot have a y intercept of zero, and it should not be forced to go through zero.

3.1.3.4. Infrared Analysis Procedure

a. Perform a background scan daily with tetrachloroethylene or before use between 3200 cm -1 and 2600 cm-1. The single beam background spectra will be used to develop absorbance spectra for analyzing samples.

b. Following the initial calibration (Section 3.2) and the background scan (a), analyze check standards daily or before sample analysis. The check standards shall represent 1mg/200 ml and 4 mg/200ml. The check standards (mineral oil in tetrachloroethylene) should read within ±10%.

c. To the residue obtained in section 2, after completion of weighing, add 7 mL of tetrachloroethylene to the residue.

d. Briefly swirl the solvent over the residue and transfer the solution to a 20 mm quartz cell. Use care during transfer to avoid loss of the sample due to spilling or overfilling of the cell.

e. Record the sample infrared absorption spectrum from 3200 to 2600 cm^{-1} using the same cell pathlength that was used to develop the calibration curve.

f. Determine the amount of NVR (mgs of hydrocarbon) in the sample by using the least squares regression calibration curve (section 3.3). If the sample concentration is greater than the highest calibration standard, a dilution of the sample with tetrachloroethylene shall be made and the appropriate dilution factor applied.

$$NVR = (M_S)\ (Vs)$$

Where:

M_S = FTIR measured sample NVR concentration (mg/200 mL)

Vs = Tetrachloroethylene volume used to reconstitute the sample residue (e.g., 7 mL)

NVR = mg of NVR (hydrocarbon) contained in the 200 mL solvent sample

3.1.4. If the result of the NVR analysis in section 3.1.2.6 is significantly higher than the result in section 3.1.3.4f, then some of the residue may not have been detected in the wavelength range of the FTIR spectrometer. This should be noted in the test report.

3.1.5. If the solvent sample as received is found to exceed 4 mg/200mL[3], the solvent must be distilled to lower the NVR level prior to testing for NVR removal efficiency, section 3.3.3.

---

[3] 10 mg/500mL (4 mg/200mL) is the maximum allowable NVR in the final rinse fluid per MSFC-SPEC-164C. KSC-SPEC-P-0022, Solvent, Cleaning, 1,3-Dichloro-1,1,2,2,3-pentafluoropropane, HCFC-225G, specifies 10 ppm by weight (max) (3.12 mg/200mL). (AK-225G has a density of 1.56 g/mL at 25^{o}C.) Commercial Item Description A-A-59743, *Cleaning Compound Solvent, Dichloropentafluoropropane (HCFC-225),* specifies 2.0 ppm by weight, (max) (0.6 mg/200mL) for both HCFC-225ca/cb and HCFC-225cb. RPSTD-8070-0001 specifies a max residue for AK-225 as 2 ppm (max) but does not indicate whether this is ppmv or ppm by weight; solvents used for final rinsing and verification are limited to 4 mg/200mL for tanks or vessels and 1 mg/200mL for all other items.

3.1.5.1. Repeat measurement of the solvent pH after distillation of solvent.

3.1.5.2. The solvent vendor shall be consulted prior to distillation to determine any special distillation requirements. Stabilizers could be removed by the distillation process, requiring addition of new stabilizer to prevent solvent deterioration.

3.1.6. Infrared analysis of the solvent

3.1.6.1. To determine the infrared signature of the solvent, for evaluation of lot to lot consistency and whether the neat solvent may be used as the FTIR carrier solvent instead of tetrachloroethylene, enabling the lab to directly test sampled solvent, transfer 7 ml of the solvent to the 20 mm quartz cell and record the infrared absorption spectrum from 3200 to 2600 cm^{-1} using the same cell pathlength as in 3.1.3.4.

3.1.6.2. Note any significant differences in the recorded absorption spectra that may be attributable to the solvent rather than the NVR content.

3.1.6.3. The infrared signatures of each solvent recorded at different test labs shall be compared to validate the consistency of the solvent samples between labs.

3.2. Test Methods for Materials Compatibility with Cleaning Solvents

3.2.1. Two classes of materials require compatibility testing, metals and nonmetals. The test methods shown below are selected for common materials used as LOX/GOX system materials of construction. If the solvent will be used on components containing materials that are not typically used in oxygen systems, such as titanium and polyvinyl chloride, additional tests and different test methods may be required to evaluate the solvent for use with those materials.

3.2.1.1. Extended exposure testing, required to verify compatibility of the solvent with nonmetallic seals used in cleaning and distillation systems, is not included as a part of this test plan. Although this data is important for implementation, this is not a selection criterion for the solvent.

3.2.1.2. Cleaning solvents that are effective at solvating hydrocarbon-based or silicone-based contaminants will be incompatible with some nonmetals. Incompatibility data is required primarily to identify limitations of the solvent.

3.2.2. Test Method for Solvent Corrosiveness with Metals

3.2.2.1. The corrosiveness of the solvent on metals shall be 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.

3.2.2.1.1. Solvent shall be tested in the as-received condition.

3.2.2.1.2. Metal coupons shall be cut from sheet stock to a nominal size of 50.8 mm by 25.4 mm (2 in. by 1 in.). Thickness of the coupons should be approx. 1.6 mm (0.06 in.) but may vary based on availability of sheet stock. Metals that are not supplied or used as sheet stock may be fabricated from other stock to a similar surface area. Coupons will be tested in the unstressed condition.

3.2.2.1.3. The thickness and stock type (sheet or plate) of the test coupons shall be recorded in the test report.

3.2.2.1.4. Metals to be tested with the candidate solvent shall be:

a. 304L stainless steel

b. A-286 PH stainless steel

c. 17-4 PH stainless steel

d. 440C stainless steel

e. AISI 4140 low alloy steel

f. Tin-Bronze

g. Brass – Admiralty brass CDA 443 (Copper Development Association)

h. Co-Cr-Ni alloy - Elgiloy® (Elgiloy Specialty Metals) or Haynes® 188 (Haynes International)

i. 2219-T6 aluminum

j. 6061-T6 aluminum

k. 2195 T8 or T3M4 aluminum-lithium (plate stock)

l. Inconel™ 718 (Specialty Metals Corp.)

m. Monel™ 400 (Specialty Metals Corp.)

3.2.2.1.5. Coupons shall be pre-cleaned with a low-NVR solvent known to be compatible with the metal, examined, immersed in the test solvent at the solvent boil temperature for 24 hours, removed to a desiccator for 30 minutes, and examined and weighed. The coupons shall then be returned to the same test solvent for an additional 144 hours, then the desiccation, examination and weighing repeated. To prevent excessive solvent evaporation during testing, a Soxhlet extractor, reflux system, or equivalent must be used for continuous solvent vapor recovery.

3.2.2.1.6. Seven test coupons, three for immersion, three for suspension in the solvent vapor, and one for a control, shall be used for each metal with each solvent.

3.2.2.1.7. Coupons shall be examined before and after exposure to the solvent for weight loss or gain, and for visual evidence of attack such as pitting, etching, or discoloration. ASTM F 483-09 contains a recommended report form.

3.2.3. Test Method for Solvent Compatibility with Nonmetals (Soft Goods)

3.2.3.1. The compatibility of the solvent with nonmetals shall be evaluated under conditions of total immersion at the boil temperature of the solvent. This test method is similar to ASTM F 483-09.

3.2.3.2. No cleaning solvent is expected to be compatible with all nonmetals therefore exposure to the baseline HCFC-225cb solvent shall be included in the test matrix for comparative purposes.

3.2.3.3. Solvent shall be tested in the as-received condition.

3.2.3.4. Test specimens shall be o-rings or gaskets with an inner diameter of 12.7 mm to 25.4 mm (0.50 in to 1.0 in, with all specimens of a specific material nominally of the same dimensions.

3.2.3.5. Four test specimens, three for immersion and one as a control, shall be used for each material with each solvent.

3.2.3.6. Nonmetals to be tested for compatibility with the solvent shall be:

a. FKM poly(hexafluoropropylene-co-vinylidene fluoride) elastomer Viton A (DuPont) or FKM Compound V0747-75 (Parker Hannafin)

b. FFKM poly(tetrafluoropropylene-co-perfluoromethylvinyl ether) elastomer - Kalrez (DuPont)

c. NBR poly(acrylonitrile-co-butadiene) rubber (Buna N) – Mil-G-21569B Class I

d. Polytetrafluoroethylene - PTFE Teflon (DuPont) or Algoflon E2 (Solvay Solexis)
e. FEP fluorinated ethylene-propylene copolymer - FEP Teflon (DuPont)
f. Polychlorotrifluoroethylene (PCTFE) – Kel-F 81 (3M) or Neoflon CTFE (Daiken) meeting the requirements of ASTM D 1430 Group 01 Class 1 Grade 3.
g. Aromatic polyimide - Vespel SP21 (DuPont)
h. PEEK polyketone – Victrex PEEK (LNP) or equivalent
i. PTFE with silica fiber – Gylon style 3502 for oxygen service (Garlock)

3.2.3.7. O-ring test specimens shall be pre-cleaned by wiping with a soft, low residue, lint free wiper and ethyl alcohol, air dried, and placed in a desiccator for approximately 24 hours. Following cleaning, specimens shall be handled only with clean stainless steel, tongs, forceps, or handling hooks.

3.2.3.8. Prior to immersion in the solvent, the specimens shall be weighed, measured for outer diameter in two perpendicular locations, and visually inspected for initial condition. Elastomers (items a. through c.) shall be measured for hardness in accordance with ASTM D 2240 Type A (Shore A durometer) at the point of maximum thickness.

3.2.3.9. Immersion of the specimens shall be performed in a glass container, fitted with a reflux condenser, on a thermostatically controlled hot plate controlled to maintain the solvent at boiling temperature. Stainless steel wire shall be used to separate and retain the specimens in the solvent.

3.2.3.10. The specimens shall be placed in the boiling solvent for 15 minutes, removed and allowed to air dry in a clean desiccator.

3.2.3.11. Thirty minutes after removal from the solvent, the specimens shall be weighed, measured, tested for hardness (elastomers only), and visually inspected for evidence of cracking, discoloration, or other evidence of attack.

3.2.3.12. Specimens exhibiting a weight gain or linear swell of greater than 1 % shall be returned to the desiccator for 24 hours and then re-measured.

3.2.3.13. Specimens continuing to exhibit a weight gain or linear swell of greater than 1 % after 24hours shall be returned to the desiccator for seven days and then re-measured.

3.3. Test Methods for Cleaning Effectiveness

3.3.1. The solvent must be effective at removing the NVR contaminants of concern for propulsion oxygen systems. A joint MSFC/SSC cleaning working group identified the following contaminants of concern:

a. Mineral oil – CAS 8042-47-5 (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, ISO grade 46 – Mobil DTE 25.
c. Synthetic hydraulic fluid, MIL-PRF-83282, fire resistant, synthetic hydrocarbon base - Castrol Brayco Micronic® 882.
d. Di-2-ethylhexyl sebacate (gauge calibration oil) - Monoplex® DOS (The C. P. Hall Co.) or equivalent.

e. Fingerprint simulated by synthetic sebum, modified Spangler soil per ASTM D4265-98 (R 2007), *Standard Guide for Evaluating Stain Removal Performance in Home Laundering*, section A2.16.2 supplied by Scientific Services S/D, Inc., Sparrow Bush , NY)

f. Fluorocarbon grease - Krytox® 240AC.

g. Heavy paraffinic grease (crane grease), Big Red

h. WD-40 from aerosol or bulk liquid (medium-heavy aliphatic hydrocarbons) – WD-40 Company

i. Particulate dust, ISO 12103-A4 course test dust (graded desert sand)

Note: The expected performance of the solvent for removal of particulate dust will be evaluated analytically by comparison of wetting index. Solvents found to be effective at removing NVR that have a wetting index equal to or higher than chlorofluorocarbon (CFC) – 113[4] are expected to adequately remove particulate from propulsion oxygen components. Wetting Index = (1000 x density) / (surface tension x viscosity). Particulate removal performance will be verified during the component level cleaning tests (section 5).

3.3.2. Quick Screen Solvency Test

3.3.2.1. Each candidate solvent shall be screened by this procedure for suitability as a solvent for common contaminants selected from the list in section 3.3.1.

3.3.2.2. Triple rinse with cb-225 five aluminum weighing dishes for each solvent to be evaluated, plus five for the baseline HCFC-225cb solvent for comparison, dry in a oven at 100°C for one hour, and weigh each dish on a calibrated balance accurate to ± 0.1 mg.

3.3.2.3. Prepare a batch of mixed contaminant by dissolving 500 mg of equal parts of the following contaminants in 100 ml of HCFC-225cb.

a. Mineral oil – CAS 8042-47-5

b. Mil-PRF-83282 hydraulic fluid – Castrol Brayco Micronic® 882

c. Di-2-ethylhexl sebacate (gauge calibration oil), Monoplex® DOS (The C. P. Hall Co.) or equivalent

d. WD-40

e. Krytox GPL103 compressor oil (representative of fluorocarbon oils and greases)

3.3.2.4. Apply one mL of this mixed contaminant with a pipette to each aluminum weighing dish, bake in an oven at 100 °C for one hour, cool in a desiccator for one hour, and reweigh.

3.3.2.5. Flush the contaminated weighing dish with 100 mL of the test solvent from a glass volumetric cylinder in three steps, capturing all of the flush solvent in a clean beaker.

a. With the contaminated dish on a flat surface, pour 30 mL of test solvent into the dish, swirl, and decant into the beaker.

b. Repeat step a. with an additional 30 mL.

---

[4] CFC-113 has a lower wetting index than HCFC-225cb. CFC-113 was used successfully for many years to remove particulate from NASA LOX/GOX systems, therefore cleaners with a wetting index equal to or higher than CFC-113 are expected to have satisfactory particulate removal performance.

c. Holding the dish, fold it slightly and slowly flush the remaining 40 mL of test solvent through each side of the fold, draining the solvent into the beaker such that the maximum surface area is exposed to the solvent.

Note: The collected test solvent effluent is not analyzed for the quick screen solvency test. If more data is desired, this effluent solvent may be evaporated to gravimetrically measure NVR removal or may be analyzed by FTIR to evaluate which constituents of the test contaminant mix were effectively solvated.

3.3.2.6. Dry, desiccate, and weigh the dish as in 3.3.2.4.

3.3.2.7. Calculate the weight of contaminant applied, weight of contaminant removed, and cleaning efficiency as the percentage of contaminant removed over contaminant applied. Note in the test report any visual anomalies.

3.3.2.8. The quick-look cleaning performance of the candidate solvents will be compared to the performance of HCFC-225cb to inform the candidate down-selection process. Solvents with removal efficiency below 90% are unlikely to be viable candidates for further testing. Historical data has shown HCFC-225ca/cb and/or HCFC-225cb to remove 96-99% of these contaminants.

3.3.3. Nonvolatile residue removal efficiency test

3.3.3.1. This test method is to assess the cleaning efficiency of a solvent to remove specific contaminants by ambient temperature flush. This method evaluates cleaning solvency without the use of heat or mechanical action beyond gravity flush. Contaminant removal efficiency is compared to the baseline solvent, HCFC-225cb. This test method is based on the NVR test method described in ASTM E 1235 and adapted by The Aerospace Corporation as described in the technical report SMC-TR-95-28.

3.3.3.2. This test shall be performed for contaminants 3.3.1 a. through h. with each candidate test solvent.

3.3.3.3. Equipment

a. NVR test panels, 304L stainless steel, fabricated per ASTM E 1235 figure 2 except that initial plate dimensions shall be 163 mm x 163 mm (6.4 in x 6.4 in) to yield a 152 mm x 152 mm (6 in x 6 in) flat, lipped surface. All dimensions are approximate. Thickness of the panel is not critical. The test panel surface shall be the as received finish, not electropolished. The surface finish of the test panels shall be measured and reported.

b. NVR test panel stand, as shown in ASTM E 1235 figure 4, to fit panel (a).

c. Drying oven, cleaned to remove background NVR, to dry the NVR test panels.

d. Calibrated analytical balance for weighing of sample contaminants prior to dissolving with solvent

e. Low form glass collection jars with Teflon sealed lid, 250 mL min.

f. Beakers and pipettes for creation and transfer of contaminant solutions

g. Oil-free aluminum foil, food service grade

h. Gloves, nitrile, low NVR clean

i. Apparatus to perform gravimetric NVR analysis in accordance with section 3.1.2.

3.3.3.4. Pre-clean the NVR test panels and glassware to remove residual NVR, dry in a clean drying oven, and protect from recontamination by wrapping with oil-free aluminum foil until use.

3.3.3.5. Separately mix each test contaminant shown listed in section 3.3.1 a. through h. in a volatile carrier solvent to create a solution with a concentration of 200 mg contaminant per 100 mL of carrier solvent. Thinner contaminants may be used without dilution.

3.3.3.6. Remove the foil from the NVR test panel and, with the plate tipped slightly away from the open corner, under a fume hood slowly apply 5 mL of solvated contaminant, or 10 mg of unsolvated contaminant, to the witness surface. If needed, tip the test panel to distribute the contaminant across the test surface while minimizing contaminant contact with the lip of the panel or loss of contaminant through the drain point. The objective is to apply the contaminant to a level of 40 mg/0.1 m^2. Air dry the panel and then bake in the horizontal position (to prevent gravity migration of the contaminant) for two hours at 55 ^oC (130 ^oF). After baking, protect the panel from particulate fallout until use with aluminum foil, a covered enclosure, or by storing the panel face down (resting on lip) on a clean surface. Do not contact the contaminated surface during handling or storage.

3.3.3.7. Under a fume hood, place the contaminated NVR test panel on the test panel stand with the open corner set to drain into a 250 mL glass collection container.

3.3.3.8. Flush the contaminated NVR test panel with 200 mL of test solvent from a Teflon wash bottle, flushing the complete contaminated surface and collecting all of the draining solvent into the glass collection container. Cover the beaker immediately to prevent particulate entry and loss of solvent.

3.3.3.9. After the test panel is visually dry, flush the NVR test panel with 200 mL of HCFC-225cb, collected in a second collection container. Cover the beaker immediately to prevent particulate entry and loss of solvent.

3.3.3.10. Perform a gravimetric NVR analysis on each collected test solvent in accordance with section 3.1.2, using 20 mL additional matching solvent to rinse the evaporating dish.

3.3.3.11. Calculate the approximate contaminant removal efficiency (Removal %) of the test solvent rinse as

$$\frac{(m_T - m_{TB})}{(m_T - m_{TB}) + (m_{AK} - m_{AKB})} \times 100 = \text{Removal \%}$$

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

3.3.3.12. This test shall be repeated three times for each contaminant with each test solvent. Removal efficiency for each set of three contaminant/solvent tests should be within 10%. If not, a retest shall be performed until at least three results are within 10%.

3.3.3.13. This test shall also be performed three times with HCFC-225cb as both the test solvent and the rinse solvent to establish a baseline removal efficiency of the HCFC-225cb alone.

3.3.3.14. Test results shall be reported as compared to the removal efficiencies of HCFC-225cb.

3.4. Test Methods for Flammability and Reactivity of Cleaning Solvents in LOX/GOX

3.4.1. For unrestricted use on existing NASA LOX/GOX propulsion systems, propulsion systems GSE, and associated test stands, the solvent shall be shown to be compatible with oxygen in accordance with these test methods. Use of solvents that are potentially reactive with LOX/GOX at the anticipated worst case end item use conditions of temperature and pressure shall be restricted to cleaning activities where complete drying and removal of the solvent can be assured.

3.4.2. LOX Mechanical Impact Test Method

3.4.2.1. The neat solvent shall be tested in accordance with ASTM G 86-98a (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 force.

3.4.2.2. Preparation of the solvent sample for test shall be in accordance with ASTM D 2512-95(2008), *Standard Test Method for Compatibility of Materials with Liquid Oxygen (Impact Sensitivity Threshold and Pass-Fail Techniques),* section 10.1 to obtain a sample thickness (depth) of 1.27 ± 0.13mm (0.050 ± 0.005 in.) in the specimen cup before freezing. (This determination is required due to variations from liquid to liquid in physical properties such as density, surface tension, and volatility.) A micrometer depth gage with leveling blocks is suggested for measurement.

3.4.2.3. If the solvent does not pass the LOX Impact Test at 98 J with no reactions in 20 impacts or not more than 1 reaction in 60 impacts, LOX impact threshold tests shall be performed (known as the “Bruceton method”) to determine the LOX reactivity of the solvent and to provide a basis for comparison of solvents that do not pass at 98 J. Solvents that react at impacts lower than 27 J (20 ft-lb) are incompatible with LOX and require no further testing.

3.4.2.4. Solvents that fail the LOX Mechanical Impact test at 98 J but pass at above 27 J or show anomalous results may be considered for further testing or evaluation.

3.4.3. Autogenous Ignition Test Method (AIT)

3.4.3.1. The neat solvent shall be 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. Tests shall be performed at 50 psi and 2000 psi.

3.4.3.2. To minimize the volatilization and loss of test solvent, step 8.4 of ASTM G72-09, flushing of the system twice with oxygen to purge the chamber of atmospheric gases, will not be performed. This will result in an oxygen concentration of approximately 81% for the test at 50 psi. At 2000 psi, the relative concentration of nitrogen will be negligible resulting in an oxygen concentration > 99%.

3.4.3.3. To decrease volatilization of the solvent prior to test, pre-chilling of the test solvent is permitted.

3.4.3.4. The following criteria have been used to determine suitability of a solvent for use with high pressure gaseous oxygen systems up to 2000 psi. Suitability of the solvent for use in gaseous oxygen at pressures greater than 2000 psi may require additional testing.

a. AIT > 400 F – Acceptable for use in oxygen systems.

b. AIT 250 F to 400 F – May be used with caution in oxygen systems.

c. AIT < 250 F – Not recommended for use in oxygen systems.

4. Evaluation of Data from Coupon-level Tests and LOX/GOX Tests

Prior to advancement to cleaning demonstration tests at the component level, test results for each solvent from section 3 will be evaluated and compared with the requirements of each user application. Some solvents may be suitable for one use but not suitable for other uses, such as use in field cleaning versus NVR verification sampling or use with stainless steel but not with aluminum. While it is preferred that a single solvent be qualified to replace all current uses of HCFC-225cb, this may not be achievable.

5. Component Level Cleaning Tests

5.1. Following down selection of candidate solvents, the selected solvent shall be tested by the users in intended final applications. The specific solvent process, contaminants, and components shall simulate expected worst case usage in launch vehicle production and test operations. These end uses will include ambient flush cleaning, ambient wipe cleaning, cleaning in an immersion system, and use as a sampling solvent for NVR cleanliness verification.

5.2. The identified users that will participate in the component level cleaning tests for MSFC and SSC propulsion test operations are:

- MSFC Valve and Component Lab - component cleaning (tubes, instruments)
- MSFC Propulsion Test Area - field cleaning
- MSFC Fabrication Shop Precision Cleaning Lab – NVR verification
- SSC Component Processing Facility – component cleaning
- SSC Calibration Lab – instrument cleaning, NVR verification
- SSC Propulsion test Area - field cleaning

5.3. Parameters to be evaluated during component level cleaning tests include:

- Cleaning of complex surfaces and blind holes (Valve lab)
- Handling of the solvent in a variety of end uses
- Ease of capture of drained solvent during NVR sampling
- Drying rate and effectiveness
- Operator acceptance (odors, handling concerns)

5.4. Components will be cleaned and/or sampled for NVR using the candidate solvent. This will be followed by verification of cleanliness using HCFC-225cb in accordance with the standard procedures for that operation to evaluate the effectiveness of the candidate solvent.

5.5. Drying will be evaluated by visual inspection and, where feasible, by a leak detector (THC analyzer) to verify removal. When drying is evaluated for a candidate solvent, this evaluation shall be performed prior to sampling of the component with HCFC-225cb.

5.6. Use of a THC analyzer to sense residual solvent will also serve to validate that this is an effective method to verify complete drying of the solvent.

Appendix A. Candidate Solvents to Test

Several new solvents and solvent azeotropes (stable blends at the solvent boiling point) have been developed since the 1990's to minimize ozone depleting potential and, in some cases, global warming potential. Some of these are now commercially available in the United States. Others are not yet approved for sale in the US, but are available from the vendor for test purposes. Two solvents that were tested in the past for use with oxygen systems (DuPont Vertrel MCA and 3M L-14780) but were not selected for use at that time are considered worthy of re-evaluation. After extensive surveys of solvent vendors and HCFC-225 (ca/cb and cb) users from NASA, DoD, and commercial aerospace companies; and review of current and historical NASA, Department of Defense, and industry literature; solvents that have been identified as potential candidates to replace HCFC-225cb in propulsion oxygen system applications are:

Single component solvents

- AGC Chemicals AE3000 (HFE-347pc-f2 [1,1,2,2-tetrafluoro-1-(2,2,2-trifluoroethoxy)-ethane])
- DuPont Capstone 4-I (> 90% perfluorobutyl iodide)
- Honeywell Solstice PF (HFO-1233zd(E) [Trans-1-chloro-3,3,3,-trifluoroprop-1-ene])
- Solvay Solkane (HFC-365mfc [1,1,1,3,3 pentafluorobutane])

Azeotropes with trans-1,2 dichloroethylene (tDCE)

- DuPont Vertrel MCA (38% tDCE / 62% HFC-43-10mee [1,1,1,2,2,3,4,5,5,5-decafluoropentane] with HQMEE stabilizer)
- AGC Chemicals AE3000AT (45% tDCE / 55% AE3000)
- 3M L-14780 developmental fluid (22% tDCE / 78% HFE-347mcc3 [methyl perfluoropropyl ether]) *HFE-347mcc3 is available from 3M as Novec 7000 Engineered Fluid. Although 3M offers other azeotropes of their Novec fluids with tDCE, the azeotrope of tDCE and Novec 7000 is not currently marketed; thus the "developmental" designation.*
- Solvay Solvokane (30% tDCE/ 70% HFC-365mfc)

Return to Menu

APPENDIX B—SOLVENT SAFETY DATA SHEETS

Various solvent safety data sheets can be found in the following sections within appendix B:

B.1 AGC Chemicals Americas (Asahi Glass Company, Japan) Asahiklin AK-225G (baseline)
B.2 Honeywell Solstice Performance Fluid
B.3 3M L-14780 Developmental Material
B.4 DuPont Capstone 4-I
B.5 AGC Chemicals Americas (Asahi Glass Company, Japan) Asahiklin AE3000
B.6 AGC Chemicals Americas (Asahi Glass Company, Japan) Asahiklin AE3000AT
B.7 Solvay Solkane 365 mfc
B.8 Solvay Solvokane
B.9 DuPont Vertrel MCA
B.10 Honeywell Solstice Spray Cleaner
B.11 Honeywell Solstice 1234ze

Return to App B

Return to Pg 2

5646

ASAHI GLASS COMPANY

MATERIAL SAFETY DATA SHEET

No.U-132 (rev. 9) **Page 1 of 6**

Product Name **Prepared:** 6-June-1994 **Revised:** 14-Nov-2001

ASAHIKLIN AK-225G

Section I - Company Identification

Manufacturer's Name
ASAHI GLASS Co., Ltd.

Address
1-12 Yurakucho 1-Chome
Chiyoda-ku, Tokyo 100-8405, JAPAN
Chemicals General Division, Fluorochemicals Division

Telephone Number for Information
011-81-3-3218-5479

Facsimile Number for Information
011-81-3-3218-7854

Supplier's Name
AGA Chemicals, Inc.

Address
2201 Water Ridge Parkway Suite 400,
Charlotte, NC28217, USA

Telephone Number for Information
704-329-7600
Facsimile Number for Information
704-357-6308

Emergency Contact (In USA, Canada)
1-800-424-9300

Section II- Composition/Information on Ingredients

Component: 1,3-Dichloro-1,1,2,2,3-pentafluoropropane (HCFC-225cb)
CAS No. 507-55-1
> 99%

Note: This product does not contain any CFCs.

Section III - Physical/Chemical Characteristics

Boiling Point: 56.1 deg.C
Specific Gravity: 1.558 @ 25 deg.C
Vapor Pressure: 0.035 MPa @ 25 deg.C
Freezing Point: -116 deg.C
Vapor Density (Air = 1): 7.0
Evaporation Rate (Diethyl ether=1): 0.85
Solubility in Water: 0.016g/100g H_2O @ 25 deg.C
Appearance and Odor: Clear, colorless liquid with slight ethereal odor.

Section IV - Fire and Explosion Hazard Data

Flash Point (Method Used): None (Tag Closed Cup & Cleveland Open Cup)
Flammable Limits: LEL = None; UEL = None
Extinguishing Media
As appropriate for combustibles in area.

Special Fire Fighting Procedures
Use water spray to cool containers. Self-contained breathing apparatus (SCBA) is required if drums rupture and contents are spilled under fire conditions.

Unusual Fire and Explosion Hazards
Containers may rupture under fire conditions. Decomposition may occur.

NFPA Hazard Code
NFPA codes are designed for use by firefighters, sheriffs, or other emergency response teams who are concerned with the hazards of burning or exploding materials. These NFPA codes are not intended to address the hazards of this product other than in a fire situation.
Decomposition of this product at temperature above 300 degree C can form hydrogen fluoride (HF), but HF will only accumulate with continuous exposure to excess heat in a sealed vessel.

a) Flammability - 0 b) Health - 2 c) Instability- 0

Section V- Reactivity Data

Stability
Stable
Condition to Avoid
Material is stable. However, avoid open flames and high temperature.
Incompatibility (Materials to Avoid)
Incompatible with alkali or alkaline earth metals-powdered Al, Zn, Be, etc.
Hazardous Decomposition or Byproducts
Decomposition products are hazardous. This compound can be decomposed by high temperatures (open flames, glowing metal surfaces, etc.) forming hydrochloric and hydrofluoric acids-possibly carbonyl halides.
Hazardous Polymerization
Will Not Occur

Section VI- Health Hazard Data

Route(s) of Entry
Inhalation: X
Skin: X
Eye: X
Ingestion: X

Health Hazard Data (Continued)

Animal Data:

1,3-Dichloro-1,1,2,2, 3-pentafluoropropane (HCFC-225cb)

Inhalation: 4-h LC50: 36,800 ppm in rats
Oral: LD50 : >5 g/kg in rats
Dermal: LD50: >2g/kg in rabbit.
Eye: Not irritant up to 0.1ml in rabbit.

Data from acute toxicity studies indicate that this compound has very low acute toxicity.
HCFC-225cb causes neither eye irritation nor dermal toxicity in standardized tests; skin application of this compound at high doses (2,000mg/kg body weight) produces no adverse effects. Therefore, the dermal LD50 is greater than 2,000mg/kg body weight. Oral administration of this compound at high doses (5,000mg/kg body weight) does not cause any mortality and the oral LD50 is greater than 5,000mg/kg body weight. This compound also has very low acute inhalation toxicity as measured by the concentration that cause 50% mortality in experimental animals, the LC50, listed above. Cardiac sensitization response in dogs is observed at approximately 20,000ppm for HCFC-225cb.

In 28-day inhalation studies with rat, the activity and responsiveness of the animals was reduced at 5,000ppm or greater. Toxicity was otherwise confined to the liver; liver enlargement and induction of peroxisomes was seen following treatment. In 28-day study with marmoset, exposure to HCFC-225cb at 5,000ppm caused somnolence during exposure and an increase of cytochrome P-450, indicative of an adaptive response to this compound. However, no liver enlargement was seen and virtually no peroxisome induction was observed.

This compound does not produce genetic damage in bacterial cell cultures (Ames Assay), CHL, and in-vivo unscheduled DNA syntheses assay. In one in-vitro study with mammalian cell cultures (human lymphocytes) genetic damage was produced but the response was only marginal.The overall evidence from these studies implies that this chemical is not genotoxic.

Carcinogenicity

HCFC-225cb is not listed by NTP, IARC or OSHA as a carcinogen.

Exposure Guidelines:

AK-225G
AEL* : 400ppm (8h-TWA)
TLV-TWA(ACGIH): Not established
PEL-TWA(OSHA): Not established
* AEL is the Acceptable Exposure Limit set by Asahi Glass Co., Ltd.
EEL* : 1000 ppm (time limit 15 min.)
2500 ppm (time limit 1 min.)
* EEL is the Emergency Exposure Limit set by Asahi Glass Co., Ltd.

Emergency Exposure Limits (EELs) are to be used for short-term emergency exposure control. They are concentrations of short periods which should not result in permanent adverse health effects or interfere with escape. They should not be confused with ACGIH TLV-TWA or TLV STEL values that are designed for repeated exposure guidelines. For the use of AK-225G, daily exposure limits such as AEL as well as EEL are to be followed. The EEL for AK-225G is needed to avoid anaesthetic effects which could prevent self-rescue. If an EEL is exceeded for specified duration, evacuation, sheltering in place or other mitigation steps should be taken.

Health Hazard Data (Continued)

Remarks
AELs (Asahi Glass Co., Ltd.) of HCFC-225cb is 400ppm. Though no ACGIH TLV or OSHA PEL are assigned, Asahi Glass temporarily recommends that workplace exposure level should be maintained at 400 ppm or less for this compound until the authorized control level such as ACGIH TLV or OSHA PEL are assigned.

Signs and Symptoms of Exposure

EYE CONTACT:
The compound may cause eye irritation.

SKIN CONTACT:
The compound may cause skin irritation.

INHALATION:
Inhalation of high concentrations of vapor is harmful and may cause hepatitis, heart irregularities, unconsciousness, or death. Intentional misuse can be fatal. Vapor reduces oxygen available for breathing and is heavier than air.

Emergency and First Aid Procedures

INHALATION:
If high concentrations are inhaled, immediately remove to fresh air. Keep persons calm. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

SKIN CONTACT:
In case of skin contact, flush with water. Get medical attention if irritation is present.

EYE CONTACT:
In case of eye contact, immediately flush eyes with plenty of water for 15minutes. Call a physician.

INGESTION:
No specific intervention is indicated as the compound is not likely to be hazardous by ingestion. Consult a physician if necessary. Do not induce vomiting because the hazard of aspirating the material into the lungs is considered greater than swallowing it.

Section VII- Precautions for Safe Handling and Use

Steps to Be Taken in Case Material is Released or Spilled

NOTES: Review “Fire and Explosion Hazard Data” and “Precautions to Be Taken in Handling and Storing” before proceeding with clean up. Use appropriate Personal Protective Equipment during clean up.

Dike spill. Prevent liquid from entering sewers, waterways or low areas. Ventilate area. Collect on absorbent material and transfer to steel drums for recovery/disposal. Comply with Federal, State, and local regulations on reporting releases.

Waste Disposal Method

Recover by distillation or remove to permitted waste disposal facility. Comply with Federal, State, and local regulations.

Precautions for Safe Handling and Use(Continued)

Precautions to Be Taken in Handling and Storing
Use with sufficient ventilation to keep employee exposure below recommended limits. Provide adequate ventilation for storage, handling, and use, especially for enclosed or low spaces. Avoid contact of liquid with eyes and prolonged skin exposure. Do not allow product to contact open flame or electrical heating elements because dangerous decomposition products may form.

Storage Conditions
Store in clean, dry, well-ventilated area. Do not heat above 30 deg.C.

Section VIII- Control Measures

Respiratory Protection
Use respiratory protection approved by NIOSH in USA or other equivalent in each country if exposure limits may be exceeded. Self-contained breathing apparatus (SCBA) is required if a large spill occurs.

Ventilation
Normal ventilation for standard manufacturing procedures is generally adequate. Local exhaust should be used when large amounts are released. Mechanical ventilation should be used in low places.

Eye/Skin Protection
Impervious gloves should be used to avoid prolonged or repeated exposure. Chemical splash goggles should be available for use as needed to prevent eye contact.

Other Precautionary Information

NPCA – HMIS (National Paint and Coating Association – Hazardous Materials Identification System) Hazard Rating

HMIS codes are intended for use in everyday workplace setting to provide a rapid indication of the occupational hazards associated with chemicals used in the workplace.

a) Flammability - 1 b) Health - 1 c) Reactivity- 0

Section IX- Regulatory Information

For European Union

EEC Classification: Not classified
Hazard Symbol: Not established
Risk phrases: Not established
Safety phrases: Not established, but recommend 23 (Don't breath gas/fumes/vapor/spray), 24/25 (Toxic in contact with skin and if swallowed), 36/37 (Irritation to eyes and respiratory system)
Council Directive 92/32/EEC Status: These chemicals are listed on the EINECS (HCFC-225cb: 208-076-9).

Regulatory Information (Continued)

For United States of America

SNAP Acceptable: HCFC-225cb is listed as SNAP acceptable substitutes for CFCs in the Solvent Cleaning Sector of the Clean Air Act.
Non-VOC: HCFC-225cb is exempted from VOC regulations in the Clean Air Act.
TSCA Status: HCFC-225cb is listed on the TSCA Inventory.
SARA Section 302: HCFC-225cb is not Section 302 hazard.
SARA Section 311, 312: Acute = Yes
Chronic = Yes
Fire = No
Reactivity = No
Pressure = No
SARA Section 313 = Yes

Section X- Transportation Information

UN No.: Not established
ADR / RID Status: Not regulated
IMDG Status: Not regulated
ICAO / IATA Status: Not regulated
US DOT Status: Not regulated

Section XI- Additional Information

This Material Safety Data Sheet is offered only for your information, consideration and investigation. Asahi Glass Co., Ltd. provides no warranties, either express or implied, and assumes no responsibility for the accuracy or completeness of the data contained herein.

WARNINGS
This substance harms public health and environment by destroying ozone in the upper atmosphere.

Return to App B | Return to Pg 3 | Return to Pg 9 | Return to Pg 36

Solstice™ Performance Fluid (PF)

000000017461

Version 1.2 Revision Date 08/12/2013 Print Date 10/08/2013

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	Solstice™ Performance Fluid (PF)
MSDS Number	:	000000017461
Product Use Description	:	Solvent
Company	:	Honeywell International, Inc. 101 Columbia Road Morristown, NJ 07962-1057
For more information call	:	800-522-8001 +1-973-455-6300 (Monday-Friday, 9:00am-5:00pm)
In case of emergency call	:	**Medical: 1-800-498-5701 or +1-303-389-1414**
	:	**Transportation (CHEMTREC): 1-800-424-9300 or +1-703-527-3887**
	:	
	:	(24 hours/day, 7 days/week)

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

Form	:	liquid, clear
Color	:	colourless
Odor	:	slight
Hazard Summary	:	This product is not flammable at ambient temperatures and atmospheric pressure. Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing. Causes asphyxiation in high concentrations. The victim will not realize that he/she is suffocating. Excessive exposure may cause central nervous system effects including drowsiness and dizziness. Excessive exposure may also cause cardiac arrhythmia. Do not breathe vapours or spray mist. Avoid contact with skin, eyes and clothing. At higher temperatures,

(>250°C), decomposition products may include hydrochloric acid (HCl), hydrofluoric acid (HF) and carbonyl halides. The ACGIH Threshold Limit Values (2007) for Hydrogen Fluoride are TLV-TWA 0.5 ppm and Ceiling Exposure Limit 2 ppm.

Potential Health Effects

Skin : Not classified as a skin irritant in animal testing.

Eyes : No adverse health effects are expected.

Ingestion : Unlikely route of exposure.
Effects due to ingestion may include:
Gastrointestinal discomfort

Inhalation : Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Causes asphyxiation in high concentrations. The victim will not realize that he/she is suffocating.
Excessive exposure may cause central nervous system effects including drowsiness and dizziness. Excessive exposure may also cause cardiac arrhythmia.

Chronic Exposure : None known.

Carcinogenicity

No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP, IARC, or OSHA.

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Chemical nature : Substance

Chemical Name	CAS-No.	Concentration
trans-1-Chloro-3,3,3-trifluoropropene	102687-65-0	>99.00 %

SECTION 4. FIRST AID MEASURES

Inhalation	: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Use oxygen as required, provided a qualified operator is present. Call a physician.
Skin contact	: After contact with skin, wash immediately with plenty of water. If symptoms persist, call a physician. Take off all contaminated clothing immediately. Wash contaminated clothing before re-use.
Eye contact	: Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. Call a physician if irritation develops or persists.
Ingestion	: If victim is fully conscious, give a cupful of water. Do not induce vomiting without medical advice. Never give anything by mouth to an unconscious person. Call a physician immediately.

Notes to physician

Treatment	: Treat symptomatically.

SECTION 5. FIREFIGHTING MEASURES

Suitable extinguishing media	: The product is not flammable. Use extinguishing measures that are appropriate to local circumstances and the surrounding environment. Water spray Carbon dioxide (CO2) Dry chemical Foam
Specific hazards during firefighting	: This product is not flammable at ambient temperatures and atmospheric pressure. However, this material can ignite when mixed with air under pressure and exposed to strong ignition sources. Container may rupture on heating. Cool closed containers exposed to fire with water spray. Do not allow run-off from fire fighting to enter drains or water courses.

Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Exposure to decomposition products may be a hazard to health.
In case of fire hazardous decomposition products may be produced such as:
Hydrogen fluoride
Gaseous hydrogen chloride (HCl).
Carbon monoxide
Carbon dioxide (CO2)
Carbonyl halides

Special protective equipment for firefighters : In the event of fire and/or explosion do not breathe fumes.
Wear self-contained breathing apparatus and protective suit.
No unprotected exposed skin areas.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Personal precautions : Immediately evacuate personnel to safe areas.
Keep people away from and upwind of spill/leak.
Wear personal protective equipment. Unprotected persons must be kept away.
Remove all sources of ignition.
Ventilate the area.
Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Avoid accumulation of vapours in low areas.
Unprotected personnel should not return until air has been tested and determined safe.
Ensure that the oxygen content is >= 19.5%.

Environmental precautions : Should not be released into the environment.
Do not flush into surface water or sanitary sewer system.
Prevent further leakage or spillage if safe to do so.
Prevent spreading over a wide area (e.g. by containment or oil barriers).

Methods for cleaning up : Contain spillage, and then collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container for disposal according to local / national regulations (see section 13).

SECTION 7. HANDLING AND STORAGE

Handling

Handling : Handle with care.
Do not use in areas without adequate ventilation.
Do not breathe vapours or spray mist.
Avoid contact with skin, eyes and clothing.
Pressurized container. Protect from sunlight and do not expose to temperatures exceeding 50 °C.
Follow all standard safety precautions for handling and use of compressed gas cylinders.
Use authorized cylinders only.
Protect cylinders from physical damage.
Do not puncture or drop cylinders, expose them to open flame or excessive heat.
Do not pierce or burn, even after use. Do not spray on a naked flame or any incandescent material.
Do not remove screw cap until immediately ready for use.
Always replace cap after use.

Advice on protection against fire and explosion : Can form a combustible mixture with air at pressures above atmospheric pressure.
Keep product and empty container away from heat and sources of ignition.

Storage

Requirements for storage areas and containers : Pressurized container: protect from sunlight and do not expose to temperatures exceeding 50 °C. Do not pierce or burn, even after use.
Keep containers tightly closed in a dry, cool and well-ventilated place.
Storage rooms must be properly ventilated.
Ensure adequate ventilation, especially in confined areas.
Protect cylinders from physical damage.
Store away from incompatible substances.

SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Protective measures	:	Ensure that eyewash stations and safety showers are close to the workstation location. Do not breathe vapours or spray mist. Avoid contact with skin, eyes and clothing.
Engineering measures	:	Use with local exhaust ventilation. Perform filling operations only at stations with exhaust ventilation facilities.
Eye protection	:	Do not wear contact lenses. Wear as appropriate: Goggles or face shield, giving complete protection to eyes
Hand protection	:	Impervious gloves Gloves must be inspected prior to use. Replace when worn.
Skin and body protection	:	Wear as appropriate: Solvent-resistant gloves Solvent-resistant apron and boots If splashes are likely to occur, wear: Protective suit
Respiratory protection	:	In case of insufficient ventilation wear suitable respiratory equipment. Wear a positive-pressure supplied-air respirator. For rescue and maintenance work in storage tanks use self-contained breathing apparatus. Use NIOSH approved respiratory protection.
Hygiene measures	:	Handle in accordance with good industrial hygiene and safety practice. Avoid contact with skin, eyes and clothing. Do not breathe vapours or spray mist. Ensure adequate ventilation, especially in confined areas. Remove and wash contaminated clothing before re-use. Contaminated work clothing should not be allowed out of the workplace. Keep working clothes separately. Wash hands before breaks and immediately after handling the product.

Exposure Guidelines

Components	CAS-No.	Value	Control parameters	Update	Basis

trans-1-Chloro-3,3,3-trifluoropropene	102687-65-0	TWA : time weighted average	(800 ppm)	2013	Honeywell:Limit established by Honeywell International Inc.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Physical state	:	liquid, clear
Color	:	colourless
Odor	:	slight
Melting point/freezing point	:	< -90 °C Method: OECD Test Guideline 102
Boiling point/boiling range	:	19 °C
Flash point	:	Method: ISO 2719 Note: not applicable
Flammability	:	The product is not flammable. Method: Flammability (gases)
Lower explosion limit	:	Note: None
Upper explosion limit	:	Note: None
Vapor pressure	:	1,516 hPa at 30 °C(86 °F)
Vapor density	:	Note: (Air = 1.0), not determined
Density	:	1.27 g/cm3

Water solubility	: 1.90 g/l at 20 °C Method: OECD Test Guideline 105
Partition coefficient: n-octanol/water	: log Pow: 2.2 at 25 °C
Ignition temperature	: 380 °C at 986.8 - 1,035.9 hPa Method: DIN 51794
Oxidizing properties	: The substance or mixture is not classified as oxidizing.
Molecular Weight	: 130.5 g/mol

SECTION 10. STABILITY AND REACTIVITY

Possibility of hazardous reactions	: Polymerization can occur.
Conditions to avoid	: Protect from heat/overheating. Keep away from direct sunlight. Heat, flames and sparks. Do not mix with oxygen or air above atmospheric pressure.
Incompatible materials to avoid	: Strong oxidizing agents Magnesium Aluminium
Hazardous decomposition products	: In case of fire hazardous decomposition products may be produced such as: Carbon monoxide Carbon dioxide (CO2) Carbonyl halides Gaseous hydrogen chloride (HCl). Gaseous hydrogen fluoride (HF).

SECTION 11. TOXICOLOGICAL INFORMATION

Acute inhalation toxicity	: LC50: 120000 ppm Exposure time: 4 h Species: rat
Skin irritation	: Species: rabbit Result: No skin irritation Classification: Not classified as a skin irritant in animal testing. Method: OECD Test Guideline 404 Exposure time: 4 h
Sensitisation	: Result: Does not cause skin sensitisation. Classification: Patch test on human volunteers did not demonstrate sensitisation properties.
	: Cardiac sensitization Species: dogs Note: Cardiac sensitisation threshold (dog): 25000 ppm.
Repeated dose toxicity	: Species: rat Application Route: Inhalation Exposure time: 4 Weeks NOEL: 4500 ppm Note: Subacute toxicity
Genotoxicity in vitro	: Test Method: Mutagenicity (Salmonella typhimurium - reverse mutation assay) Result: negative
Genotoxicity in vivo	: Species: rat Cell type: Bone marrow Method: Mutagenicity (micronucleus test) Result: negative
Genotoxicity in vivo	: Test Method: Unscheduled DNA synthesis Species: rat Result: negative

Genotoxicity in vivo	:	Species: mouse Cell type: Bone marrow Method: Mutagenicity (micronucleus test) Result: negative
Reproductive toxicity	:	Species: rabbit Note: No-observed-effect level - 15,000 ppm
	:	Species: rat Note: No-observed-effect level - 10,000 ppm
Teratogenicity	:	Species: rabbit Note: No-observed-effect level - 15,000 ppm
	:	Species: rat Note: No-observed-effect level - 10,000 ppm

SECTION 12. ECOLOGICAL INFORMATION

Ecotoxicity effects

Toxicity to fish	:	LC50: 38 mg/l Exposure time: 96 h Species: Oncorhynchus mykiss (rainbow trout) Method: OECD Test Guideline 203
Toxicity to daphnia and other aquatic invertebrates	:	Immobilization EC50: 82 mg/l Exposure time: 48 h Species: Daphnia magna (Water flea) Method: OECD Test Guideline 202
Toxicity to algae	:	Growth inhibition EC50: 106.7 mg/l Exposure time: 72 h Species: Pseudokirchneriella subcapitata (green algae)

Method: OECD Test Guideline 201

: Growth rate
NOEC: 115 mg/l
Exposure time: 72 h
Species: Pseudokirchneriella subcapitata (green algae)
Method: OECD Test Guideline 201

Elimination information (persistence and degradability)

Biodegradability : Result: Not readily biodegradable.
Value: 0 %
Method: OECD 301 D

Further information on ecology

SECTION 13. DISPOSAL CONSIDERATIONS

Disposal methods : Observe all Federal, State, and Local Environmental regulations.

Note : Where possible recycling is preferred to disposal or incineration.

SECTION 14. TRANSPORT INFORMATION

DOT
UN/ID No. : UN 3163
Proper shipping name : LIQUEFIED GAS, N.O.S. (Trans-1-Chloro-3,3,3-trifluoropropene)
Class 2.2
Packing group
Hazard Labels 2.2

IATA
UN/ID No. : UN 3163
Description of the goods : LIQUEFIED GAS, N.O.S. (Trans-1-Chloro-3,3,3-trifluoropropene)
Class : 2.2
Hazard Labels : 2.2
Packing instruction (cargo : 200

	aircraft)	
	Packing instruction (passenger aircraft)	: 200
IMDG	UN/ID No.	: UN 3163
	Description of the goods	: LIQUEFIED GAS, N.O.S. (TRANS-1-CHLORO-3,3,3-TRIFLUOROPROPENE)
	Class	: 2.2
	Hazard Labels	: 2.2
	EmS Number	: F-C, S-V
	Marine pollutant	: no

SECTION 15. REGULATORY INFORMATION

Inventories

US. Toxic Substances Control Act	: On TSCA Inventory	
Australia. Industrial Chemical (Notification and Assessment) Act	: Not in compliance with the inventory	
	: trans-1-Chloro-3,3,3-trifluoropropene	102687-65-0
Canada. Canadian Environmental Protection Act (CEPA). Domestic Substances List (DSL)	: All components of this product are on the Canadian DSL.	
Japan. Kashin-Hou Law List	: On the inventory, or in compliance with the inventory	
Korea. Toxic Chemical Control Law (TCCL) List	: On the inventory, or in compliance with the inventory	
Philippines. The Toxic Substances and Hazardous and Nuclear Waste Control Act	: Not in compliance with the inventory	
	: trans-1-Chloro-3,3,3-trifluoropropene	102687-65-0

China. Inventory of Existing Chemical Substances : Not in compliance with the inventory

: trans-1-Chloro-3,3,3-trifluoropropene 102687-65-0

New Zealand. Inventory of Chemicals (NZIoC), as published by ERMA New Zealand : Not in compliance with the inventory

: trans-1-Chloro-3,3,3-trifluoropropene 102687-65-0

National regulatory information

SARA 302 Components : SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components : SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards : Acute Health Hazard
Sudden Release of Pressure Hazard

California Prop. 65 : This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

WHMIS Classification : A: Compressed Gas
This product has been classified according to the hazard criteria of the CPR and the MSDS contains all of the information required by the CPR.

SECTION 16. OTHER INFORMATION

		HMIS III	NFPA
Health hazard	:	2	2
Flammability	:	0	0
Physical Hazard	:	0	
Instability	:		0

Hazard rating and rating systems (e.g. HMIS® III, NFPA): This information is intended solely for the use of individuals trained in the particular system.

Further information

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text. Final determination of suitability of any material is the sole responsibility of the user. This information should not constitute a guarantee for any specific product properties.

Changes since the last version are highlighted in the margin. This version replaces all previous versions.
Previous Issue Date: 04/23/2013
Prepared by: Honeywell Performance Materials and Technologies Product Stewardship Group

Return to App B Return to Pg 3 Return to Pg 9

Safety Data Sheet

Document Group:	08-1222-2	**Version Number:**	12.02
Issue Date:	01/21/14	**Supercedes Date:**	02/28/13

SECTION 1: Identification

1.1. Product identifier

L-14780 Developmental Material (Experimental)

LB-I000-1478-0

1.2. Recommended use and restrictions on use

Recommended use
Research Sample
Restrictions on use
One or more components in this material are approved for specific commercial use(s) under a U.S. EPA TSCA Significant New Use Rule (SNUR) or Consent Order. Approved uses under SNUR: refrigerant or heat transfer fluid. Additional U.S. EPA approved uses: aerosol spray cleaner, medium for low-temperature immersion baths (e.g., histology baths), deposition coating solvent, vapor degreaser, specialty foam blowing additive, and / or line flush for substances requiring special handling (e.g., liquid oxygen). Please contact 3M for additional regulatory information.

1.3. Supplier's details

MANUFACTURER:	3M
DIVISION:	Electronics Markets Materials Division
ADDRESS:	3M Center, St. Paul, MN 55144-1000, USA
Telephone:	1-888-3M HELPS (1-888-364-3577)

1.4. Emergency telephone number

1-800-364-3577 or (651) 737-6501 (24 hours)

SECTION 2: Hazard identification

2.1. Hazard classification

Serious Eye Damage/Irritation: Category 2B.
Specific Target Organ Toxicity (central nervous system): Category 3.

2.2. Label elements

Signal word
Warning

Symbols
Exclamation mark |

Pictograms

Hazard Statements
Causes eye irritation.
May cause drowsiness or dizziness.

Precautionary Statements

Prevention:
Avoid breathing dust/fume/gas/mist/vapors/spray.
Use only outdoors or in a well-ventilated area.
Wash thoroughly after handling.

Response:
IF INHALED: Remove person to fresh air and keep comfortable for breathing.
IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.
If eye irritation persists: Get medical advice/attention.
Call a POISON CENTER or doctor/physician if you feel unwell.

Storage:
Store in a well-ventilated place. Keep container tightly closed.
Store locked up.

Disposal:
Dispose of contents/container in accordance with applicable local/regional/national/international regulations.

Notes to Physician
Not applicable

2.3. Hazards not otherwise classified
None.

SECTION 3: Composition/information on ingredients

Ingredient	C.A.S. No.	% by Wt
Methyl perfluoropropyl ether	375-03-1	75 - 80
1,2-Trans-dichloroethylene	156-60-5	20 - 25

SECTION 4: First aid measures

4.1. Description of first aid measures

Inhalation:
Remove person to fresh air. If you feel unwell, get medical attention.

Skin Contact:
Wash with soap and water. If you feel unwell, get medical attention.

Eye Contact:
Flush with large amounts of water. Remove contact lenses if easy to do. Continue rinsing. If signs/symptoms persist, get medical attention.

If Swallowed:
Rinse mouth. If you feel unwell, get medical attention.

4.2. Most important symptoms and effects, both acute and delayed
See Section 11.1. Information on toxicological effects.

4.3. Indication of any immediate medical attention and special treatment required
Not applicable

SECTION 5: Fire-fighting measures

5.1. Suitable extinguishing media
In case of fire: Use a fire fighting agent suitable for ordinary combustible material such as water or foam to extinguish.

5.2. Special hazards arising from the substance or mixture
Exposure to extreme heat can give rise to thermal decomposition.

Hazardous Decomposition or By-Products

Substance	Condition
Carbon monoxide	During Combustion
Carbon dioxide	During Combustion
Hydrogen Chloride	During Combustion
Hydrogen Fluoride	During Combustion
Toxic Vapor, Gas, Particulate	During Combustion

5.3. Special protective actions for fire-fighters
When fire fighting conditions are severe and total thermal decomposition of the product is possible, wear full protective clothing, including helmet, self-contained, positive pressure or pressure demand breathing apparatus, bunker coat and pants, bands around arms, waist and legs, face mask, and protective covering for exposed areas of the head.

SECTION 6: Accidental release measures

6.1. Personal precautions, protective equipment and emergency procedures
Evacuate area. Ventilate the area with fresh air. For large spill, or spills in confined spaces, provide mechanical ventilation to disperse or exhaust vapors, in accordance with good industrial hygiene practice. Warning! A motor could be an ignition source and could cause flammable gases or vapors in the spill area to burn or explode. Refer to other sections of this SDS for information regarding physical and health hazards, respiratory protection, ventilation, and personal protective equipment.

6.2. Environmental precautions
Avoid release to the environment. For larger spills, cover drains and build dikes to prevent entry into sewer systems or bodies of water.

6.3. Methods and material for containment and cleaning up
Contain spill. Working from around the edges of the spill inward, cover with bentonite, vermiculite, or commercially available inorganic absorbent material. Mix in sufficient absorbent until it appears dry. Remember, adding an absorbent material does not remove a physical, health, or environmental hazard. Collect as much of the spilled material as possible. Place in a closed container approved for transportation by appropriate authorities. Clean up residue with detergent and water. Seal the container. Dispose of collected material as soon as possible.

SECTION 7: Handling and storage

7.1. Precautions for safe handling
Do not breathe thermal decomposition products. Avoid breathing dust/fume/gas/mist/vapors/spray. Do not get in eyes, on skin, or on clothing. Do not eat, drink or smoke when using this product. Wash thoroughly after handling. Avoid contact with oxidizing agents (eg. chlorine, chromic acid etc.)

7.2. Conditions for safe storage including any incompatibilities
Store in a well-ventilated place. Keep container tightly closed. Store away from strong bases. Store away from oxidizing agents.

SECTION 8: Exposure controls/personal protection

8.1. Control parameters

Occupational exposure limits

Ingredient	C.A.S. No.	Agency	Limit type	Additional Comments
1,2-Trans-dichloroethylene	156-60-5	Amer Conf of Gov. Indust. Hyg.	TWA:200 ppm	
Ethene, 1,2-dichloro-	156-60-5	US Dept of Labor - OSHA	TWA:790 mg/m3(200 ppm)	
Methyl perfluoropropyl ether	375-03-1	Manufacturer determined	TWA:250 ppm	

Amer Conf of Gov. Indust. Hyg. : American Conference of Governmental Industrial Hygienists
American Indust. Hygiene Assoc : American Industrial Hygiene Association
Chemical Manufacturer Rec Guid : Chemical Manufacturer's Recommended Guidelines
US Dept of Labor - OSHA : United States Department of Labor - Occupational Safety and Health Administration
TWA: Time-Weighted-Average
STEL: Short Term Exposure Limit
CEIL: Ceiling

8.2. Exposure controls

8.2.1. Engineering controls
Provide appropriate local exhaust when product is heated. Use general dilution ventilation and/or local exhaust ventilation to control airborne exposures to below relevant Exposure Limits and/or control dust/fume/gas/mist/vapors/spray. If ventilation is not adequate, use respiratory protection equipment.

8.2.2. Personal protective equipment (PPE)

Eye/face protection

Select and use eye/face protection to prevent contact based on the results of an exposure assessment. The following eye/face protection(s) are recommended:
Indirect Vented Goggles

Skin/hand protection
No chemical protective gloves are required.

Respiratory protection

An exposure assessment may be needed to decide if a respirator is required. If a respirator is needed, use respirators as part of a full respiratory protection program. Based on the results of the exposure assessment, select from the following respirator

type(s) to reduce inhalation exposure:
During heating:
Use a positive pressure supplied-air respirator if there is a potential for over exposure from an uncontrolled release, exposure levels are not known, or under any other circumstances where air-purifying respirators may not provide adequate protection.

Half facepiece or full facepiece supplied-air respirator

For questions about suitability for a specific application, consult with your respirator manufacturer.

SECTION 9: Physical and chemical properties

9.1. Information on basic physical and chemical properties

General Physical Form:	Liquid
Odor, Color, Grade:	Clear, colorless with chlorinated solvent odor
Odor threshold	*No Data Available*
pH	*Not Applicable*
Melting point	*Not Applicable*
Boiling Point	28 - 30 ºC
Flash Point	No flash point
Evaporation rate	*No Data Available*
Flammability (solid, gas)	Not Applicable
Flammable Limits(LEL)	*Not Applicable*
Flammable Limits(UEL)	*Not Applicable*
Vapor Pressure	665 mmHg [@ 20 ºC]
Vapor Density	3.3 [*Ref Std:* AIR=1]
Density	1.4 g/ml
Specific Gravity	1.4 [*Ref Std:* WATER=1]
Solubility- non-water	*No Data Available*
Partition coefficient: n-octanol/ water	*No Data Available*
Autoignition temperature	*No Data Available*
Decomposition temperature	*No Data Available*
Viscosity	<=10 centipoise
Volatile Organic Compounds	302 g/l [*Test Method:* South Cost Air Qual Mgmt Dist]
Percent volatile	100 %
VOC Less H2O & Exempt Solvents	*Not Applicable*

SECTION 10: Stability and reactivity

10.1. Reactivity
This material may be reactive with certain agents under certain conditions - see the remaining headings in this section.

10.2. Chemical stability
Stable.

10.3. Possibility of hazardous reactions
Hazardous polymerization will not occur.

10.4. Conditions to avoid
Not determined

10.5. Incompatible materials
Strong bases
Strong oxidizing agents

10.6. Hazardous decomposition products

Substance	Condition
None known.	

Refer to section 5.2 for hazardous decomposition products during combustion.

Extreme heat arising from situations such as misuse or equipment failure can generate hydrogen fluoride as a decomposition product.

SECTION 11: Toxicological information

The information below may not be consistent with the material classification in Section 2 if specific ingredient classifications are mandated by a competent authority. In addition, toxicological data on ingredients may not be reflected in the material classification and/or the signs and symptoms of exposure, because an ingredient may be present below the threshold for labeling, an ingredient may not be available for exposure, or the data may not be relevant to the material as a whole.

11.1. Information on Toxicological effects

Signs and Symptoms of Exposure

Based on test data and/or information on the components, this material may produce the following health effects:

Inhalation:
Respiratory Tract Irritation: Signs/symptoms may include cough, sneezing, nasal discharge, headache, hoarseness, and nose and throat pain.

Skin Contact:
Contact with the skin during product use is not expected to result in significant irritation.

Eye Contact:
Moderate Eye Irritation: Signs/symptoms may include redness, swelling, pain, tearing, and blurred or hazy vision.

Ingestion:
Gastrointestinal Irritation: Signs/symptoms may include abdominal pain, stomach upset, nausea, vomiting and diarrhea.

May cause target organ effects after ingestion.

Target Organ Effects:

Single exposure may cause:
Central Nervous System (CNS) Depression: Signs/symptoms may include headache, dizziness, drowsiness, incoordination, nausea, slowed reaction time, slurred speech, giddiness, and unconsciousness.

Toxicological Data
If a component is disclosed in section 3 but does not appear in a table below, either no data are available for that endpoint or the data are not sufficient for classification.

Acute Toxicity

Name	Route	Species	Value
Overall product	Ingestion		No data available; calculated ATE > 5,000 mg/kg
Methyl perfluoropropyl ether	Inhalation-Vapor (4 hours)	Mouse	LC50 > 820 mg/l
Methyl perfluoropropyl ether	Ingestion	Rat	LD50 > 2,000 mg/kg

1,2-Trans-dichloroethylene	Dermal	Rabbit	LD50 > 5,000 mg/kg
1,2-Trans-dichloroethylene	Inhalation-Vapor (4 hours)	Rat	LC50 95.6 mg/l
1,2-Trans-dichloroethylene	Ingestion	Rat	LD50 7,902 mg/kg

ATE = acute toxicity estimate

Skin Corrosion/Irritation

Name	Species	Value
Methyl perfluoropropyl ether	Rabbit	No significant irritation
1,2-Trans-dichloroethylene	Rabbit	Minimal irritation

Serious Eye Damage/Irritation

Name	Species	Value
Methyl perfluoropropyl ether	Rabbit	No significant irritation
1,2-Trans-dichloroethylene	Rabbit	Moderate irritant

Skin Sensitization

Name	Species	Value
Methyl perfluoropropyl ether	Mouse	Not sensitizing
1,2-Trans-dichloroethylene		Data not available or insufficient for classification

Respiratory Sensitization

Name	Species	Value
Methyl perfluoropropyl ether		Data not available or insufficient for classification
1,2-Trans-dichloroethylene		Data not available or insufficient for classification

Germ Cell Mutagenicity

Name	Route	Value
Methyl perfluoropropyl ether	In Vitro	Not mutagenic
1,2-Trans-dichloroethylene	In Vitro	Not mutagenic
1,2-Trans-dichloroethylene	In vivo	Not mutagenic

Carcinogenicity

Name	Route	Species	Value
Methyl perfluoropropyl ether			Data not available or insufficient for classification
1,2-Trans-dichloroethylene			Data not available or insufficient for classification

Reproductive Toxicity

Reproductive and/or Developmental Effects

Name	Route	Value	Species	Test Result	Exposure Duration
Methyl perfluoropropyl ether	Inhalation	Not toxic to reproduction and/or development	Rat	NOAEL 246 mg/l	30 days
1,2-Trans-dichloroethylene	Ingestion	Not toxic to female reproduction	Rat	NOAEL 3,000 mg/kg/day	90 days
1,2-Trans-dichloroethylene	Inhalation	Not toxic to female reproduction	Rat	NOAEL 16 mg/l	90 days
1,2-Trans-dichloroethylene	Ingestion	Not toxic to male reproduction	Rat	NOAEL 3,000 mg/kg/day	90 days
1,2-Trans-dichloroethylene	Inhalation	Not toxic to male reproduction	Rat	NOAEL 16 mg/l	90 days
1,2-Trans-dichloroethylene	Inhalation	Some positive developmental data exist, but the data are not sufficient for classification	Rat	NOAEL 24 mg/l	during organogenesis

Target Organ(s)

Specific Target Organ Toxicity - single exposure

Name	Route	Target Organ(s)	Value	Species	Test Result	Exposure Duration
Methyl perfluoropropyl ether			Data not available or insufficient for classification			
1,2-Trans-dichloroethylene	Inhalation	central nervous system depression	Some positive data exist, but the data are not sufficient for classification	Human	NOAEL Not available	occupational exposure
1,2-Trans-dichloroethylene	Inhalation	respiratory irritation	Some positive data exist, but the data are not sufficient for classification		NOAEL Not available	
1,2-Trans-dichloroethylene	Ingestion	central nervous system depression	May cause drowsiness or dizziness	Rat	LOAEL 4,500 mg/kg	not applicable

Specific Target Organ Toxicity - repeated exposure

Name	Route	Target Organ(s)	Value	Species	Test Result	Exposure Duration
Methyl perfluoropropyl ether	Inhalation	kidney and/or bladder | blood | liver	Some positive data exist, but the data are not sufficient for classification	Rat	NOAEL 246 mg/l	30 days
Methyl perfluoropropyl ether	Inhalation	heart | endocrine system | hematopoietic system | nervous system | respiratory system	All data are negative	Rat	NOAEL 246 mg/l	30 days
1,2-Trans-dichloroethylene	Inhalation	endocrine system | liver | kidney and/or bladder | respiratory system	All data are negative	Rat	NOAEL 16 mg/l	90 days
1,2-Trans-dichloroethylene	Ingestion	kidney and/or bladder	Some positive data exist, but the data are not sufficient for classification	Rat	NOAEL 2,000 mg/kg/day	14 weeks
1,2-Trans-dichloroethylene	Ingestion	blood | liver	Some positive data exist, but the data are not sufficient for classification	Rat	NOAEL 125 mg/kg/day	14 weeks
1,2-Trans-dichloroethylene	Ingestion	heart | immune system | respiratory system	All data are negative	Rat	NOAEL 2,000 mg/kg/day	14 weeks

Aspiration Hazard

Name	Value
Methyl perfluoropropyl ether	Not an aspiration hazard
1,2-Trans-dichloroethylene	Not an aspiration hazard

Please contact the address or phone number listed on the first page of the SDS for additional toxicological information on this material and/or its components.

SECTION 12: Ecological information

Ecotoxicological information

Please contact the address or phone number listed on the first page of the SDS for additional ecotoxicological information on this material and/or its components.

Chemical fate information

Please contact the address or phone number listed on the first page of the SDS for additional chemical fate information on this material and/or its components.

SECTION 13: Disposal considerations

13.1. Disposal methods

Dispose of contents/ container in accordance with the local/regional/national/international regulations.

Dispose of waste product in a permitted industrial waste facility. As a disposal alternative, incinerate in a permitted waste incineration facility. Proper destruction may require the use of additional fuel during incineration processes. Combustion products will include halogen acid (HCl/HF/HBr). Facility must be capable of handling halogenated materials. Empty drums/barrels/containers used for transporting and handling hazardous chemicals (chemical substances/mixtures/preparations classified as Hazardous as per applicable regulations) shall be considered, stored, treated & disposed of as hazardous wastes unless otherwise defined by applicable waste regulations. Consult with the respective regulating authorities to determine the available treatment and disposal facilities.

EPA Hazardous Waste Number (RCRA): Not regulated

SECTION 14: Transport Information

For Transport Information, please visit **http://3M.com/Transportinfo** or call 1-800-364-3577 or 651-737-6501.

SECTION 15: Regulatory information

15.1. US Federal Regulations

Contact 3M for more information.

311/312 Hazard Categories:

Fire Hazard - No Pressure Hazard - No Reactivity Hazard - No Immediate Hazard - Yes Delayed Hazard - No

Section 313 Toxic Chemicals subject to the reporting requirements of that section and 40 CFR part 372 (EPCRA):

Ingredient	C.A.S. No	% by Wt
1,2-Trans-dichloroethylene (Ethene, 1,2-dichloro-)	156-60-5	20 - 25

This material contains a chemical which requires export notification under TSCA Section 12[b]:

Ingredient (Category if applicable)	C.A.S. No	Regulation	Status
Methyl perfluoropropyl ether	375-03-1	Toxic Substances Control Act (TSCA) 5 SNUR or Consent Order Chemicals	Applicable

This material contains a chemical regulated by an EPA Significant New Use Rule (TSCA Section 5)

Ingredient (Category if applicable)	C.A.S. No	Reference
Methyl perfluoropropyl ether	375-03-1	40CFR721.8145

15.2. State Regulations

Contact 3M for more information.

15.3. Chemical Inventories

The components of this product are in compliance with the chemical notification requirements of TSCA.

Contact 3M for more information.

15.4. International Regulations

Contact 3M for more information.

This SDS has been prepared to meet the U.S. OSHA Hazard Communication Standard, 29 CFR 1910.1200.

SECTION 16: Other information

NFPA Hazard Classification
Health: 3 **Flammability:** 1 **Instability:** 0 **Special Hazards:** None

National Fire Protection Association (NFPA) hazard ratings are designed for use by emergency response personnel to address the hazards that are presented by short-term, acute exposure to a material under conditions of fire, spill, or similar emergencies. Hazard ratings are primarily based on the inherent physical and toxic properties of the material but also include the toxic properties of combustion or decomposition products that are known to be generated in significant quantities.

HMIS Hazard Classification
Health: *2 **Flammability:** 1 **Physical Hazard:** 0 **Personal Protection:** X - See PPE section.

Hazardous Material Identification System (HMIS® III) hazard ratings are designed to inform employees of chemical hazards in the workplace. These ratings are based on the inherent properties of the material under expected conditions of normal use and are not intended for use in emergency situations. HMIS® III ratings are to be used with a fully implemented HMIS® III program. HMIS® is a registered mark of the American Coatings Association (ACA).

Document Group:	08-1222-2	**Version Number:**	12.02
Issue Date:	01/21/14	**Supercedes Date:**	02/28/13

DISCLAIMER: The information in this Safety Data Sheet (SDS) is believed to be correct as of the date issued. 3M MAKES NO WARRANTIES, EXPRESSED OR IMPLIED, INCLUDING, BUT NOT LIMITED TO, ANY IMPLIED WARRANTY OF MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE OR COURSE OF PERFORMANCE OR USAGE OF TRADE. User is responsible for determining whether the 3M product is fit for a particular purpose and suitable for user's method of use or application. Given the variety of factors that can affect the use and application of a 3M product, some of which are uniquely within the user's knowledge and control, it is essential that the user evaluate the 3M product to determine whether it is fit for a particular purpose and suitable for user's method of use or application.

3M provides information in electronic form as a service to its customers. Due to the remote possibility that electronic transfer may have resulted in errors, omissions or alterations in this information, 3M makes no representations as to its completeness or accuracy. In addition, information obtained from a database may not be as current as the information in the SDS available directly from 3M

Return to App B Return to Pg 3 Return to Pg 9

Material Safety Data Sheet

Capstone® 4-I

Version 2.2

Revision Date 12/20/2011 Ref. 130000042690

This SDS adheres to the standards and regulatory requirements of the United States and may not meet the regulatory requirements in other countries.

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name : Capstone® 4-I
Tradename/Synonym : Zonyl® PFBI

MSDS Number : 130000042690

Product Use : Chemical intermediate

Manufacturer : DuPont
1007 Market Street
Wilmington, DE 19898

Product Information : 1-800-441-7515 (outside the U.S. 1-302-774-1000)
Medical Emergency : 1-800-441-3637 (outside the U.S. 1-302-774-1139)
Transport Emergency : CHEMTREC: 1-800-424-9300 (outside the U.S. 1-703-527-3887)

Other information : professional use

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

The hazards of this product are associated mainly with its processing. Inhalation of decomposition products in high concentration may cause shortness of breath (lung oedema). Inhalation of aerosol or fine spray mist may cause serious respiratory problems.

Warning symptoms:
Irregular cardiac activity Pain, Itching, Rash, Sensitisation, Irritation, Ulceration

Potential Health Effects

Skin : Causes skin irritation. May cause:, Pain, Burning sensation, Itching, Redness, Swelling, Rash.

Eyes : Causes eye irritation. May cause:, Pain, tearing, Swelling, Redness, Temporary, visual impairment.

Inhalation	:	May cause irritation of respiratory tract. May cause:, Cough, Shortness of breath, Central nervous system depression, dizziness, confusion, incoordination, drowsiness, or unconsciousness, Prolonged contact may cause:, irregular heartbeat with a strange sensation in the chest, heart thumping, apprehension, feeling of fainting, dizziness or weakness, Fluid in the lungs (pulmonary oedema) with cough, wheezing, abnormal lung sounds, possibly progressing to severe shortness of breath and bluish discoloration of the skin (symptoms might be delayed).
Ingestion	:	Gross overexposure may cause: Thyroid effects
Repeated exposure	:	May cause harm to the unborn child.

Target Organs

Perfluorobutyl iodide	:	Respiratory Tract
Iodine	:	Thyroid

Carcinogenicity

None of the components present in this material at concentrations equal to or greater than 0.1% are listed by IARC, NTP, or OSHA, as a carcinogen.

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Component	CAS-No.	Concentration
Perfluorobutyl iodide	423-39-2	>90%
Perfluorohexyl Iodide	355-43-1	<0.4 %
Perfluoroethyl iodide	354-64-3	<0.5 %
Iodine	7553-56-2	<0.1 %

SECTION 4. FIRST AID MEASURES

Skin contact	: Wash off immediately with soap and plenty of water. Wash contaminated clothing before re-use.
Eye contact	: Rinse immediately with plenty of water for at least 15 minutes. Seek medical advice.
Inhalation	: Move to fresh air. Oxygen or artificial respiration if needed. Symptoms may be delayed. Call a physician immediately.
Ingestion	: Call a physician or poison control centre immediately. If swallowed, DO NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person.
General advice	: When symptoms persist or in all cases of doubt seek medical advice.

SECTION 5. FIREFIGHTING MEASURES

Flammable Properties	
Flash point	: closed cup does not flash
Thermal decomposition	: > 200 °C (> 392 °F) To avoid thermal decomposition, do not overheat. Thermal decomposition can lead to release of irritating gases and vapours.
Fire and Explosion Hazard	: The product itself does not burn. Hazardous decomposition products formed under fire conditions. Hazardous combustion products Hydrogen fluoride Carbon dioxide (CO2) Carbon monoxide Other hazardous decomposition products may be formed.
Suitable extinguishing media	: Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.
Firefighting Instructions	: Wear self contained breathing apparatus for fire fighting if necessary. Evacuate personnel to safe areas. Do not allow run-off from fire fighting to enter drains or water courses.

SECTION 6. ACCIDENTAL RELEASE MEASURES

NOTE: Review FIRE FIGHTING MEASURES and HANDLING (PERSONNEL) sections before proceeding with clean-up. Use appropriate PERSONAL PROTECTIVE EQUIPMENT during clean-up.

Safeguards (Personnel)	:	Evacuate personnel to safe areas. Use personal protective equipment. Ventilate the area.
Spill Cleanup	:	Dam up. Soak up with inert absorbent material (e.g. sand, silica gel, acid binder, universal binder, sawdust). Pick up and transfer to properly labelled containers.
Accidental Release Measures	:	Do not discharge to streams, ponds, lakes or sewers. Avoid subsoil penetration.

SECTION 7. HANDLING AND STORAGE

Handling (Personnel)	:	Avoid formation of respirable particles. Do not breathe vapours or spray mist. Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product. Wash contaminated clothing before re-use.
Handling (Physical Aspects)	:	To avoid thermal decomposition, do not overheat. Thermal decomposition can lead to release of irritating gases and vapours. Do not spray near open flame or heated surface.
Storage	:	Keep tightly closed in a dry, cool and well-ventilated place.

SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Personal protective equipment		
Respiratory protection	:	In case of mist, spray or aerosol exposure wear suitable personal respiratory protection and protective suit.
Hand protection	:	Additional protection: Impervious gloves

Eye protection : Wear safety glasses or coverall chemical splash goggles.

Skin and body protection : Lightweight protective clothing

Exposure Guidelines

Exposure Limit Values

Perfluorobutyl iodide

AEL *	(DUPONT)	375 ppm		8 & 12 hr. TWA

Iodine

PEL:	(OSHA)	0.1 ppm	1 mg/m3	TLV-C	
TLV	(ACGIH)	0.01 ppm		TWA	Inhalable fraction and vapor.
TLV	(ACGIH)	0.1 ppm		STEL	Vapor and aerosol.

* AEL is DuPont's Acceptable Exposure Limit. Where governmentally imposed occupational exposure limits which are lower than the AEL are in effect, such limits shall take precedence.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Form	liquid
Color	clear, colourless, to, violet
Odor	slight, acidic
Melting point	< -70 °C (< -94 °F)
Boiling point	69.1 °C (156.4 °F)
% Volatile	> 98 %
Vapour Pressure	148 Torr at 25 °C (77 °F)
Specific gravity	2.00 at 25 °C (77 °F)
Water solubility	negligible

SECTION 10. STABILITY AND REACTIVITY

Stability : Stable under normal conditions.

Conditions to avoid	: To avoid thermal decomposition, do not overheat.
Hazardous decomposition products	: Carbon dioxide , Carbon monoxide, Hydrofluoric acid...% Incompletely burned carbon products Other hazardous decomposition products may be formed.

SECTION 11. TOXICOLOGICAL INFORMATION

Perfluorobutyl iodide

Inhalation 4 h LC50	: 14000 ppm , rat Target Organs: Respiratory Tract Respiratory tract irritation
Inhalation	: 6000 ppm , dog Cardiac sensitization
Skin irritation	: Skin irritation, guinea pig
Eye irritation	: Eye irritation, animals (unspecified species)
Skin sensitization	: Did not cause sensitization on laboratory animals., guinea pig
Repeated dose toxicity	: Inhalation rat Respiratory tract irritation, Nasal irritation
Mutagenicity	: Evidence suggests this substance does not cause genetic damage in animals. Tests on bacterial or mammalian cell cultures did not show mutagenic effects.
Further information	: Cardiac sensitisation threshold limit : 84000 mg/m3

Perfluorohexyl Iodide

Inhalation NOEC	: 7500 ppm , dog Not a cardiac sensitizer.
Skin irritation	: Non-corrosive

Perfluoroethyl iodide

Inhalation 4 h LC50 : 40000 ppm , rat
Central nervous system depression
Incoordination
Hypoactivity
Anaesthetic effects
Cardiac sensitization

Further information : Cardiac sensitisation threshold limit : 5030 mg/m3

Iodine

Dermal Acute toxicity estimate : 1,100 mg/kg , rabbit

Oral Acute toxicity estimate : 500 mg/kg , human

Inhalation 4 h Acute toxicity estimate : 1.5 mg/l , rat

Skin irritation : Skin irritation, Not tested on animals

Eye irritation : Corrosive, multiple species

Skin sensitization : Did not cause sensitization on laboratory animals., mouse
There are rare or inconclusive reports of human skin sensitization.

Repeated dose toxicity : Oral
human

Target Organs: Thyroid
Thyroid effects

Carcinogenicity : Overall weight of evidence indicates that the substance is not carcinogenic.

Mutagenicity : Overall weight of evidence indicates that the substance is not mutagenic.
Genetic damage in cultured mammalian cells was observed in some laboratory tests but not in others.

Did not cause genetic damage in animals.
Information given is based on data obtained from similar substances.

Reproductive toxicity : Effects on or via lactation

Teratogenicity	: Has been reported to cause: Reduced embryo-foetal viability Reduced growth

SECTION 12. ECOLOGICAL INFORMATION

Aquatic Toxicity

Perfluorobutyl iodide

96 h LC50	: Pimephales promelas (fathead minnow) 2.3 mg/l
48 h EC50	: Daphnia magna (Water flea) 1.7 mg/l

Iodine

96 h LC50	: Oncorhynchus mykiss (rainbow trout) 1.67 mg/l
72 h ErC50	: Desmodesmus subspicatus (green algae) 0.13 mg/l
48 h LC50	: Daphnia magna (Water flea) 0.55 mg/l

Environmental Fate

Perfluorobutyl iodide

Biodegradability	: Not readily biodegradable.
Additional ecological information	: No data is available on the product itself.

SECTION 13. DISPOSAL CONSIDERATIONS

Waste Disposal : Collect spent solvent and either reclaim or incinerate. Collect materials containing or contaminated with solvent, including rags, disposable wipes, and gloves in a sealed container for incineration. Prevent wastewater, spent solvent, and materials containing or contaminated with solvent from entering waterways. Wastewater should be sent to a wastewater treatment facility.

SECTION 14. TRANSPORT INFORMATION

IATA_C	UN number	: 3082
	Proper shipping name	: Environmentally hazardous substance, liquid, n.o.s. (Perfluorobutyl Iodide)
	Class	: 9
	Packing group	: III
	Labelling No.	: 9
IMDG	UN number	: 3082
	Proper shipping name	: Environmentally hazardous substance, liquid, n.o.s. (Perfluorobutyl Iodide)
	Class	: 9
	Packing group	: III
	Labelling No.	: 9
	Marine pollutant	: yes (Perfluorobutyl Iodide)

SECTION 15. REGULATORY INFORMATION

TSCA Status	: On the inventory, or in compliance with the inventory
SARA 313 Regulated Chemical(s)	: SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.
Title III hazard classification	: Acute Health Hazard: Yes Chronic Health Hazard: No Fire: No Reactivity/Physical hazard: No Pressure: No
California Prop. 65	: Chemicals known to the State of California to cause cancer, birth defects or any other harm: none known

SECTION 16. OTHER INFORMATION

HMIS

Health	:	2
Flammability	:	0
Reactivity/Physical hazard	:	0
PPE	:	Personal Protection rating to be supplied by user depending on use conditions.

The DuPont Oval Logo® and Capstone®
are registered trademarks or trademarks of E. I. du Pont de Nemours and Company or its affiliates.

Contact person : MSDS Coordinator, DuPont Chemicals and Fluoroproducts, Wilmington, DE 19898, (800) 441-7515

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

Significant change from previous version is denoted with a double bar.

Return to App B

Return to Pg 9

AGC

: AGC Chemicals Americas, Inc.

Effective Date: 03/28/2013
Version: 15

SAFETY DATA SHEET

ASAHIKLIN AE3000

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: ASAHIKLIN AE3000
General Use: Solvent

Manufacturer

ASAHI GLASS CO., LTD
Chemicals Company
1-5-1, Marunouchi, Chiyoda-ku,Tokyo 100-8405, JAPAN

Telephone Number for Information

+81-3-3218-5574

Facsimile Number for Information

+81-3-3218-7854

Supplier

AGC Chemicals Americas, Inc.
55 E. Uwchlan Avenue
Suite 201
Exton, PA 19341

24 Hours Medical Emergency Telephone #: 800-420-8479
24 Hours Transportation Emergency # (CHEMTREC): 800-424-9300
Customer Service #: 800-424-7833

2. HAZARDS IDENTIFICATION

This product is hazardous under OSHA.

Emergency Overview:

WARNING!
May cause eye and skin irritation.
Prolonged or repeated skin contact with liquid may cause drying, cracking, redness, itching, and/or swelling (dermatitis).

Aspiration hazard if swallowed - can enter lungs and cause damage.
Inhalation of high concentration of vapors is harmful and may cause heart irregularites, unconsciousness, or death.

Potential Health Effects:

Inhalation: Inhalation of vapor may cause coughing, dizziness, dullness, drowsiness, and headache. Inhalation of higher concentrations of vapor is harmful and may cause heart irregularities, central nervous system depression, narcosis, unconsciousness, respiratory failure, or death. Intentional misuse can be fatal. Vapor reduces oxygen available for breathing and is heavier than air.

Skin Contact: May cause skin irritation.

Eye Contact: May cause eye irritation.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Component Name	CAS-NO	Weight %
1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether	406-78-0	99.9-100

OSHA - 29 CFR 1910 Specifically Regulated Chemicals

1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether

4. FIRST AID MEASURES

Eye Contact: Immediately flush eyes with plenty of water for at least 15 minutes. Remove contact lenses, if worn. Get medical attention if irritation persists.

Skin Contact: In case of skin contact, flush with water. Remove contaminated clothing and shoes. Get medical attention if irritation persists.

Inhalation: If inhalation symptoms occur, immediately remove to fresh air. Keep person calm. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if cough or other symptoms develop.

Ingestion: No specific intervention is indicated as the compound is not likely to be hazardous by ingestion. Do not induce vomiting because the hazard of aspirating the material into the lungs is considered greater than swallowing it. Get medical attention if symptoms develop. Never give anything by mouth to an unconscious person.

5. FIRE FIGHTING MEASURES

Suitable Extinguishing Media: Dry chemical, Carbon dioxide (CO2) Water spray mist or foam.

Unsuitable Extinguish Media/Methods: None

Hazardous Combustion Product or Gases: Containers may rupture under fire conditions. Decomposition of this product at temperature above 300deg.C (572deg.F) can form hydrogen fluoride (HF), but HF will only accumulate with continuous exposure to excess heat in a sealed vessel.

Special Protective Equipment For Fire Fighters: Self-contained breathing apparatus (SCBA) is required if drums rupture and contents are spilled under fire conditions.

Additional Information: Use water spray to cool containers. Move containers from fire areas if it can be done without risk.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions:
Ensure adequate ventilation.
Use personal respiratory protection, impermeable gloves, chemical splash goggles and protective clothing.

Environmental precautions:
Collect contaminated water/firefighting water separately. Do not wash away into drain or waterway.

Methods for cleaning up/taking up:
Absorb or contain liquid with inert material and dispose of in accordance with applicable regulations.

In case of spill or other release:
NOTES: Review chapter 5, chapter 7, and chapter 8 before proceeding with clean up. Use appropriate Personal Protective Equipment during clean up.

Extinguish flames in area to avoid forming dangerous decomposition products (See chapter 5). Dike spill. Prevent liquid from entering sewers, waterways or low areas. Ventilate area. Collect on absorbent material and transfer to steel drums for recovery/disposal. Comply with Federal, State, and local regulations on reporting releases.

Additional information:
Additional information: Review chapter 7 for safe handling information.
Review chapter 13 for disposal information.

7. HANDLING AND STORAGE

Handling:
Use with sufficient ventilation to keep employee exposure below recommended limits. Provide adequate ventilation for storage, handling, and use, especially for enclosed or low spaces. Avoid contact of liquid with eyes and prolonged skin exposure. Do not allow product to contact open flame or electrical heating elements because dangerous decomposition products may form.

Storage
Store in clean, dry, well-ventilated area. Do not store product in direct sunlight.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Exposure Guidelines:

Component Name	AEL:	CEC:	EEL:	ECE:
1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether	Daily exposure limit: 50ppm (8h-TWA) *AEL is the Acceptable Exposure Limit set by Asahi Glass Co., Ltd.	Ceiling exposure concentration (non-emergency): 150ppm Exposure Limit recommended by US EPA	Emergency exposure limit: 150ppm (time limit 15min) Emergency Exposure Limit (EEL) set by Asahi Glass Co., Ltd.	Emergency ceiling exposure concentration: 500ppm Emergency ceiling exposure concentration set by Asahi Glass Co., Ltd.

EEL: Emergency Exposure Limits (EELs) are to be used for short-term emergency exposure control. They are concentrations of short periods which should not result in permanent adverse health effects or interfere with escape. They should not be confused with daily exposure limits (such as AEL's and EPA recommended exposure limits) that are designed for repeated exposure guidelines. For the use of AE3000, daily exposure limits such as AEL as well as EEL are to be followed. The EEL for AE3000 is needed to avoid anaesthetic effects, which could prevent self-rescue. If an EEL is exceeded for specified duration, evacuation, sheltering in place or other mitigatin steps should be taken.

Occupational Exposure Controls.

Engineering Controls:

Normal ventilation for standard manufacturing procedures is generally adequate. Local exhaust should be used when large amounts are released. Vapors are heavier than air. Use with adequate ventilation to prevent vapor buildup in low lying areas.

Personal Protection:

Respiratory Protection Use respiratory protection approved by NIOSH in USA or other equivalent in each country if exposure limits may be exceeded. Self-contained breathing apparatus (SCBA) is required if a large spill occurs.

Hand Protection: Impervious gloves

Eye Protection: Safety glasses and/or chemical tight goggles if splashing is likely to occur.

Additional advice:

Provide eyewash stations Impermeable apron and boots to prevent skin contact.

NPCA - HMIS (National Paint and Coating Association - Hazardous Materials Identification System) Hazard Rating

HMIS codes are intended for use in everyday workplace setting to provide a rapid indication of the occupational hazards associated with chemicals used in the workplace.
a) Flammability - 0, b) Health - 1, c) Reactivity - 0

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance and Odor:	Clear, colorless liquid with slight ethereal odor
Boiling Point (°F/C):	134/56.2
Melting Point/Range:	-138 deg.F (-94 degC)
Flash Point (Method Used):	None (Pensky-Martens Closed Cup & Cleveland Open Cup)
Lower Explosive Limit:	None
Autoignition Temp. (°F/C):	1094deg.F (590deg.C)
Upper Explosive Limit:	None
Vapor Pressure:	31kPa @25deg.C
Relative Density:	1.47 @25deg.C
Solubility in Water:	0.01g/100g H2O @20deg.C
Solubility in Solvent	0.09g H2O/100g
pH Value	N/D @20 deg.C
Viscosity:	6.5 x 10^-4 Pa @25deg.C
Partition Coefficient	n-octanol/water: log Pow=2.18
Other:	(Vapor density, miscibility, evaporation rate, conductivity, etc.): N/D

10. STABILITY AND REACTIVITY

Conditions to Avoid: Material is stable however, avoid open flames and high temperature. Do not allow product to contact open flame or electrical heating elements because dangerous decomposition products may form.

Stability: Stable under recommended storage conditions.

Materials to Avoid:
Incompatible with alkali or alkaline earth metals-powdered Al, Zn, Be, etc.

Avoid contamination with caustic soda, caustic potash, or oxidizing materials. Shock sensitive compounds may be formed.

Hazardous Decomposition Products:
Decomposition products are hazardous. This compound can be decomposed by high temperatures (open flames, glowing metal surfaces, etc.) forming hydrochloric and hydrofluoric acids-possibly carbonyl halides.

Hazardous Polymerization: Will Not Occur

11. TOXICOLOGICAL INFORMATION

Animal Data

Component Name	Inhalation	Oral	Dermal	Eyes
1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether	LC50: >24.8mg/L (3010ppm) in rat	LD50: >2,000mg/kg in rat	LD50: >2,000mg/kg in rat	

1,1,2,2-tetrafluoroethyl-2,2,2-trifluoroethyl ether

Acute Toxicity:
Oral LD50 (rat): >2000mg/kg
Inhalation LC50 (rat): >24.8mg/L (3010ppm)
Dermal LD50 (rat): >2000mg/kg

Skin and eye irritation:
Slight irritation to eye and mucous membranes
Skin irritation (rabbit): none
Eye irritation (rabbit): slight

Sensitization:
Skin (rat): none

Genetic Studies:
Ames Assay: Negative (OECD 471 & 472)
Chromosomal Aberration Test: Negative (CHL Cell) (OECD 473)

Repeated Dose Oral Toxicity (28 Day): NOEL 1000mg/kg/d
Repeated Dose Inhalation Toxicity (5 day): NOEL 1800ppm
Rats exposed to 2500 or 5000 ppm for 6 hours per day for 5 days showed convulsions.

Repeated Dose Inhalation Toxicity (90 day): NOEL 1000ppm
Rats exposed to 1000ppm for 6 hours per day, 5 days per week for 90 days showed no adverse effects.

Carcinogenicity
(ACGIH): N/E

12. ECOLOGICAL INFORMATION

Biodegradability:	Not biodegraded (OECD 301C)
Bioaccumulation:	N/D
Activated Sludge Study:	>100mg/L (OECD 209)
Fish Toxicity:	LC50 (96hr) (Carp): >76mg/L
Other Information:	Algal growth inhibition: ErC50>213mg/L ---- EbC50>213mg/L Mobility inhibition (Daphnia magna): 48hr-EC50>94mg/L

13. DISPOSAL CONSIDERATIONS

Waste treatment:	Recover by distillation or remove to permitted waste disposal facility.
Packaging treatment:	Dispose of waste containers to authorized landfill, in accordance with local laws and regulations.
Disposal methods	Comply with all federal, state, and local regulations. Do not dump this product into sewers, on the ground, or into any body of water. Reuse the residual product when possible. Send waste product for thermal destruction, using high-temperature incinerators designed to burn fluorine compounds. Dispose of waste containers in accordance with local laws and regulations. Comply with all federal, state, and local regulations. Do not dump this product into sewers, on the ground, or into any body of water.

14. TRANSPORT INFORMATION

US DEPARTMENT OF TRANSPORTATION (DOT)

Hazardous Materials: N/A
Hazard Class or Division: Not classified
Identification Number: Not regulated
Packing Group: Not classified
Hazardous Materials Description and Proper Shipping Name: N/A
Label(s) Required: Not classified

UN/Id No.: N/A
IMDG Status: Not restricted
Marine Pollutant: No

ICAO/IATA Status: Not restricted

15. REGULATORY INFORMATION

For European Union

EEC Classification	Not classified
Hazard Symbol	Not established
Risk Phrases:	Not established

Safety Phrases:
Not established

For United States of America

SNAP Acceptable:
Acceptable for precision cleaning, electronics cleaning, & aerosol solvents.

OSHA
This product is hazardous under 29 CFR 1910.1200

TSCA Status:
This chemical is listed on the TSCA inventory.

TSCA - Sect. 5(a)(2) - Chemicals with SNUR
This material will be the subject of a significant new use rule (SNUR) under 40 CFR Part 721. Accordingly, this material can only be used for the following industrial-use applications: (i) cleaning electronic components; (ii) precision cleaning; (iii) dewatering of electronic components and other parts following aqueous cleaning; and (iv) carrier/lubricant coating for hard disk drives and other precision parts.

SARA Section 302:
N/A

Sara (311, 312) Hazard Class:
Acute Health Hazard,

SARA Section 313: N/A

16. ADDITIONAL INFORMATION

N/E: Not Established

N/A: Not Applicable

N/D: No Data

C.O.C.: Cleveland Open Cup

NFPA Hazard Code
NFPA codes are designed for use by firefighters, sheriffs, or other emergency response teams who are concerned with the hazards of burning or exploding materials. These NFPA codes are not intended to address the hazards of this product other than in a fire situation.

NFPA Rating:
Health 2, Flammability 0 , Reactivity 0

Revision Summary - MSDS Section(s) Updated: 15

AGC

: AGC Chemicals Americas, Inc.

Effective Date: 07/22/2013
Version: 15

SAFETY DATA SHEET

AE3000AT

1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

Product Name: AE3000AT
General Use: Solvent

Manufacturer
ASAHI GLASS CO., LTD
Chemicals Company
1-5-1, Marunouchi, Chiyoda-ku,Tokyo 100-8405, JAPAN

Telephone Number for Information
+81-3-3218-5574

Facsimile Number for Information
+81-3-3218-7854

Supplier
AGC Chemicals Americas, Inc.
55 E. Uwchlan Avenue
Suite 201
Exton, PA 19341

24 Hours Medical Emergency Telephone #: 800-420-8479
24 Hours Transportation Emergency # (CHEMTREC): 800-424-9300
Customer Service #: 800-424-7833

2. HAZARDS IDENTIFICATION

This product is hazardous under OSHA.

Emergency Overview:

WARNING!
May cause eye and skin irritation.
Prolonged or repeated skin contact with liquid may cause drying, cracking, redness, itching, and/or swelling (dermatitis).

Aspiration hazard if swallowed - can enter lungs and cause damage.
Inhalation of high concentration of vapors is harmful and may cause heart irregularites, unconsciousness, or death.

Potential Health Effects:

Inhalation:	Inhalation of vapor may cause coughing, dizziness, dullness, drowsiness, and headache. Inhalation of higher concentrations of vapor is harmful and may cause heart irregularities, central nervous system depression, narcosis, unconsciousness, respiratory failure, or death. Intentional misuse can be fatal. Vapor reduces oxygen available for breathing and is heavier than air.
Skin Contact:	May cause skin irritation.
Eye Contact:	May cause eye irritation.

3. COMPOSITION/INFORMATION ON INGREDIENTS

Component Name	CAS-NO	Weight %
1,2-trans-Dichloroethylene	156-60-5	46-54
1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether	406-78-0	43-52

OSHA - 29 CFR 1910 Specifically Regulated Chemicals

1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether and 1,2-trans-Dichloroethylene are hazardous components.

4. FIRST AID MEASURES

Eye Contact:	Immediately flush eyes with plenty of water for at least 15 minutes. Remove contact lenses, if worn. Get medical attention if irritation persists.
Skin Contact:	In case of skin contact, flush with water. Remove contaminated clothing and shoes. Get medical attention if irritation persists.
Inhalation:	If inhalation symptoms occur, immediately remove to fresh air. Keep person calm. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if cough or other symptoms develop.
Ingestion:	No specific intervention is indicated as the compound is not likely to be hazardous by ingestion. Do not induce vomiting because the hazard of aspirating the material into the lungs is considered greater than swallowing it. Get medical attention if symptoms develop. Never give anything by mouth to an unconscious person.

5. FIRE-FIGHTING MEASURES

Suitable Extinguishing Media:	Dry chemical Carbon dioxide (CO2) Water spray mist or foam.

Unsuitable Extinguish Media/Methods: None

Unusual Fire and Explosion Hazards: None known

Hazardous Combustion Product or Gases: Containers may rupture under fire conditions. Decomposition of this product at temperature above 300deg.C (572deg.F) can form hydrogen fluoride (HF), but HF will only accumulate with continuous exposure to excess heat in a sealed vessel.

Special Protective Equipment For Fire Fighters: Self-contained breathing apparatus (SCBA) is required if drums rupture and contents are spilled under fire conditions.

Additional Information: Use water spray to cool containers. Move containers from fire areas if it can be done without risk.

6. ACCIDENTAL RELEASE MEASURES

Personal precautions:
Ensure adequate ventilation.
Use personal respiratory protection, impermeable gloves, chemical splash goggles and protective clothing.

Environmental precautions:
Collect contaminated water/firefighting water separately. Do not wash away into drain or waterway.

Methods for cleaning up/taking up:
Absorb or contain liquid with inert material and dispose of in accordance with applicable regulations.

In case of spill or other release:
NOTES: Review chapter 5, chapter 7, and chapter 8 before proceeding with clean up. Use appropriate Personal Protective Equipment during clean up.

Extinguish flames in area to avoid forming dangerous decomposition products (See chapter 5). Dike spill. Prevent liquid from entering sewers, waterways or low areas. Ventilate area. Collect on absorbent material and transfer to steel drums for recovery/disposal. Comply with Federal, State, and local regulations on reporting releases.

Additional information:
Additional information: Review chapter 7 for safe handling information.
Review chapter 13 for disposal information.

7. HANDLING AND STORAGE

Handling:
Use with sufficient ventilation to keep employee exposure below recommended limits. Provide adequate ventilation for storage, handling, and use, especially for enclosed or low spaces. Avoid contact of liquid with eyes and prolonged skin exposure. Do not allow product to contact open flame or electrical heating elements because dangerous decomposition products may form. Trans-1,2-dichlorethylene can react with air to form explosive peroxides.

Storage
Store in clean, dry, well-ventilated area. Do not store product in direct sunlight.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Component Name	ACGIH TLV:	OSHA PEL:	NIOSH - Pocket Guide - OELs
1,2-trans-Dichloroethylene	200 ppm TWA	200 ppm TWA 790 mg/m^3 TWA	REL-TWA: 200ppm 790mg/m^3

Exposure Guidelines:

Component Name	AEL:	CEC:	EEL:	ECE:

1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether	Daily exposure limit: 50ppm (8h-TWA) *AEL is the Acceptable Exposure Limit set by Asahi Glass Co., Ltd.	Ceiling exposure concentration (non-emergency): 150ppm Exposure Limit recommended by US EPA	Emergency exposure limit: 150ppm (time limit 15min) Emergency Exposure Limit (EEL) set by Asahi Glass Co., Ltd.	Emergency ceiling exposure concentration: 500ppm Emergency ceiling exposure concentration set by Asahi Glass Co., Ltd.

EEL: Emergency Exposure Limits (EELs) are to be used for short-term emergency exposure control. They are concentrations of short periods which should not result in permanent adverse health effects or interfere with escape. They should not be confused with daily exposure limits (such as AEL's and EPA recommended exposure limits) that are designed for repeated exposure guidelines. For the use of AE3000, daily exposure limits such as AEL as well as EEL are to be followed. The EEL for AE3000 is needed to avoid anaesthetic effects, which could prevent self-rescue. If an EEL is exceeded for specified duration, evacuation, sheltering in place or other mitigatin steps should be taken.

Occupational Exposure Controls.

Engineering Controls:

Normal ventilation for standard manufacturing procedures is generally adequate. Local exhaust should be used when large amounts are released. Vapors are heavier than air. Use with adequate ventilation to prevent vapor buildup in low lying areas.

Personal Protection:

Respiratory Protection Use respiratory protection approved by NIOSH in USA or other equivalent in each country if exposure limits may be exceeded. Self-contained breathing apparatus (SCBA) is required if a large spill occurs.

Hand Protection: Impervious gloves

Eye Protection: Safety glasses and/or chemical tight goggles if splashing is likely to occur.

Additional advice:

Provide eyewash stations Impermeable apron and boots to prevent skin contact.

NPCA - HMIS (National Paint and Coating Association - Hazardous Materials Identification System) Hazard Rating

HMIS codes are intended for use in everyday workplace setting to provide a rapid indication of the occupational hazards associated with chemicals used in the workplace.
a) Flammability - 0, b) Health - 1, c) Reactivity - 0

9. PHYSICAL AND CHEMICAL PROPERTIES

Appearance and Odor:	Clear, colorless liquid with slight ethereal odor
Boiling Point (°F/C):	100.4/38.0
Melting Point/Range:	-56.74 deg.F (-49.3 degC)
Flash Point (Method Used):	None (Pensky-Martens Closed Cup & Cleveland Open Cup)
Lower Explosive Limit:	9.8%
Autoignition Temp. (°F/C):	N/D
Upper Explosive Limit:	13.3%
Vapor Pressure:	0.048MPa @ 20deg.C
Specific Gravity:	1.36 @ 25deg.C

Solubility in Water: N/D
pH Value Neutral
Vapor Density: N/D
Evaporation Rate: (Ethyl ether = 1) : 0.9

10. STABILITY AND REACTIVITY

Conditions to Avoid: Material is stable however, avoid open flames and high temperature. Do not allow product to contact open flame or electrical heating elements because dangerous decomposition products may form. Trans-1,2-dichlorethylene can react with air to form explosive peroxides.

Stability: Stable under recommended storage conditions.

Materials to Avoid:

Incompatible with alkali or alkaline earth metals-powdered Al, Zn, Be, etc.

Avoid contamination with caustic soda, caustic potash, or oxidizing materials. Shock sensitive compounds may be formed.

Hazardous Decomposition Products:

Decomposition products are hazardous. This compound can be decomposed by high temperatures (open flames, glowing metal surfaces, etc.) forming hydrochloric and hydrofluoric acids-possibly carbonyl halides.

Hazardous Polymerization: Will Not Occur

11. TOXICOLOGICAL INFORMATION

Animal Data

Component Name	Inhalation	Oral	Dermal	Eyes
1,2-trans-Dichloroethylene	6-h LC50: 21,700ppm in mouse			
1,1,2,2-Tetrafluoroethyl-2,2,2-trifluoroethyl ether	LC50: >24.8mg/L (3010ppm) in rat	LD50: >2,000mg/kg in rat	LD50: >2,000mg/kg in rat	

1,2-trans-Dichloroethylene

Other Toxicity Information:

Administration of trans-1,2-dichloroethylene in the drinking water to male and female mice at doses of 21,210 or 420 mg/kg/d for 90 days produced few adverse findings. In female, there was a decrease in lung weight (11%) at the high dose, in thymus weight (33 and 18%) at the high and intermediate dose levels, respectively. Decreases in SGPT, SGOT, and aniline hydroxylase activity were observed at all levels. In males, there was an increase of SAP and a decrease of glutathione. Glucose levels were increased in both sexes at all doses.

1,1,2,2-tetrafluoroethyl-2,2,2-trifluoroethyl ether

Acute Toxicity:
Skin and eye irritation:
Slight irritation to eye and mucous membranes
Skin irritation (rabbit): none
Eye irritation (rabbit): slight

Sensitization:
Skin (rat): none

Genetic Studies:
Ames Assay: Negative (OECD 471 & 472)
Chromosomal Aberration Test: Negative (CHL Cell) (OECD 473)

Repeated Dose Oral Toxicity (28 Day): NOEL 1000mg/kg/d
Repeated Dose Inhalation Toxicity (5 day): NOEL 1800ppm
Rats exposed to 2500 or 5000 ppm for 6 hours per day for 5 days showed convulsions.

Repeated Dose Inhalation Toxicity (90 day): NOEL 1000ppm
Rats exposed to 1000ppm for 6 hours per day, 5 days per week for 90 days showed no adverse effects.

12. ECOLOGICAL INFORMATION

Biodegradability:	Not biodegraded (OECD 301C)
Bioaccumulation:	N/D
Activated Sludge Study:	>100mg/L (OECD 209)
Fish Toxicity:	LC50 (96hr) (Carp): >76mg/L
Other Information:	Algal growth inhibition: ErC50>213mg/L ---- EbC50>213mg/L Mobility inhibition (Daphnia magna): 48hr-EC50>94mg/L

13. DISPOSAL CONSIDERATIONS

Waste treatment:	Recover by distillation or remove to permitted waste disposal facility.
Packaging treatment:	Dispose of waste containers in accordance with local laws and regulations.
Disposal methods	Comply with all federal, state, and local regulations. Do not dump this product into sewers, on the ground, or into any body of water. Reuse the residual product when possible. Send waste product for thermal destruction, using high-temperature incinerators designed to burn fluorine compounds. Dispose of waste containers in accordance with local laws and regulations. Comply with all federal, state, and local regulations. Do not dump this product into sewers, on the ground, or into any body of water.

14. TRANSPORT INFORMATION

US DEPARTMENT OF TRANSPORTATION (DOT)

Hazardous Materials: N/A
Hazard Class or Division: Not classified

Identification Number: Not regulated
Packing Group: Not classified
Hazardous Materials Description and Proper Shipping Name: N/A
Label(s) Required: Not classified

UN/Id No.: N/A
IMDG Status: Not restricted
Marine Pollutant: No

ICAO/IATA Status: Not restricted

15. REGULATORY INFORMATION

For European Union

EEC Classification	Not classified
Hazard Symbol	Not established
R-Phrases	Not established

Safety Phrases:
Not established

Council Directive 92/32/EEC Status:
These chemicals are listed on the EINECS: (trans-1,2-dichloroethylene: 205-860-2)

For United States of America

SNAP Acceptable:
Acceptable for precision cleaning, electronics cleaning, & aerosol solvents.

OSHA
This product is hazardous under 29 CFR 1910.1200

TSCA Status:
This chemical is listed on the TSCA inventory.

TSCA - Sect. 5(a)(2) - Chemicals with SNUR
This material will be the subject of a significant new use rule (SNUR) under 40 CFR Part 721. Accordingly, this material can only be used for the following industrial-use applications: (i) cleaning electronic components; (ii) precision cleaning; (iii) dewatering of electronic components and other parts following aqueous cleaning; and (iv) carrier/lubricant coating for hard disk drives and other precision parts.

SARA Section 302:
N/A

Sara (311, 312) Hazard Class:
Acute Health Hazard,

SARA Section 313: Yes trans-1,2-Dichloroethylene)
State Regulations (United States)

Component Name	U.S. - CERCLA/SARA - Hazardous Substances and their Reportable Quantities
1,2-trans-Dichloroethylene 156-60-5	1000 lb 454 kg 1 lb 0.454 kg

CERCLA Section 103: Yes (trans-1,2-Dichloroethylene: 1000lbs RQ)
User should ensure that this material is in compliance with federal requirements and ensure conformity to local regulations.

16. ADDITIONAL INFORMATION

N/E: Not Established
N/A: Not Applicable
N/D: No Data
C.O.C.: Cleveland Open Cup

NFPA Hazard Code

NFPA codes are designed for use by firefighters, sheriffs, or other emergency response teams who are concerned with the hazards of burning or exploding materials. These NFPA codes are not intended to address the hazards of this product other than in a fire situation.

NFPA Rating:

Health 2, Flammability 0 , Reactivity 0

Revision Summary - MSDS Section(s) Updated: 15

Return to App B

Return to Pg 9

Solkane® 365 mfc

Material Safety Data Sheet

Chemical: Pentafluorobutane

NFPA: H=0 F=4 I=1 S=None
HMIS: H=0 F=4 R=1 PPE= Supplied by user; dependent on conditions

MSDS Number: S365-1204
Effective Date: 03 December 2004
Issued by: Solvay Chemicals, Inc. Regulatory Affairs Department

Not valid three years after effective date or after issuance of superseding MSDS, whichever is earlier. French or Spanish translations of this MSDS may be available. Check www.solvaychemicals.us or call Solvay Fluorides, LLC to verify the latest version or translation availability.

Material Safety Data Sheets contain country specific regulatory information. Therefore, the MSDS's provided are for use only by customers of Solvay Fluorides, LLC in North America. If you are located in a country other than Canada, Mexico or the United States, please contact the Solvay Group company in your country for MSDS information applicable to your location.

1. Company and Product Identification

1.1 **Product Name:** Solkane® 365 mfc

Chemical Name: 1,1,1,3,3-pentafluorobutane

Synonyms: Pentafluorobutane, HFC-365 mfc

Chemical Formula: $C_4H_5F_5$

Molecular Weight: 148

CAS Number: 406-58-6

EINECS Number: 430-250-1

1.2 **Recommended Uses:** Foaming agents; solvents

1.3 **Supplier:**
Solvay Fluorides, LLC
PO BOX 27328 Houston, TX 77227-7328
3333 Richmond Ave. Houston, Texas 77098

1.4 **Emergency Telephone Numbers**
Emergencies (USA): 1-800-424-9300 (CHEMTREC®)
Transportation Emergencies (INTERNATIONAL/MARITIME): 1-703-527-3887 (CHEMTREC®)
Transportation Emergencies (CANADA): 1-613-996-6666 (CANUTEC)
Transportation Emergencies (MEXICO-SETIQ): 01-800-00-214-00 (MEX. REPUBLIC)
525-559-1588 (Mexico City and metro area)

2. Composition/Information on Ingredients

INGREDIENTS	FORMULA	WT. PERCENT	CAS #
1,1,1,3,3-pentafluorobutane	$C_4H_5F_5$	≥ 99.50	406-58-6

3. Hazards Identification

Emergency Overview:
- Highly flammable.
- In case of decomposition, releases hydrogen fluoride.

3.1 Route of Entry: Inhalation: Yes Skin: Yes Ingestion: Unlikely

3.2 Potential Effects of exposure:

Inhalation:
- No reported cases of intoxication in humans.
- Risk of moderate consequences experimentally observed or under certain conditions.
- At high concentrations, risk of narcosis or asphyxia by lack of oxygen.

Eyes: Slight irritation.

Skin contact: In case of repeated contact: dry and chapped skin.

Ingestion: No data available for humans.

Carcinogenicity: See section 11.3.

4. First-Aid Measures

General Recommendations:

4.1 Inhalation:
- Remove the subject from the contaminated area.
- Administer oxygen or cardiopulmonary resuscitation if necessary.
- Consult a physician in case of respiratory and nervous symptoms.

Eyes:
- Flush with running water for several minutes, while keeping the eyelids wide open.
- Consult an ophthalmologist in case of persistent pain.

Skin:
- Wash the affected skin with soap and water.
- Consult a physician in case of persistent pain or redness.

Ingestion: Unknown symptoms: consult a physician for advice.

If the subject is completely conscious: Rinse mouth and administer fresh water.

If the subject is unconscious: Not applicable.

4.2 Medical Treatment/Notes to Physician: None.

5. Fire-Fighting Measures

5.1 **Flash point:** ≤ -27°C (-17°F); Highly Flammable.

5.2 **Auto-ignition Temperature**: 580°C (1076°F).

5.3 **Flammability Limits:** Upper limit 13.3% (V/V); Lower limit 3.6% (V/V).

5.4 **Unusual Fire and Explosion Hazards:** Explosion possible with gas vapor and air mixture (See section 10).

5.5 **Extinguishing Methods**

Common:
- Powder.
- Foam, AFFF.
- CO_2.

Inappropriate extinguishing means: Water.

5.6 **Fire Fighting Procedures**

Specific hazards:
- Highly flammable (see section 9).
- Formation of dangerous gas/vapors in case of decomposition (see section 10).
- Gas/vapors are heavier than air and so may travel along the ground; remote ignition possible.
- Gas/vapors explosion possible in presence of air.

Protective measures in case of intervention:
- Evacuate all non-essential personnel.
- Wear self-contained breathing apparatus when in close proximity or in confined spaces.
- After intervention, take a shower, remove clothing carefully, clean and check the equipment.
- Intervention only by capable personnel who are trained and aware of the hazards of the product.
- When intervention in close proximity wear acid-resistant over-suit.

Other precautions:
- If safe to do so, remove the exposed containers, or cool with large quantities of water.
- Approach from upwind.
- As for any fire, ventilate and clean the rooms before reentry.
- After the fire, proceed rapidly to clean the surfaces exposed to the fumes in order to limit the damage to the equipment.
- Avoid propagating the fire, when directing the extinguishing means in a jet on the surface of the burning liquid.

6. Accidental Release Measures

6.1 Precautions:

- Follow the protective measures given in sections 5 and 8.
- Keep away materials and products which are incompatible with the product (see section 10).
- Eliminate all sources of ignition, and do not generate flames or sparks.
- If safe to do so, without overexposing anyone, try to stop the leak.
- Approach from upwind.
- Disperse gas/vapors with water spray.
- Protect intervention team with water spray.

6.2 Cleanup methods:

- If possible, dam large quantities of liquid with sand or earth.
- Prevent the product from entering sewers or confined places.
- Place everything in a closed, labeled container compatible with the product.
- Store the product in a safe and isolated place.
- For disposal methods, refer to section 13.
- Collect the product with suitable means.
- Clean the area with large quantities of water.

6.3 Precautions for protection of the environment: Prevent discharges into the environment (atmosphere…..).

7. Handling and Storage

7.1 Handling:

- Carry out industrial operations in closed piping circuits and equipment.
- Operate in a well-ventilated area.
- Prevent product vapors' decomposition from contacting hot spots.
- Keep away from heat sources.
- Keep away from reactive products (see section 10).
- Handle small quantities under a lab hood.
- Do not use tools that produce sparks.
- Use only equipment and materials which are compatible with the product.

7.2 Storage:

- Store in a ventilated, cool area.
- Keep away from heat sources.
- Keep away from reactive products (see section 10).
- Provide containment bund around storage containers and transfer installation.
- For bulk storage, consult the producer.

7.3 Specific Uses: For any particular use, please contact the supplier.

7.4 Other precautions:
- No open flames or sparks, no smoking.
- Follow the protective measures given in section 8.
- Use grounded equipment.
- Warn people about the dangers of the product.
- Prevent electrostatic discharges.
- Provide electrical equipment safe for hazardous locations.

7.5 Packaging:
- Ordinary steel.

8. Exposure Controls/Personal Protection

8.1 Exposure Limit Values -

Authorized limit Values

None listed

8.2 Exposure Controls:
- Follow the protective measures given in section 7.
- Provide premises ventilation.
- Respect local, state and national regulations for aqueous emissions (see section 15).

8.2.1 Occupational Exposure Controls:

8.2.1.1 Ventilation: Provide local ventilation suitable for the emission risk.

8.2.1.2 Respiratory protection:
- Minimum need if the local exhaust ventilation is adequate.
- Self-contained breathing apparatus in medium confinement/insufficient oxygen/ in case of large uncontrolled emissions / in all circumstances when the mask and cartridge do not give adequate protection.
- Use only respiratory protection that conforms to international /national standards.

8.2.1.3 Hand protection: Protective gloves - chemical-resistant (Neoprene).

8.2.1.4 Eye protection: Wear protective goggles for all industrial operations.

8.2.1.5 Skin protection:
- Overalls.
- Apron/boots of butyl rubber if risk of splashing.

8.3 Other precautions:
- Provide shower and eye wash stations.
- Consult the industrial hygienist or the safety manager for the selection of personal protective equipment suitable for the working conditions.
- Do not smoke, eat and drink in the working area.

9. Physical and Chemical Properties

9.1 **Appearance:** volatile liquid.
Color: colorless.
Odor: ethereal.

9.2 **Important Health, Safety and Environmental information:**
pH: 6 Concentration: 1.7 g/l.

Change of state:
Melting point: Not applicable.
Boiling point: 40°C (104°F).
Decomposition Temperature: No data.

Flash Point: ≤ -27°C (-17°F) - Remark: Highly Flammable; Method: DIN 51755 T2.

Flammability: Upper Limit: 13.3% (V/V); Lower limit: 3.6%(V/V).

Explosive Properties: Explosion possible with gas/vapor and air mixtures. (See also Section 10).

Oxidizing Properties: Not applicable.

Vapor Pressure:
- 43.3 kPa @ 20°C (68°F).
- 1.42 bar (abs) @ 50°C (122°F).

Relative Density:
Specific gravity (H_2O=1): 1.27.

Solubility:
Water: 1.7 g/l @ 21°C (70°F).
Fat: No data.

Partition coefficient: P (n-octanol/water): 1.6 Method: Measured value.

Viscosity: 0.4 mPa.s @ 25°C (77°F).

Vapor Density (air=1): 5.11.

10. Stability and Reactivity

10.1 **Conditions to avoid:** Heat/Sources of heat.

10.2 **Materials and substances to avoid:**
- Oxidizing agents.
- Metallic powders.
- Alkaline metals.

10.3 **Hazardous decomposition products:**
- Hydrogen fluoride.
- Fluorophosgene.

10.4 **Hazardous Polymerization:** Will not occur.

10.5 **Other information:** The vapor is heavier than air and disperses at ground level.

11. Toxicological Information

11.1 Acute toxicity:
Inhalation: LC_{50}, 4h, rat, > 10%.

Oral: LD_{50}, rat, > 2,000 mg/kg.

Dermal: No data.

Irritation:
- Rabbit, non-irritant (skin).
- Rabbit, slight irritant (eyes).

Sensitization: Guinea Pig, Non sensitizing (skin).

Comments: No appreciable toxic effect.

11.2 Chronic toxicity:
- Inhalation, after repeated exposure, rat, 30,000 ppm, observed effect (body weight).
- No mutagenic effect.
- Inhalation, after a single exposure, dog, ≥ 7.5%, cardiac sensitization following adrenergic stimulation.

11.3 Carcinogenic Designation: None.

12. Ecological Information

12.1 Acute ecotoxicity:
- Fishes, Brachydanio rerio, LC_{50}, 96h, >200 mg/l.
- Crustaceans, Daphnia magna, NOEC, 48 h, > 200 mg/l.
- Algae, Selenastrum capricornutum, NOEC, 72 h, = 113 mg/l.
- Terrestrial plants, NOEC, 1,000 ppm.

12.2 Chronic ecotoxicity: No data.

12.3 Mobility:
- Air, Henry's law constant (H) ca. 3.8kPa.m^3/mol - considerable volatility.
  Conditions: 20°C (68°F) / calculated value.
- Soil/sediments, adsorption, log KOC ca. 1.8.
  Conditions: calculated value.

12.4 Degradation
Abiotic:
- Air, indirect photo-oxidation, $t_{1/2}$ ca. 7.04 year(s).
- Air, photolysis, ODP =0 - Result: no effect on stratospheric zone.
  Reference value for CFC 11: ODP =1.
- Air, greenhouse effect, GWP = 890.
  Reference value for carbon dioxide: GWP = 1. (SOLKANE® 365mfc).

Biotic: Aerobic, test ready biodegradability/closed bottle, = 13%, 28 day(s)
Non-readily biodegradable.

12.5 Potential for bioaccumulation: Bioconcentration: log Po/w ca. 1.61.
Improbable bioaccumulation - Conditions: measured value.

12.6 Other adverse effects /Comments:

- Product is persistent in air (atmospheric lifetime: 16-19 years).
- Hazard for the aquatic environment is limited due to product properties: Considerable volatility, Low bioaccumulation potential.

13. Disposal Considerations

13.1 Waste treatment:

- Dispose in compliance with local, state and national regulations.
- It is recommended to contact the producer for recycling /recovery.
- Send the product to an authorized industrial waste incinerator.
- The incinerator must be equipped with a system for the neutralization of HF.

13.2 Packaging treatment: To avoid treatment, use dedicated containers.

13.3 RCRA Hazardous Waste: D001 (Ignitability).

14. Transport Information

Mode	DOT	IMDG	IATA
UN Number	1993	1993	1993
Class (Subsidiary)	3	3	3
Proper Shipping Name	Flammable Liquid, N.O.S. (Pentafluorobutane)	Flammable Liquid, N.O.S. (Pentafluorobutane)	Flammable Liquid, N.O.S. (Pentafluorobutane)
Hazard label (Subsidiary)	Flammable Liquid	Flammable Liquid	Flammable Liquid
Marine Pollutant			
Placard [Subsidiary]			
Packing Group	II		
MFAG			
Emergency Info	ERG: 123	EmS: 3-07	ERG Code: 3HP

15. Regulatory Information

National Regulations (US)

TSCA Inventory 8(b): Yes.

SARA Title III Sec. 302/303 Extremely Hazardous Substances (40 CFR 355): No.

SARA Title III Sec. 311/312 (40 CFR 370):
Hazard Category: Fire Hazard.
Threshold planning quantity: 10,000 lbs.

SARA Title III Sec. 313 Toxic Chemical Emissions Reporting (40 CFR 372): No.

CERCLA Hazardous Substance (40 CFR Part 302)
Listed Substance: No.
Unlisted Substance: Yes, Reportable Quantity- 100 lbs.
Characteristic: D001- Ignitability.

State Component Listing:
State Comment: None identified.

National Regulations (Canada)

Canadian DSL Registration: No.

WHMIS Classification: B2 - Flammable Liquid.

This product has been classified in accordance with the hazard criteria of the ***Controlled Products Regulations,*** and the MSDS contains all the information required by the ***Controlled Products Regulations.***

Labeling according to Directive 1999/45/EC.

Category	ID	Phrase
Symbols	F	Highly flammable.
Phrases R	11	Highly flammable.
Phrases S	16	Keep away from sources of ignition --- No smoking.

16. Other Information

16.1 Ratings:

NFPA (NATIONAL FIRE PROTECTION ASSOCIATION)
Health = 0 Flammability = 4 Instability = 1 Special = None

HMIS (HAZARDOUS MATERIAL INFORMATION SYSTEM)
Health = 0 Fire = 4 Reactivity = 1 PPE = Supplied by User; dependent on local conditions

16.2 Other Information:

The previous information is based upon our current knowledge and experience of our product and is not exhaustive. It applies to the product as defined by the specifications. In case of combinations or mixtures, one must confirm that no new hazards are likely to exist. In any case, the user is not exempt from observing all legal, administrative and regulatory procedures relating to the product, personal hygiene, and integrity of the work environment. (Unless noted to the contrary, the technical information applies only to pure product).

To our actual knowledge, the information contained herein is accurate as of the date of this document. However, neither Solvay Fluorides, LLC nor any of its affiliates makes any warranty, express or implied, or accepts any liability in connection with this information or its use. This information is for use by technically skilled persons at their own discretion and risk and does not relate to the use of this product in combination with any other substance or any other process. This is not a license under any patent or other proprietary right. The user alone must finally determine suitability of any information or material for any contemplated use, the manner of use and whether any patents are infringed. This information gives typical properties only and is not to be used for specification purposes.

Trademarks and/or other Solvay Fluorides, LLC products referenced herein are either trademarks or registered trademarks of Solvay Fluorides, LLC or its affiliates, unless otherwise indicated.

16.3 Reason for revision:

Supersedes edition: Solvay Fluorides, LLC MSDS, S365-1103 dated 11-11-03.
Purpose of revision: update information, correct typos.

Return to App B   Return to Pg 9

SAFETY DATA SHEET
North American Version

Solvokane (TM)

1. PRODUCT AND COMPANY IDENTIFICATION

1.1. Identification of the substance or mixture

Product name : Solvokane (TM)
Molecular formula : $C_2H_2Cl_2$; $C_4H_5F_5$

1.2. Use of the Substance/Mixture

Recommended use :
- Aerosol propellants
- Cleaning agent
- Detergent
- Electrical industry
- Electronic industry

1.3. Company/Undertaking Identification

Address : SOLVAY FLUORIDES, LLC
3333 RICHMOND AVENUE
HOUSTON TX 77098-3099
United States

1.4. Emergency and contact telephone numbers

Emergency telephone : **1 (800) 424-9300 CHEMTREC ® (USA & Canada)**
01-800-00-214-00 (MEX. REPUBLIC)

Contact telephone number (product information): : **US: +1-800-765-8292 (Product information)**
US: +1-713-525-6500 (Product information)

2. HAZARDS IDENTIFICATION

2.1. Emergency Overview:

NFPA : H= 0 F= 0 I= 0 S= none
HMIS : H= 0 F= 0 R= 0 PPE = Supplied by User; dependent on local conditions

General Information

Appearance : liquid
Colour : colourless
Odour : ether-like

Main effects
- The product is not flammable.
- In use, may form flammable/explosive vapour-air mixture.
- Harmful by inhalation.
- Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
- Hazardous decomposition products formed under fire conditions.
- Gaseous hydrogen fluoride (HF).

2.2. Potential Health Effects:

Inhalation
- May be harmful by inhalation (after often repeated exposure).

Eye contact
- Contact with eyes may cause irritation.

Skin contact
- The product may be absorbed through the skin.

Ingestion
- No information available.
- Ingestion may cause gastrointestinal irritation, nausea, vomiting and diarrhoea.

Other toxicity effects
- See section 11: Toxicological Information

2.3. Environmental Effects:

- See section 12: Ecological Information

3. COMPOSITION/INFORMATION ON INGREDIENTS

1,1,1,3,3-pentafluorobutane
CAS-No. : 406-58-6
ELINCS No. : 430-250-1
Concentration : >= 65,0 %

trans-Dichloro ethylene
CAS-No. : 156-60-5
Concentration : < 35,0 %

4. FIRST AID MEASURES

4.1. Inhalation

- Oxygen or cardiopulmonary resuscitation if necessary
- Remove to fresh air.
- If symptoms persist, call a physician.

4.2. Eye contact

- Flush eyes with running water for several minutes, while keeping the eyelids wide open.
- If eye irritation persists, consult a specialist.

4.3. Skin contact

- Wash off with soap and water.
- Remove and wash contaminated clothing before re-use.
- If symptoms persist, call a physician.

4.4. Ingestion

- If symptoms persist, call a physician.

If victim is conscious:
- Clean mouth with water and drink afterwards plenty of water.

If victim is unconscious but breathing:
- Oxygen or artificial respiration if needed.

5. FIRE-FIGHTING MEASURES

5.1. Suitable extinguishing media

- powder
- Foam, AFFF
- Carbon dioxide (CO2)

5.2. Extinguishing media which shall not be used for safety reasons

- Water may be ineffective.

5.3. Special exposure hazards in a fire

- The product is not flammable.
- Vapours are heavier than air and may spread along floors.
- Risk of ignition.
- Vapours may form explosive mixtures with air.
- Hazardous decomposition products formed under fire conditions.

5.4. Hazardous decomposition products

- Gaseous hydrogen fluoride (HF).
- Fluorophosgene
- Gaseous hydrogen chloride (HCl).
- Phosgene
- Carbon monoxide
- The release of other hazardous decomposition products is possible.

5.5. Special protective equipment for fire-fighters

- Wear self-contained breathing apparatus and protective suit.
- Fire fighters must wear fire resistant personnel protective equipment.
- Wear chemical resistant oversuit
- Clean contaminated surface thoroughly.

5.6. Other information

- Evacuate personnel to safe areas.
- Keep containers and surroundings cool with water spray.
- After the fire, proceed rapidly with cleaning of surfaces exposed to the fumes in order to limit equipment damage.
- Approach from upwind.
- Avoid propagating the fire when directing the extinguishing agent as a jet onto the surface of the burning liquid.

6. ACCIDENTAL RELEASE MEASURES

6.1. Personal precautions

- Refer to protective measures listed in sections 7 and 8.
- Approach from upwind.
- Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
- Suppress (knock down) gases/vapours/mists with a water spray jet.
- Avoid spraying the leak source.
- Cover the spreading liquid with foam in order to slow down the evaporation.
- Prevent further leakage or spillage if safe to do so.
- Keep away from open flames, hot surfaces and sources of ignition.
- Ventilate the area.

6.2. Environmental precautions

- Should not be released into the environment.

6.3. Methods for cleaning up

- Dam up.

- Soak up with inert absorbent material.
- Prevent product from entering drains.
- Keep in properly labelled containers.
- Keep in suitable, closed containers for disposal.
- Treat recovered material as described in the section "Disposal considerations".

7. HANDLING AND STORAGE

7.1. Handling

- Use only in well-ventilated areas.
- Use only equipment and materials which are compatible with the product.
- Keep away from Incompatible products.
- Keep away from heat/sparks/open flames/hot surfaces. - No smoking.
- Ensure all equipment is electrically grounded before beginning transfer operations.
- Take measures to prevent the build up of electrostatic charge.

7.2. Storage

- Store in original container.
- Keep container closed.
- Keep in a cool, well-ventilated place.
- Keep away from heat and sources of ignition.
- Keep away from incompatible products
- Keep in a bunded area.
- Do not freeze.
- Keep in a dry place.

7.3. Other information

- To avoid thermal decomposition, do not overheat.

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

8.1. Exposure Limit Values

1,1,1,3,3-pentafluorobutane

- SAEL (Solvay Acceptable Exposure Limit) 2006
  TWA = 1.000 ppm

trans-Dichloro ethylene

- Canada. Alberta OELs (Occupational Health & Safety Code, Schedule 1, Table 2) 10 2006
  time weighted average = 200 ppm
  time weighted average = 793 mg/m3
- Canada. British Columbia OELs. (Occupational Exposure Limits for Chemical Substances, Occupational Health and Safety Regulation 296/97, as amended) 07 2007
  time weighted average = 200 ppm
- US. ACGIH Threshold Limit Values 2009
  time weighted average = 200 ppm
- Canada. Quebec OELS. (Ministry of Labor - Regulation Respecting the Quality of the Work Environment) 2006
  time weighted average = 200 ppm
  time weighted average = 793 mg/m3
- Canada. Ontario OELs. (Ministry of Labor - Control of Exposure to Biological or Chemical Agents) 08 2008
  time weighted average = 200 ppm
  time weighted average = 790 mg/m3
- Canada. Ontario OELs. (Ministry of Labor - Control of Exposure to Biological or Chemical Agents) 08 2008

Short term exposure limit = 250 ppm
Short term exposure limit = 990 mg/m3

- <u>Canada. Quebec OELS. (Ministry of Labor - Regulation Respecting the Quality of the Work Environment) 12 2008</u>
  time weighted average = 200 ppm
  time weighted average = 793 mg/m3

8.2. Engineering controls

- Provide appropriate exhaust ventilation at machinery.
- Apply technical measures to comply with the occupational exposure limits.
- Refer to protective measures listed in sections 7 and 8.

8.3. Personal protective equipment

8.3.1. Respiratory protection

- Use NIOSH approved respiratory protection.
- Comply with OSHA respiratory protection requirements.
- Self-contained breathing apparatus in medium confinement/insufficient oxygen/in case of large uncontrolled emissions/in all circumstances when the mask and cartridge do not give adequate protection.
- Use only respiratory protection that conforms to international/ national standards.

8.3.2. Hand protection

- Protective gloves - impervious chemical resistant:
- Neoprene
- Take note of the information given by the producer concerning permeability and break through times, and of special workplace conditions (mechanical strain, duration of contact).

8.3.3. Eye protection

- Chemical resistant goggles must be worn.

8.3.4. Skin and body protection

- Wear suitable protective clothing.

8.3.5. Hygiene measures

- Consult the industrial hygienist or the safety manager for the selection of personal protective equipment suitable for the working conditions.

9. PHYSICAL AND CHEMICAL PROPERTIES

9.1. General Information

Appearance	:	liquid
Colour	:	colourless
Odour	:	ether-like

9.2. Important health safety and environmental information

pH	:	= 6 (SOLKANE ® 365mfc) *Concentration*: 1,7 g/l
Boiling point/boiling range	:	36 °C (97 °F)
Flash point	:	*Remarks*: does not flash, The product is not flammable. *Method*: closed cup
Flammability	:	*<u>Upper explosion limit</u>*: 9,4 %(V)

		Lower explosion limit: 5,4 %(V) *Remarks*: The product is not flammable.
Explosive properties	:	*Explosion danger*: *Remarks*: Vapours may form explosive mixture with air.
Oxidizing properties	:	*Remarks*: Non oxidizer
Vapour pressure	:	540 hPa *Temperature*: 20 °C (68 °F)
Relative density / **Density**	:	1,225
Solubility	:	3,34 g/l
Partition coefficient: n-octanol/water	:	*log Pow*: < 3 *Method*: calculated value
Viscosity	:	0,53 mPa.s *Temperature*: 25 °C (77 °F)
Vapour density	:	> 1 *Method*: calculated value

9.3. Other data

Freezing point: : -43 °C (-45 °F)

10. STABILITY AND REACTIVITY

10.1. Stability

- Stable under recommended storage conditions.
- Strong oxidizers, alkali metals and alkaline earth metals may cause fires or explosions.
- Vapours are heavier than air and may spread along floors.

10.2. Conditions to avoid

- Direct sources of heat.
- Keep away from direct sunlight.
- Do not freeze.

10.3. Materials to avoid

- Oxidizing agents, Powdered metals, Reducing agents, Alkali metals

10.4. Hazardous decomposition products

- Gaseous hydrogen fluoride (HF)., Fluorophosgene, Gaseous hydrogen chloride (HCl)., Phosgene, Carbon monoxide, The release of other hazardous decomposition products is possible.

11. TOXICOLOGICAL INFORMATION

Toxicological data

Acute oral toxicity

- LD50, rat, > 2.000 mg/kg (SOLKANE ® 365mfc)
- LD50, rat, > 2.000 mg/kg (trans-Dichloro ethylene)

Acute inhalation toxicity

- LC50, 4 h, rat, > 0,605 g/l (SOLKANE ® 365mfc)

- LD50, 4 h, rat, 0,0955 g/l (trans-Dichloro ethylene)

Skin irritation

- rabbit, No skin irritation (SOLKANE ® 365mfc)

Eye irritation

- rabbit, Mild eye irritation (SOLKANE ® 365mfc)

Sensitisation

- guinea pig (SOLKANE ® 365mfc)

Chronic toxicity

- Inhalation, after a single exposure, dog, NOEL**:** >= 7,5 %, cardiac sensitization following adrenergic stimulation, (SOLKANE ® 365mfc)
- Inhalation, Repeated exposure, rat, NOEL**:** 30000 ppm, (SOLKANE ® 365mfc)
- Inhalation, 90-day, rat, NOEL**:** > 4000 ppm, (trans-Dichloro ethylene)
- Oral, rat, NOEL**:** 190 mg/kg, (trans-Dichloro ethylene)

Remarks

- Harmful by inhalation.
- Information given is based on data on the components and the toxicology of similar products.
- There is no data available for this product.
- May cause eye and skin irritation.
- May cause irritation of the mucous membranes.

12. ECOLOGICAL INFORMATION

12.1. Ecotoxicity effects

Acute toxicity

- Fishes, Brachydanio rerio, LC50, 96 h, > 200 mg/l
- Crustaceans, Daphnia magna, NOEC, 48 h, > 200 mg/l

Chronic toxicity

- Algae, Selenastrum capricornutum, NOEC, 72 h, = 113 mg/l

12.2. Mobility

- Air, Henry's law constant (H) ca. 38 hPa**.**m³/mol
  Conditions: 20 °C / calculated value
  Remarks: considerable volatility
- Soil/sediments, adsorption, log KOC:ca. 1,8
  Conditions: calculated value

12.3. Persistence and degradability

Abiotic degradation

- Air, indirect photo-oxidation, t 1/2 ca. 7,04 y

Biodegradation

- aerobic, Tested according to: Closed Bottle test
  Remarks: Not readily biodegradable.

12.4. Bioaccumulative potential

- Bioaccumulative potential: log Pow ca. 1,61
  Result: Bioaccumulation is unlikely.
  Remarks: measured value

12.5. Other adverse effects

- Ozone Depletion Potential :
  Ozone depletion potential; ODP; (R-11 = 1) = 0
  Result: no effect on stratospheric ozone

- Global Warming Potential :
  Global warming potential (GWP) = 890
  Remarks: Reference value for carbon dioxide: GWP = 1

12.6. Remarks

- Information refers to the main component.
- The product contains following substances which are hazardous for the environment:
- Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.

13. DISPOSAL CONSIDERATIONS

13.1. Waste from residues / unused products

- In accordance with local and national regulations.
- Refer to manufacturer/supplier for information on recovery/recycling.
- or
- Must be incinerated in a suitable incineration plant holding a permit delivered by the competent authorities.
- The incinerator must be equipped with a system for the neutralisation or recovery of HF.

13.2. Packaging treatment

- To avoid treatments, as far as possible, use dedicated containers.

13.3. RCRA Hazardous Waste

- Listed RCRA Hazardous Waste (40 CFR 302) - Yes

14. TRANSPORT INFORMATION

- not regulated
- It is recommended that ERG Guide number 111 be used for all non-regulated material.

15. REGULATORY INFORMATION

15.1. Inventory Information

Australian Inventory of Chemical Substances (AICS)	: -	In compliance with inventory.
Canadian Domestic Substances List (DSL)	: -	In compliance with inventory.
Inventory of Existing Chemical Substances (China) (IECS)	: -	In compliance with inventory.
Japan (ENCS) List (ENCS (JP))	: -	In compliance with inventory.
New Zealand Interim Inventory of Chems. (NZ CLSC)	: -	In compliance with inventory.
Toxic Substance Control Act list (TSCA)	: -	In compliance with inventory.
EU list of existing chemical substances (EINECS)	: -	In compliance with inventory, The formulation contains ELINCS substances..
Korea Existing Chemicals Inv. (KECI) (KECI (KR))	: -	In compliance with inventory.

Philippines PICCS (PICCS (PH))	: -	One or more components not listed on inventory.

15.2. Other regulations

US. EPA Emergency Planning and Community Right-To-Know Act (EPCRA) SARA Title III Section 302 Extremely Hazardous Substance (40 CFR 355, Appendix A)

- not regulated.

US. EPA Emergency Planning and Community Right-To-Know Act (EPCRA) SARA Title III Section 313 Toxic Chemicals (40 CFR 372.65) - Supplier Notification Required

- no.

US. EPA CERCLA Hazardous Substances (40 CFR 302)

- This product is reportable under 40 CFR Part 302.4 because it contains the following substance(s):, trans-dichloroethylene.

US. New Jersey Worker and Community Right-to-Know Act (New Jersey Statute Annotated Section 34:5A-5)

- yes.

US. Pennsylvania Worker and Community Right-to-Know Law (34 Pa. Code Chap. 301-323)

- yes.

US. California Safe Drinking Water & Toxic Enforcement Act (Proposition 65)

- not regulated.

15.3. Classification and labelling

Canada. Canadian Environmental Protection Act (CEPA). WHMIS Ingredient Disclosure List (Can. Gaz., Part II, Vol. 122, No. 2)

- Toxic Material Causing Other Toxic Effects

Remarks: This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all the information required by the Controlled Products Regulations.

EC Label

- Hazardous components which must be listed on the label: 1,1,1,3,3-pentafluorobutane / trans-Dichloro ethylene
- The product is classified and labelled in accordance with Directive 1999/45/EC.

Symbol(s)	Xn	Harmful
R-phrase(s)	R18	In use, may form flammable/explosive vapour-air mixture.
	R20	Harmful by inhalation.
	R52/53	Harmful to aquatic organisms, may cause long-term adverse effects in the aquatic environment.
S-phrase(s)	S16	Keep away from sources of ignition - No smoking.
	S29	Do not empty into drains.
	S36/37/39	Wear suitable protective clothing, gloves and eye/face protection.
	S61	Avoid release to the environment. Refer to special instructions/ Safety data sheets.

16. OTHER INFORMATION

Ratings :

NFPA (National Fire Protection Association)
Health = 0 Flammability = 0 Instability = 0 Special =none

HMIS (Hazardous Material Information System)
Health = 0 Fire = 0 Reactivity = 0 PPE : Supplied by User; dependent on local conditions

Further information

- Update
  This data sheet contains changes from the previous version in section(s): 7.1, 7.2, 7.4, 10.1 ,10.2
- Distribute new edition to clients

Material Safety Data Sheets contain country specific regulatory information; therefore, the MSDS's provided are for use only by customers of the company mentioned in section 1 in North America. If you are located in a country other than Canada, Mexico or the United States, please contact the Solvay Group company in your country for MSDS information applicable to your location.

The previous information is based upon our current knowledge and experience of our product and is not exhaustive. It applies to the product as defined by the specifications. In case of combinations or mixtures, one must confirm that no new hazards are likely to exist. In any case, the user is not exempt from observing all legal, administrative and regulatory procedures relating to the product, personal hygiene, and integrity of the work environment. (Unless noted to the contrary, the technical information applies only to pure product).

To our actual knowledge, the information contained herein is accurate as of the date of this document. However, neither the company mentioned in section 1 nor any of its affiliates makes any warranty, express or implied, including merchantability or fitness for use, or accepts any liability in connection with this information or its use. This information is for use by technically skilled persons at their own discretion and risk and does not relate to the use of this product in combination with any other substance or any other process. This is not a license under any patent or other proprietary right. The user alone must finally determine suitability of any information or material for any contemplated use in compliance with applicable law, the manner of use and whether any patents are infringed. This information gives typical properties only and is not to be used for specification purposes. The company mentioned in section 1 reserves the right to make additions, deletions or modifications to the information at any time without prior notification.

Trademarks and/or other products of the company mentioned in section 1 referenced herein are either trademarks or registered trademarks of the company mentioned in section 1 or its affiliates, unless otherwise indicated.

This product has been classified in accordance with the hazard criteria of the Controlled Products Regulations and the MSDS contains all the information required by the Controlled Products Regulations.

Return to App B

Return to Pg 9

Material Safety Data Sheet

DuPont™ VERTREL® MCA specialty fluid

Version 2.1

Revision Date 07/13/2012 Ref. 130000000634

This SDS adheres to the standards and regulatory requirements of the United States and may not meet the regulatory requirements in other countries.

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name	:	DuPont™ VERTREL® MCA specialty fluid
MSDS Number	:	130000000634
Product Use	:	Cleaning agent
Manufacturer	:	DuPont 1007 Market Street Wilmington, DE 19898
Product Information	:	1-800-441-7515 (outside the U.S. 1-302-774-1000)
Medical Emergency	:	1-800-441-3637 (outside the U.S. 1-302-774-1139)
Transport Emergency	:	CHEMTREC: 1-800-424-9300 (outside the U.S. 1-703-527-3887)

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing. Prolonged skin contact may defat the skin and produce dermatitis. May cause cardiac arrhythmia.

Potential Health Effects

Skin

trans-Dichloroethylene : Causes skin irritation. May cause:, Pain, burning sensation, itching, redness, swelling, or rash..

Eyes

trans-Dichloroethylene : May cause eye irritation. May cause:, Tearing, redness, or discomfort..

Inhalation

trans-Dichloroethylene : May cause: Central nervous system depression with dizziness, confusion, incoordination, drowsiness, or unconsciousness.

1,1,1,2,2,3,4,5,5,5-Decafluoropentane	:	Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing. Effects of breathing high concentrations of vapour may include:, Tiredness or drowsiness, Central nervous system effects, Convulsions.

Repeated exposure

1,1,1,2,2,3,4,5,5,5-Decafluoropentane	:	Adverse effects from repeated inhalation may include: Central nervous system effects

Target Organs

trans-Dichloroethylene	:	Central nervous system

Carcinogenicity

None of the components present in this material at concentrations equal to or greater than 0.1% are listed by IARC, NTP, or OSHA, as a carcinogen.

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Component	CAS-No.	Concentration
trans-Dichloroethylene	156-60-5	37 - 39 %
1,1,1,2,2,3,4,5,5,5-Decafluoropentane	138495-42-8	61 - 63 %

SECTION 4. FIRST AID MEASURES

Skin contact	:	Take off all contaminated clothing immediately. Wash off with warm water.
Eye contact	:	In case of eye contact Hold eyelids apart and flush eyes with plenty of water for at least 15 minutes. Get medical attention.

Inhalation	: Remove from exposure, lie down. Move to fresh air. Keep patient warm and at rest. Artificial respiration and/or oxygen may be necessary. Consult a physician.
Ingestion	: Do not induce vomiting without medical advice. Never give anything by mouth to an unconscious person. Drink 1 or 2 glasses of water. If vomiting occurs, have victim lean forward to reduce the risk of aspiration. Consult a physician.
General advice	: Never give anything by mouth to an unconscious person. Victim to lie down in the recovery position, cover and keep him warm. Give oxygen or artificial respiration if needed. When symptoms persist or in all cases of doubt seek medical advice.
Notes to physician	: Do not give adrenaline or similar drugs.

SECTION 5. FIREFIGHTING MEASURES

Flammable Properties	
Flash point	: Method : Pensky-Martens closed cup - PMCC does not flash
Lower explosion limit	: Method : None per ASTM E681
Upper explosion limit	: Method : None per ASTM E681
Fire and Explosion Hazard	: Fire or intense heat may cause violent rupture of packages. Hazardous combustion products: Hydrogen fluoride Fluorinated hydrocarbons Carbonyl halides Carbon oxides Hydrogen chloride The product is not flammable.
Suitable extinguishing media	: Water spray, Water mist, Dry chemical, Carbon dioxide (CO2)

Firefighting Instructions	: In the event of fire, wear self-contained breathing apparatus. Use personal protective equipment. Wear neoprene gloves during cleaning up work after a fire. Exposure to decomposition products may be a hazard to health. Use extinguishing measures that are appropriate to local circumstances and the surrounding environment. Evacuate personnel to safe areas. Cool containers / tanks with water spray. Fire residues and contaminated fire extinguishing water must be disposed of in accordance with local regulations.

SECTION 6. ACCIDENTAL RELEASE MEASURES

NOTE: Review FIRE FIGHTING MEASURES and HANDLING (PERSONNEL) sections before proceeding with clean-up. Use appropriate PERSONAL PROTECTIVE EQUIPMENT during clean-up.

Safeguards (Personnel)	: Evacuate personnel to safe areas. Ventilate area, especially low or enclosed places where heavy vapours might collect. In case of insufficient ventilation, wear suitable respiratory equipment. Refer to protective measures listed in sections 7 and 8.
Spill Cleanup	: Contain spillage, and then collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container for disposal according to local / national regulations (see section 13).
Accidental Release Measures	: Prevent further leakage or spillage. Prevent spreading over a wide area (e.g. by containment or oil barriers). Should not be released into the environment.

SECTION 7. HANDLING AND STORAGE

Handling (Personnel)	: Avoid contact with skin, eyes and clothing. Avoid breathing vapours or mist. Provide sufficient air exchange and/or exhaust in work rooms. For personal protection see section 8. Handle in accordance with good industrial hygiene and safety practice. When using do not eat, drink or smoke. Do not breathe vapours or spray mist. Avoid contact with skin, eyes and clothing. Wash hands before breaks and at the end of workday.
Handling (Physical Aspects)	: Material should not be dispensed from its container by pouring, except for small sample containers where fume hoods or where other ventilation is used to manage the exposure limits. The use of a drum pump is recommended for

		dispensing from shipping containers.
Storage	:	Protect from contamination. Keep container tightly closed in a dry and well-ventilated place. Store in original container. Avoid freezing temperatures. If stored below -10°C (14°F), mix prior to use.
Storage temperature	:	< 52 °C (< 126 °F)

SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Engineering controls	:	Use sufficient ventilation to keep employee exposure below recommended limits.
Personal protective equipment		
Respiratory protection	:	For rescue and maintenance work in storage tanks use self-contained breathing apparatus. Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Hand protection	:	Material: Solvent-resistant gloves
Eye protection	:	Safety glasses with side-shields Additionally wear a face shield where the possibility exists for face contact due to splashing, spraying or airborne contact with this material.
Skin and body protection	:	Protective suit

Exposure Guidelines

Exposure Limit Values

1,1,1,2,2,3,4,5,5,5-Decafluoropentane				
AEL *	(DUPONT)	200 ppm		8 & 12 hr. TWA
AEL *	(DUPONT)	400 ppm		Ceiling Limit Value:
trans-Dichloroethylene				
PEL:	(OSHA)	200 ppm	790 mg/m3	8 hr. TWA

TLV	(ACGIH)	200 ppm	TWA
AEL *	(DUPONT)	200 ppm	8 & 12 hr. TWA

* AEL is DuPont's Acceptable Exposure Limit. Where governmentally imposed occupational exposure limits which are lower than the AEL are in effect, such limits shall take precedence.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Form	: liquid
Color	: colourless
Odor	: ether-like
pH	: neutral
Melting point/range	: < -50.0 °C (< -58.0 °F)
Boiling point/boiling range	: 39.0 °C (102.2 °F) at 1,013 hPa
Vapour Pressure	: 216 hPa at 0 °C (32 °F)
	: 619 hPa at 25 °C (77 °F)
	: 1,481 hPa at 50 °C (122 °F)
Density	: 1.47 g/cm3 at 0 °C (32 °F)
Density	: 1.41 g/cm3 at 25 °C (77 °F)
Density	: 1.35 g/cm3 at 50 °C (122 °F)
Water solubility	: 15 g/l at 25 °C (77 °F)
Viscosity	: 0.49 mPa.s at 25 °C (77 °F)
Vapour density	: 5.4

SECTION 10. STABILITY AND REACTIVITY

Stability	: No decomposition if stored and applied as directed.
Incompatibility	: Alkali metals Alkaline earth metals, Powdered metals, Powdered metal salts, Strong bases
Hazardous decomposition products	: Hazardous decomposition products formed under fire conditions.: Fluorinated hydrocarbons, Hydrogen fluoride, Carbon dioxide (CO2), Carbon monoxide

SECTION 11. TOXICOLOGICAL INFORMATION

DuPont™ VERTREL® MCA specialty fluid

Further information : Aspiration may cause pulmonary oedema and pneumonitis.

trans-Dichloroethylene

Dermal LD50 : > 5,000 mg/kg , rabbit

Oral LD50 : 7,902 mg/kg , rat

Inhalation 4 h LC50 : 96.4 mg/l , rat
Target Organs: Central nervous system
narcosis

Inhalation Low Observed Adverse Effect Concentration (LOAEC) : 250000 ppm , rat
Cardiac sensitization

Skin irritation : Skin irritation, rabbit

Eye irritation : Mild eye irritation, rabbit

Repeated dose toxicity : Inhalation
rat
90 d
No toxicologically significant effects were found.

Oral - feed
rat
90 d
No toxicologically significant effects were found.

Mutagenicity : Did not cause genetic damage in animals.
Tests on bacterial or mammalian cell cultures did not show mutagenic effects.

Reproductive toxicity : Animal testing showed no reproductive toxicity.

Teratogenicity : Animal testing showed no developmental toxicity.

1,1,1,2,2,3,4,5,5,5-Decafluoropentane

Dermal LD50 : > 5,000 mg/kg , rabbit

Oral LD50 : > 5,000 mg/kg , rat

Inhalation 4 h LC50 : 114 mg/l , rat
Central nervous system effects
Convulsions

Skin irritation : No skin irritation, rabbit

Eye irritation : No eye irritation, rabbit

Skin sensitization : Did not cause sensitization on laboratory animals., guinea pig

Repeated dose toxicity : Inhalation
rat

No toxicologically significant effects were found.

Mutagenicity : Did not cause genetic damage in animals.
Did not cause genetic damage in cultured mammalian cells.
Did not cause genetic damage in cultured bacterial cells.

Reproductive toxicity : Animal testing showed no reproductive toxicity.

Teratogenicity : Animal testing showed no developmental toxicity.

SECTION 12. ECOLOGICAL INFORMATION

Aquatic Toxicity

trans-Dichloroethylene

96 h LC50 : Lepomis macrochirus (Bluegill sunfish) 74 mg/l

96 h EC50 : Pseudokirchneriella subcapitata (green algae) 798 mg/l

48 h LC50 : Daphnia magna (Water flea) 79 mg/l

1,1,1,2,2,3,4,5,5,5-Decafluoropentane

96 h LC50 : Oncorhynchus mykiss (rainbow trout) 13.9 mg/l

96 h LC50 : Pimephales promelas (fathead minnow) 27.2 mg/l

96 h LC50	:	Danio rerio (zebra fish) 13 mg/l
72 h EC50	:	Pseudokirchneriella subcapitata (green algae) > 120 mg/l
48 h LC50	:	Daphnia magna (Water flea) 11.7 mg/l
21 d	:	NOEC Daphnia magna (Water flea) 1.72 mg/l

Environmental Fate

trans-Dichloroethylene

Biodegradability	:	95 % Readily biodegradable.
Bioaccumulation	:	Bioaccumulation is unlikely.

1,1,1,2,2,3,4,5,5,5-Decafluoropentane

Biodegradability	:	Not readily biodegradable.
Bioaccumulation	:	Bioaccumulation is unlikely.

SECTION 13. DISPOSAL CONSIDERATIONS

Waste Disposal	:	Can be used after re-conditioning. If recycling is not practicable, dispose of in compliance with local regulations. The product should not be allowed to enter drains, water courses or the soil.
Environmental Hazards	:	If recycling is not practicable, dispose of in compliance with local regulations.

SECTION 14. TRANSPORT INFORMATION

Not classified as dangerous in the meaning of transport regulations.
DOT only - when shipped in packages with > 2640 lbs., use: UN3082, Environmentally Hazardous Substance, Liquid, N.O.S. (Dichloroethylene), 9, PGIII RQ (Dichloroethylene)
Not classified as dangerous in the meaning of transport regulations.

SECTION 15. REGULATORY INFORMATION

TSCA : 1,1,1,2,2,3,4,5,5,5-DECAFLUOROPENTANE (CAS# 138495-42-8) is controlled by TSCA Section 5, Significant New Use Rule (SNUR; 40 CFR 721.5645) The approved uses are: precision and general cleaning, carrier fluid, displacement drying, printed circuit board cleaning, particulate removal and film cleaning, process medium, heat transfer fluid (dielectric and non-dielectric), and test fluid. Processors and users of this substance must also comply with the applicable general SNUR requirements set forth in 40 CFR 721 subpart A, including export notification requirements if applicable (40 CFR 721.20), and the applicable record keeping requirements set forth at 40 CFR 721.125.

SARA 313 Regulated Chemical(s) : trans-Dichloroethylene

CERCLA Reportable Quantity : 2,639 lbs
Based on the percentage composition of this chemical in the product.: trans-Dichloroethylene

California Prop. 65 : Chemicals known to the State of California to cause cancer, birth defects or any other harm: none known

PA Right to Know Regulated Chemical(s) : Substances on the Pennsylvania Hazardous Substances List present at a concentration of 1% or more (0.01% for Special Hazardous Substances): trans-Dichloroethylene

NJ Right to Know Regulated Chemical(s) : Substances on the New Jersey Workplace Hazardous Substance List present at a concentration of 1% or more (0.1% for substances identified as carcinogens, mutagens or teratogens): trans-Dichloroethylene

SECTION 16. OTHER INFORMATION

Before use read DuPont's safety information.
For further information contact the local DuPont office or DuPont's nominated distributors.
DuPont™ Vertrel® are registered trademarks or trademarks of E. I. du Pont de Nemours and Company or its affiliates.

Contact person : MSDS Coordinator, DuPont Chemicals and Fluoroproducts, Wilmington, DE 19898, (800) 441-7515

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

Significant change from previous version is denoted with a double bar.

Return to App B   Return to Pg 9   Return to Pg 36

Solstice™ PF Spray Cleaner

000000017627

Version 1.1   Revision Date 04/23/2013   Print Date 04/25/2013

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name : Solstice™ PF Spray Cleaner

MSDS Number : 000000017627

Product Use Description : Solvent

Company : Honeywell International, Inc.
101 Columbia Road
Morristown, NJ 07962-1057

For more information call : 800-522-8001
+1-973-455-6300
(Monday-Friday, 9:00am-5:00pm)

In case of emergency call : **Medical: 1-800-498-5701 or +1-303-389-1414**
: **Transportation (CHEMTREC): 1-800-424-9300 or +1-703-527-3887**
:
: (24 hours/day, 7 days/week)

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

Form : Liquefied gas

Color : clear and colourless

Odor : slight

Hazard Summary : This product is not flammable at ambient temperatures and atmospheric pressure. Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing. Causes asphyxiation in high concentrations. The victim will not realize that he/she is suffocating. Excessive exposure may cause central nervous system effects including drowsiness and dizziness. Excessive exposure may also cause cardiac arrhythmia. May cause eye irritation. Do not breathe vapours or spray mist. Avoid contact with skin, eyes and clothing. At higher temperatures, (>250°C), decomposition products may include hydrochloric acid (HCl), hydrofluoric acid (HF) and carbonyl halides. The ACGIH Threshold Limit Values (2007) for Hydrogen Fluoride are TLV-TWA 0.5 ppm and Ceiling Exposure Limit 2 ppm.

Potential Health Effects

Skin : Not classified as a skin irritant in animal testing.

Eyes : May cause eye irritation.

Ingestion : Unlikely route of exposure.
Effects due to ingestion may include:
Gastrointestinal discomfort

Inhalation : Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Causes asphyxiation in high concentrations. The victim will not realize that he/she is suffocating.
Excessive exposure may cause central nervous system effects including drowsiness and dizziness. Excessive exposure may also cause cardiac arrhythmia.

Chronic Exposure : None known.

Carcinogenicity

No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP, IARC, or OSHA.

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Chemical Name	CAS-No.	Concentration
trans-1-Chloro-3,3,3-trifluoropropene	102687-65-0	80.00 %
trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9	20.00 %

SECTION 4. FIRST AID MEASURES

Inhalation	: Remove to fresh air. If breathing is irregular or stopped, administer artificial respiration. Use oxygen as required, provided a qualified operator is present. Call a physician.
Skin contact	: Rapid evaporation of the liquid may cause frostbite. If there is evidence of frostbite, bathe (do not rub) with lukewarm (not hot) water. If water is not available, cover with a clean, soft cloth or similar covering. Call a physician if irritation develops or persists.
Eye contact	: Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. In case of frostbite water should be lukewarm, not hot. If symptoms persist, call a physician.
Ingestion	: Unlikely route of exposure. As this product is a gas, refer to the inhalation section. Do not induce vomiting without medical advice. Call a physician immediately.

Notes to physician

Treatment	: Treat frost-bitten areas as needed.

SECTION 5. FIREFIGHTING MEASURES

Suitable extinguishing media	: Use extinguishing measures that are appropriate to local circumstances and the surrounding environment. Water mist Dry powder Foam Carbon dioxide (CO2)
Specific hazards during firefighting	: This product is not flammable at ambient temperatures and atmospheric pressure. However, this material can ignite when mixed with air under pressure and exposed to strong ignition sources. Container may rupture on heating. Cool closed containers exposed to fire with water spray. Do not allow run-off from fire fighting to enter drains or water courses.

Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Exposure to decomposition products may be a hazard to health.
In case of fire hazardous decomposition products may be produced such as:
Hydrogen fluoride
Gaseous hydrogen chloride (HCl).
Carbon monoxide
Carbon dioxide (CO2)
Carbonyl halides

Special protective equipment for firefighters	: In the event of fire and/or explosion do not breathe fumes. Wear self-contained breathing apparatus and protective suit. No unprotected exposed skin areas. Exposure to decomposition products may be a hazard to health.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Personal precautions	: Immediately evacuate personnel to safe areas. Keep people away from and upwind of spill/leak. Wear personal protective equipment. Unprotected persons must be kept away. Remove all sources of ignition. Avoid skin contact with leaking liquid (danger of frostbite). Ventilate the area. After release, disperses into the air. Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing. Avoid accumulation of vapours in low areas. Unprotected personnel should not return until air has been tested and determined safe.
Environmental precautions	: Prevent further leakage or spillage if safe to do so. The product evaporates readily. Prevent spreading over a wide area (e.g. by containment or oil barriers).
Methods for cleaning up	: Do not direct water spray at the point of leakage. Allow to evaporate.

SECTION 7. HANDLING AND STORAGE

Handling

Handling	: Handle with care. Do not breathe vapours or spray mist. Avoid contact with skin, eyes and clothing. Wear personal protective equipment. Pressurized container. Protect from sunlight and do not expose to temperatures exceeding 50 °C. Follow all standard safety precautions for handling and use of compressed gas cylinders. Use authorized cylinders only. Protect cylinders from physical damage. Do not puncture or drop cylinders, expose them to open flame or excessive heat. Do not pierce or burn, even after use. Do not spray on a naked flame or any incandescent material. Do not remove screw cap until immediately ready for use. Always replace cap after use.
Advice on protection against fire and explosion	: Do not spray on a naked flame or any incandescent material. Keep away from direct sunlight. Fire or intense heat may cause violent rupture of packages. Vapours may form explosive mixtures with air. The product is not easily combustible.

Storage

Requirements for storage areas and containers	: Keep containers tightly closed in a cool, well-ventilated place. Keep away from direct sunlight. Protect cylinders from physical damage. Store away from incompatible substances.
Further information on storage conditions	: Keep only in the original container at temperature not exceeding 50°C
Advice on common storage	: Do not store together with: Oxidizing agents

SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Protective measures	:	Ensure that eyewash stations and safety showers are close to the workstation location.
Engineering measures	:	Local exhaust
Eye protection	:	Goggles
Hand protection	:	Protective gloves
Skin and body protection	:	impervious clothing Wear cold insulating gloves/ face shield/ eye protection.
Respiratory protection	:	In case of insufficient ventilation wear suitable respiratory equipment. Wear a positive-pressure supplied-air respirator.
Hygiene measures	:	Keep working clothes separately. Do not smoke. Do not breathe vapours or spray mist. Avoid contact with skin, eyes and clothing.

Exposure Guidelines

Components	CAS-No.	Value	Control parameters	Update	Basis
trans-1-Chloro-3,3,3-trifluoropropene	102687-65-0	TWA : time weighted average	(800 ppm)	2013	Honeywell:Limit established by Honeywell International Inc.
trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9	TWA : time weighted average	(800 ppm)	2012	WEEL:US. AIHA Workplace Environmental Exposure Level (WEEL) Guides

trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9	TWA : time weighted average	(800 ppm)	31.03.11	Honeywell:Limit established by Honeywell International Inc.

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Physical state	: Liquefied gas
Color	: clear and colourless
Odor	: slight
pH	: Note: not applicable
Boiling point/boiling range	: Note: not determined
Flash point	: Note: does not flash
Lower explosion limit	: Note: no data available
Upper explosion limit	: Note: no data available
Vapor pressure	: 234 kPa at 30 °C(86 °F)
Vapor density	: Note: not determined, (Air = 1.0)
Density	: Note: not determined
Water solubility	: Note: not determined

Ignition temperature	: Note: no data available
Decomposition temperature	: Note: Hazardous decomposition products formed under fire conditions., To avoid thermal decomposition, do not overheat.

SECTION 10. STABILITY AND REACTIVITY

Chemical stability	: Stable under normal conditions.
Possibility of hazardous reactions	: Hazardous polymerisation does not occur.
Conditions to avoid	: Pressurized container. Protect from sunlight and do not expose to temperatures exceeding 50 °C. Can form a combustible mixture with air at pressures above atmospheric pressure. Do not mix with oxygen or air above atmospheric pressure.
Incompatible materials to avoid	: Reactions with alkali metals.
Hazardous decomposition products	: In case of fire hazardous decomposition products may be produced such as: Carbon monoxide Carbon dioxide (CO2) Carbonyl halides Gaseous hydrogen chloride (HCl). Gaseous hydrogen fluoride (HF).

SECTION 11. TOXICOLOGICAL INFORMATION

Acute inhalation toxicity

trans-1-Chloro-3,3,3-trifluoropropene	: LC50: 120000 ppm Exposure time: 4 h Species: rat
trans-1,3,3,3-Tetrafluoroprop-1-ene	: 100000 ppm Species: mouse Note: Acute (4-Hour) Inhalation Toxicity Screening Study

(mouse): No lethality at >100,000 ppm.

LC50: > 207000 ppm
Exposure time: 4 h
Species: rat

Skin irritation

trans-1-Chloro-3,3,3-trifluoropropene : Species: rabbit
Result: No skin irritation
Classification: Not classified as a skin irritant in animal testing.
Method: OECD Test Guideline 404
Exposure time: 4 h

trans-1,3,3,3-Tetrafluoroprop-1-ene : Species: rabbit
Result: No skin irritation
Method: OECD Test Guideline 404

Sensitisation

trans-1-Chloro-3,3,3-trifluoropropene : Cardiac sensitization
Species: dogs
Note: Cardiac sensitisation threshold (dog): 25000 ppm.

Result: Does not cause skin sensitization.
Classification: Patch test on human volunteers did not demonstrate sensitization properties.

trans-1,3,3,3-Tetrafluoroprop-1-ene : Cardiac sensitization
Species: dogs
Note: Did not cause sensitization on laboratory animals.

Repeated dose toxicity

trans-1-Chloro-3,3,3-trifluoropropene : Species: rat
Application Route: Inhalation
Exposure time: (4 Weeks)
NOEL: 4500 ppm
Subacute toxicity

trans-1,3,3,3-Tetrafluoroprop-1-ene : Species: rat
Application Route: Inhalation
Exposure time: (13 Weeks)
NOEL: 5000 ppm
Causes mild effects on the heart.

Genotoxicity in vitro

trans-1-Chloro-3,3,3-trifluoropropene	: Test Method: Mutagenicity (Salmonella typhimurium - reverse mutation assay) Result: negative
trans-1,3,3,3-Tetrafluoroprop-1-ene	: Test Method: Chromosome aberration test in vitro Cell type: Human lymphocytes Result: negative
	: Test Method: Ames test Result: negative

Genotoxicity in vivo

trans-1-Chloro-3,3,3-trifluoropropene	: Species: rat Cell type: Bone marrow Method: Mutagenicity (micronucleus test) Result: negative
	: Test Method: Unscheduled DNA synthesis Species: rat Result: negative
	: Species: mouse Cell type: Bone marrow Method: Mutagenicity (micronucleus test) Result: negative
trans-1,3,3,3-Tetrafluoroprop-1-ene	: Test Method: Mutagenicity (in vivo mammalian bone-marrow cytogenetic test, chromosomal analysis) Species: mouse Cell type: Micronucleus Application Route: Inhalation Result: negative

Reproductive toxicity

trans-1-Chloro-3,3,3-trifluoropropene	: Species: rabbit Note: No-observed-effect level - 15,000 ppm Species: rat Note: No-observed-effect level - 10,000 ppm

Teratogenicity

trans-1-Chloro-3,3,3-trifluoropropene	: Species: rabbit Note: No-observed-effect level - 15,000 ppm Species: rat Note: No-observed-effect level - 10,000 ppm
trans-1,3,3,3-Tetrafluoroprop-1-ene	: Species: rabbit Method: Prenatal Developmental Inhalation Toxicity Study Note: Did not show teratogenic effects in animal experiments. Species: rat Method: Prenatal Developmental Inhalation Toxicity Study Note: Did not show teratogenic effects in animal experiments.

SECTION 12. ECOLOGICAL INFORMATION

Toxicity to fish

trans-1-Chloro-3,3,3-trifluoropropene	: LC50: 38 mg/l Exposure time: 96 h Species: Oncorhynchus mykiss (rainbow trout) Method: OECD Test Guideline 203
trans-1,3,3,3-Tetrafluoroprop-1-ene	: NOEC: > 117 mg/l Exposure time: 96 h Species: Cyprinus carpio (Carp)

Toxicity to daphnia and other aquatic invertebrates

trans-1-Chloro-3,3,3-trifluoropropene	: Immobilization EC50: 82 mg/l Exposure time: 48 h Species: Daphnia magna (Water flea) Method: OECD Test Guideline 202
trans-1,3,3,3-Tetrafluoroprop-1-ene	: EC50: > 160 mg/l Exposure time: 48 h Species: Daphnia magna (Water flea)

Toxicity to algae

trans-1-Chloro-3,3,3-trifluoropropene	:	Growth inhibition EC50: 106.7 mg/l Exposure time: 72 h Species: Pseudokirchneriella subcapitata (green algae) Method: OECD Test Guideline 201
		Growth rate NOEC: 115 mg/l Exposure time: 72 h Species: Pseudokirchneriella subcapitata (green algae) Method: OECD Test Guideline 201
trans-1,3,3,3-Tetrafluoroprop-1-ene	:	Growth inhibition NOEC: > 170 mg/l Exposure time: 72 h Species: Algae

Biodegradability

trans-1-Chloro-3,3,3-trifluoropropene	:	Result: Not readily biodegradable. Value: 0 % Method: OECD 301 D
trans-1,3,3,3-Tetrafluoroprop-1-ene	:	aerobic Result: Not readily biodegradable.

Further information on ecology

SECTION 13. DISPOSAL CONSIDERATIONS

Disposal methods : Observe all Federal, State, and Local Environmental regulations.

SECTION 14. TRANSPORT INFORMATION

DOT	UN/ID No.	:	UN 3163
	Proper shipping name	:	LIQUEFIED GAS, N.O.S. (Trans-1-Chloro-3,3,3-trifluoropropene, trans-1,3,3,3-Tetrafluoroprop-1-ene)

	Class	2.2
	Packing group	
	Hazard Labels	2.2
IATA	UN/ID No.	: UN 3163
	Description of the goods	: LIQUEFIED GAS, N.O.S. (Trans-1-Chloro-3,3,3-trifluoropropene, trans-1,3,3,3-Tetrafluoroprop-1-ene)
	Class	: 2.2
	Hazard Labels	: 2.2
	Packing instruction (cargo aircraft)	: 200
	Packing instruction (passenger aircraft)	: 200
IMDG	UN/ID No.	: UN 3163
	Description of the goods	: LIQUEFIED GAS, N.O.S. (TRANS-1-CHLORO-3,3,3-TRIFLUOROPROPENE, TRANS-1,3,3,3-TETRAFLUOROPROP-1-ENE)
	Class	: 2.2
	Hazard Labels	: 2.2
	EmS Number	: F-C, S-V
	Marine Pollutant	: no

SECTION 15. REGULATORY INFORMATION

Inventories

US. Toxic Substances Control Act	: On TSCA Inventory
Australia. Industrial Chemical (Notification and Assessment) Act	: Not in compliance with the inventory
Canada. Canadian Environmental Protection Act (CEPA). Domestic Substances List (DSL)	: This product contains one or several components that are not on the Canadian DSL nor NDSL.
Japan. Kashin-Hou Law	: On the inventory, or in compliance with the inventory

List

Korea. Toxic Chemical Control Law (TCCL) List : On the inventory, or in compliance with the inventory

Philippines. The Toxic Substances and Hazardous and Nuclear Waste Control Act : Not in compliance with the inventory

China. Inventory of Existing Chemical Substances : Not in compliance with the inventory

New Zealand. Inventory of Chemicals (NZIoC), as published by ERMA New Zealand : Not in compliance with the inventory

National regulatory information

SARA 302 Components : SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components : SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards : Acute Health Hazard
Sudden Release of Pressure Hazard

California Prop. 65 : This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

WHMIS Classification : A: A: Compressed gas
This product has been classified according to the hazard criteria of the CPR and the MSDS contains all of the information required by the CPR.

SECTION 16. OTHER INFORMATION

		HMIS III	NFPA
Health hazard	:	1	2
Flammability	:	0	0
Physical Hazard	:	0	
Instability	:		0

Hazard rating and rating systems (e.g. HMIS® III, NFPA): This information is intended solely for the use of individuals trained in the particular system.

Further information

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text. Final determination of suitability of any material is the sole responsibility of the user. This information should not constitute a guarantee for any specific product properties.

Changes since the last version are highlighted in the margin. This version replaces all previous versions.
Previous Issue Date: 04/02/2013
Prepared by: Honeywell Performance Materials and Technologies Product Stewardship Group

Return to App B
Return to Pg 36

Material Safety Data Sheet

Honeywell

Solstice™ 1234ze

000000012546

Version 2 Revision Date 04/25/2012 Print Date 11/12/2013

SECTION 1. PRODUCT AND COMPANY IDENTIFICATION

Product name : Solstice™ 1234ze

MSDS Number : 000000012546

Product Use Description : Aerosol propellant, Foam blowing agent, Refrigerant

Company : Honeywell International, Inc.
101 Columbia Road
Morristown, NJ 07962-1057

For more information call : 800-522-8001
(Monday-Friday, 9:00am-5:00pm)

In case of emergency call : **Medical (PROSAR): 1-800-498-5701 or +1-651-523-0309**
: **Transportation (CHEMTREC): 1-800-424-9300 or +1-703-527-3887**
:
: (24 hours/day, 7 days/week)

SECTION 2. HAZARDS IDENTIFICATION

Emergency Overview

Form : Liquefied gas

Color : colourless

Odor : slight ether-like

Hazard Summary : Warning! Container under pressure. Gas reduces oxygen available for breathing. Causes asphyxiation in high concentrations. The victim will not realize that he/she is suffocating. Rapid evaporation of the liquid may cause frostbite. Excessive exposure may cause central nervous system effects including drowsiness and dizziness. Excessive exposure may also cause cardiac arrhythmia. May cause eye irritation.

Potential Health Effects

Skin : No skin irritation

Eyes : May cause eye irritation.

Ingestion : Unlikely route of exposure.

Inhalation : Excessive exposure may cause central nervous system effects including drowsiness and dizziness. Excessive exposure may also cause cardiac arrhythmia.

Chronic Exposure : None known.

Carcinogenicity

No component of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP, IARC, or OSHA.

SECTION 3. COMPOSITION/INFORMATION ON INGREDIENTS

Chemical nature : Substance

Chemical Name	CAS-No.	Concentration
trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9	100.00%

SECTION 4. FIRST AID MEASURES

Inhalation : Remove to fresh air. If breathing is irregular or stopped, administer artificial respiration. Use oxygen as required, provided a qualified operator is present. Call a physician.

Skin contact : Rapid evaporation of the liquid may cause frostbite. If there is evidence of frostbite, bathe (do not rub) with lukewarm (not hot) water. If water is not available, cover with a clean, soft cloth or similar covering. Call a physician if irritation develops or persists.

Eye contact : Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. In case of frostbite water should be lukewarm, not hot. If symptoms persist, call a physician.

Ingestion : Unlikely route of exposure. As this product is a gas, refer to the inhalation section. Do not induce vomiting without medical advice. Call a physician immediately.

Notes to physician

Treatment : Treat frost-bitten areas as needed.

SECTION 5. FIREFIGHTING MEASURES

Suitable extinguishing media : Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

Water mist
Dry powder
Foam
Carbon dioxide (CO_2)

Specific hazards during firefighting : Contents under pressure.
Heating will cause pressure rise with risk of bursting
Cool closed containers exposed to fire with water spray.
Product is not combustible under normal conditions.
However, this material can ignite when mixed with air under pressure and exposed to strong ignition sources.
Do not allow run-off from fire fighting to enter drains or water courses.
Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Some risk may be expected of corrosive and toxic decomposition products.
Fire may cause evolution of:
Hydrogen fluoride

Special protective equipment for firefighters : In the event of fire and/or explosion do not breathe fumes.
Wear self-contained breathing apparatus and protective suit.
No unprotected exposed skin areas.
Exposure to decomposition products may be a hazard to health.

SECTION 6. ACCIDENTAL RELEASE MEASURES

Personal precautions : Immediately evacuate personnel to safe areas.
Keep people away from and upwind of spill/leak.
Wear personal protective equipment. Unprotected persons must be kept away.
Remove all sources of ignition.
Avoid skin contact with leaking liquid (danger of frostbite).
Ventilate the area.
After release, disperses into the air.
Vapours are heavier than air and can cause suffocation by reducing oxygen available for breathing.
Avoid accumulation of vapours in low areas.
Unprotected personnel should not return until air has been tested and determined safe.

Environmental precautions : Prevent further leakage or spillage if safe to do so.
The product evaporates readily.
Prevent spreading over a wide area (e.g. by containment or oil barriers).

Methods for cleaning up : Do not direct water spray at the point of leakage.

Allow to evaporate.

SECTION 7. HANDLING AND STORAGE

Handling

Handling	:	Handle with care. Avoid inhalation of vapour or mist. Do not get in eyes, on skin, or on clothing. Wear personal protective equipment. Pressurized container. Protect from sunlight and do not expose to temperatures exceeding 50 °C. Follow all standard safety precautions for handling and use of compressed gas cylinders. Use authorized cylinders only. Protect cylinders from physical damage. Do not puncture or drop cylinders, expose them to open flame or excessive heat. Do not pierce or burn, even after use. Do not spray on a naked flame or any incandescent material. Do not remove screw cap until immediately ready for use. Always replace cap after use.
Advice on protection against fire and explosion	:	Do not spray on a naked flame or any incandescent material. Keep away from direct sunlight. Fire or intense heat may cause violent rupture of packages. Vapours may form explosive mixtures with air. The product is not easily combustible.

Storage

Requirements for storage areas and containers	:	Keep containers tightly closed in a cool, well-ventilated place. Keep away from direct sunlight.
Further information on storage conditions	:	Keep only in the original container at temperature not exceeding 50°C
Advice on common storage	:	Do not store together with: Oxidizing agents

SECTION 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Protective measures	:	Do not breathe vapour. Avoid contact with skin, eyes and clothing. Ensure that eyewash stations and safety showers are close to

the workstation location.

Engineering measures : Local exhaust

Eye protection : Goggles

Hand protection : Protective gloves

Skin and body protection : impervious clothing
Wear cold insulating gloves/ face shield/ eye protection.

Respiratory protection : In case of insufficient ventilation wear suitable respiratory equipment.
Wear a positive-pressure supplied-air respirator.

Hygiene measures : Avoid breathing vapors, mist or gas.
Keep working clothes separately.
Do not smoke.

Exposure Guidelines–

Components	CAS-No.	Value	Control parameters	Update	Basis
trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9	TWA : time weighted average	(800 ppm)	31.03.11	Honeywell:Limit established by Honeywell International Inc.
		TWA : time weighted average	(800 ppm)	2009	WEEL:US. Workplace Environmental Exposure Level (WEEL) Guides

SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES

Physical state : Liquefied gas

Colour : colourless

Odour : slight ether-like

Boiling point/boiling range : -19 °C

Flash point	:	Note: does not flash
Lower explosion limit	:	Note: Exhibits flame limits at temperatures in excess of 28˚ C.
Upper explosion limit	:	Note: Exhibits flame limits at temperatures in excess of 28˚ C.
Vapour pressure	:	4,192 hPa at 20 °C(68 °F) 10,998 hPa at 54.4 °C(129.9 °F)
Vapour density	:	4 Note: (Air = 1.0)
Density	:	1.17 g/cm3 at 21.1 °C
Water solubility	:	0.373 g/l
Partition coefficient: n-octanol/water	:	log Pow: 1.6
Ignition temperature	:	368 °C Method: Autoignition temperature
Decomposition temperature	:	Note: Hazardous decomposition products formed under fire conditions., To avoid thermal decomposition, do not overheat.

SECTION 10. STABILITY AND REACTIVITY

Conditions to avoid	:	Pressurized container. Protect from sunlight and do not expose to temperatures exceeding 50 °C. Can form a combustible mixture with air at pressures above atmospheric pressure. Do not mix with oxygen or air above atmospheric pressure.

Materials to avoid	: Reactions with alkali metals.
Hazardous decomposition products	: Pyrolysis products containing fluoride Fluorocarbons Hydrogen fluoride
Thermal decomposition	: Hazardous decomposition products formed under fire conditions., To avoid thermal decomposition, do not overheat.
Hazardous reactions	: Hazardous polymerisation does not occur. Stable under normal conditions.

SECTION 11. TOXICOLOGICAL INFORMATION

Acute inhalation toxicity	: Species: mouse Note: Acute (4-Hour) Inhalation Toxicity Screening Study (mouse): No lethality at >100,000 ppm.
	: LC50: > 207000 ppm Exposure time: 4 h Species: rat
Skin irritation	: Species: rabbit Result: No skin irritation Method: OECD Test Guideline 404
Sensitisation	: Cardiac sensitization Species: dogs Result: Did not cause sensitization on laboratory animals.
Repeated dose toxicity	: Species: rat Application Route: Inhalation Exposure time: 13 Weeks Note: Causes mild effects on the heart. NOEL 5,000 ppm
Genotoxicity in vitro	: Test Method: Chromosome aberration test in vitro Cell type: Human lymphocytes Result: negative
	: Test Method: Ames test Result: negative
Genotoxicity in vivo	: Test Method: Mutagenicity (in vivo mammalian bone-marrow cytogenetic test, chromosomal analysis)

Species: mouse
Cell type: Micronucleus
Application Route: Inhalation
Result: negative

Teratogenicity : Species: rabbit
Method: Prenatal Developmental Inhalation Toxicity Study
Note: Did not show teratogenic effects in animal experiments.

: Species: rat
Method: Prenatal Developmental Inhalation Toxicity Study
Note: Did not show teratogenic effects in animal experiments.

SECTION 12. ECOLOGICAL INFORMATION

Ecotoxicity effects

Toxicity to fish : NOEC: > 117 mg/l
Exposure time: 96 h
Species: Cyprinus carpio (Carp)

Toxicity to daphnia and other aquatic invertebrates. : EC50: > 160 mg/l
Exposure time: 48 h
Species: Daphnia magna (Water flea)

Toxicity to algae : Growth inhibition
NOEC: > 170 mg/l
Exposure time: 72 h
Species: Algae

Elimination information (persistence and degradability)

Biodegradability : aerobic
Result: Not readily biodegradable.

Further information on ecology

Additional ecological information : no data available

SECTION 13. DISPOSAL CONSIDERATIONS

Disposal methods : Observe all Federal, State, and Local Environmental regulations.

SECTION 14. TRANSPORT INFORMATION

DOT	UN/ID No.	:	UN 3163
	Proper shipping name	:	LIQUEFIED GAS, N.O.S. (trans-1,3,3,3-Tetrafluoroprop-1-ene)
	Class		2.2
	Packing group		
	Hazard Labels		2.2
IATA	UN/ID No.	:	UN 3163
	Description of the goods	:	LIQUEFIED GAS, N.O.S. (trans-1,3,3,3-Tetrafluoroprop-1-ene)
	Class	:	2.2
	Hazard Labels	:	2.2
	Packing instruction (cargo aircraft)	:	200
	Packing instruction (passenger aircraft)	:	200
IMDG	UN/ID No.	:	UN 3163
	Description of the goods	:	LIQUEFIED GAS, N.O.S. (TRANS-1,3,3,3-TETRAFLUOROPROP-1-ENE)
	Class	:	2.2
	Hazard Labels	:	2.2
	EmS Number	:	F-C
	Marine pollutant	:	no

SECTION 15. REGULATORY INFORMATION

Inventories

US. Toxic Substances Control Act	:	On TSCA Inventory	
Australia. Industrial Chemical (Notification and Assessment) Act	:	Not in compliance with the inventory	
	:	trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9
Canada. Canadian Environmental Protection Act (CEPA). Domestic Substances List (DSL)	:	All components of this product are on the Canadian DSL list.	
Japan. Kashin-Hou Law	:	On the inventory, or in compliance with the inventory	

List

Korea. Existing Chemicals Inventory (KECI)	:	Not in compliance with the inventory	
	:	trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9
Philippines. The Toxic Substances and Hazardous and Nuclear Waste Control Act	:	Not in compliance with the inventory	
	:	trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9
China. Inventory of Existing Chemical Substances	:	On the inventory, or in compliance with the inventory	
New Zealand. Inventory of Chemicals (NZIoC), as published by ERMA New Zealand	:	Not in compliance with the inventory	
	:	trans-1,3,3,3-Tetrafluoroprop-1-ene	29118-24-9

National regulatory information

SARA 302 Components : SARA 302: No chemicals in this material are subject to the reporting requirements of SARA Title III, Section 302.

SARA 313 Components : SARA 313: This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

SARA 311/312 Hazards : Acute Health Hazard
Sudden Release of Pressure Hazard

California Prop. 65 : This product does not contain any chemicals known to State of California to cause cancer, birth defects, or any other reproductive harm.

WHMIS Classification	: A: Compressed Gas This product has been classified according to the hazard criteria of the CPR and the MSDS contains all of the information required by the CPR.

SECTION 16. OTHER INFORMATION

		HMIS III	**NFPA**
Health hazard	:	1	2
Flammability	:	0	0
Physical Hazard	:	0	
Instability	:		0

Hazard rating and rating systems (e.g. HMIS® III, NFPA): This information is intended solely for the use of individuals trained in the particular system.

Further information

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text. Final determination of suitability of any material is the sole responsibility of the user. This information should not constitute a guarantee for any specific product properties.

Changes since the last version are highlighted in the margin. This version replaces all previous versions.
Previous Issue Date: 11/10/2011
Prepared by: Honeywell Performance Materials and Technologies Product Stewardship Group

Return to Menu

Return to Pg 11

APPENDIX C—TEST REPORTS FOR NONVOLATILE RESIDUE BACKGROUND IN SOLVENTS

The following test reports for NVR background in solvents can be found in sections C.1 and C.2:

C.1 SSC—Gravimetric and FTIR NVR Analysis of the Candidate Solvents
C.2 MSFC—NVR Analysis of Solvent Study Samples (EM10 memo 013-605)

Return to App C

Gravimetric and FTIR NVR Analysis of the Candidate Solvents (per 200ml)

Solvent	Lot #	Initial (g)	Pan + NVR (g)	Grav. NVR (mg)	FTIR NVR (mg)
HCFC225cb	91207300	2.3289	2.3289	<0.1	0.12
Solstice PF	BB103A	2.3359	2.3362	0.3	0.19
3M L-14780	No Lot # Bottle dated 2/3/2011	2.3099	2.3100	0.1	0.09
Vertrel MCA	CT13AG092	2.3520	2.3535	1.5	1.97
Solvokane	No Lot # Drum dated 08/01/01	2.3348	2.3390	4.2	3.4
Solkane	16136	2.2781	2.2787	0.6	0.32
Capstone 4-I	18	2.2780	2.3740	96.0	1.34*

* Capstone 4I contains high levels of inorganic components.

Capstone 4I (PFBI) Issues

PFBI After 4 hrs Exposure to Light

PFBI - Immediately Poured from an Amber Bottle

Photo reactivity

Liberates iodine when exposed to light

Stability / Corrosion

Reacts with iron, steel and aluminum alloys. Product purity varies from lot to lot.

Contamination

High particulate levels are generated after exposure to iron / steel. Filtered particulate matter draws moisture in from the atmosphere.

Limited Availability

OEM has shown no commercial interest to produce PFBI as a precision cleaning solvent

High Cost

>4X than HCFC225

Particulate Matter Collected from Capstone 4I (PFBI), Lot #18

Heavy Silting Observed in Unused Capstone 4I rec'd from USAF / WPAB

SSC Lab 8/30/2013

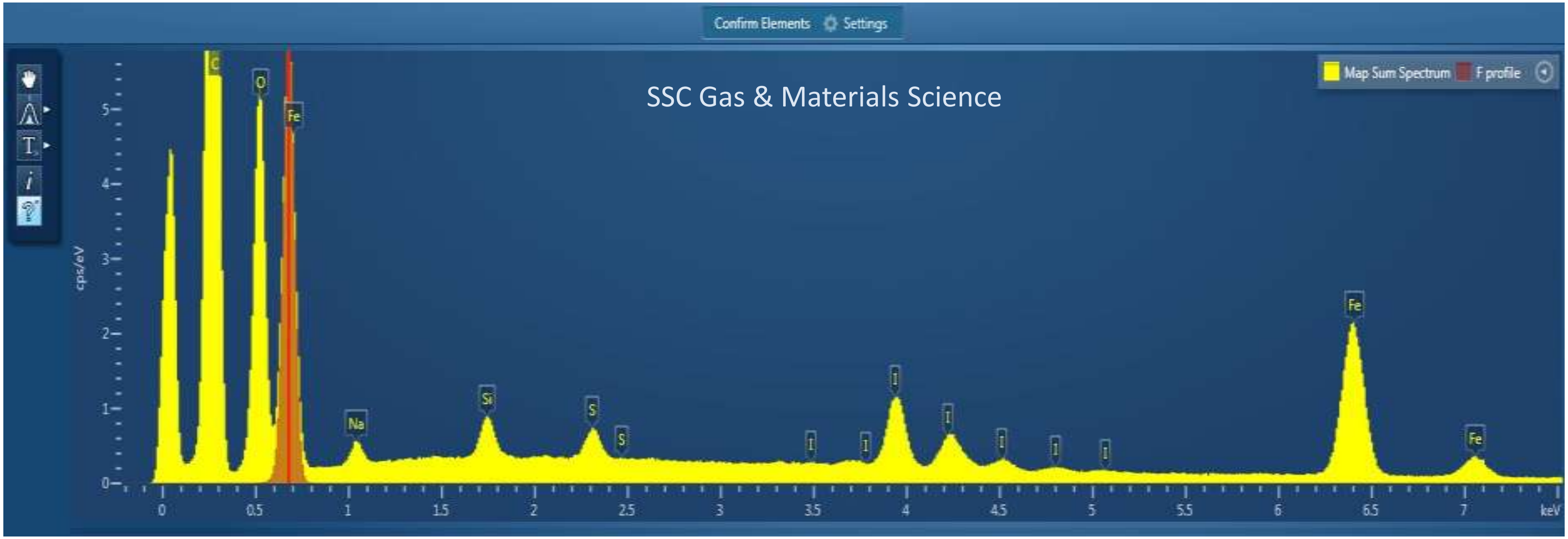
Filtrate Mass After Filtering 100mL Capstone 4I (PFBI)Solvent

DuPont

Initial Weight of Filter (mg)	**73.4 mg**
Weight after filtration of 100mL Capstone 4-I (mg)	77.5mg
Total weight of filtered matter (mg)	4.1mg

Filtered 100ml of Capstone 4I through a 47 mm dia. filter with a 0.45 micron pore size.

X-Ray Spectrum of the PFBI Particulate Matter from Lot #18



Per email response from Raymond Davenport /Dupont, “Lot 18 has a lot of iron in your tests which suggests that it was probably stored in mild steel drums which caused some corrosion prior to it being re-packaged into amber bottles. It is not a high purity lot and the corrosion could have been accelerated by an impurity. The pink / violet / purple color change is to be expected and is unavoidable and is generated by the liberated iodine on exposure to light.”

Lab Observations: Glassware is difficult to clean - heavy stains, may lightly etch the glassware under the right conditions. Sodium and Silicon in the X-Ray spectrum suggest PFBI is leaching these elements from the glass.

Filtered NVR associated with Capstone 4I from Lot 18

Forms moisture on the filter pad as it sits in the lab.

Return to App C

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

September 6, 2013

Reply to Attn of: EM10 (013-605)

TO: EM50/Mark Mitchell

FROM: EM10/Gail H. Gordon

SUBJECT: NVR Analysis of Solvent Study Samples

The nonvolatile residue analyses on the solvent study samples that were furnished by EM50 have been completed. Results of the analyses are enclosed.

If there are questions, please contact Bobby Graves at 544-3072.

Gail H Gordon
Gail H. Gordon
Branch Chief
Materials Test Branch

Enclosure

cc:
EM10/S. Androlake
EM10/E. Davis
EM10/File copy
EM50/ESSSA/N. Lowrey
EM10/METTS/B. Graves

AK-225 G Control

Date of analysis- 7/25/2013
NVR- <=0.1mg/ 200ml.
pH- 6.76
H2O bath temp.- start 60 deg.C end 60 deg.C
condenser H2O temp.- room temp.
distillation time- on- 8:30 off- 10:00
oven temp.- 60 deg.C
distillate recovery- ~160ml.

Solstice PF

Date of analysis- 7/26/2013
NVR- 0.3mg/ 200ml.
pH- 7.0-7.5
H2O bath temp.- 21 deg. C
condenser H2O temp.- room temp.
distillation time- times not recorded
oven temp.- room temp.
distillate recovery- 0 ml. **Note 1**

Note 1: No chilled H2O running through condenser coils.

3M L- 14780

Date of analysis- 8/28/13
NVR- 4.20 mg/ 200ml.
pH- 7.0-7.5
H2O bath temp.- start - 29 deg.C end- 31 deg.C
condenser H2O temp.- start- 19.6 deg.C end- 10 deg.C
distillation time- on- 7:10 off- 9:08
oven temp.- start- 105 +- 5 deg.C **Note 1 & Note 2**
distillate recovery- ~150ml.

Note 1: After the sample was placed in the oven, the oven temp. jumped to 120 deg.C. The oven door was opened to bring the temp. back to 105 deg. Shortly after the door was closed, the oven temp. shot back up to 120 deg. After the sample was removed from the oven, the oven temp. dropped back down to 105 deg.

Note 2: A vacuum pump was attached to the oven and the oven temp. remained at a constant 100 deg through the evaporation process for the remainder of the solvents.

Capstone 4-1

Date of analysis- 8/28/2013
NVR- 6.40 mg/ 200ml.
pH- ~5.5 **Note 1**
H2O bath temp.- start - 68 deg.C end- 71 deg.C
condenser H2O temp.- start- 10 deg.C end- 10 deg.C
distillation time- on- 11:20 off- 1:05
oven temp.- start- 100 +- 5 deg.C end 100 deg.C +- 5 deg.
distillate recovery- ~150ml.

Note 1: The solvent was transferred from an oxidized container to a glass bottle before I received it.

Vertrel MCA

Date of analysis- 8/29/2013
NVR- 2.80 mg/ 200ml.
pH- 7.0-7.5
H2O bath temp.- start - 41 deg.C end- 55 deg.C **Note 1**:
condenser H2O temp.- start- 10 deg.C end- 10 deg.C
distillation time- on- 7:20 off- 9:25
oven temp.- start- 100 +- 5 deg.C end 100 deg.C +- 5 deg.
distillate recovery- ~160ml.

Note 1: At 45 deg.C, the condensation stopped with a good portion of the sample still remaining The temp. was raised to 48 deg.C, condensation resumed, and then stopped again with ~70 ml. remaining. The temp. was raised 1 deg. at a time and at 55 deg.C, the sample had finally evaporated down to the required 10-20 ml. prescribed by the procedure.

Solkane

Date of analysis- 8/29/2013
NVR- 4.30 mg/ 200ml.
pH- 6.32
H2O bath temp.- start - 42 deg.C end- 43 deg.C
condenser H2O temp.- start- 10 deg.C end- 10 deg.C
distillation time- on- 12:35 off- 1:20
oven temp.- start- 100 +- 5 deg.C end 100 deg.C +- 5 deg.
distillate recovery- ~150ml.

Solvokane

Date of analysis- 9/3/2013
NVR- 4.60 mg/ 200ml.
pH- 6.89
H2O bath temp.- start - 36 deg.C end- 38 deg.C
condenser H2O temp.- start- 10 deg.C end- 10 deg.C
distillation time- on- 8:45 off- 11:47
oven temp.- start- 100 +- 5 deg.C end 100 deg.C +- 5 deg.
distillate recovery- ~150ml.

Equipment Used

Buchi Rotvapor R-205
Buchi heating bath B-490
Fisher Scientific Vacuum Oven 281-A
A & D Analytical Balance HR-200 with a detection of +- 0.1mg.
Millipore LWSP Teflon filter pads, 47mm., porosity of 5u

Variances from test plan

50 ml. NVR beakers were used for the AK-225 G control sample and the Solstice PF sample. Due to the amount of time it took to get constant weights on the beakers, 30 ml.NVR beakers were used on the rest of the solvents except the Vertrel MCA due to observation found in **Note 1** under the above Vertrel MCA data. The 30ml. NVR beakers enabled quicker, constant weights. As stated under Solstice PF, **Note 1** above, there was no condensation into distillate probably due to the fact that I did not have a water chiller at that time. A water chiller was obtained and used in the testing of the remaining sovents. There was a reaction observed on the filter pad while filtering the Solstice PF. Further test on the pad, (weight loss difference) showed no difference in pre and post weights of the pad. A vacuum pump was attached to the oven and used on all the solvents tested following the 3ML-14780 due to the anomaly observed in **Note 1** under 3ML-14780. The Capstone 4-1 had a deep purple appearance and after filtration filtration of the solvent, the filter pad was silted.

The above solvents were analyzed per 3.1.1-3.1.2.8 of the Solvent Study Replacement Test except for those listed under **Variances from test plan** stated above.

Bobby Graves/ Metts in support of EM 10, MSFC.

Return to Menu

Return to Pg 12

APPENDIX D—QUICK SCREEN SOLVENCY TEST RESULTS

Appendix D contains the SSC Solvent Screening Test Results, excerpt from: HCFC225cb (AK225g) Replacement Study, 1st Technical Interchange Meeting, September 19–20, 2013, Updated December 2013.

SSC Solvent Screening Test Results

Excerpt from:

HCFC225cb (AK225g) Replacement Study
1st Technical Interchange Meeting
Sept. 19-20, 2013/Updated Dec. 2013

Bruce Farner - NASA/SSC RA 20

H. Richard Ross - A2R / SSC Gas & Materials Science

Outline

- Lab Results of Solvent Screening Tests for NVR Removal (Cleaning Efficiency)
- Candidate Solvents Tested
  - Neat Solvents
    - AGC Chemicals HCFC225 cb (AK225g) Control Solvent
    - Honeywell Solstice PF (HFO 1233zd)
    - Solvay Solkane (HFC-365)
    - Dupont Capstone 4I (Perfluorobutyl iodide (PFBI) or Nonafluoro-1-iodobutane)
  - Azeotropes
    - 3M L-14780 (22%tDCE/78% HFE-347mcc3)
    - Solvay Solvokane (30% tDCE/70% HFC-365mfc)
    - Dupont Vertrel MCA (38%tDCE/62%HFE-43-10mee)
- NVR Miscibility / Saturation Tests
- Solvent NVR Data

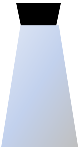
SSC Replacement Study Team

- NASA /SSC
  - Bruce Farner, NASA Project Lead

- SSC Lab (Quick Screen Cleaning Tests and Doping Flex Hoses for the Field Cleaning Demonstration with Honeywell using Solstice PF)
  - H. Richard Ross , Solvent Test Lead /A2R
  - Taylor Davie / A2R - Chemist
  - Eric Guttierrez / A2R - Chemist
  - Marry Kerschbaum / A2R - Chemist

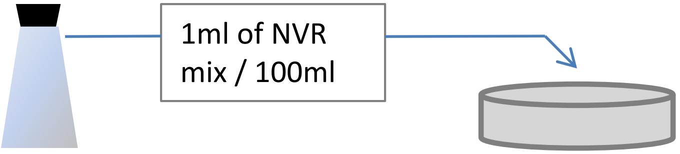
Quick Screening Test Details

- Triple rinse with HCFC225cb, ten aluminum weighing dishes (pans) per solvent to be tested
- Next, immerse the pans in a beaker filled with HCFC225cb for a min. of two hrs.
- Place the aluminum pans in a preheated (100 deg. C) oven for one hour.
- Prepare a batch of mixed contaminants by dissolving 0.5240g of equal parts of the following contaminants in100ml of HCFC225cb.
  - 0.1023g of Mineral Oil – pharmaceutical grade – mixed aliphatic
  - 0.1018g of MIL-PRF-8382 – ester based hydraulic fluid
  - 0.1025g of Di-2-ethylhexylsebacate (gauge calibration oil), Monoplex DOS
  - 0.1112g of WD 40 (medium-heavy hydrocarbons)
  - 0.1062g of Krytox GPL103 (fluorocarbon lubricant for oxygen systems)



0.5240 g. NVR
mix / 100ml

- Remove the pans from the oven and place in a desiccator for a min. of one hour.
- Weigh each dish on a calibrated balance accurate to ± 0.1mg. This weight is reported as the "clean pan" in the results table. Apply 1 ml of the mixed NVR std. to each aluminum weighing dish.



1ml of NVR
mix / 100ml

NVR Transferred to
the Al Weighing Pan

- Return the pans to the oven and bake for one hr. at 100 deg. C. Transfer pans to the desiccator and cool for 1 hr. and reweigh each pan. This is reported as the "Pan + NVR " in the results table.

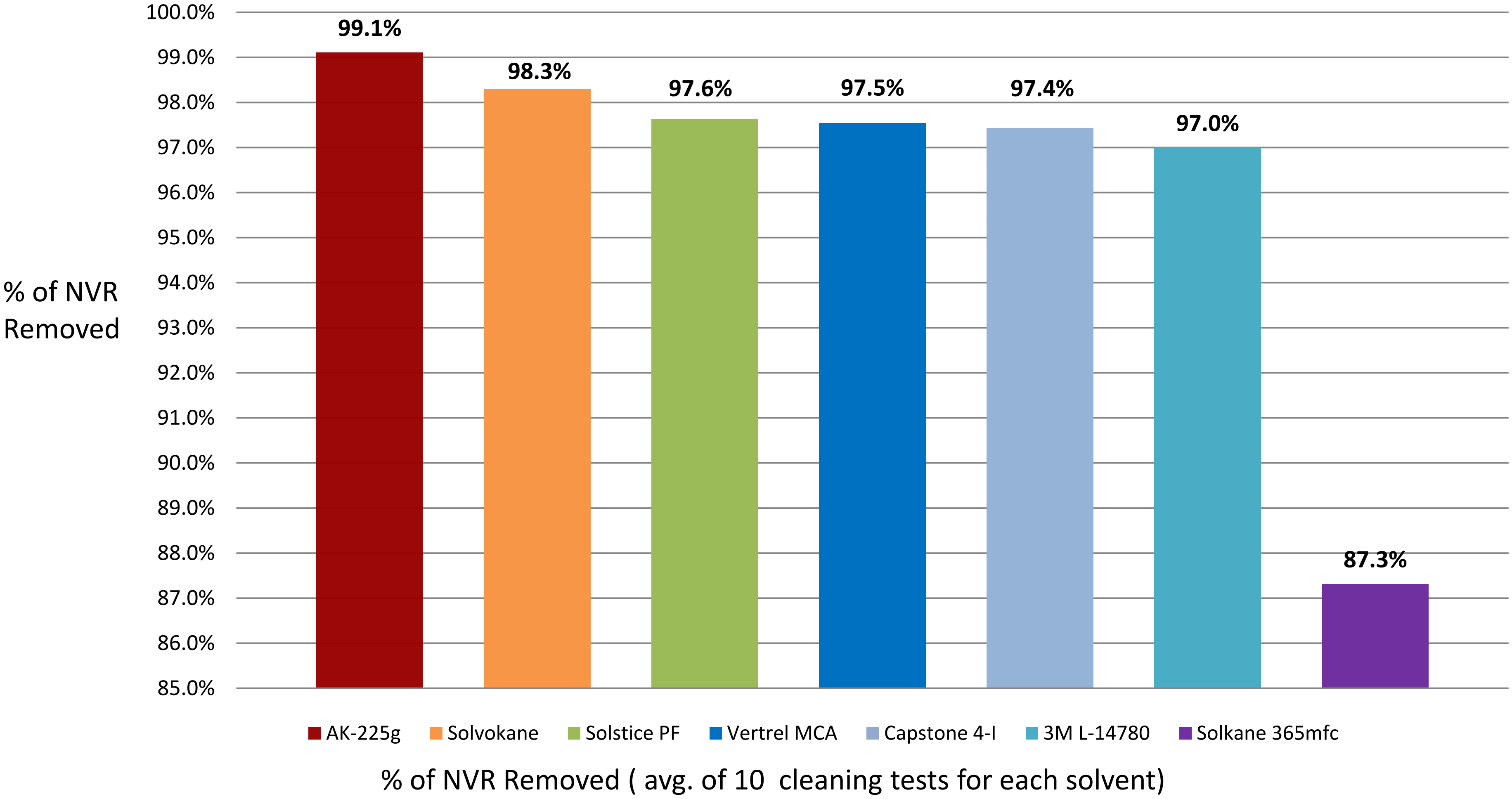
Quick Screen Test Details (Continuation)

- The mass difference between the "Pan + NVR" and "Clean Pan" is the "Initial Pan NVR" (i.e., NVR added to the pan).
  - Note. Each pan was doped with 5 mg; however, only 3.3mg (avg. for 10 pans doped with NVR for each test solvent) remained after the 1 hr. bakeout at 100C.
  - Pan surface area = 5.31in^2 w/3.4mg of NVR is ≈ 92.2mg / ft ^{2}.
- Flush each NVR doped weighing pan with 100ml of the test solvent from a glass volumetric cylinder in three steps, capturing all of the flush solvent in a clean beaker.
  - With the NVR doped dish on a flat surface, pour 30 ml of test solvent into the dish, swirl and decant into a beaker
  - Repeat the above step with an additional 30ml
  - Holding the dish, fold it slightly and slowly flush the remaining 40 ml of test solvent through each side of the fold, draining the solvent into the beaker such that the maximum surface area is exposed.
- Dry in the oven at 100 deg.C for 1 hr., cool in the desiccator for 1 hr. and reweigh. This is reported as "NVR Removed From the Pan, Post Solvent Flush".
- % Cleaning Efficiency = (mgs. of NVR Removed from the Pan After the Post Solvent Flush / Doped Pan NVR mgs.) (100)

Solvent Screening Test Results for Cleaning Efficiency

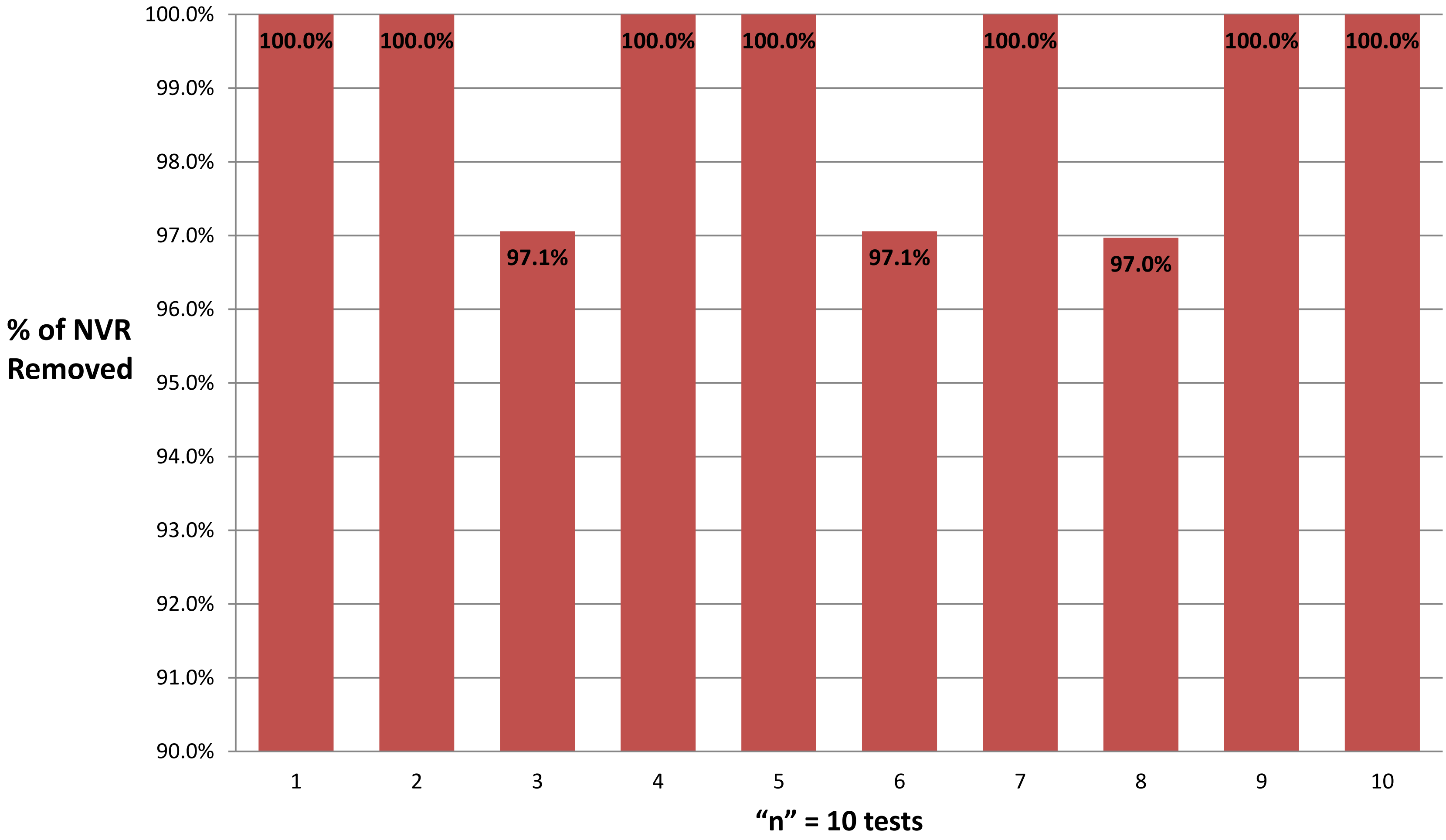
Avg. NVR Removal of Test Solvents

Contamination Level = 3.4 mg /5.31 in^2 ≈ 89.5mg / ft^2



Cleaning Efficiency Tests Using HCFC225cb (AK225g)
NVR Contamination Level = 3.3 mg /5.31 in^2 ≈ 89.5mg/ ft.^{2}

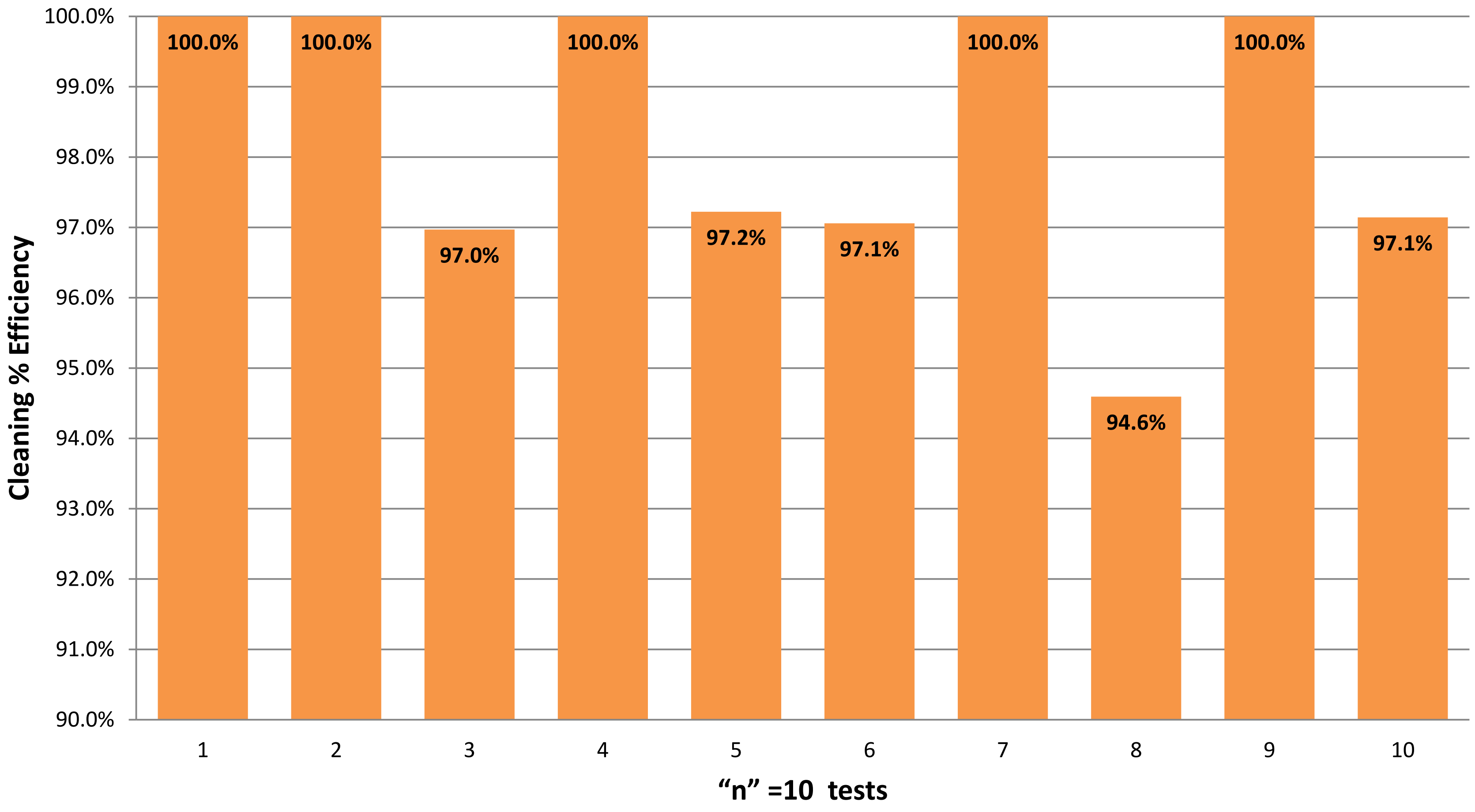
HCFC225cb (Control/Baseline Solvent)



Avg. NVR Removal Efficiency = 99.1 %
RSD = 1.45%

Cleaning Efficiency Tests Using Solvokane
NVR Contamination Level = 3.4 mg /5.31 in^2 ≈ 92.2 mg/ ft.^{2}

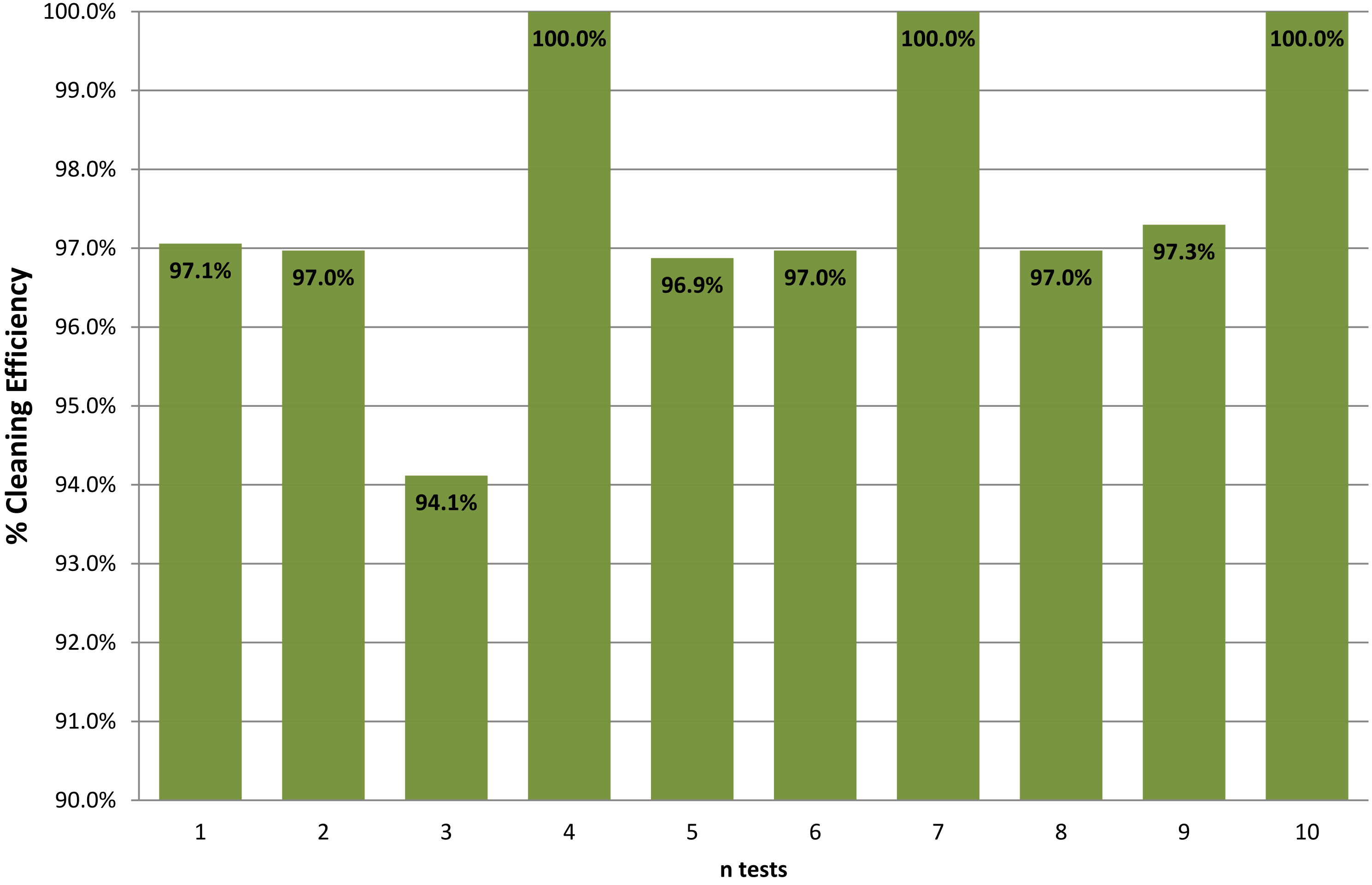
Solvay Solvokane



Avg. NVR Removal Efficiency = 98.3%
RSD = 1.98%

Cleaning Efficiency Tests Using Solstice PF
NVR Contamination Level = 3.4 mg /5.31 in^2 ≈ 92.2 mg / ft^2

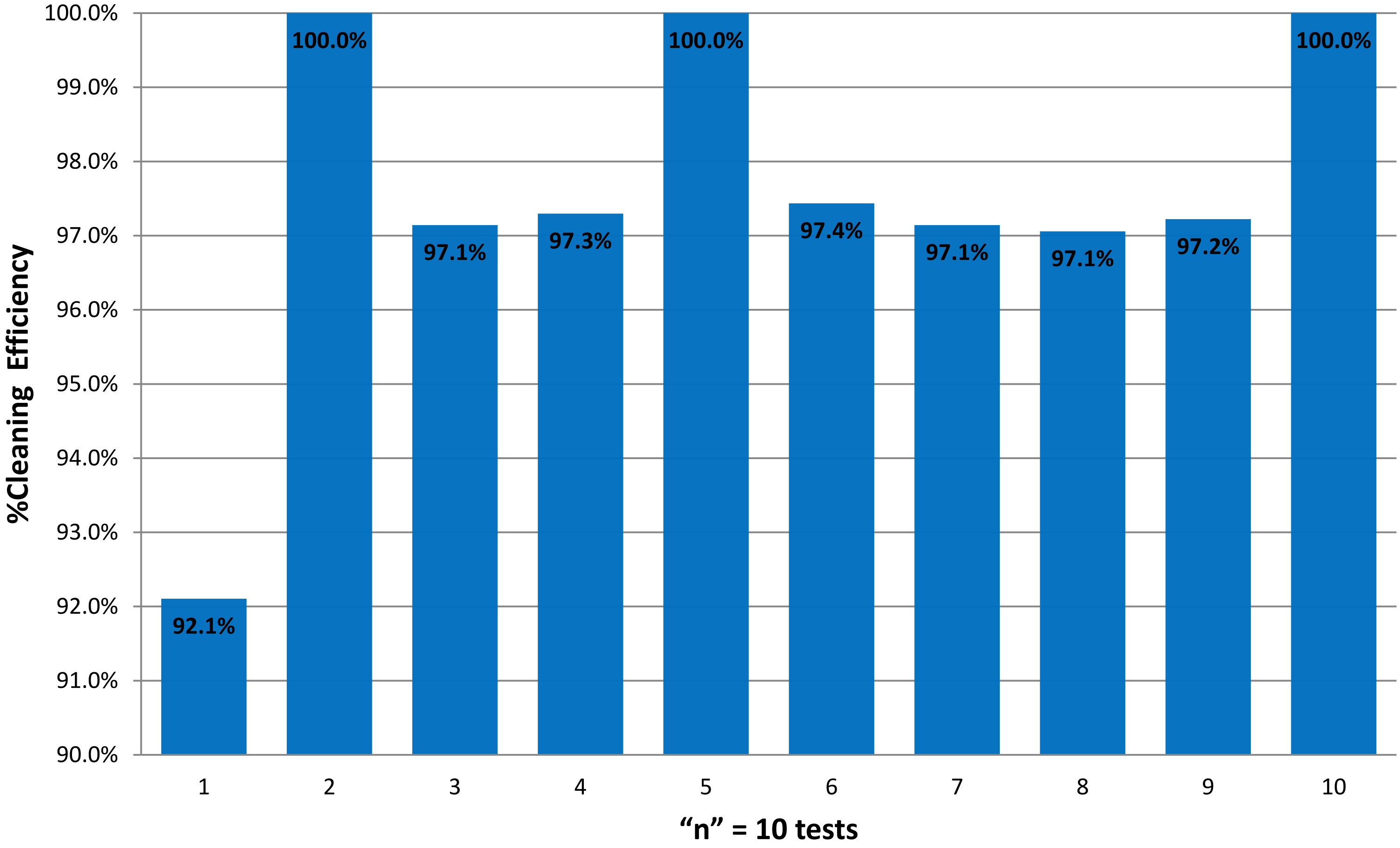
Honeywell Solstice PF



Avg. NVR Removal Efficiency = 97.6%
RSD = 1.92%

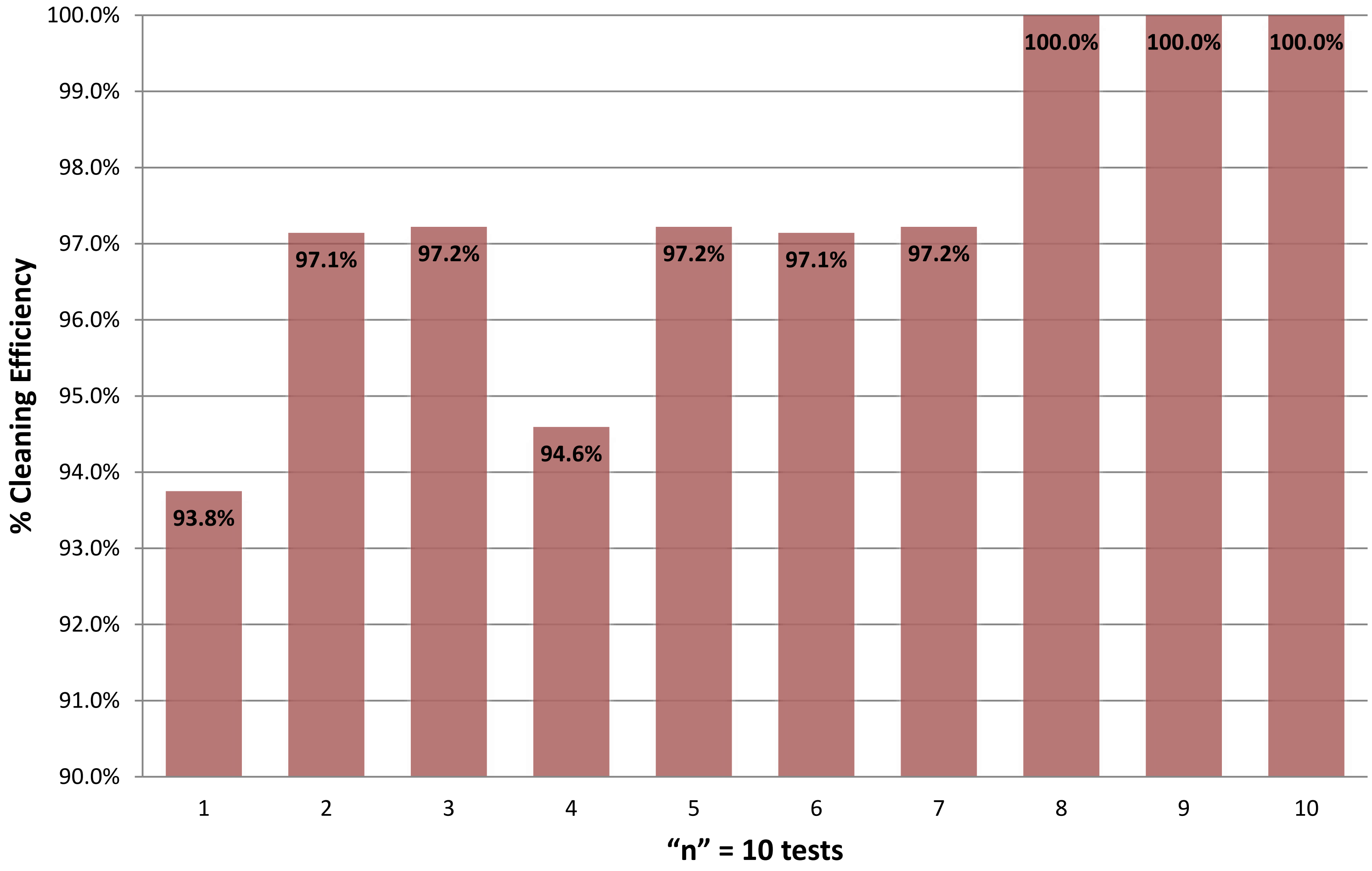
Cleaning Efficiency Tests Using Vertrel MCA
NVR Contamination Level = 3.5mg /5.31 $in^2 \approx$ 94.7mg / ft^2

DuPont Vertrel MCA



Avg. NVR Removal Efficiency = 97.5%
RSD = 2.38%

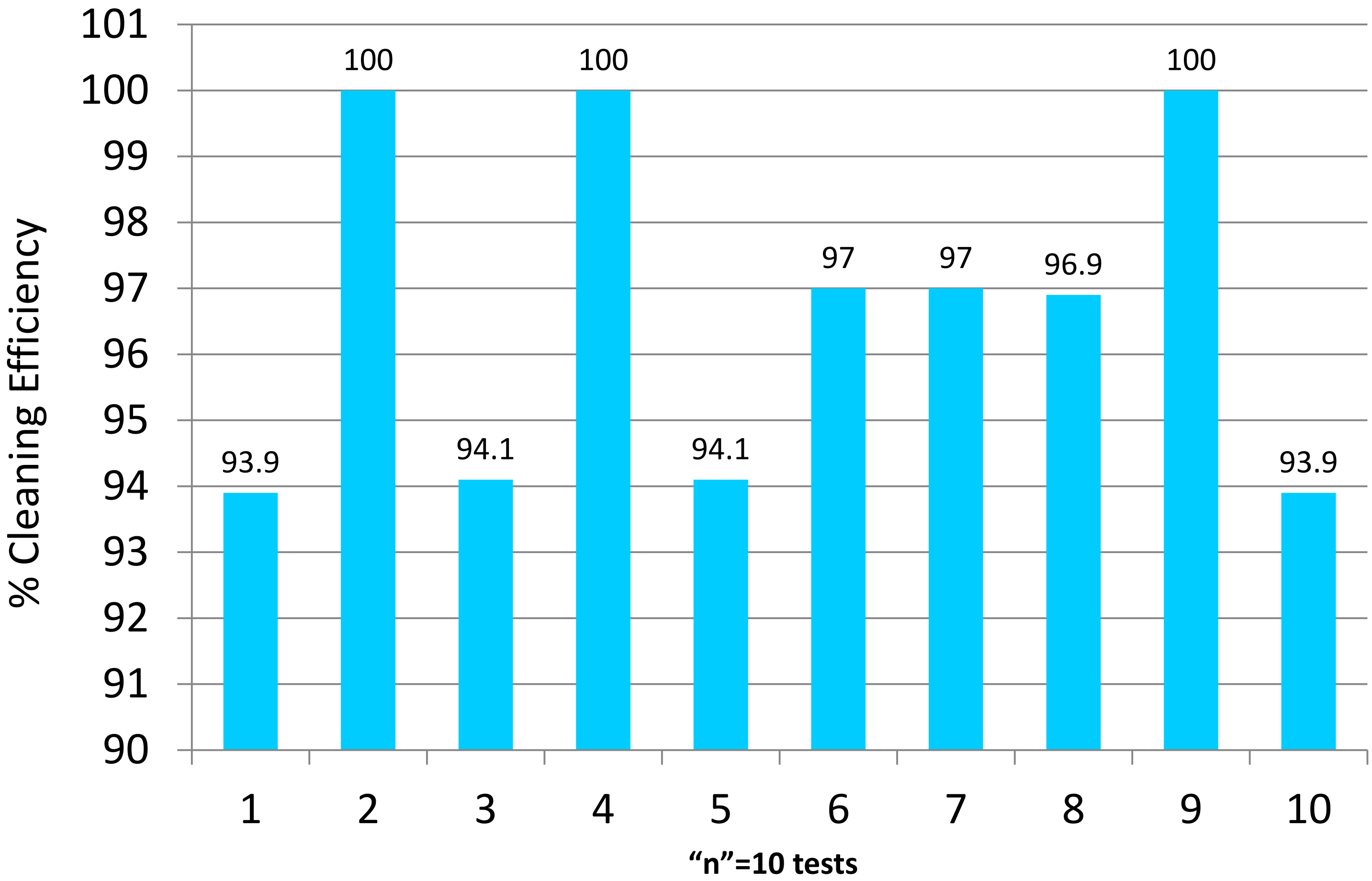
Cleaning Efficiency Tests Using Capstone 4-I
NVR Contamination Level = 3.3 mg /5.31 in^2 ≈ 89.5mg / ft^2
DuPont Capstone 4-I (PFBI)



Avg. NVR Removal Efficiency = 97.4%
RSD= 2.21%

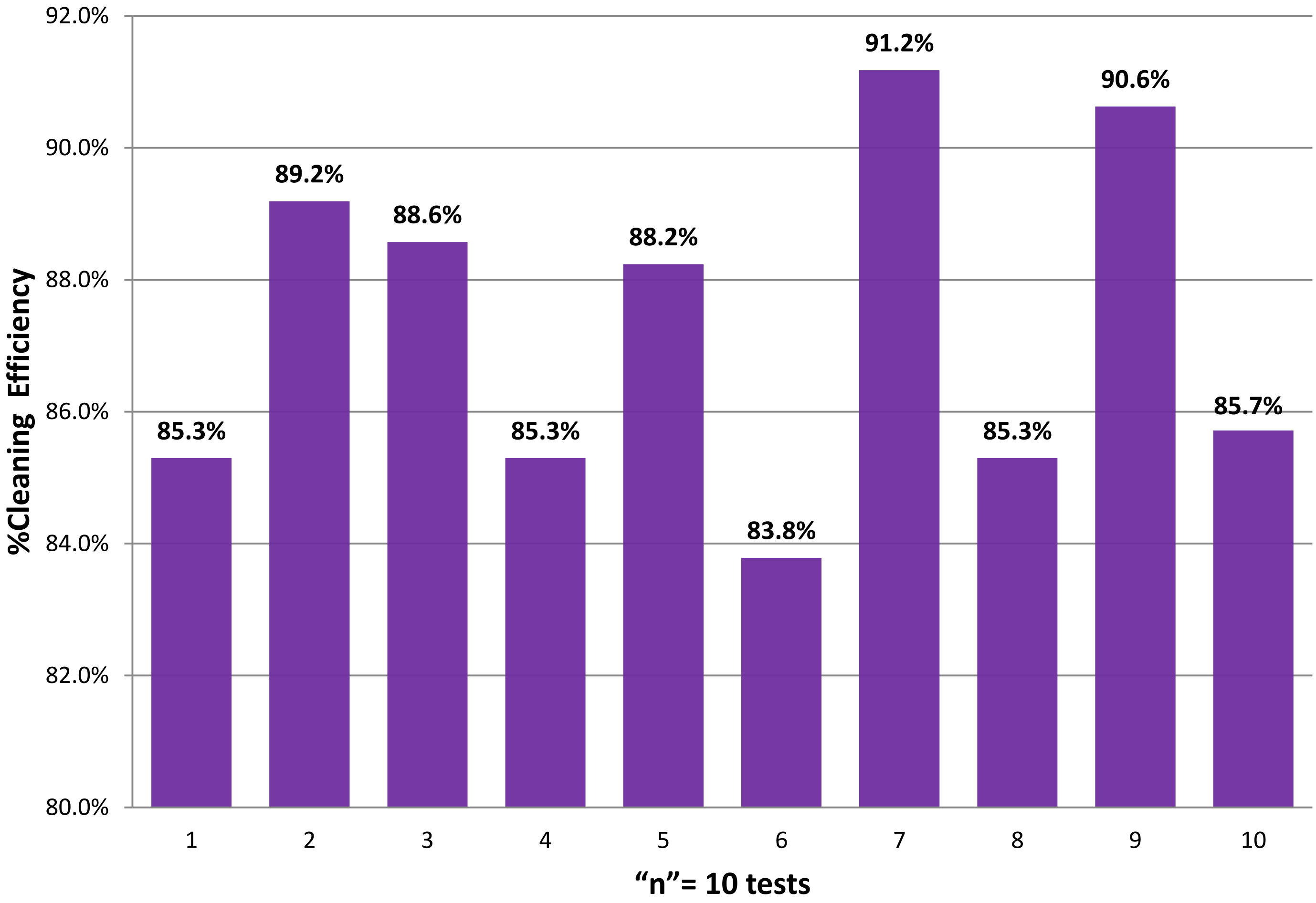
Cleaning Efficiency Tests Using L-14780
NVR Contamination Level = 3.2 mg /5.31 in^2 ≈ 86.8mg / ft^2

3M L-14780



Avg. NVR Removal Efficiency = 97.5%
RSD = 2.38%

Cleaning Efficiency Tests Using Solkane
NVR Contamination Level = 3.0 mg /5.31 in^2 ≈ 81.4mg / ft^2
Solvay Solkane



Solvay Solkane 365mfc

Avg. NVR Removal Efficiency = 87.3%
RSD = 2.93%

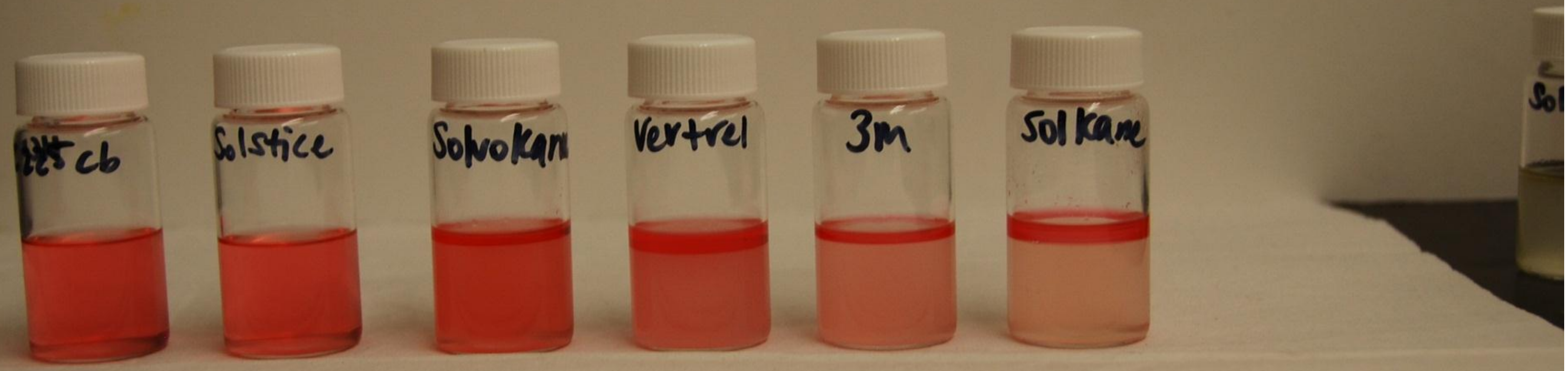
Miscibility /Saturation Tests of the Candidate Solvents

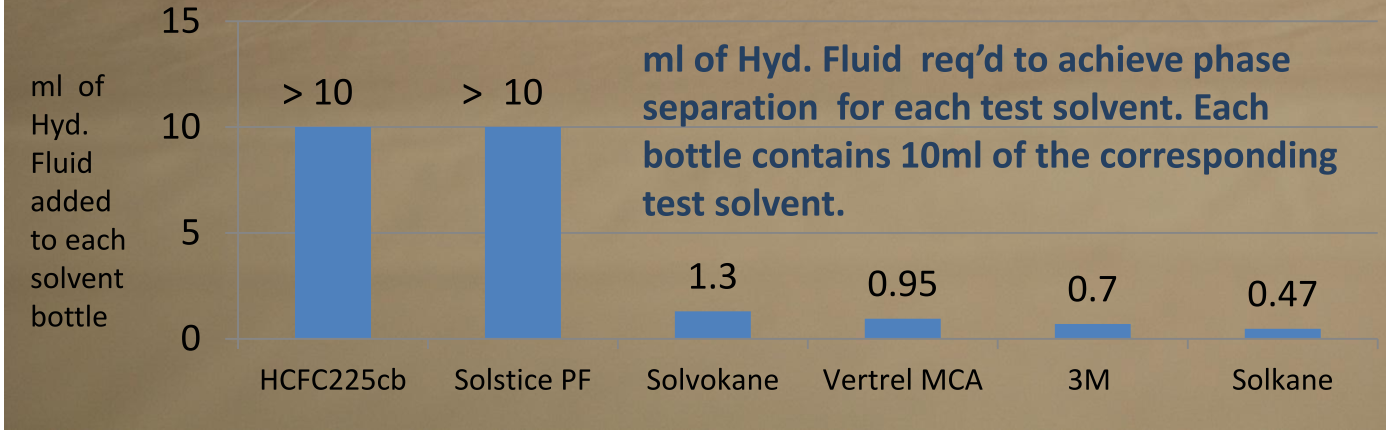
The solubility of a substance (e.g., hydraulic fluid) in a solvent can be measured as the saturation concentration or by the miscibility of the substance with the solvent.

NVR Miscibility /Saturation Tests for the Candidate Solvents

Milliliters of Hydraulic Fluid (NVR) required to achieve saturation (separation). Each bottle contains "10ml" of solvent.

MIL-PRF-8382 – ester based hydraulic fluid





ml of Hyd. Fluid req'd to achieve phase separation for each test solvent. Each bottle contains 10ml of the corresponding test solvent.

Note: The Hyd. Fluid was still soluble in AK225g & Solstice PF at equal volumes (1:1). Solstice PF surpassed the other candidate solvents.

NVR Miscibility Range of the Candidate Solvents

0.95ml of Hydraulic Fluid was added to each bottle containing 10ml of solvent)

MIL-PRF-8382 – ester based hydraulic fluid

Conc. for each bottle = 0.95ml Hyd. Fluid /10 ml Solvent

Vertrel MCA is partially miscible, the remaining three solvents are fully miscible (no stratification or phase separation were observed with the three remaining solvents).

NVR Miscibility Range of the Candidate Solvents

Next, 1.3 ml of Hydraulic Fluid was added to each bottle containing 10ml of solvent

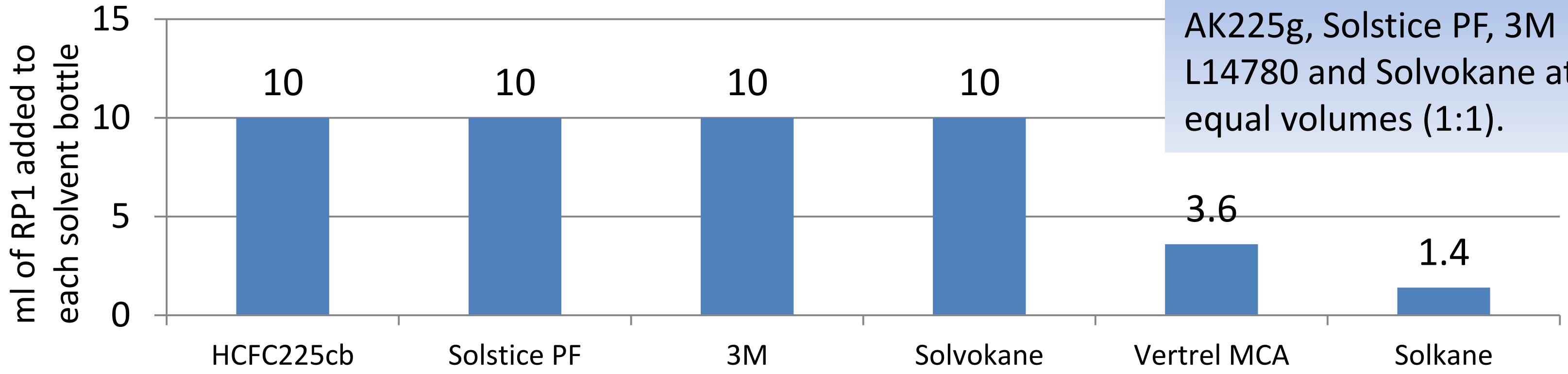
MIL-PRF-8382 – ester based hydraulic fluid

Conc. for each bottle = 1.3ml Hyd. Fluid /10 ml Solvent

Solvokane is partially miscible w/1.3ml of hydraulic fluid, the Solstice PF and the Control (AK225g) are fully miscible (no stratification or phase separation were observed).

NVR Miscibility /Saturation Tests for the Candidate Solvents

Vol of RP1 (NVR) required to achieve saturation (separation) is reported in the below graph. Each bottle contains 10ml of solvent. RP1 is predominantly C9-C16 (w/ sulfur < 30 ppm) & aromatics 5% Max.



RP1 was still soluble in AK225g, Solstice PF, 3M L14780 and Solvokane at equal volumes (1:1).

Miscibility /Saturation Response of the Candidate Solvents to Heavy Weight (85-140W) Gear Oil (Petroleum-Based)

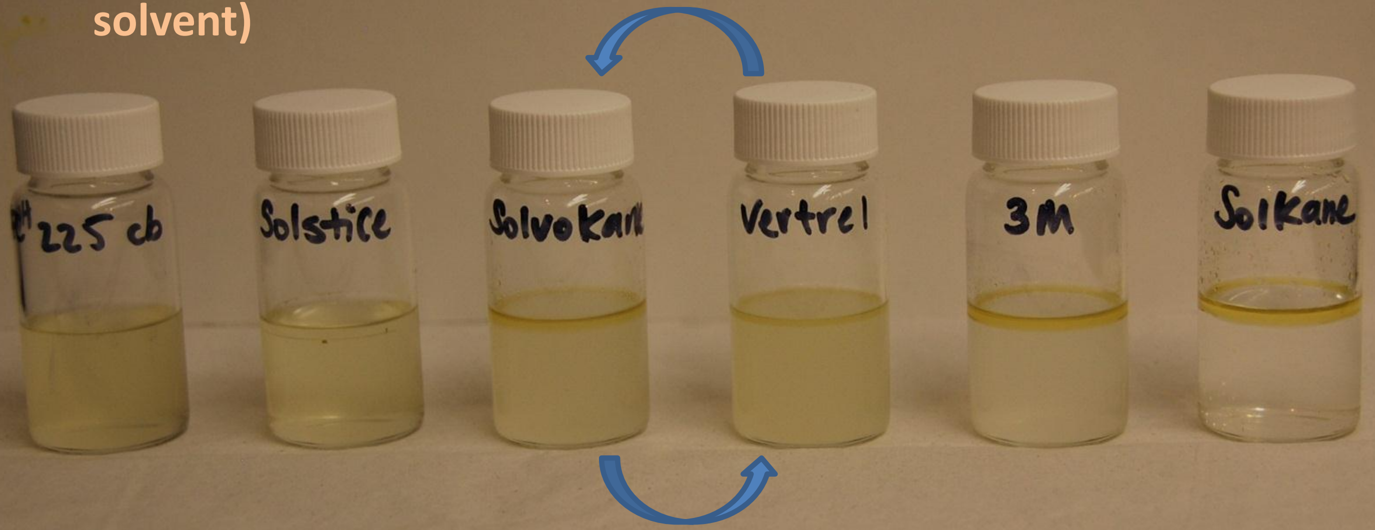
Six drops (150mg) of Gear Oil was added to each bottle (10ml solvent)

The Gear Oil was fully miscible in Solstice & HCFC225cb (AK225g)

3ML14780 & Solkane were the least miscible solvents

Miscibility Response of the Candidate Solvents to Heavy Weight (85-140W) Gear Oil (Petroleum-Based)

Six drops (150mg) of Gear Oil was added to each bottle (10ml solvent)



Solvokane and Vertrel MCA were placed out of order to highlight the miscibility / saturation rank

The Gear Oil was fully miscible in Solstice & HCFC225cb (AK225g) 3ML14780 & Solkane were the least effective solvents

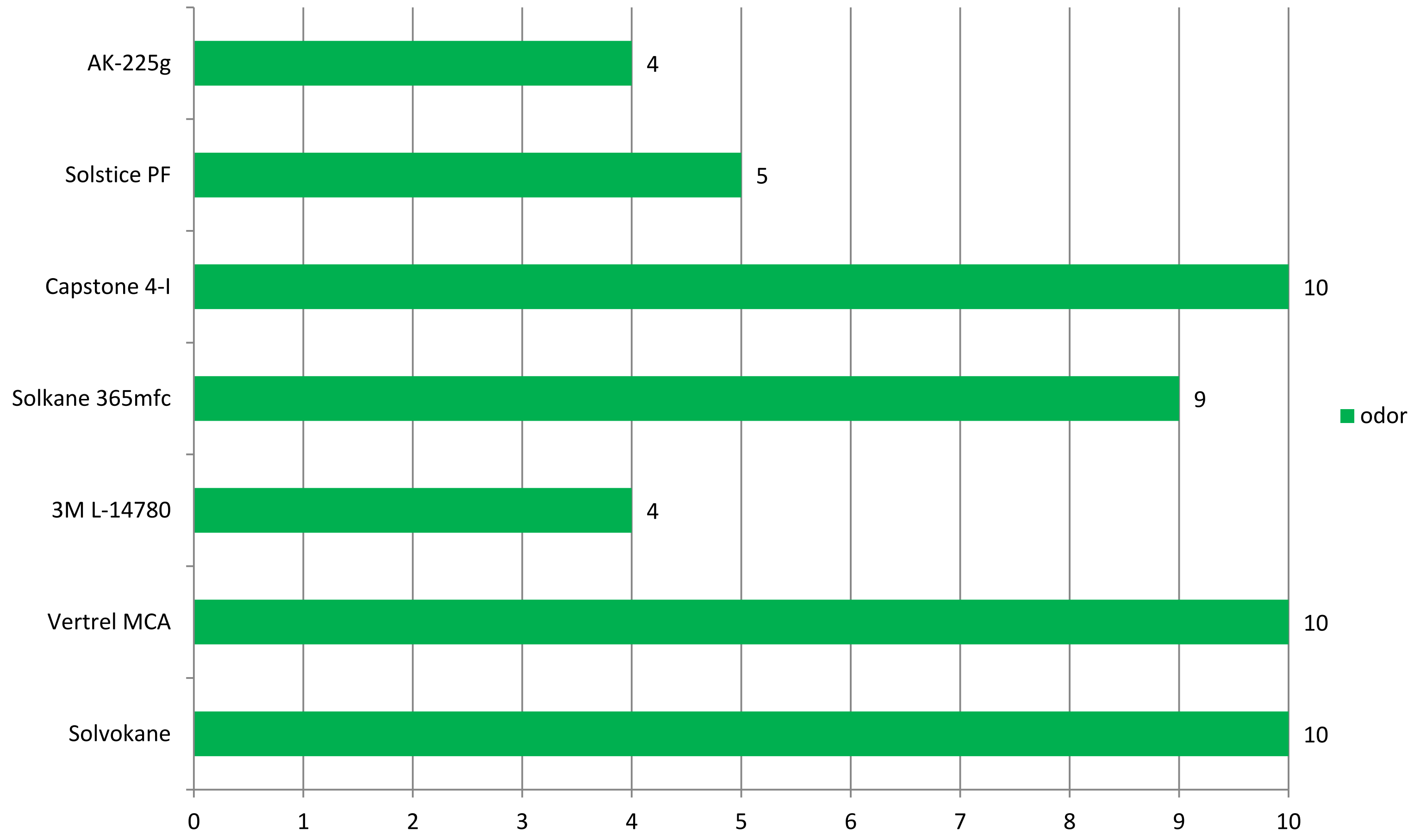
Solvent Cleaning Performance Rankings Based on SSC Lab Tests

	Quick Screen	Hyd Fluid Solubility	RP1 Solubility	85-140W Solubility	Rank (Test Avg.)[1]
HCFC225cb	1	1	1	1	1
Solstice PF	3 [2]	1	1	1	1.5
Solvokane	2	2	1	3	2
Vertrel MCA	4	3	2	2	2.8
3M L14780	5	4	1	4	3.5
Solkane	6	5	3	5	4.8

[1] HCFC225cb (AK 225g) has a value of "1" (best solvency and cleaning efficiency). The closer the candidate solvent is to "1", the better the fit is to performing similar to HCFC225 cb.

[2] The quick screening cleaning tests show Solstice PF has a 97.6% NVR Removal Efficiency. AK225g has a 99.1% NVR removal efficiency.

Solvent Odor Strength



Solvent Efficiency for NVR Removal

3.3mg NVR

HCFC225cb (Control /Baseline Solvent)

Cleaned Pan (g.)	Pan + NVR (g.)	Initial Pan NVR (g.)	Pan + NVR Post Solvent Flush (g.)	NVR Removed from Pan, Post Solvent Flush (g.)	% Solvent Efficiency NVR Removal
2.2783	2.2817	0.0034	2.2783	0.0034	100.0%
2.3163	2.3198	0.0035	2.3163	0.0035	100.0%
2.2569	2.2603	0.0034	2.2570	0.0033	97.1%
2.2718	2.2750	0.0032	2.2718	0.0032	100.0%
2.3042	2.3075	0.0033	2.3042	0.0033	100.0%
2.2613	2.2647	0.0034	2.2614	0.0033	97.1%
2.3265	2.3298	0.0033	2.3265	0.0033	100.0%
2.3343	2.3376	0.0033	2.3344	0.0032	97.0%
2.2789	2.2823	0.0034	2.2789	0.0034	100.0%
2.3243	2.3275	0.0032	2.3243	0.0032	100.0%

Kb = 37

Avg. Efficiency = 99.1 %

Solvent Efficiency for NVR Removal

3.4 mg NVR

Solvay Solvokane (Test Solvent)

Cleaned Pan (g.)	Pan + NVR (g.)	Initial Pan NVR (g.)	Pan + NVR Post Solvent Flush (g.)	NVR Removed from Pan, Post Solvent Flush (g.)	% Solvent Efficiency NVR Removal
2.4423	2.4453	0.0030	2.4423	0.0030	100.0%
2.3880	2.3912	0.0032	2.3880	0.0032	100.0%
2.4083	2.4116	0.0033	2.4084	0.0032	97.0%
2.4123	2.4157	0.0034	2.4123	0.0034	100.0%
2.4379	2.4415	0.0036	2.4380	0.0035	97.2%
2.3895	2.3929	0.0034	2.3896	0.0033	97.1%
2.3925	2.3959	0.0034	2.3925	0.0034	100.0%
2.3880	2.3917	0.0037	2.3882	0.0035	94.6%
2.4358	2.4392	0.0034	2.4358	0.0034	100.0%
2.3595	2.3630	0.0035	2.3596	0.0034	97.1%

Kb = 25

Avg. Efficiency = 98.3%

Solvent Efficiency for NVR Removal

3.4 mg NVR

Honeywell Solstice PF (Test Solvent)

Cleaned Pan (g.)	Pan + NVR (g.)	Initial Pan NVR (g.)	Pan + NVR Post Solvent Flush (g.)	NVR Removed from Pan, Post Solvent Flush (g.)	% Solvent Efficiency NVR Removal
2.3237	2.3271	0.0034	2.3238	0.0033	97.1%
2.3390	2.3423	0.0033	2.3391	0.0032	97.0%
2.3654	2.3688	0.0034	2.3656	0.0032	94.1%
2.3365	2.3399	0.0034	2.3365	0.0034	100.0%
2.3269	2.3301	0.0032	2.3270	0.0031	96.9%
2.3942	2.3975	0.0033	2.3943	0.0032	97.0%
2.4031	2.4064	0.0033	2.4031	0.0033	100.0%
2.3567	2.3600	0.0033	2.3568	0.0032	97.0%
2.3776	2.3813	0.0037	2.3777	0.0036	97.3%
2.3588	2.3626	0.0038	2.3588	0.0038	100.0%

Kb = 25

Avg. Efficiency = 97.6%

Solvent Efficiency for NVR Removal

3.6mg NVR

DuPont Vertrel MCA (Test Solvent)

Cleaned Pan (g.)	Pan + NVR (g.)	Initial Pan NVR (g.)	Pan + NVR Post Solvent Flush (g.)	NVR Removed from Pan, Post Solvent Flush (g.)	% Solvent Efficiency NVR Removal
2.3778	2.3816	0.0038	2.3781	0.0035	92.1%
2.3799	2.3832	0.0033	2.3799	0.0033	100.0%
2.3748	2.3783	0.0035	2.3749	0.0034	97.1%
2.3865	2.3902	0.0037	2.3866	0.0036	97.3%
2.3701	2.3736	0.0035	2.3701	0.0035	100.0%
2.3762	2.3801	0.0039	2.3763	0.0038	97.4%
2.4100	2.4135	0.0035	2.4101	0.0034	97.1%
2.4522	2.4556	0.0034	2.4523	0.0033	97.1%
2.3903	2.3939	0.0036	2.3904	0.0035	97.2%
2.3643	2.3677	0.0034	2.3643	0.0034	100.0%

Kb = 20

Avg. Efficiency = 97.8%

Solvent Efficiency for NVR Removal

3.5mg NVR

DuPont Capstone 4-I (Test Solvent)

Cleaned Pan (g.)	Pan + NVR (g.)	Initial Pan NVR (g.)	Pan + NVR Post Solvent Flush (g.)	NVR Removed from Pan, Post Solvent Flush (g.)	% Solvent Efficiency NVR Removal
2.3660	2.3692	0.0032	2.3662	0.0030	93.8%
2.3510	2.3545	0.0035	2.3511	0.0034	97.1%
2.3807	2.3843	0.0036	2.3808	0.0035	97.2%
2.4340	2.4377	0.0037	2.4342	0.0035	94.6%
2.2808	2.2844	0.0036	2.2809	0.0035	97.2%
2.4554	2.4589	0.0035	2.4555	0.0034	97.1%
2.3824	2.3860	0.0036	2.3825	0.0035	97.2%
2.3908	2.3943	0.0035	2.3908	0.0035	100.0%
2.3619	2.3655	0.0036	2.3619	0.0036	100.0%
2.4313	2.4348	0.0035	2.4313	0.0035	100.0%

Kb = Similar to
AK-225g (37)

Avg. Efficiency = 97.4%

Solvent Efficiency for NVR Removal

3.3mg NVR

3M L-14780 (Test Solvent)

Cleaned Pan (g.)	Pan + NVR (g.)	Initial Pan NVR (g.)	Pan + NVR Post Solvent Flush (g.)	NVR Removed from Pan, Post Solvent Flush (g.)	% Solvent Efficiency NVR Removal
2.5269	2.5302	0.0033	2.5271	0.0031	93.9%
2.5035	2.5069	0.0034	2.5035	0.0034	100.0%
2.5007	2.5041	0.0034	2.5009	0.0032	94.1%
2.5244	2.5276	0.0032	2.5244	0.0032	100.0%
2.4982	2.5016	0.0034	2.4984	0.0032	94.1%
2.5183	2.5216	0.0033	2.5184	0.0032	97.0%
2.4972	2.5005	0.0033	2.4973	0.0032	97.0%
2.5422	2.5454	0.0032	2.5423	0.0031	96.9%
2.5111	2.5143	0.0032	2.5111	0.0032	100.0%
2.5269	2.5302	0.0033	2.5271	0.0031	93.9%

Kb = Similar to MCA (20)

Avg. Efficiency = 97.0%

Solvent Efficiency for NVR Removal

3.5mg NVR

Solvay Solkane 365mfc (Test Solvent)

Cleaned Pan (g.)	Pan + NVR (g.)	Initial Pan NVR (g.)	Pan + NVR Post Solvent Flush (g.)	NVR Removed from Pan, Post Solvent Flush (g.)	% Solvent Efficiency NVR Removal
2.3943	2.3977	0.0034	2.3948	0.0029	85.3%
2.3565	2.3602	0.0037	2.3569	0.0033	89.2%
2.3507	2.3542	0.0035	2.3511	0.0031	88.6%
2.3414	2.3448	0.0034	2.3419	0.0029	85.3%
2.3870	2.3904	0.0034	2.3874	0.0030	88.2%
2.3077	2.3114	0.0037	2.3083	0.0031	83.8%
2.3671	2.3705	0.0034	2.3674	0.0031	91.2%
2.4233	2.4267	0.0034	2.4238	0.0029	85.3%
2.3577	2.3609	0.0032	2.3580	0.0029	90.6%
2.3748	2.3783	0.0035	2.3753	0.0030	85.7%

Kb = 12

Avg. Efficiency = 87.3%

Return to Menu

Return to Pg 16

APPENDIX E—METALS COMPATIBILITY DATA

Appendix E contains the Metals Corrosion Test Results for the Candidate Solvents, HCF-C225cb (AK225g) Replacement Study, 2nd Technical Interchange Meeting (TIM), February 19–20, 2014.

HCFC225cb (AK225g) Replacement Study

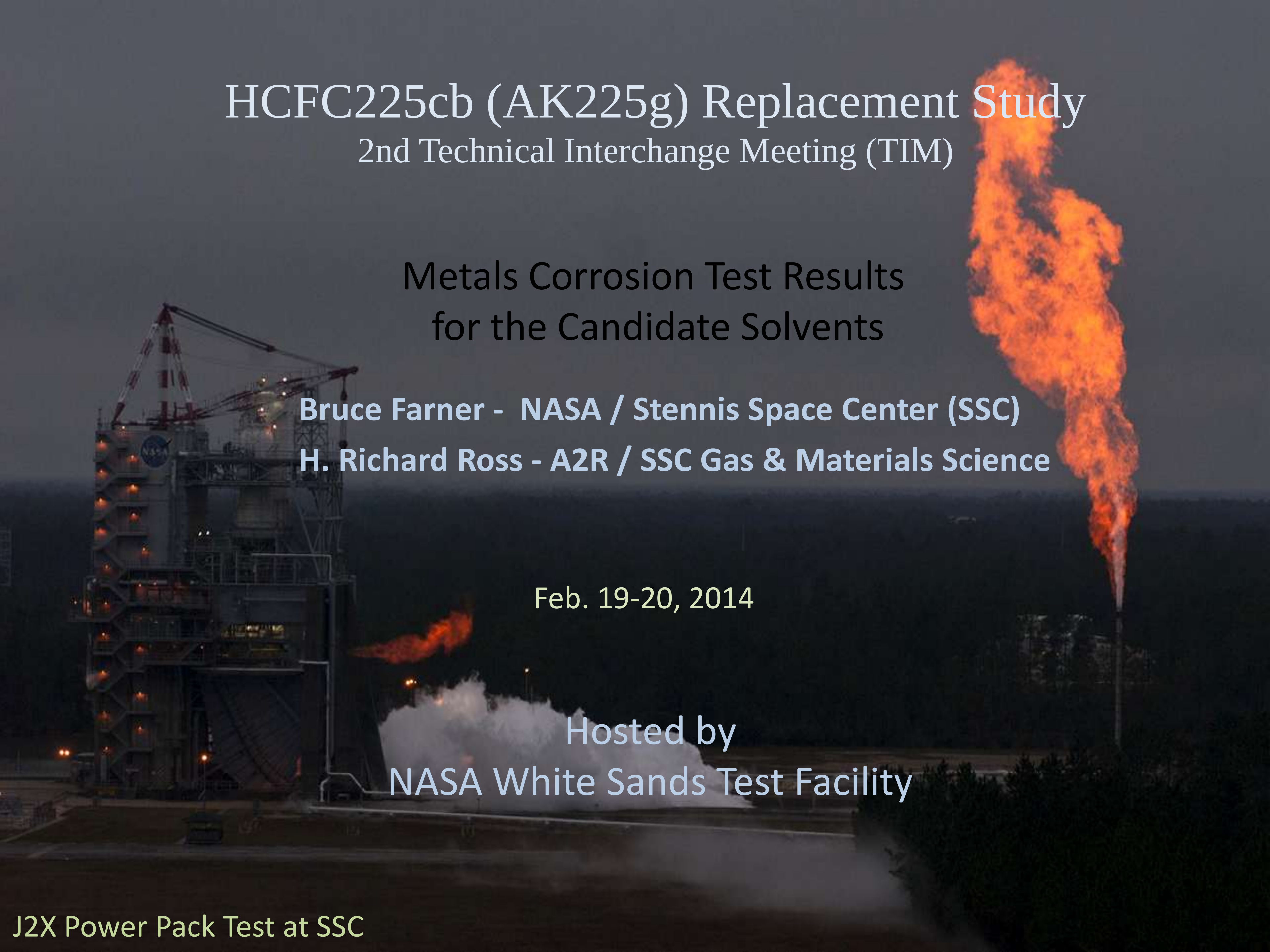
2nd Technical Interchange Meeting (TIM)

Metals Corrosion Test Results for the Candidate Solvents

Bruce Farner - NASA / Stennis Space Center (SSC)
H. Richard Ross - A2R / SSC Gas & Materials Science

Feb. 19-20, 2014

Hosted by
NASA White Sands Test Facility



J2X Power Pack Test at SSC

Phase II Test Objective

To determine the reactivity (corrosiveness) of the three candidate cleaning solvents” that were selected from the Phase 1 study with thirteen different alloys that were exposed to solvent vapor and total liquid immersion by measuring a combination of weight changes and visual qualitative changes.

Solvents & Alloys Tested

- Solvents
  - AGC Chemicals HCFC225 cb (AK225g) Baseline Solvent
  - Honeywell Solstice PF (HFO 1233zd)
  - 3M L-14780 (22%tDCE/78% HFE-347mcc3)
  - Solvay Solvokane (30% tDCE/70% HFC-365mfc)
- Metals
  - Carbon Steel (4140)
  - Stainless Steels (17-4PH, A286, 304 & 440C)
  - Nickel Alloys (Monel 400 & Inconel 718)
  - Co Cr Ni Alloy (Elgiloy)
  - Tin Bronze
  - Brass (Naval Brass)
  - Aluminum (6061 -T6, 2195 -T8 & 2219 -T6)

Acknowledgements

NASA /SSC Project Lead

Bruce Farner / RA 20

NASA /MSFC

Mark Mitchell for providing the aluminum alloy coupons, 2195 and 2219

SSC Laboratory Team

H. Rick Ross - Lab Lead / A2Research
Mary Kerschbaum - Lab Scientist / A2Research
Erick Guttierrez - Lab Scientist/ A2Research
Taylor Davie – Lab Scientist / A2 Research

Test Protocol

Test coupons for each given alloy were fabricated from new or fresh stock – the fabricated coupons measured approximately 2” x 0.75”

All fabricated coupons were cleaned with AK225g (rinsed 2X, soaked for 1 hr, rec’d a final rinse after soaking, dried with forced GN and were immediately placed in Teflon holders that separated each coupon and were stored in a laboratory desiccator.

The corrosion tests were performed at the BP temperature for each candidate solvent while ensuring the coupons had free contact with the solvent and were isolated from each other using Teflon support devices

The boiling point temperature for each solvent was maintained using a constant temperature water bath

Test Protocol (Continuation)

Coupon set in HP Glass Tube. Heavy Wall Glass Tube Rated at 150 psig, Tube Vol. 210 ml.

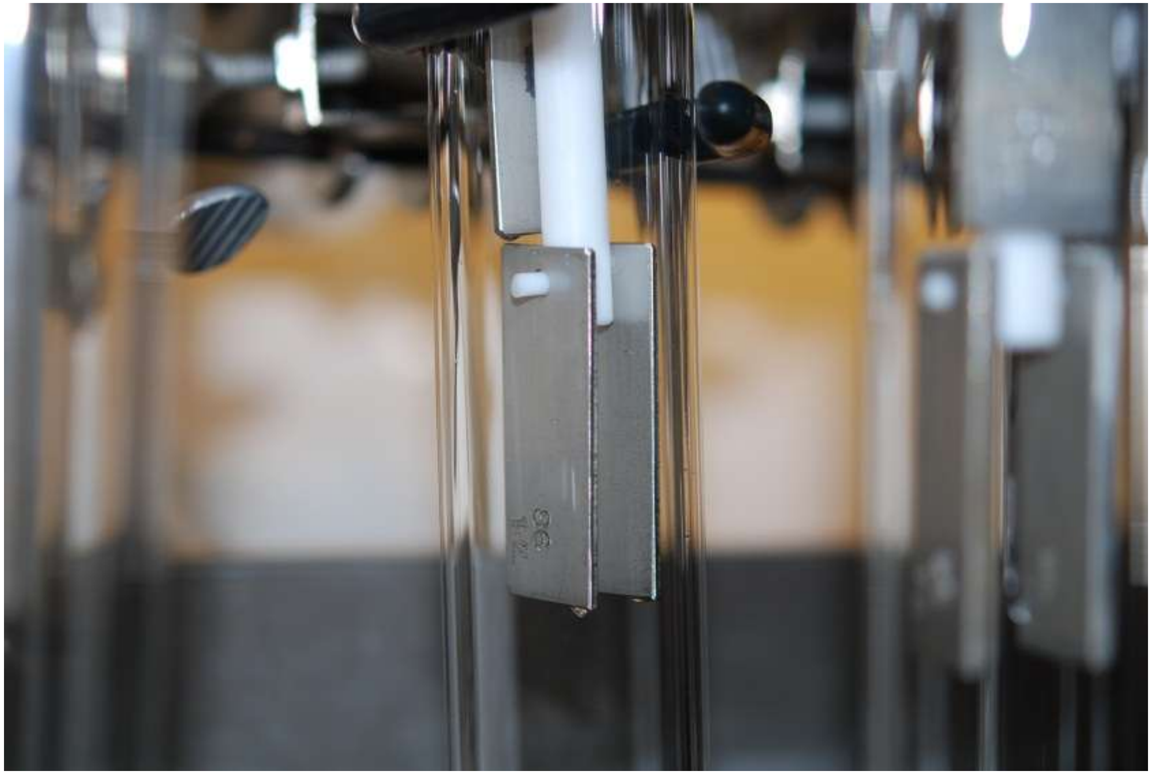
Alloy coupon set secured in HP Glass Tube w/ 80 ml of test solvent prior to placing in the water bath.

Six replicate test coupons for each alloy were placed in ACE High Pressure Borosilicate Glass Tube w/ a # 25 TFE threaded plug (rated @ 150 psi).

Three replicates were totally immersed in the solvent and three replicates were placed above the liquid for exposure to the solvent vapor.

(6 coupons + 1 control / alloy) (13 alloys/solvent) (3 tests/ coupon @ before solvent exposure, @ 24 hrs & for additional 144hrs / solvent) = 273 coupon tests / solvent. For 4 solvents, **"n"** = 948 tests

Test Protocol (Continuation)



Close up view of an alloy coupon set coming out the water bath after solvent exposure. The coupons are secured to a TFE rod.

Side View

Coupons for each alloy were placed in the Teflon holder and stored in the desiccator.

Top View of the Teflon Coupon Holder

The radial slots secure & separate each alloy coupon.

Analytical Process

Solvent compatibility testing for the alloy coupons were performed for weight changes, corrosion, etching, pitting, discoloration and other signs of degradation.

Weight lost and surface corrosion is based on the appearance of the surfaces compared to the control coupon.

Total solvent immersion and solvent vapor tests were performed in triplicate for each alloy coupon and analyzed in accordance with ASTM F-483-09.

Coupon samples and the control coupon were re-weighed after each solvent exposure test to the nearest 0.1mg

Metal Coupon Changes After 168 hrs in Solstice PF (HFO 1233zd) at 72 deg F

Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
A286 SST	-0.1mg	-0.3 mg	0.0 mg	No Change
304L	-0.1mg	-0.1mg	-0.1mg	No Change
440C SST	0.2 mg	0.0 mg	0.1mg	No Change
17-4PH	0.2 mg	0.1 mg	0.0 mg	No Change
AISI 4140	0.2 mg	-0.5 mg	-0.4 mg	No Change

Non Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
Eligiloy	0.0 mg	0.0 mg	0.0 mg	No Change
Inconel 718	0.0 mg	0.0 mg	0.1mg	No Change
Monel 400	0.0 mg	-0.1 mg	-0.1mg	No Change
Naval Brass	-0.1 mg	-0.1mg	0.0 mg	No Change
Tin Bronze 510	-0.2 mg	-0.2 mg	-0.2 mg	No Change
6061 T6 Al	0.0 mg	-0.1mg	0.0 mg	No Change
2219 T6 Al	0.0 mg	0.0 mg	0.1 mg	No Change
2195 T8(Al Li)	0.1 mg	0.0 mg	0.1 mg	No Change

Metal Coupon Change After 168 hrs in L14780 at 82 deg F

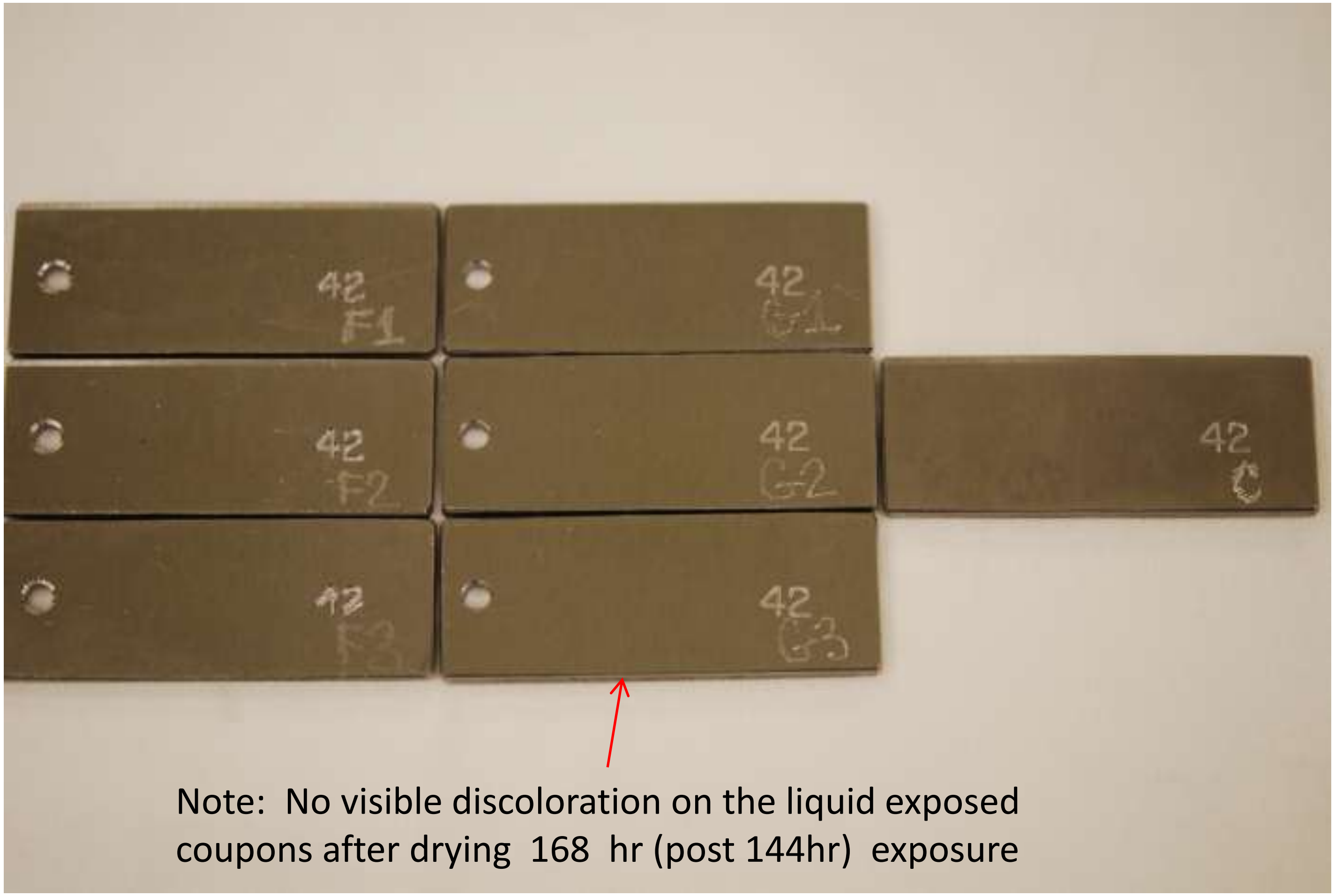
Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
A286 SST	0.0 mg	-0.2 mg	0.0 mg	No Change
304L	0.0 mg	-0.1mg	-0.2 mg	No Change
440C SST	-0.1 mg	-0.2 mg	- 0.1mg	No Change
17-4PH	0.0 mg	0.0 mg	-0.1 mg	No Change
AISI 4140	-0.1 mg	-0.5 mg	-0.3 mg	**Delayed Rust** [1]

Non Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
Eligiloy	0.1 mg	0.0 mg	-0.1 mg	No Change
Inconel 718	0.1 mg	0.0 mg	0.0 mg	No Change
Monel 400	-0.1 mg	0.0 mg	0.0 mg	No Change
Naval Brass	0.0 mg	0.0 mg	0.0 mg	No Change
Tin Bronze 510	-0.2 mg	-0.2 mg	0.0 mg	No Change
6061 T6 Al	0.0 mg	-0.1mg	0.1 mg	No Change
2219 T6 Al	0.0 mg	0.0 mg	0.1 mg	No Change
2195 T8(Al Li)	-0.1 mg	0.0 mg	0.0 mg	No Change

[1] No rust or discoloration were present after the 168 hr exposure test was performed

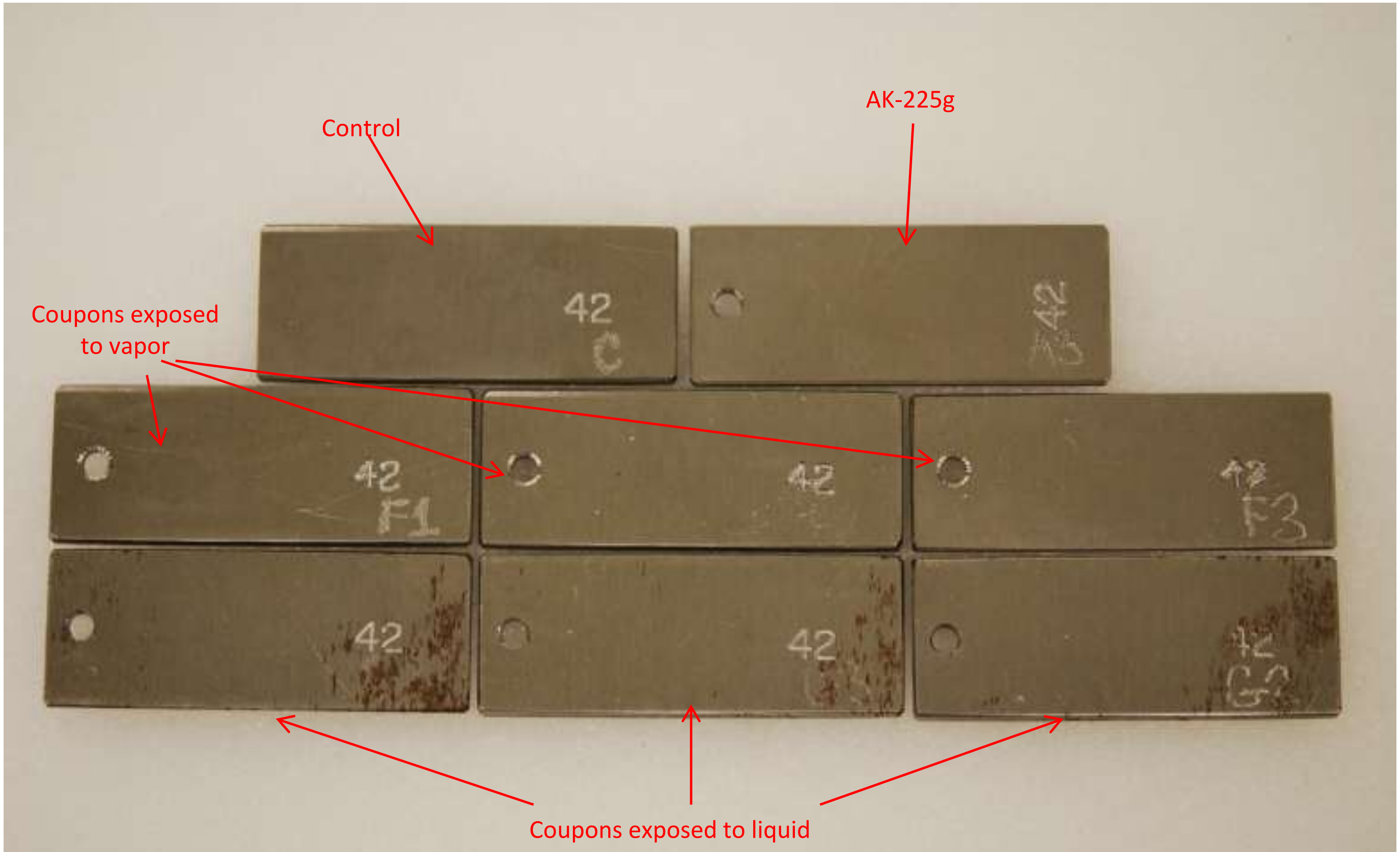
AISI 4140 Low Alloy Steel

3M L-14780 After 168 hr exposure



Note: No visible discoloration on the liquid exposed coupons after drying 168 hr (post 144hr) exposure

AISI 4140 Low Alloy Steel Exposed to 3M L-14780 4 Weeks After the 168 hr Exposure Test



Note: All three coupons that were exposed to the 3M liquid show discoloration, while all three coupons exposed to the 3M vapor show no discoloration.

Metal Coupon Change After 168 hrs in Solvokane at 93 deg F

Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
A286 SST	0.1 mg	0.0 mg	0.1 mg	No Change
304L	0.0 mg	-0.1mg	0.0 mg	No Change
440C SST	-0.1 mg	-0.1 mg	0.0 mg	No Change
17-4PH	0.0 mg	-0.1 mg	0.1 mg	No Change
AISI 4140	0.0 mg	-0.1 mg	0.2 mg	No Change

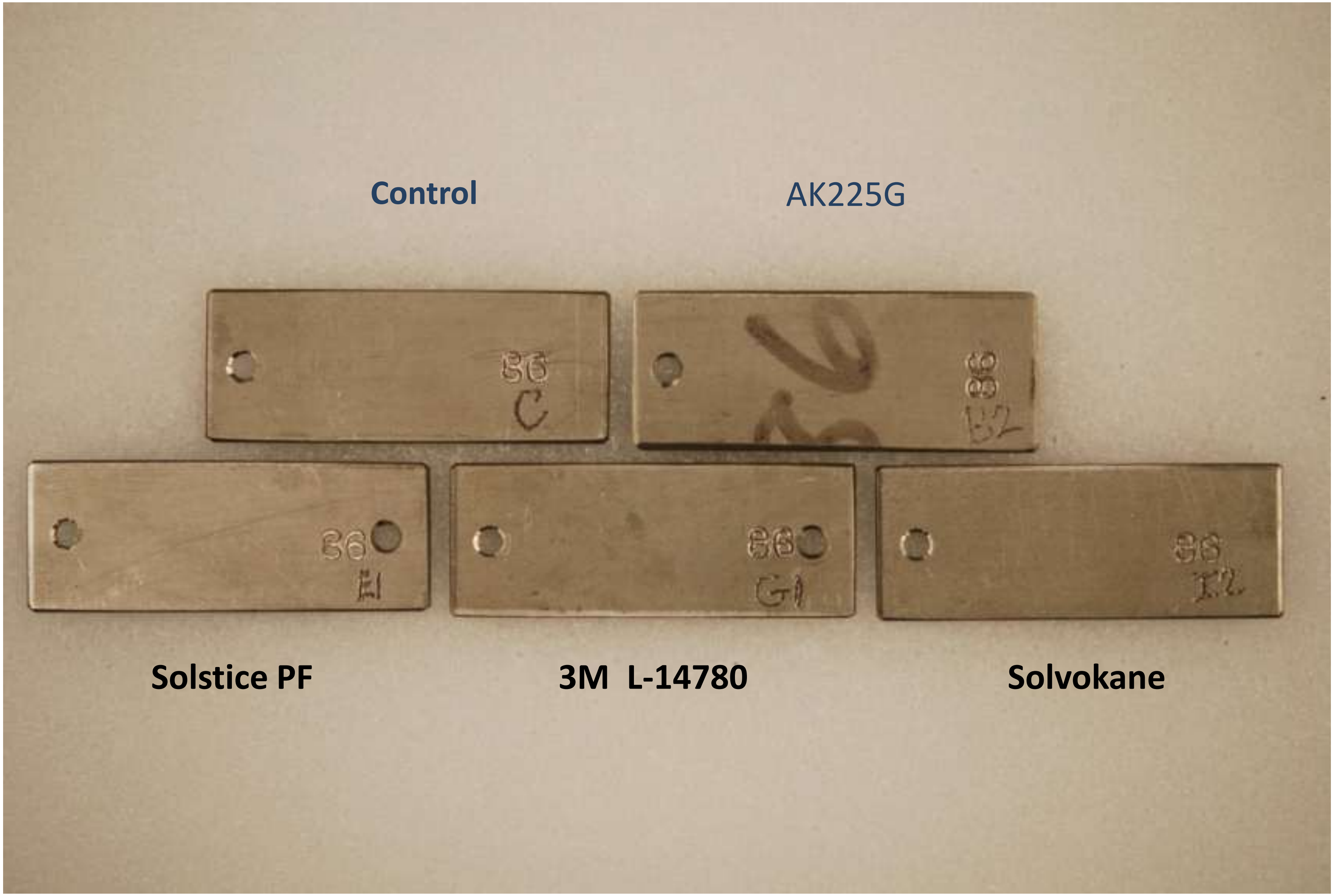
Non Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
Eligiloy	0.1 mg	0.1 mg	0.0 mg	No Change
Inconel 718	0.1 mg	0.0 mg	0.1 mg	No Change
Monel 400	0.1 mg	0.0 mg	0.0 mg	No Change
Naval Brass	0.0 mg	0.0mg	0.0 mg	No Change
Tin Bronze 510	0.1 mg	0.0 mg	0.0 mg	No Change
6061 T6 Al	0.0 mg	0.0 mg	0.0 mg	No Change
2219 T6 Al	0.0 mg	0.0 mg	0.0 mg	No Change
2195 T8(Al Li)	0.0 mg	0.0 mg	0.1 mg	No Change

Metal Coupon Change After 168 hrs in AK225G at 133 deg F

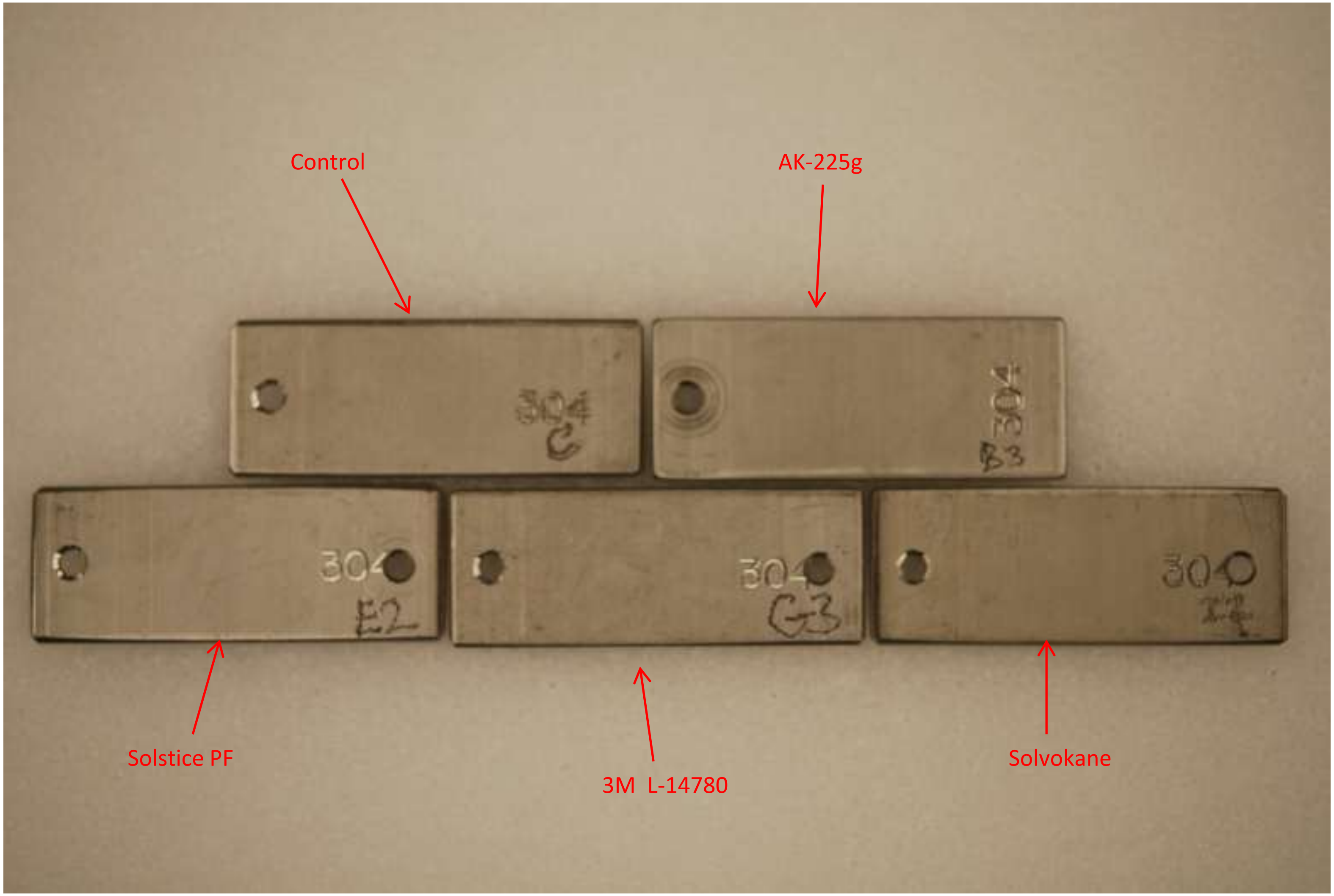
Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
A286 SST	0.0 mg	-0.1 mg	-0.1 mg	No Change
304L	-0.1 mg	-0.1mg	-0.1 mg	No Change
440C SST	-0.3 mg	-0.3 mg	-0.1 mg	No Change
17-4PH	0.0 mg	-0.1 mg	-0.1 mg	No Change
AISI 4140	0.5 mg	-0.3 mg	0.0 mg	No Change

Non Ferrous Based Coupons	**Vapor Coupon Wt Change**	**Liquid Coupon Wt Change**	**Control Coupon Wt Change**	**Sample vs. Control Appearance**
Eligiloy	-0.2 mg	-0.1 mg	-0.1 mg	No Change
Inconel 718	-0.1 mg	-0.1 mg	0.0 mg	No Change
Monel 400	-0.1 mg	-0.2 mg	-0.1 mg	No Change
Naval Brass	-0.2 mg	-0.2mg	-0.1 mg	No Change
Tin Bronze 510	-0.1 mg	-0.1 mg	-0.1 mg	No Change
6061 T6 Al	-0.1 mg	0.0 mg	0.0 mg	No Change
2219 T6 Al	0.0 mg	-0.1 mg	0.0 mg	No Change
2195 T8(Al Li)	0.0 mg	0.0 mg	0.1 mg	No Change

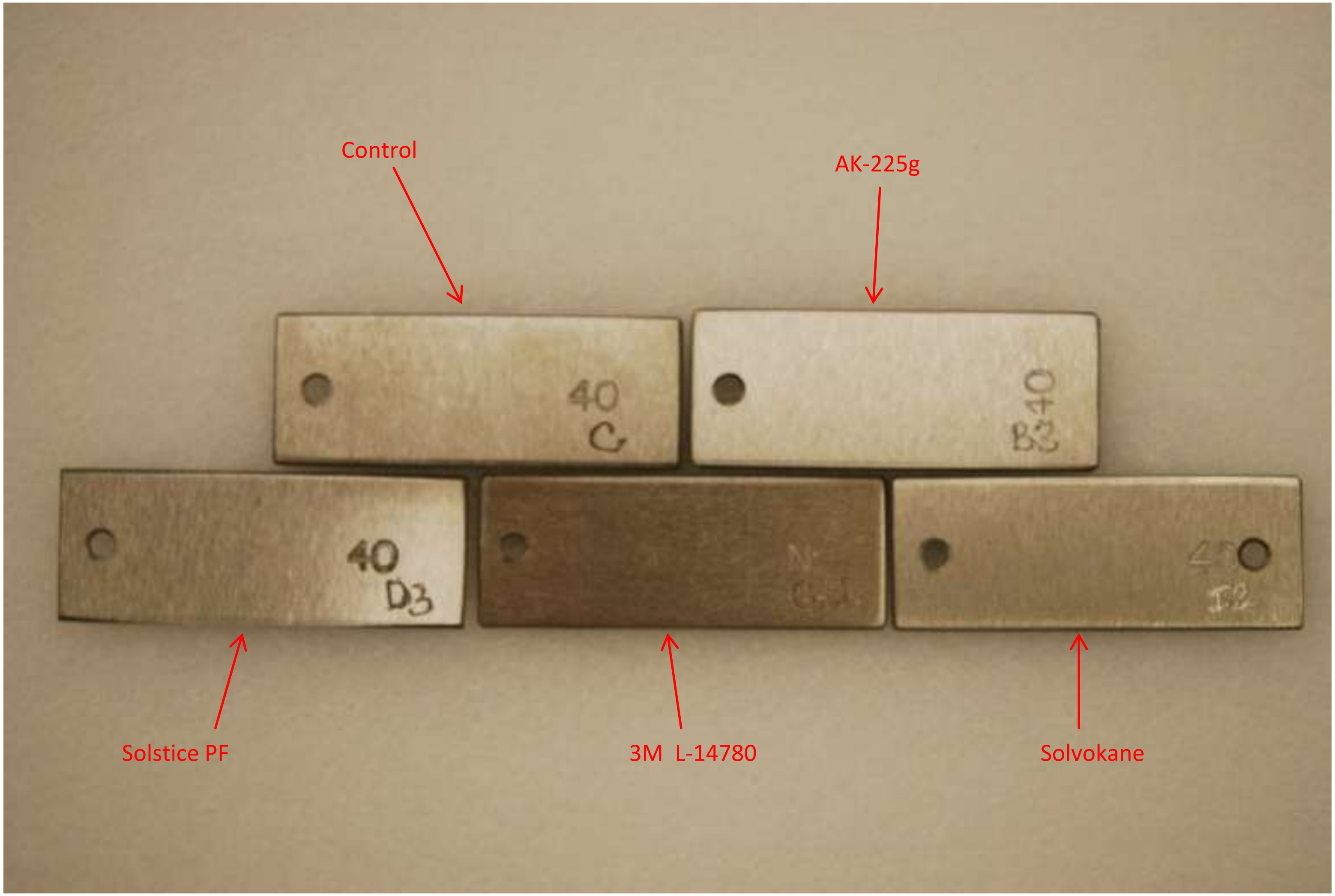
A-286 Stainless Steel
Representative Coupon from Liquid Immersion Tests



304L Stainless Steel



440C Stainless Steel



Control

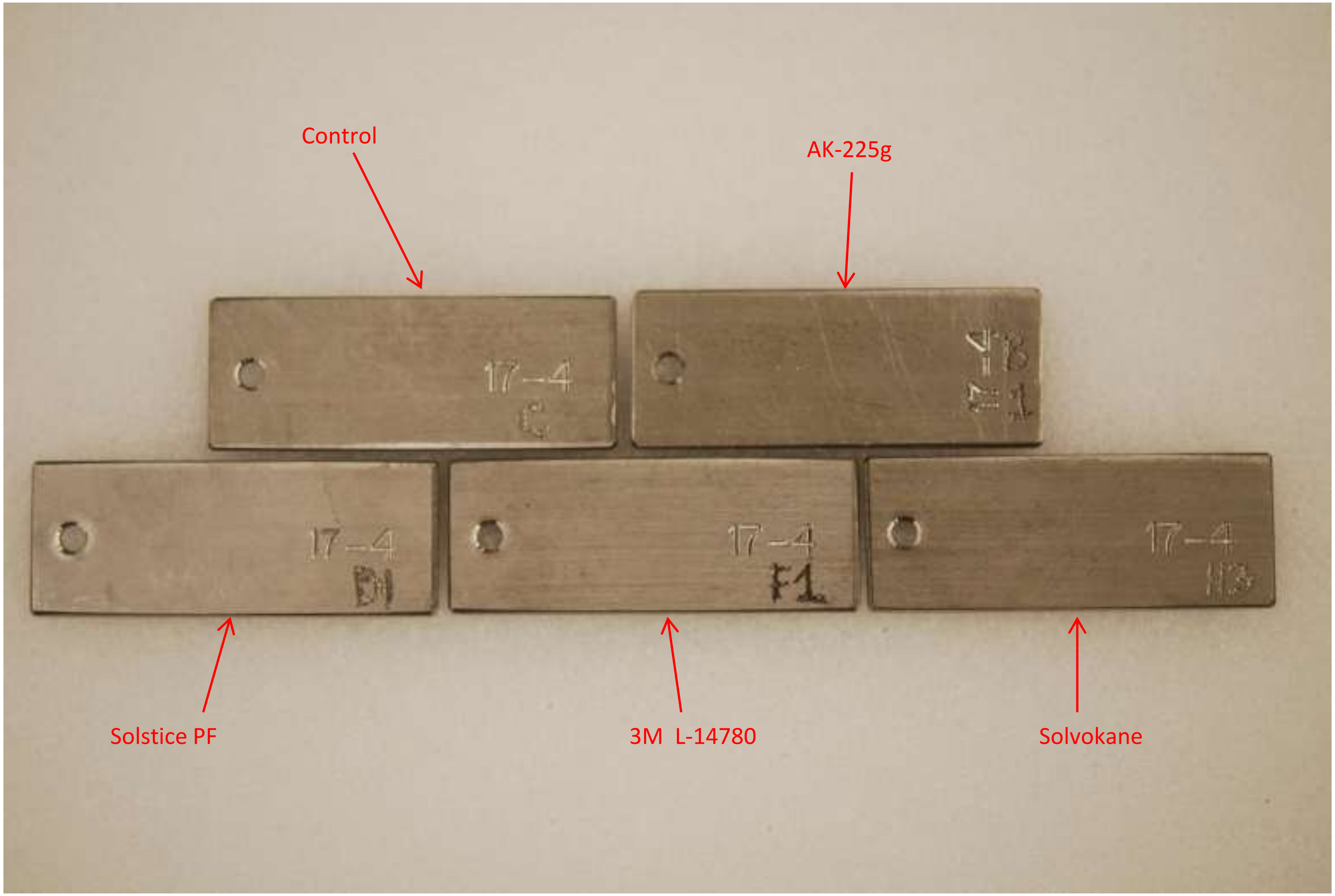
AK-225g

Solstice PF

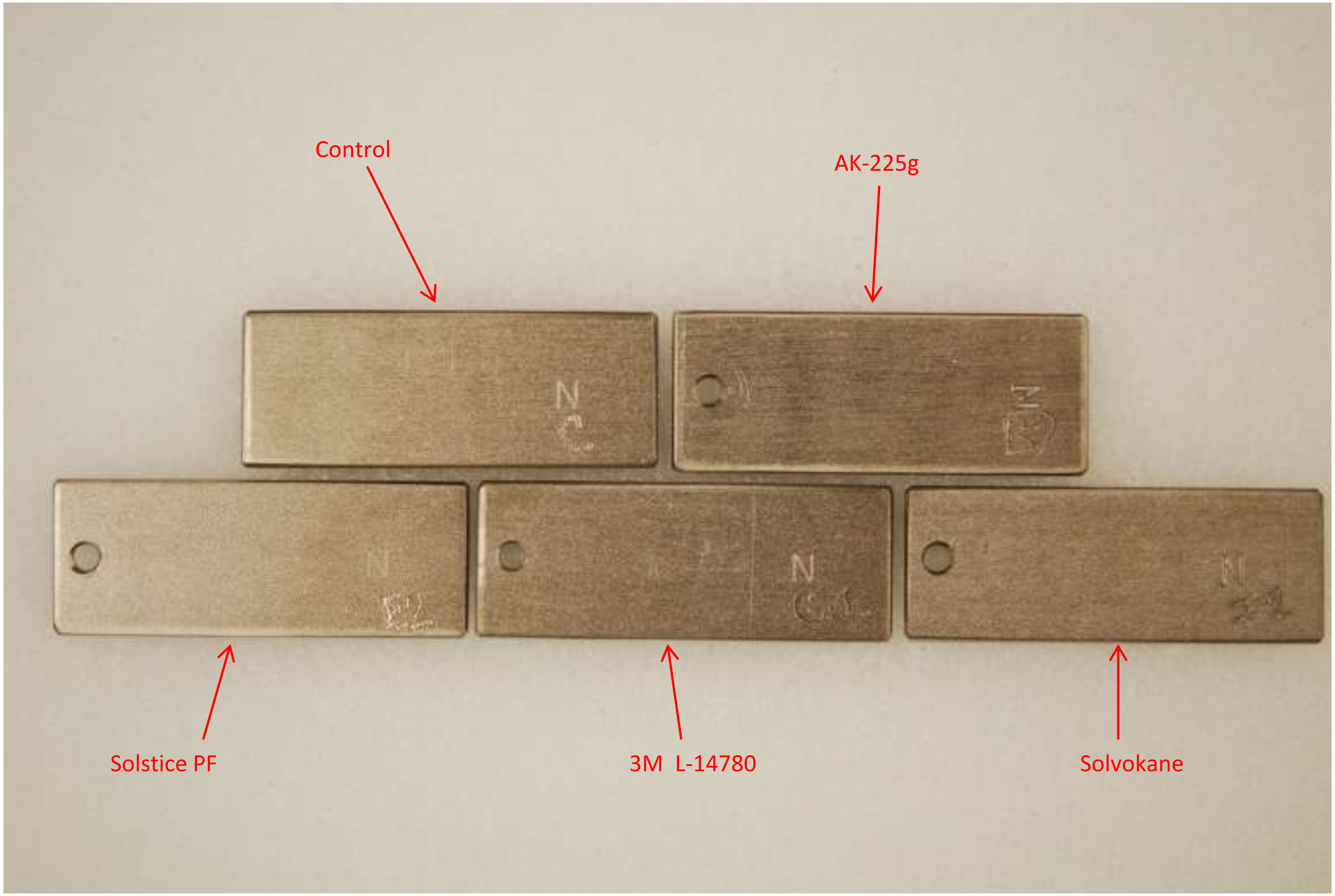
3M L-14780

Solvokane

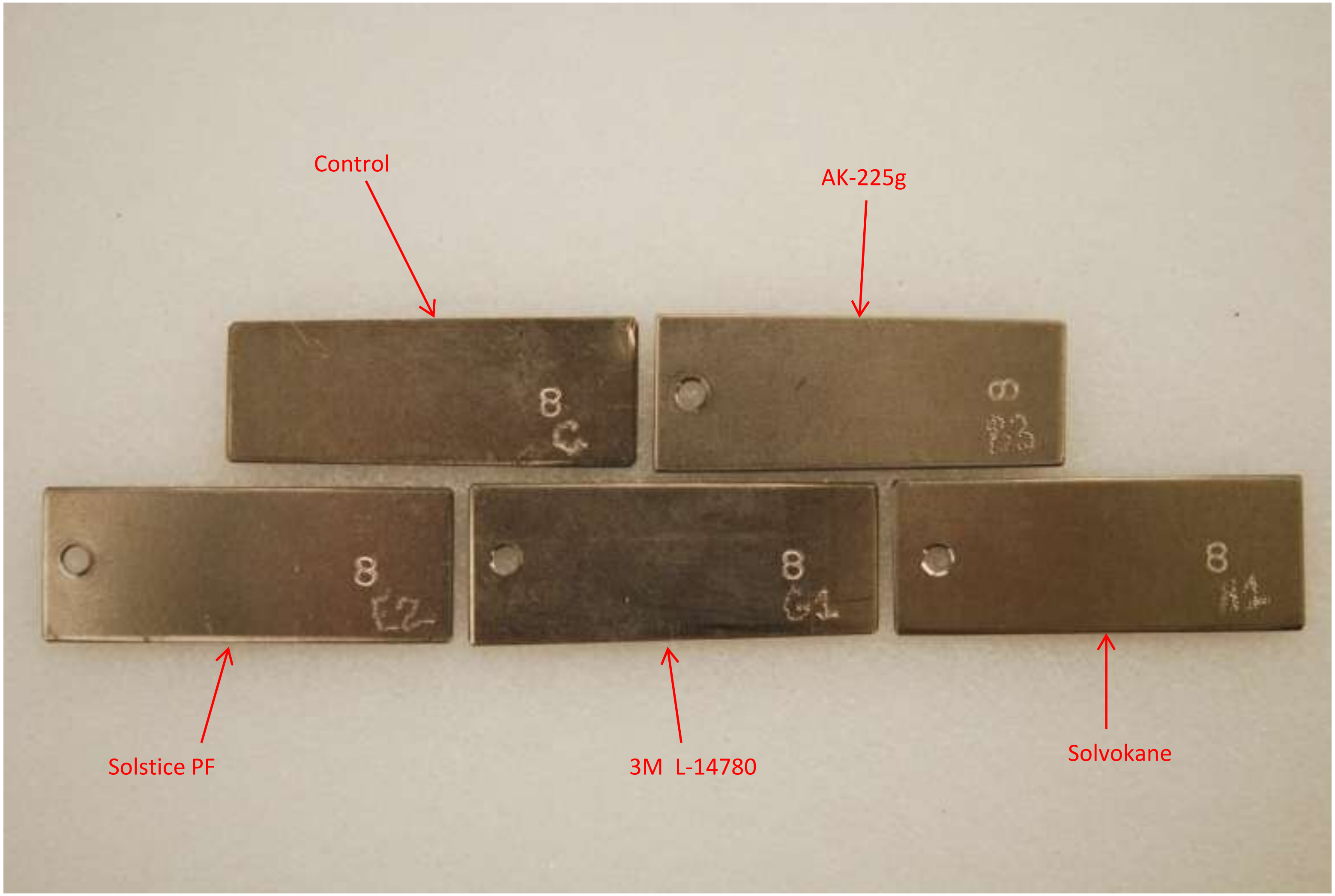
17-4 PH Stainless Steel



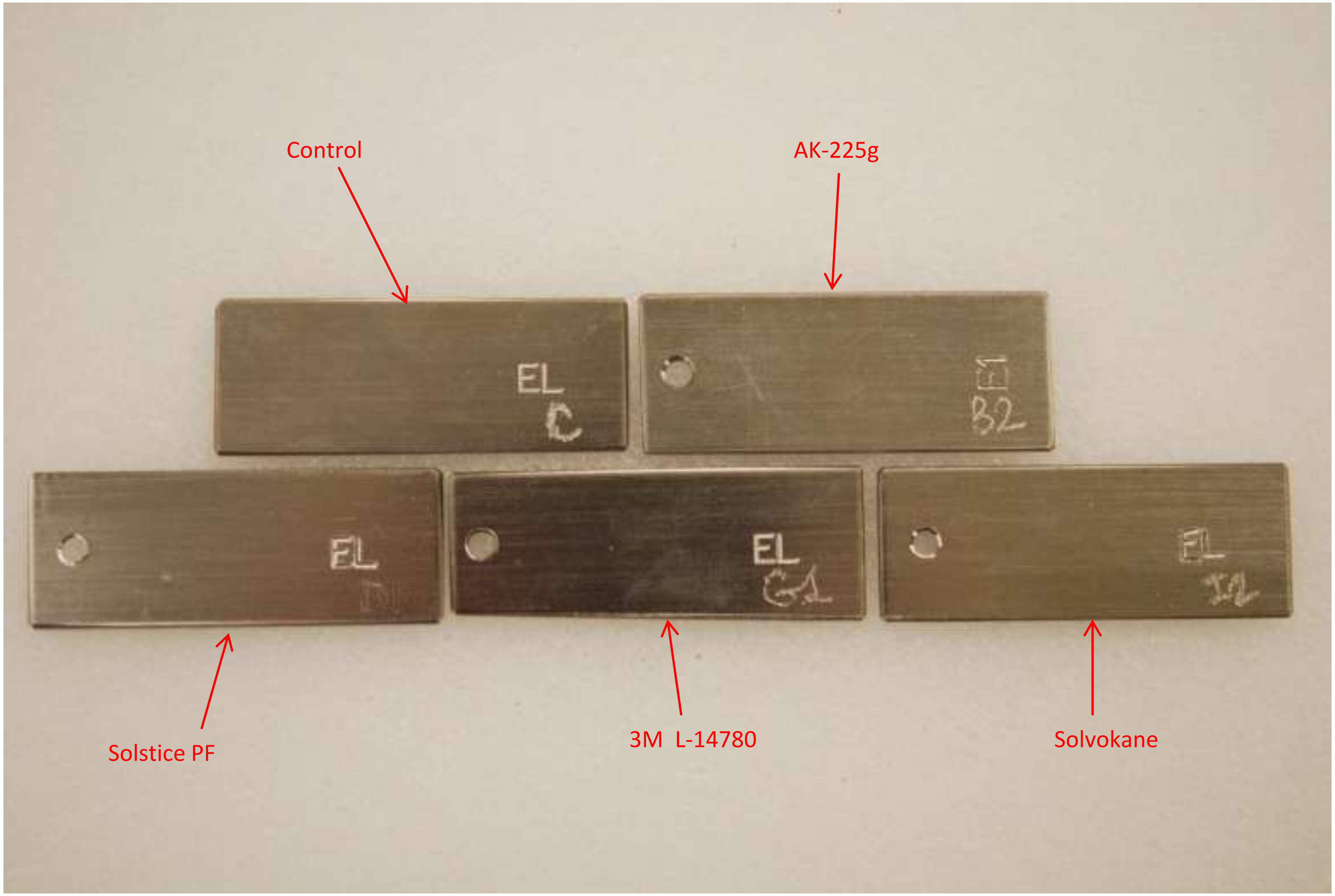
Monel 400 (Nickel)



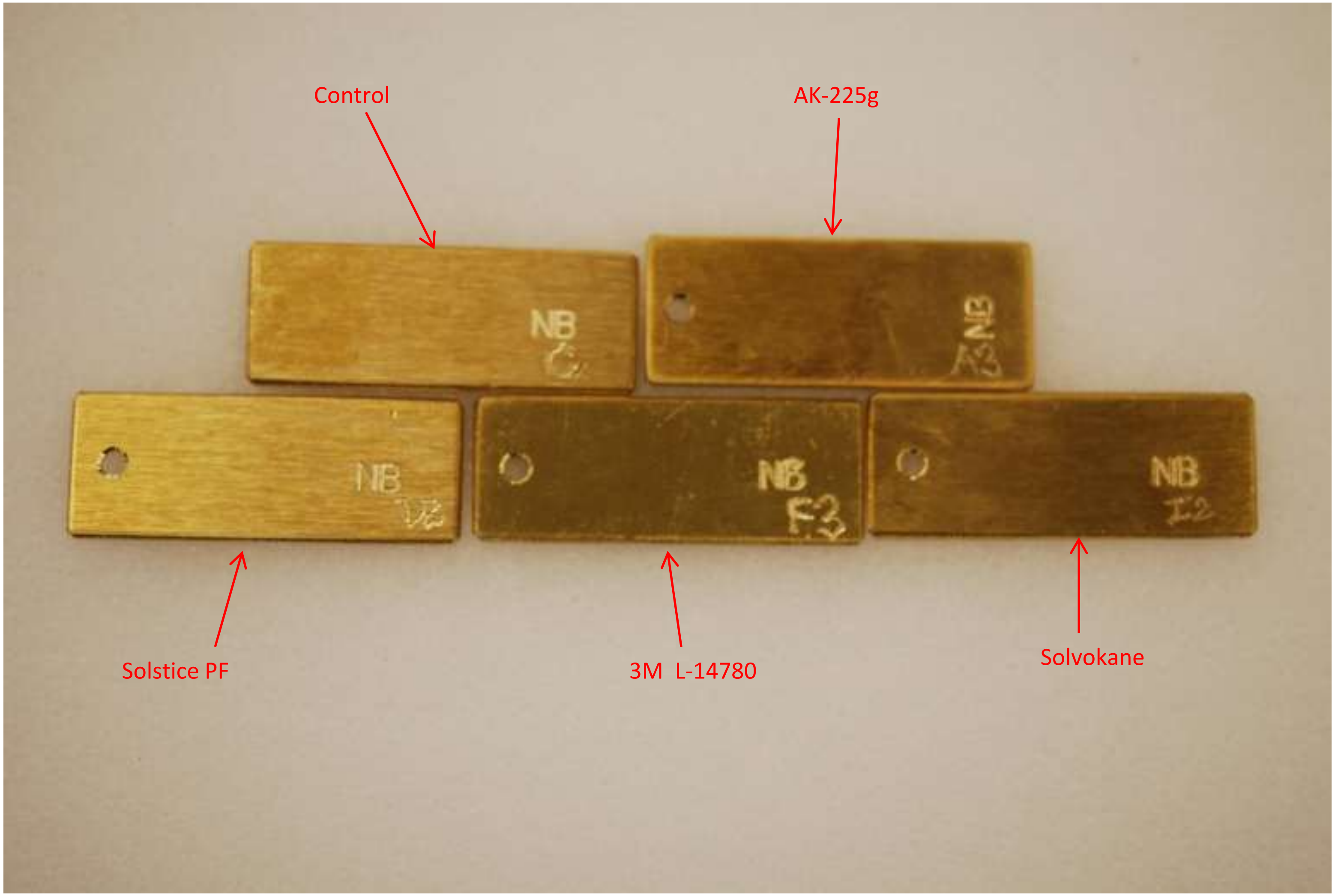
Inconel 718



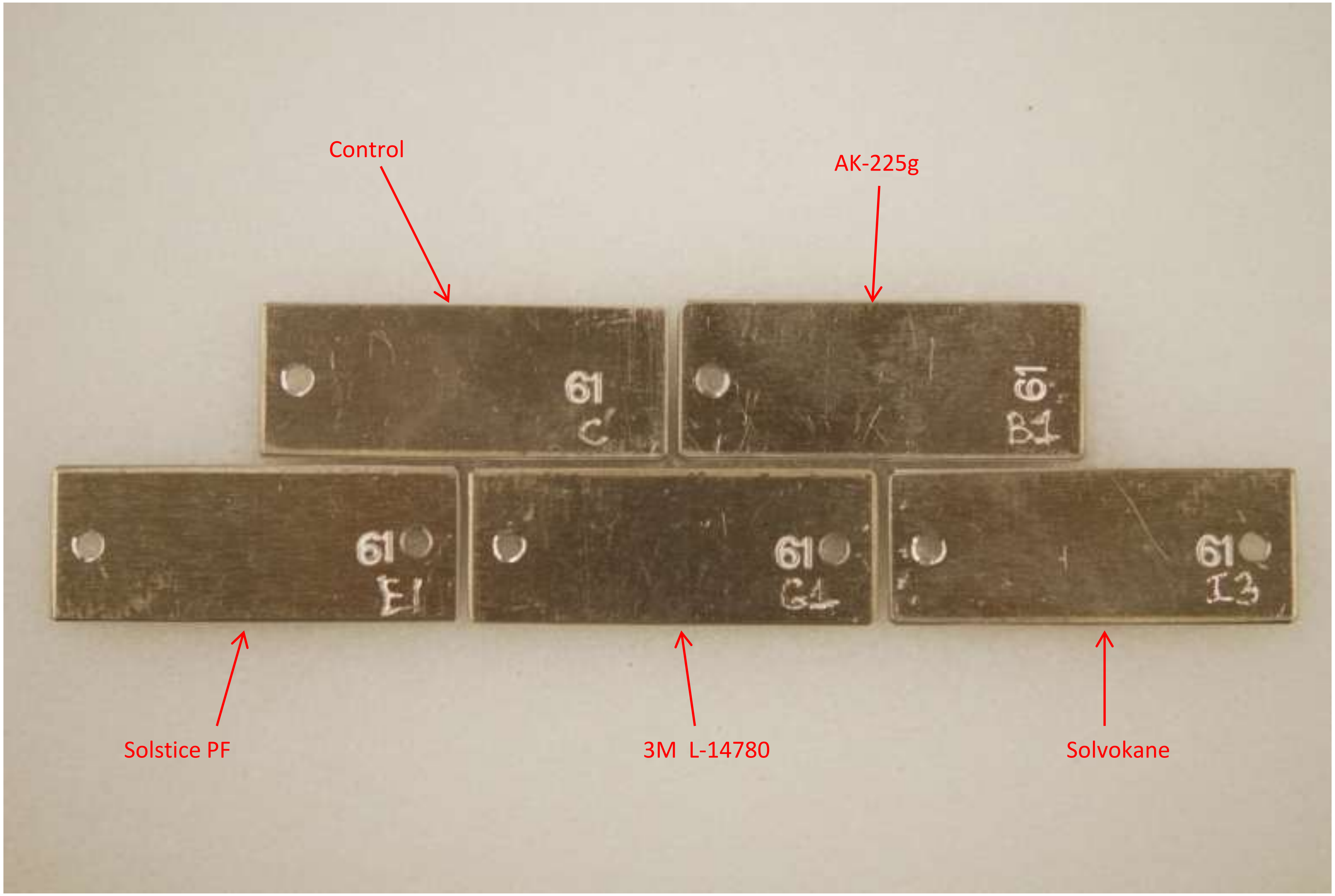
Co-Cr-Ni Alloy - Elgiloy



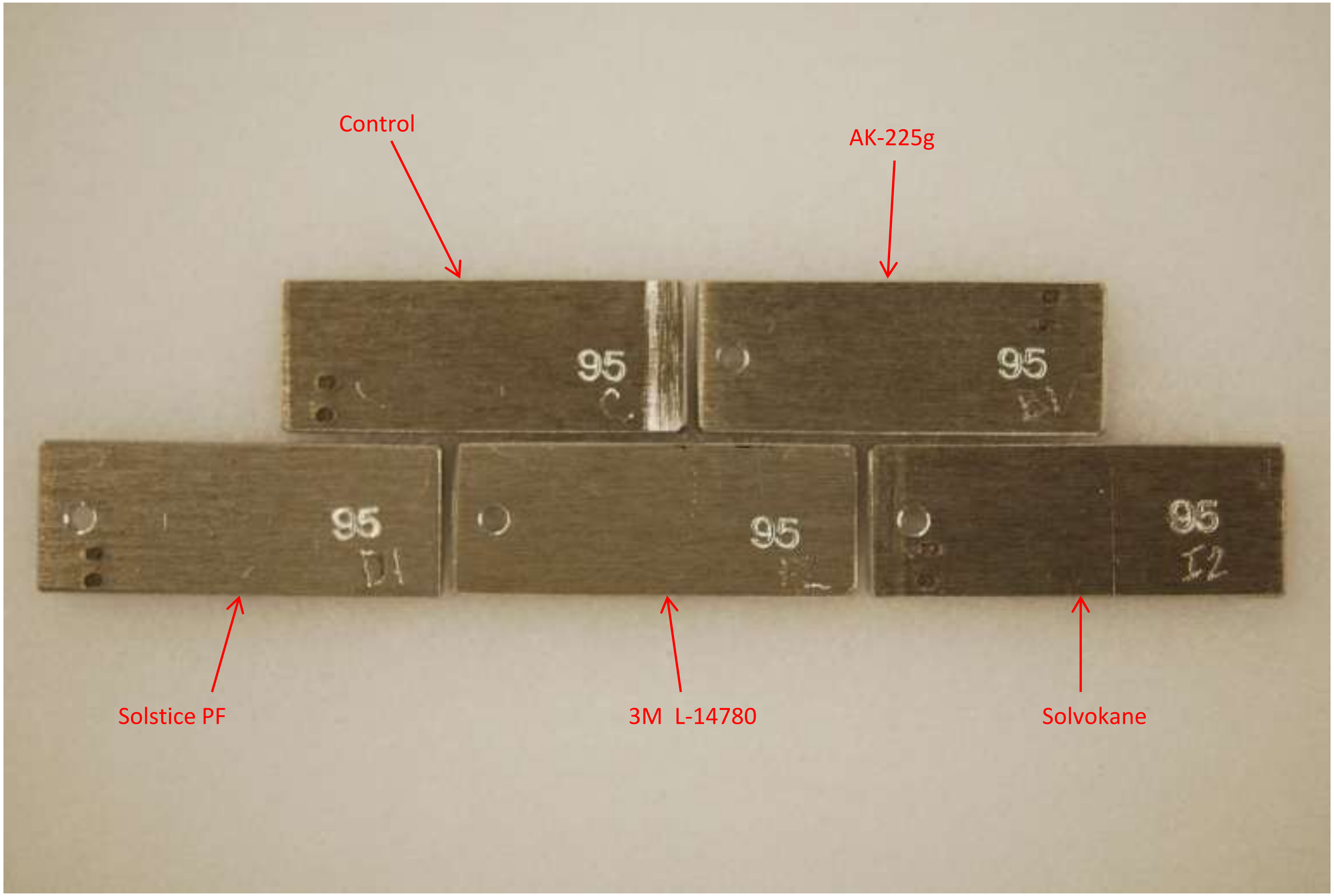
Naval Brass


Control
AK-225g
Solstice PF
3M L-14780
Solvokane
NB
NB
NB
NB
NB

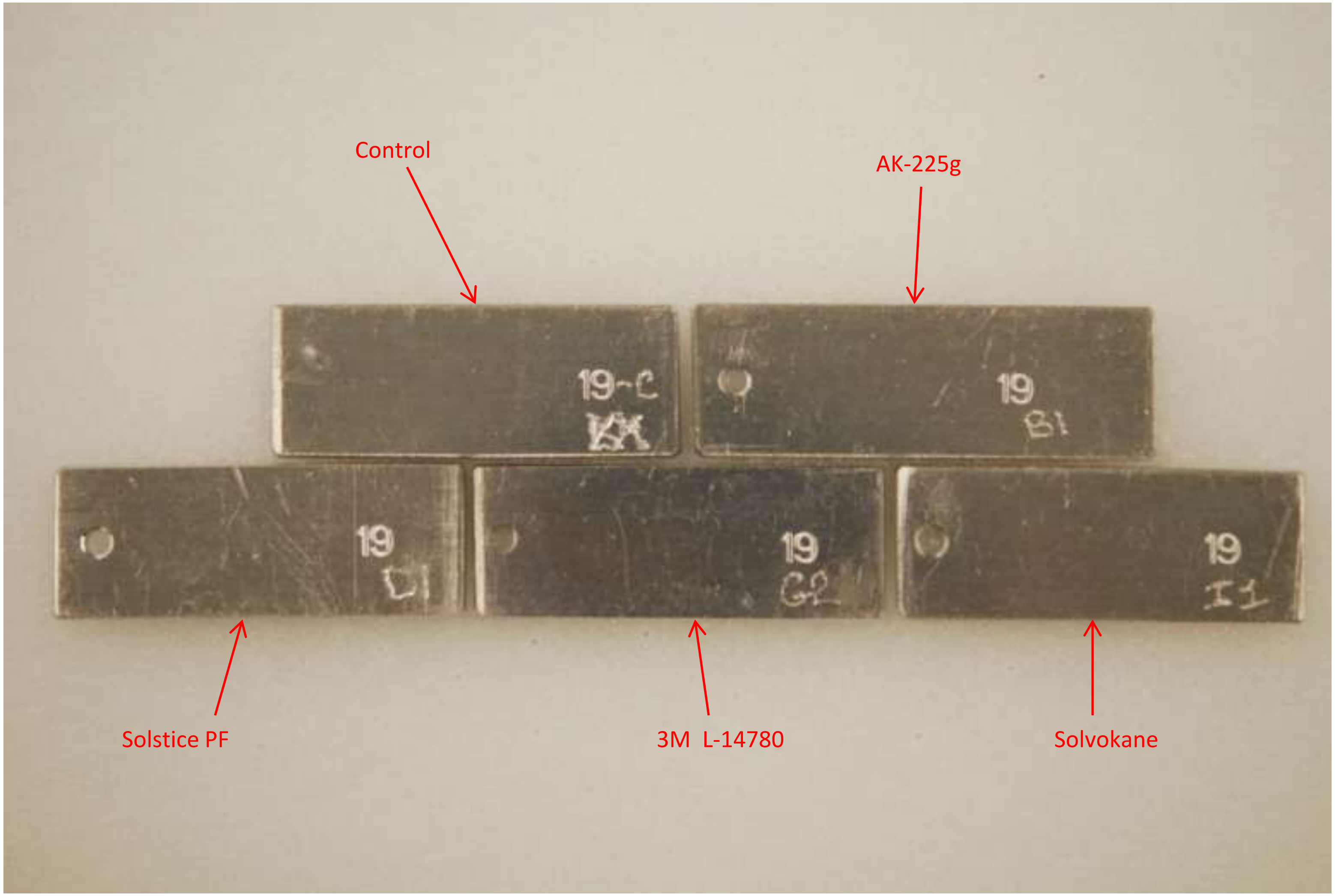
6061-T6 Aluminum



2195-T8 Aluminum-Lithium



2219-T6 Aluminum



Control

AK-225g

Solstice PF

3M L-14780

Solvokane

Return to Menu Return to Pg 19

APPENDIX F—NONMETALS COMPATIBILITY TEST RESULTS

Appendix F contains the Nonmetals Compatibility Immersion Test—Immersion data sheets by solvent and bar charts by nonmetal.

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
A. FKM Cpd. V0747-75 (Viton A) 11/21/13	1	401.29	20.17	91	453.64	21.35	92	13.0	5.9	1.1	weight unstable after 15 min exposure
	2	399.11	20.12	88	449.68	21.46	90	12.7	6.7	2.3	weight unstable after 15 min exposure
	3	406.00	20.32	93	454.84	21.43	94	12.0	5.5	1.1	weight unstable after 15 min exposure
	Control	407.24	20.45	93	407.25	20.65	93	0.0	1.0	0.0	
B. FFKM (Kalrez) 11/25/13	1	388.53	24.66	92	444.39	25.50	90	14.4	3.4	-2.2	weight unstable after 15 min exposure
	2	389.27	24.61	88	447.41	25.43	93	14.9	3.3	5.7	weight unstable after 15 min exposure
	3	390.37	24.70	88	446.38	25.89	93	14.3	4.8	5.7	weight unstable after 15 min exposure
	Control	388.61	24.69	87	388.63	24.66	90	0.0	-0.1	3.4	
C. NBR (Buna N) 11/25/13	1	856.89	24.82	94	984.35	26.06	96	14.9	5.0	2.1	weight unstable after 15 min exposure
	2	841.22	24.59	92	965.59	26.39	95	14.8	7.3	3.3	weight unstable after 15 min exposure
	3	851.06	24.37	97	974.70	26.22	95	14.5	7.6	-2.1	weight unstable after 15 min exposure
	Control	841.11	24.01	95	841.19	25.26	96	0.0	5.2	1.1	

Notes

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
A. FKM Cpd. V0747-75 (Viton A) 11/21/13	1	433.04	20.71	7.9	2.7	423.63	20.86	5.6	3.4	
	2	430.50	20.85	7.9	3.6	421.20	20.72	5.5	3.0	
	3	436.58	20.90	7.5	2.9	427.58	20.85	5.3	2.6	
	Control	407.24	20.47	0.0	0.1	407.16	20.51	0.0	0.3	
B. FFKM (Kalrez) 11/25/13	1	418.08	25.98	7.6	5.4	407.83	25.27	5.0	2.5	
	2	419.76	25.88	7.8	5.1	408.91	25.87	5.0	5.1	
	3	420.25	25.93	7.7	5.0	409.85	25.59	5.0	3.6	
	Control	388.62	25.29	0.0	2.4	388.57	25.19	0.0	2.0	
C. NBR (Buna N) 11/25/13	1	913.65	25.76	6.6	3.8	883.14	25.29	3.1	1.9	
	2	896.61	25.90	6.6	5.3	866.63	25.40	3.0	3.3	
	3	906.93	25.85	6.6	6.1	877.10	25.41	3.1	4.3	
	Control	841.29	25.59	0.0	6.6	841.32	25.27	0.0	5.2	

Notes

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	**Sample**	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
A. FKM Cpd. V0747-75 (Viton A) 11/21/13	1	12.6	5.0	7.8	3.0	5.5	2.7
	2						
	3						
	Control						
B. FFKM (Kalrez) 11/25/13	1	14.5	4.0	7.7	2.7	5.0	1.7
	2						
	3						
	Control						
C. NBR (Buna N) 11/25/13	1	14.7	1.4	6.6	-1.5	3.0	-2.1
	2						
	3						
	Control						

Notes

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
D. PTFE Algoflon 11/25/13	1	541.33	20.56		542.46	20.93		0.2	1.8		
	2	527.96	20.69		529.18	20.74		0.2	0.2		
	3	518.95	20.64		520.15	20.74		0.2	0.5		
	Control	527.01	20.84		526.99	20.79		0.0	-0.2		
E. FEP Teflon 11/25/13	1	411.73	25.58		413.63	24.95		0.5	-2.5		
	2	402.48	25.40		404.30	25.39		0.5	-0.1		
	3	414.73	25.50		416.60	25.31		0.5	-0.8		
	Control	412.84	25.55		412.86	25.56		0.0	0.1		
F. PCTFE 11/25/13	1	602.93	20.83		603.95	20.79		0.2	-0.2		
	2	605.56	20.62		606.60	20.71		0.2	0.4		
	3	594.88	20.65		595.99	20.67		0.2	0.1		
	Control	594.01	20.74		594.02	20.66		0.0	-0.4		

Notes: Hardness measurements not applicable to materials on this sheet.

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	**Sample**	**24 hour weight**	**24 hour diameter**	**24 hour % change weight**	**24 hour % change diameter**	**7 day weight**	**7 day diameter**	**7 day % change weight**	**7 day % change diameter**	**Observed Visual Changes**
D. PTFE Algoflon 11/25/13	1		20.65		0.4					
	2									
	3									
	Control									
E. FEP Teflon 11/25/13	1		25.45		-0.5					
	2									
	3									
	Control									
F. PCTFE 11/25/13	1									
	2									
	3									
	Control									

Notes: Ha

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

HCFC-225cb Replacement Study

Material:	**Sample**	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
D. PTFE Algoflon 11/25/13	1	0.2	1.1		0.4		
	2						
	3						
	Control						
E. FEP Teflon 11/25/13	1	0.5	-1.2		-0.5		
	2						
	3						
	Control						
F. PCTFE 11/25/13	1	0.2	0.5				
	2						
	3						
	Control						

Notes: Ha

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
G. Vespel SP21 11/25/13	1	427.58	20.65		427.61	20.78		0.0	0.6		Floated in tube. Did NOT float after boil.
	2	426.40	20.77		426.42	20.85		0.0	0.4		Floated in tube. Did NOT float after boil.
	3	425.96	20.77		425.98	20.77		0.0	0.0		Floated in tube. Did NOT float after boil.
	Control	423.68	20.78		423.74	20.81		0.0	0.1		Floated in tube.
H. PEEK 11/25/13	1	366.71	20.83		366.73	20.76		0.0	-0.3		Floated in tube before and after boil.
	2	374.34	20.78		374.33	20.78		0.0	0.0		Floated in tube before and after boil.
	3	367.69	20.80		367.69	20.88		0.0	0.4		Floated in tube before and after boil.
	Control	371.19	20.83		371.21	20.87		0.0	0.2		Floated in tube.
I. Gylon 11/25/13	1	500.73	18.95		501.22	19.14		0.1	1.0		
	2	495.21	19.07		496.12	18.99		0.2	-0.4		
	3	529.86	19.08		531.11	19.29		0.2	1.1		
	Control	532.55	19.32		532.56	19.16		0.0	-0.8		

Notes: Hardness measurements not applicable to materials on this sheet.

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

HCFC-225cb Replacement Study

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
G. Vespel SP21 11/25/13	1									
	2									
	3									
	Control									
H. PEEK 11/25/13	1									
	2									
	3									
	Control									
I. Gylon 11/25/13	1		18.97		0.1					
	2									
	3		19.00		-0.4					
	Control									

Notes: Har

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

HCFC-225cb Replacement Study

Material:	**Sample**	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
G. Vespel SP21 11/25/13	1	0.0	0.2				
	2						
	3						
	Control						
H. PEEK 11/25/13	1	0.0	-0.2				
	2						
	3						
	Control						
I. Gylon 11/25/13	1	0.2	1.4		-0.2		
	2						
	3						
	Control						

Notes: Har

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

HCFC-225cb Replacement Study

Material:	**Sample**	**Initial weight mg**	**Initial diameter mm**	**Initial Shore A hardness**	**Final weight**	**Final diameter mm**	**Final Shore A hardness**	**% change weight**	**% change diameter**	**% change hardness**	**Observed Visual Changes**
Repeat of A. FKM V0747-75 (Viton A) 12/11/13	1	403.31	20.59	75	468.44	21.13	70	16.1	2.6	-6.7	weight unstable after 15 min exposure
	2	399.57	20.42	72	463.64	21.64	71	16.0	6.0	-1.4	weight unstable after 15 min exposure
	3	398.94	20.32	75	462.46	21.54	73	15.9	6.0	-2.7	weight unstable after 15 min exposure
	Control	404.13	20.65	73	404.16	20.65	77	0.0	0.0	5.5	
	1										
	2										
	3										
	Control										
	1										
	2										
	3										
	Control										

Notes

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
Repeat of A. FKM V0747-75 (Viton A) 12/11/13	1	441.18	21.30	9.4	3.4	430.20	20.95	6.7	1.7	
	2	437.17	21.19	9.4	3.8	426.25	20.89	6.7	2.3	
	3	436.27	21.19	9.4	4.3	425.46	20.82	6.6	2.5	
	Control	404.08	20.70	0.0	0.2	403.99	20.35	0.0	-1.5	
	1									
	2									
	3									
	Control									
	1									
	2									
	3									
	Control									

Notes

Solvent: AK-225G

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	**Sample**	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
Repeat of A. FKM V0747-75 (Viton A) 12/11/13	1	16.0	4.9	9.4	3.6	6.7	3.7
	2						
	3						
	Control						
	1						
	2						
	3						
	Control						
	1						
	2						
	3						
	Control						

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
A. FKM Cpd. V0747-75 (Viton A) 12/4/13	1	399.31	20.49	97	449.00	21.46	96	12.4	4.7	-1.0	weight unstable after 15 min exposure
	2	399.27	20.47	97	448.48	21.48	97	12.3	5.0	0.0	weight unstable after 15 min exposure
	3	400.38	20.48	91	450.03	21.61	94	12.4	5.5	3.3	weight unstable after 15 min exposure
	Control	400.75	20.45	92	400.75	20.54	100	0.0	0.5	8.7	
B. FFKM (Kalrez) 12/4/13	1	387.90	25.09	88	404.19	25.47	90	4.2	1.5	2.3	
	2	390.64	24.75	94	407.17	24.77	91	4.2	0.1	-3.2	
	3	391.34	25.35	94	407.52	25.61	98	4.1	1.0	4.3	
	Control	391.20	25.41	95	391.23	25.36	99	0.0	-0.2	4.2	
C. NBR (Buna N) 12/4/13	1	849.05	25.57	93	897.43	25.82	94	5.7	1.0	1.1	weight unstable after 15 min exposure
	2	850.67	25.56	94	899.33	25.85	95	5.7	1.1	1.1	weight unstable after 15 min exposure
	3	854.01	25.48	94	901.63	25.87	97	5.6	1.6	3.2	weight unstable after 15 min exposure
	Control	853.24	25.42	96	853.39	25.56	95	0.0	0.6	-1.0	

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
A. FKM Cpd. V0747-75 (Viton A) 12/4/13	1	424.07	21.01	6.2	2.5	413.45	20.71	3.5	1.1	
	2	423.97	20.93	6.2	2.2	413.36	20.55	3.5	0.4	
	3	425.29	20.90	6.2	2.1	414.55	20.71	3.5	1.1	
	Control					400.68	20.53	0.0	0.4	
B. FFKM (Kalrez) 12/4/13	1	396.12	24.89	2.1	-0.8	392.87	25.25	1.3	0.6	
	2	398.99	25.13	2.1	1.5	395.67	25.14	1.3	1.6	
	3	399.56	24.83	2.1	-2.1	396.30	25.61	1.3	1.0	
	Control					391.07	25.22	0.0	-0.8	
C. NBR (Buna N) 12/4/13	1	862.83		1.6		847.96	25.52	-0.1	-0.2	
	2	864.79	25.45	1.7	-0.4	849.73	25.48	-0.1	-0.3	
	3	867.85	25.36	1.6	-0.5	853.01	25.51	-0.1	0.1	
	Control					852.56	25.41	-0.1	0.0	

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
A. FKM Cpd. V0747-75 (Viton A) 12/4/13	1	12.4	4.6	6.2	2.3	3.6	0.4
	2						
	3						
	Control						
B. FFKM (Kalrez) 12/4/13	1	4.2	1.1	2.1	-0.4	1.3	1.8
	2						
	3						
	Control						
C. NBR (Buna N) 12/4/13	1	5.6	0.6	1.6	-0.5	0.0	-0.1
	2						
	3						
	Control						

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
D. PTFE Algoflon 12/4/13	1	520.94	20.78		521.77	20.86		0.2	0.4		
	2	520.16	20.80		520.95	20.83		0.2	0.2		
	3	522.24	20.81		523.03	20.82		0.2	0.0		
	Control	507.14	20.83		507.15	20.77		0.0	-0.3		
E. FEP Teflon 12/4/13	1	405.54	25.39		406.75	25.34		0.3	-0.2		
	2	412.96	25.53		414.23	25.44		0.3	-0.3		
	3	409.02	25.51		410.26	25.40		0.3	-0.5		
	Control	410.85	25.57		410.86	25.52		0.0	-0.2		
F. PCTFE 12/4/13	1	595.35	20.63		595.74	20.64		0.1	0.1		
	2	599.11	20.58		599.39	20.61		0.0	0.1		
	3	581.47	20.25		581.86	20.58		0.1	1.7		
	Control	589.64	20.67		589.68	20.66		0.0	0.0		

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

HCFC-225cb Replacement Study

Material:	**Sample**	**24 hour weight**	**24 hour diameter**	**24 hour % change weight**	**24 hour % change diameter**	**7 day weight**	**7 day diameter**	**7 day % change weight**	**7 day % change diameter**	**Observed Visual Changes**
D. PTFE Algoflon 12/4/13	1									
	2									
	3									
	Control									
E. FEP Teflon 12/4/13	1									
	2									
	3									
	Control									
F. PCTFE 12/4/13	1									
	2									
	3	581.76	20.59	0.0	1.7	581.68	20.65	0.0	2.0	
	Control					589.66	20.61	0.0	-0.3	

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

HCFC-225cb Replacement Study

Material:	**Sample**	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
D. PTFE Algoflon 12/4/13	1	0.2	0.5	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
E. FEP Teflon 12/4/13	1	0.3	-0.2	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
F. PCTFE 12/4/13	1	0.1	0.7	0.0	1.7	0.0	2.3
	2						
	3						
	Control						

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
G. Vespel SP21 12/4/13	1	426.16	20.77		426.55	20.66		0.1	-0.6		
	2	426.26	20.80		426.64	20.58		0.1	-1.1		
	3	425.91	20.73		426.25	20.71		0.1	-0.1		
	Control	429.62	20.81		429.79	20.77		0.0	-0.2		
H. PEEK 12/4/13	1	366.29	20.65		366.36	20.80		0.0	0.7		
	2	353.07	20.70		353.12	20.81		0.0	0.5		
	3	359.29	20.76		359.36	20.78		0.0	0.1		
	Control	370.26	20.90		370.32	20.80		0.0	-0.5		
I. Gylon 12/4/13	1	517.79	19.20		517.76	19.27		0.0	0.4		
	2	535.26	17.21		535.28	19.38		0.0	12.6		
	3	499.78	17.74		499.77	19.16		0.0	8.0		
	Control	549.20	19.19		549.16	19.04		0.0	-0.8		

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
G. Vespel SP21 12/4/13	1									
	2	426.36	20.82	0.0	0.1					
	3									
	Control									
H. PEEK 12/4/13	1									
	2									
	3									
	Control									
I. Gylon 12/4/13	1									
	2	535.22	19.19	0.0	11.5	535.16	19.12	0.0	11.1	
	3	499.73	19.12	0.0	7.8	499.67	19.06	0.0	7.5	
	Control					549.11		0.0	0.0	

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
G. Vespel SP21 12/4/13	1	0.0	-0.4	0.0	0.1		
	2						
	3						
	Control						
H. PEEK 12/4/13	1	0.0	0.9				
	2						
	3						
	Control						
I. Gylon 12/4/13	1	0.0	7.8	0.0	9.6	0.0	9.3
	2						
	3						
	Control						

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
Rerun of I. Gylon 12/10/13	1	505.44	19.22		505.37	18.95		0.0	-1.4		
	2	518.35	19.12		518.31	19.16		0.0	0.2		
	3	523.05	19.21		523.03	19.14		0.0	-0.4		
	Control	482.91	19.16		482.87	18.98		0.0	-0.9		
Second Rerun of I. Gylon 12/17/13	1	500.65	19.02		500.64	19.07		0.0	0.2		
	2	500.80	19.05		500.78	18.77		0.0	-1.5		
	3	496.88	19.12		496.84	18.95		0.0	-0.9		
	Control	520.23	19.09		520.22	19.11		0.0	0.1		
	1										
	2										
	3										
	Control										

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter		6 day diameter		6 day % change diameter	Observed Visual Changes
Rerun of I. Gylon 12/10/13	1						19.10		-0.6	
	2									
	3									
	Control						19.04		-0.6	
Second Rerun of I. Gylon 12/17/13	1	500.72	18.88	0.0	-0.7					
	2									
	3									
	Control	520.21	19.08	0.0	-0.1					
	1									
	2									
	3									
	Control									

Notes

Solvent: Solstice PF

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
Rerun of I. Gylon 12/10/13	1	0.0	0.4				
	2						
	3						
	Control						
Second Rerun of I. Gylon 12/17/13	1	0.0	-0.8	0.0	-0.7		
	2						
	3						
	Control						
	1						
	2						
	3						
	Control						

Notes

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
A. FKM Cpd. V0747-75 (Viton A) 12/10 /13	1	400.81	20.61	71	424.44	20.97	63	5.9	1.7	-11.3	weight unstable after 15 min exposure
	2	402.04	20.61	72	425.87	20.85	65	5.9	1.1	-9.7	weight unstable after 15 min exposure
	3	397.99	20.52	73	421.29	20.95	68	5.9	2.1	-6.8	weight unstable after 15 min exposure
	Control	402.73	20.48	75	402.75	20.44	71	0.0	-0.2	-5.3	
B. FFKM (Kalrez) 12/10 /13	1	387.42	24.95	81	410.83	25.51	71	6.0	2.2	-12.3	weight unstable after 15 min exposure
	2	391.42	24.68	79	414.84	25.62	71	6.0	3.8	-10.1	weight unstable after 15 min exposure
	3	387.23	24.59	79	410.56	25.35	72	6.0	3.1	-8.9	weight unstable after 15 min exposure
	Control	390.93	24.79	79	390.94	24.84	81	0.0	0.2	2.5	
C. NBR (Buna N) 12/10 /13	1	855.45	25.45	67	910.29	25.75	66	6.4	1.2	-1.5	weight unstable after 15 min exposure
	2	848.72	25.32	70	902.88	25.78	67	6.4	1.8	-4.3	weight unstable after 15 min exposure
	3	846.92	24.90	70	899.78	25.90	66	6.2	4.0	-5.7	weight unstable after 15 min exposure
	Control	852.16	25.35	72	852.22	25.42	70	0.0	0.3	-2.8	

Notes

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
A. FKM Cpd. V0747-75 (Viton A) 12/10 /13	1	415.13	20.85	3.6	1.2	410.87	20.70	2.5	0.4	
	2	416.56	20.78	3.6	0.8	412.23	20.67	2.5	0.3	
	3	412.24	20.84	3.6	1.6	408.00	20.61	2.5	0.4	
	Control	402.65	20.51	0.0	0.1	402.57	20.41	0.0	-0.3	
B. FFKM (Kalrez) 12/10 /13	1	401.43	25.42	3.6	1.9	396.98	25.27	2.5	1.3	
	2	405.55	25.56	3.6	3.6	401.10	25.47	2.5	3.2	
	3	401.23	25.66	3.6	4.3	396.72	25.44	2.5	3.5	
	Control	390.86	25.14	0.0	1.4	390.72	25.11	-0.1	1.3	
C. NBR (Buna N) 12/10 /13	1	869.92	25.48	1.7	0.1	853.35	25.36	-0.2	-0.4	
	2	863.30	25.52	1.7	0.8	846.90	25.37	-0.2	0.2	
	3	861.26	25.37	1.7	1.9	845.24	25.15	-0.2	1.0	
	Control	852.14	25.43	0.0	0.3	852.53	25.32	0.0	-0.1	

Notes

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
A. FKM Cpd. V0747-75 (Viton A) 12/10 /13	1	5.9	1.8	3.6	1.0	2.6	0.7
	2						
	3						
	Control						
B. FFKM (Kalrez) 12/10 /13	1	6.0	2.9	3.6	1.9	2.5	1.4
	2						
	3						
	Control						
C. NBR (Buna N) 12/10 /13	1	6.3	2.0	1.7	0.6	-0.3	0.4
	2						
	3						
	Control						

Notes

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
D. PTFE Algoflon 12/10 /13	1	516.57	20.73		517.10	20.73		0.1	0.0		
	2	515.17	20.72		515.71	20.73		0.1	0.0		
	3	521.58	20.85		522.13	20.85		0.1	0.0		
	Control	521.45	20.79		521.44	20.81		0.0	0.1		
E. FEP Teflon 12/10 /13	1	411.60	25.56		412.79	25.58		0.3	0.1		
	2	413.00	25.56		414.17	25.38		0.3	-0.7		
	3	410.53	25.58		411.68	25.41		0.3	-0.7		
	Control	411.24	25.56		411.23	25.50		0.0	-0.3		
F. PCTFE 12/10 /13	1	588.73	20.63		589.00	20.62		0.0	0.0		
	2	599.71	20.71		599.93	20.71		0.0	0.0		
	3	583.58	20.69		583.91	20.74		0.1	0.2		
	Control	589.29	20.63		589.29	20.66		0.0	0.1		
	Notes										

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
D. PTFE Algoflon 12/10 /13	1									
	2									
	3									
	Control									
E. FEP Teflon 12/10 /13	1									
	2									
	3									
	Control									
F. PCTFE 12/10 /13	1									
	2									
	3									
	Control									
	Notes									

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
D. PTFE Algoflon 12/10 /13	1	0.1	0.0	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
E. FEP Teflon 12/10 /13	1	0.3	-0.2	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
F. PCTFE 12/10 /13	1	0.0	-0.1	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						

Notes

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
G. Vespel SP21 12/10/13	1	428.62	20.88		429.27	20.78		0.2	-0.5		
	2	429.71	20.83		430.32	20.83		0.1	0.0		
	3	421.13	20.80		421.65	20.79		0.1	0.0		
	Control	428.79	20.78		428.83	20.75		0.0	-0.1		
H. PEEK 12/10/13	1	367.51	20.80		367.72	20.79		0.1	0.0		
	2	368.44	20.81		368.65	20.83		0.1	0.1		
	3	371.61	20.80		371.85	20.77		0.1	-0.2		
	Control	372.07	20.80		372.07	20.76		0.0	-0.2		
I. Gylon 12/10/13	1	520.54	19.23		521.15	19.04		0.1	-1.0		
	2	495.97	19.15		496.45	19.01		0.1	-0.7		
	3	537.41	19.02		538.02	19.07		0.1	0.2		
	Control	498.75	19.11		498.73	19.10		0.0	-0.1		

Notes

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
G. Vespel SP21 12/10/13	1									
	2									
	3									
	Control									
H. PEEK 12/10/13	1									
	2									
	3									
	Control									
I. Gylon 12/10/13	1									
	2									
	3									
	Control									

Notes

Solvent: L-14780

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	**Sample**	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
G. Vespel SP21 12/10/13	1	0.1	0.0	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
H. PEEK 12/10/13	1	0.1	0.2	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
I. Gylon 12/10/13	1	0.1	-0.4	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						

Notes

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
A. FKM V0747-75 (Viton A) 12/12/13	1	400.35	20.37	73	472.79	22.17	71	18.1	8.8	-2.7	weight unstable after 15 min exposure
	2	400.11	20.40	73	471.60	22.26	69	17.9	9.1	-5.5	weight unstable after 15 min exposure
	3	406.79	20.53	75	477.34	22.27	68	17.3	8.5	-9.3	weight unstable after 15 min exposure
	Control	407.51	20.45	74	407.51	20.55	75	0.0	0.5	1.4	
B. FFKM (Kalrez) 12/12/13	1	389.70	25.07	82	395.86	25.09	79	1.6	0.1	-3.7	
	2	387.49	24.97	81	394.58	25.07	79	1.8	0.4	-2.5	
	3	388.81	25.32	80	395.92	25.30	80	1.8	-0.1	0.0	
	Control	390.89	24.83	79	390.89	24.76	80	0.0	-0.3	1.3	
C. NBR (Buna N) 12/12/13	1	838.93	25.41	69	947.80	26.06	62	13.0	2.6	-10.1	weight unstable after 15 min exposure
	2	850.75	25.36	70	958.73	26.26	64	12.7	3.5	-8.6	weight unstable after 15 min exposure
	3	855.40	25.29	70	967.03	26.35	62	13.1	4.2	-11.4	weight unstable after 15 min exposure
	Control	833.72	25.32	68	833.75	25.46	69	0.0	0.5	1.5	
	Notes										

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
A. FKM V0747-75 (Viton A) 12/12/13	1	438.02	21.36	9.4	4.8	424.26	20.95	6.0	2.8	
	2	437.57	21.47	9.4	5.2	423.93	21.03	6.0	3.1	
	3	444.25	21.53	9.2	4.9	430.91	21.16	5.9	3.1	
	Control	407.43	20.48	0.0	0.2	407.36	20.45	0.0	0.0	
B. FFKM (Kalrez) 12/12/13	1	393.42	25.85	1.0	3.1	392.06	25.05	0.6	-0.1	
	2	391.72	25.94	1.1	3.9	390.24	25.00	0.7	0.1	
	3	393.05	25.56	1.1	0.9	391.56	25.08	0.7	-0.9	
	Control	390.86	24.89	0.0	0.2	390.73	25.01	0.0	0.7	
C. NBR (Buna N) 12/12/13	1	871.96	25.77	3.9	1.4	843.91	25.20	0.6	-0.8	
	2	883.60	25.68	3.9	1.3	855.50	25.18	0.6	-0.7	
	3	889.50	25.88	4.0	2.3	860.64	25.25	0.6	-0.2	
	Control	833.52	25.58	0.0	1.0	833.00	25.25	-0.1	-0.3	

Notes

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
A. FKM V0747-75 (Viton A) 12/12/13	1	17.8	8.3	9.3	4.8	6.0	3.0
	2						
	3						
	Control						
B. FFKM (Kalrez) 12/12/13	1	1.7	0.4	1.1	2.4	0.7	-1.0
	2						
	3						
	Control						
C. NBR (Buna N) 12/12/13	1	12.9	2.9	4.0	0.6	0.7	-0.3
	2						
	3						
	Control						

Notes

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
D. PTFE Algoflon 12/12/13	1	524.80	20.80		525.11	20.86		0.1	0.3		
	2	523.50	20.78		523.83	20.78		0.1	0.0		
	3	528.00	20.83		528.34	20.84		0.1	0.0		
	Control	528.67	20.84		528.64	20.64		0.0	-1.0		
E. FEP Teflon 12/12/13	1	418.41	25.52		418.95	25.47		0.1	-0.2		
	2	412.81	25.43		413.34	25.46		0.1	0.1		
	3	414.88	25.46		415.41	25.29		0.1	-0.7		
	Control	412.62	25.45		412.63	25.48		0.0	0.1		
F. PCTFE 12/12/13	1	598.29	20.63		598.52	20.66		0.0	0.1		
	2	602.07	20.65		602.27	20.73		0.0	0.4		
	3	596.43	20.67		596.65	20.67		0.0	0.0		
	Control	596.38	20.65		596.37	20.64		0.0	0.0		

Notes

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
D. PTFE Algoflon 12/12/13	1									
	2									
	3									
	Control									
E. FEP Teflon 12/12/13	1									
	2									
	3									
	Control									
F. PCTFE 12/12/13	1									
	2									
	3									
	Control									

Notes

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
D. PTFE Algoflon 12/12/13	1	0.1	1.1	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
E. FEP Teflon 12/12/13	1	0.1	-0.4	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
F. PCTFE 12/12/13	1	0.0	0.2	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						

Notes

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial weight mg	Initial diameter mm	Initial Shore A hardness	Final weight	Final diameter mm	Final Shore A hardness	% change weight	% change diameter	% change hardness	Observed Visual Changes
G. Vespel SP21 12/12/13	1	432.36	20.81		433.19	20.79		0.2	-0.1		
	2	425.11	20.80		426.94	20.79		0.4	0.0		
	3	428.12	20.78		428.86	20.77		0.2	0.0		
	Control	428.58	20.80		428.60	20.81		0.0	0.0		
H. PEEK 12/12/13	1	366.72	20.78		367.00	20.74		0.1	-0.2		
	2	368.59	20.79		368.80	20.79		0.1	0.0		
	3	371.99	20.79		372.18	20.79		0.1	0.0		
	Control	369.42	20.76		369.40	20.77		0.0	0.0		
I. Gylon 12/12/13	1	495.27	18.91		495.45	19.13		0.0	1.2		
	2	527.50	19.04		527.76	19.02		0.0	-0.1		
	3	497.68	19.14		497.89	19.19		0.0	0.2		
	Control	492.45	19.10		492.47	19.09		0.0	-0.1		

Notes

Solvent: Solvokane

NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	24 hour weight	24 hour diameter	24 hour % change weight	24 hour % change diameter	7 day weight	7 day diameter	7 day % change weight	7 day % change diameter	Observed Visual Changes
G. Vespel SP21 12/12/13	1									
	2									
	3									
	Control									
H. PEEK 12/12/13	1									
	2									
	3									
	Control									
I. Gylon 12/12/13	1	495.25	18.93	0.0	0.1					
	2									
	3									
	Control	492.46	19.12	0.0	0.1					

Notes

Solvent: Solvokane

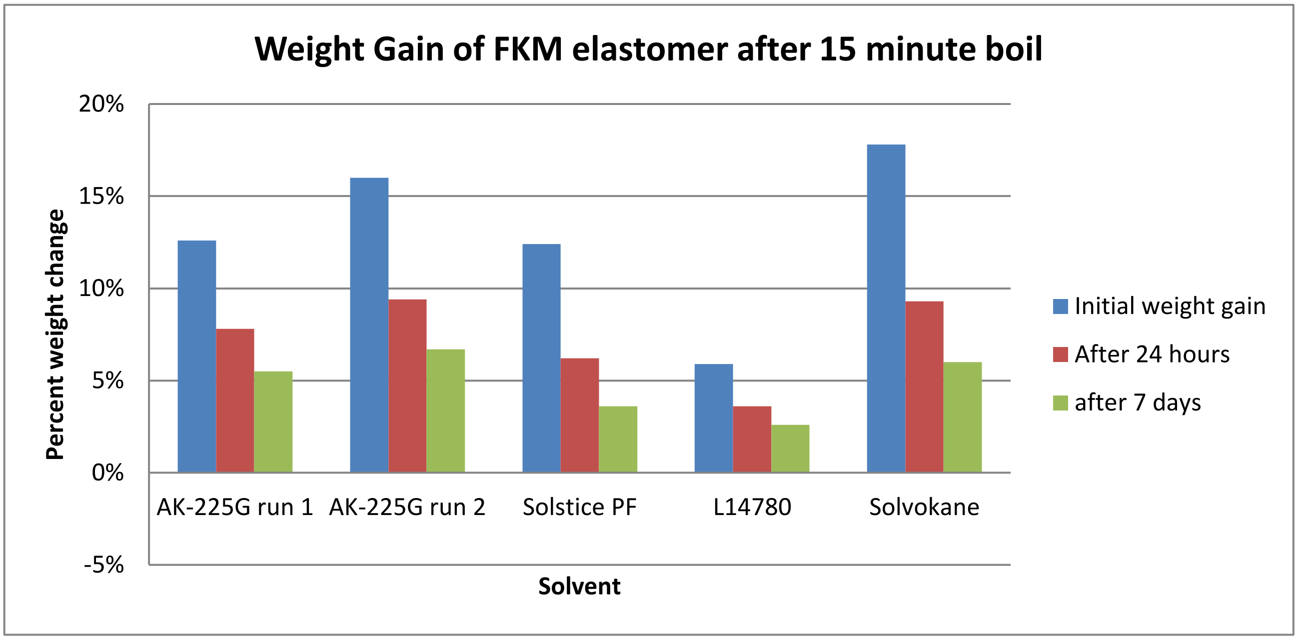
NONMETALS COMPATIBILITY IMMERSION TEST

Material:	Sample	Initial ave. weight % change	Initial ave. diameter % change	24 hour ave. weight % change	24 hour ave. diameter % change	7 day ave. weight % change	7 day ave. diameter % change
G. Vespel SP21 12/12/13	1	0.3	-0.1	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
H. PEEK 12/12/13	1	0.1	-0.1	#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!
	2						
	3						
	Control						
I. Gylon 12/12/13	1	0.0	0.5	0.0	0.1	#DIV/0!	#DIV/0!
	2						
	3						
	Control						

Notes

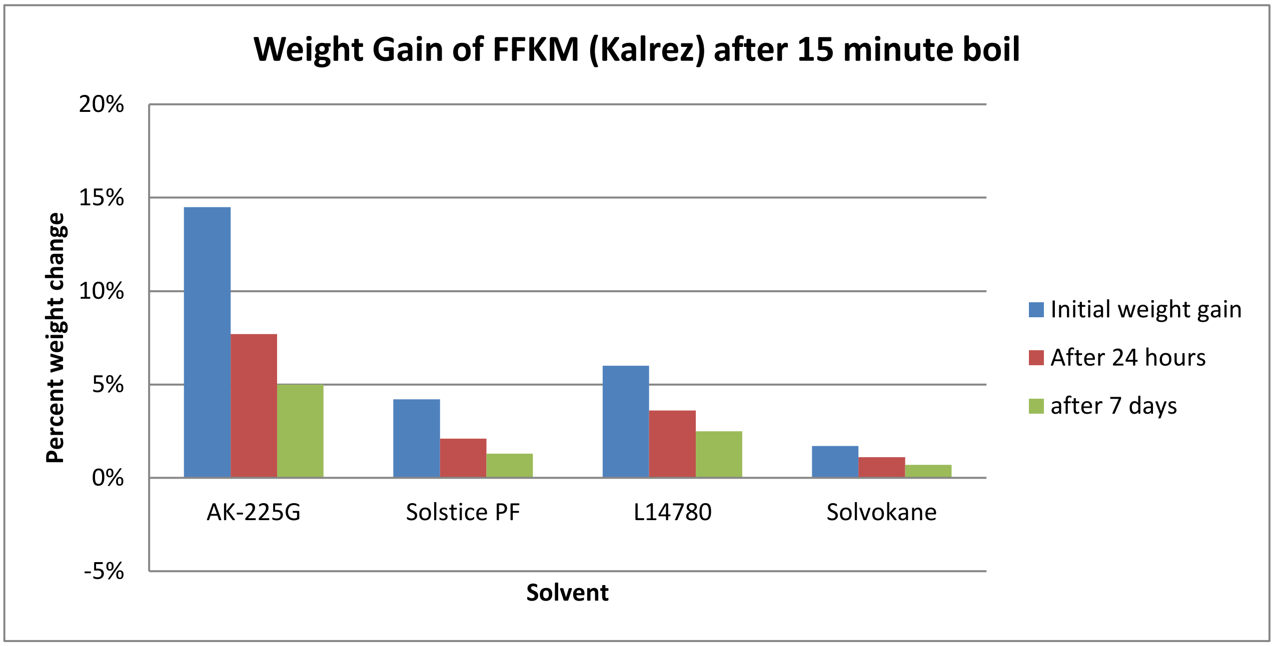
NONMETALS COMPATIBILITY IMMERSION TEST

	% weight change	24 hour weight change	7 day weight change
AK-225G run 1	12.6%	7.8%	5.5%
AK-225G run 2	16.0%	9.4%	6.7%
Solstice PF	12.4%	6.2%	3.6%
L14780	5.9%	3.6%	2.6%
Solvokane	17.8%	9.3%	6.0%



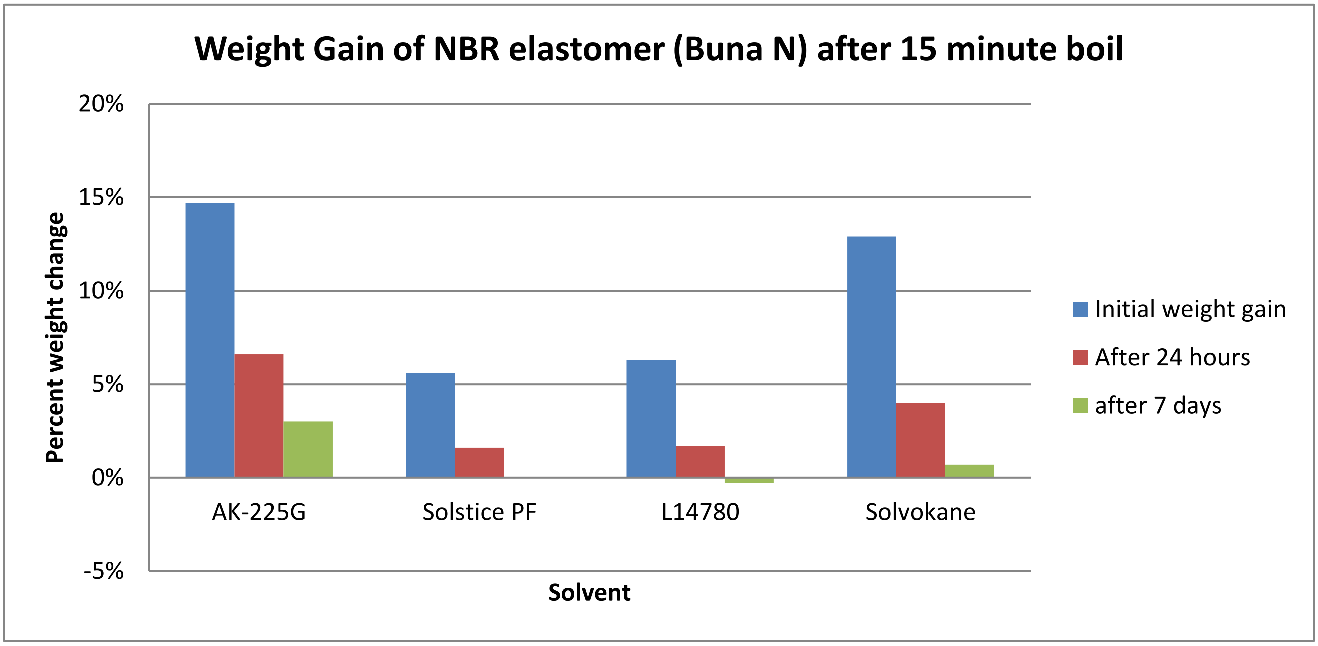
NONMETALS COMPATIBILITY IMMERSION TEST

	% weight change	24 hour weight change	7 day weight change
AK-225G	14.5%	7.7%	5.0%
Solstice PF	4.2%	2.1%	1.3%
L14780	6.0%	3.6%	2.5%
Solvokane	1.7%	1.1%	0.7%

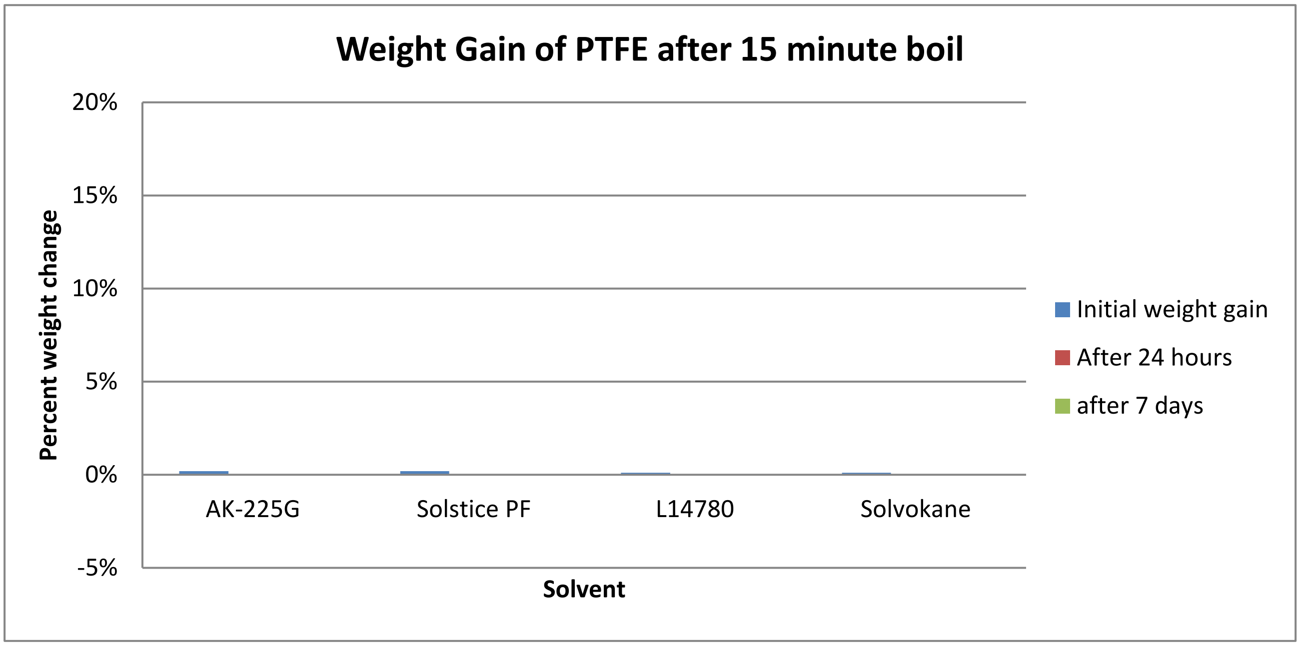


NONMETALS COMPATIBILITY IMMERSION TEST

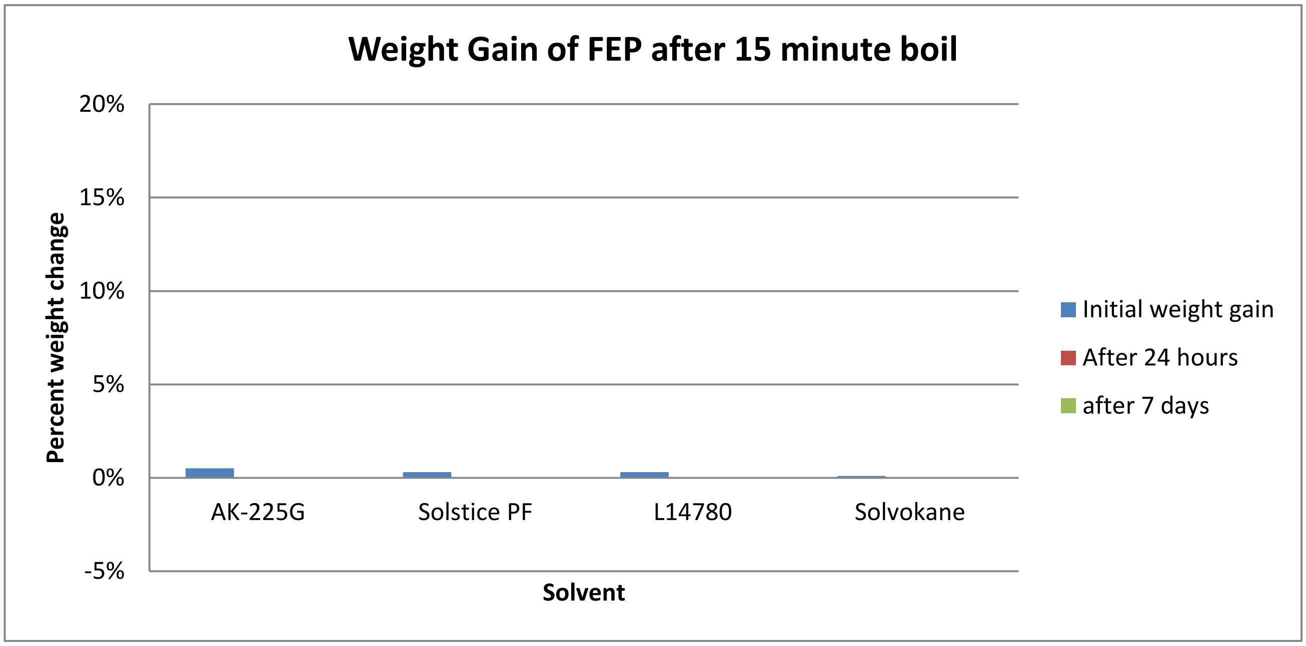
	% weight change	24 hour weight change	7 day weight change
AK-225G	14.7%	6.6%	3.0%
Solstice PF	5.6%	1.6%	0.0%
L14780	6.3%	1.7%	-0.3%
Solvokane	12.9%	4.0%	0.7%



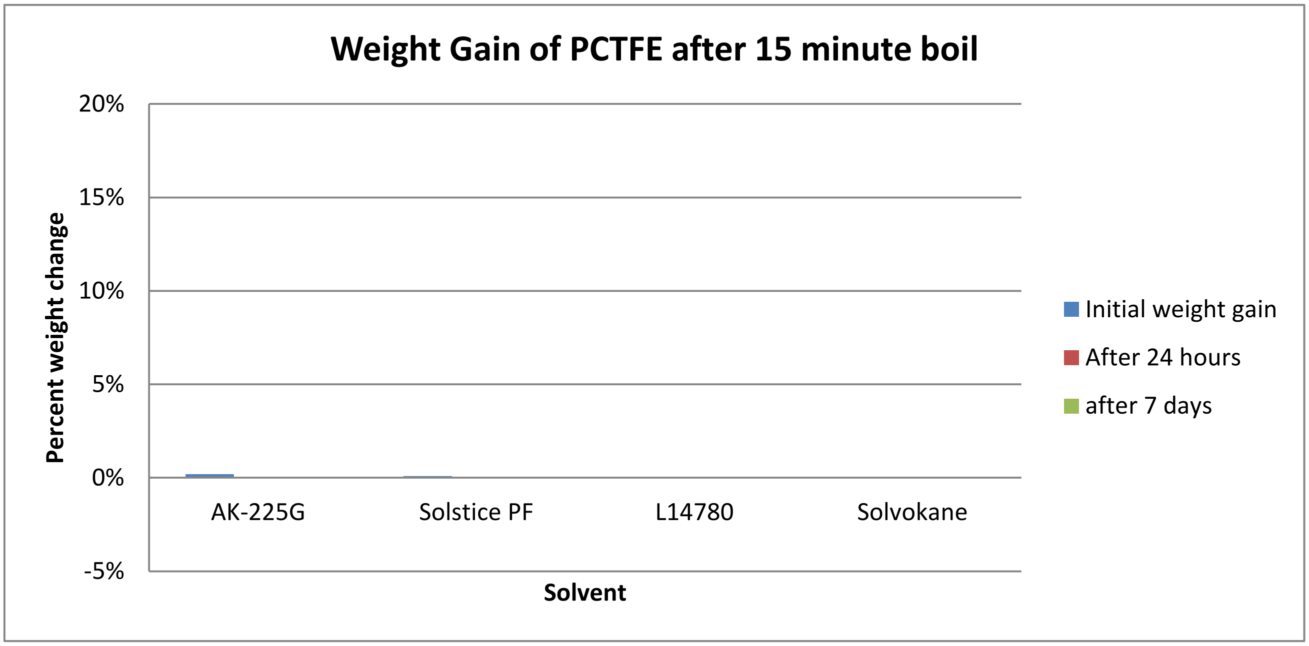
	% weight change	24 hour weight change	7 day weight change
AK-225G	0.2%		
Solstice PF	0.2%		
L14780	0.1%		
Solvokane	0.1%		



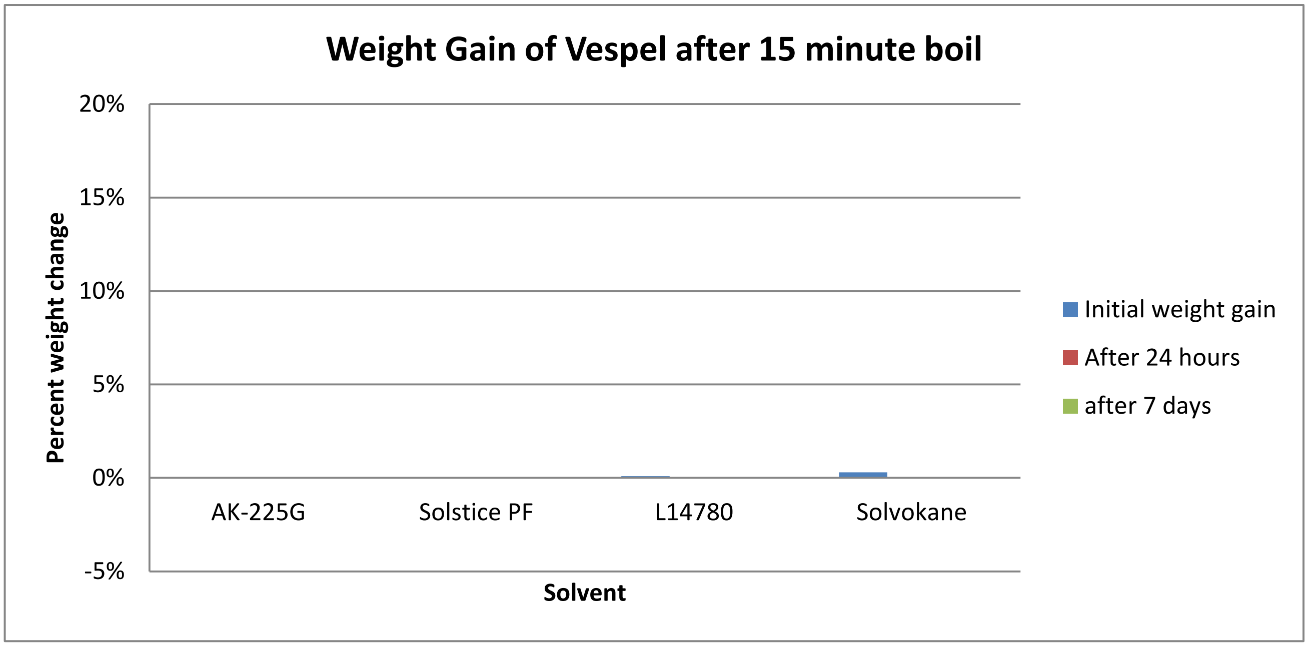
	% weight change	24 hour weight change	7 day weight change
AK-225G	0.5%		
Solstice PF	0.3%		
L14780	0.3%		
Solvokane	0.1%		



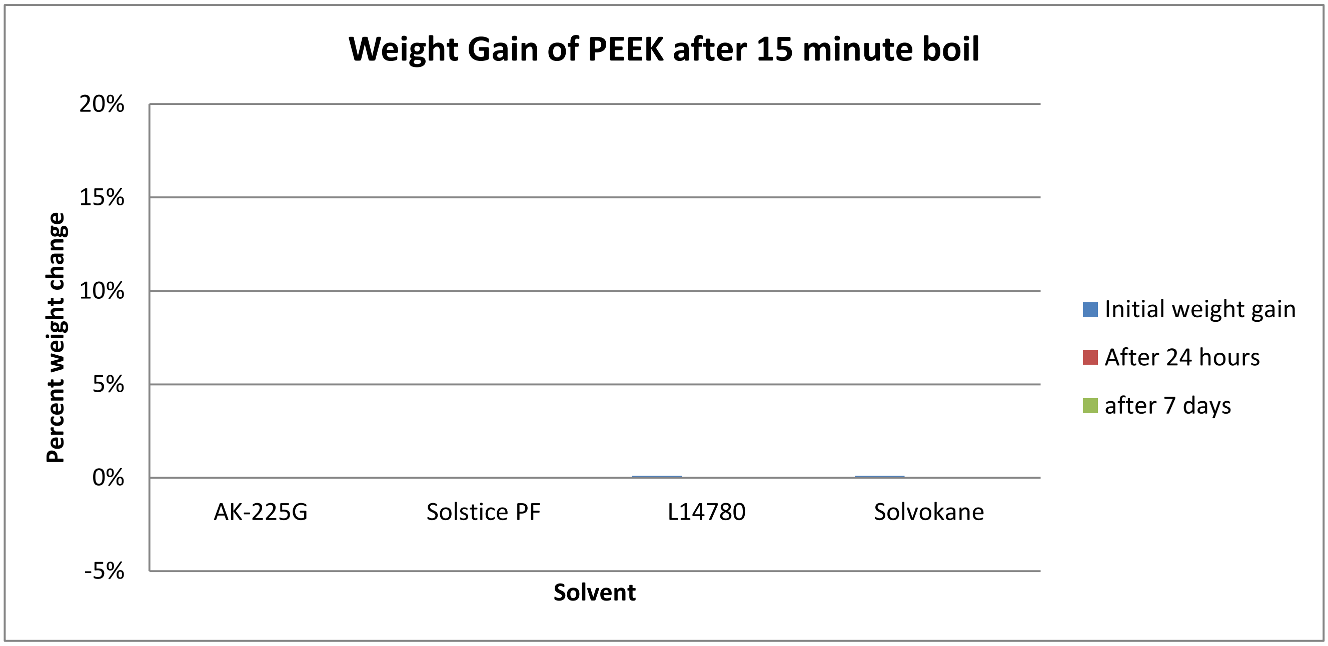
	% weight change	24 hour weight change	7 day weight change
AK-225G	0.2%		
Solstice PF	0.1%		
L14780	0.0%		
Solvokane	0.0%		



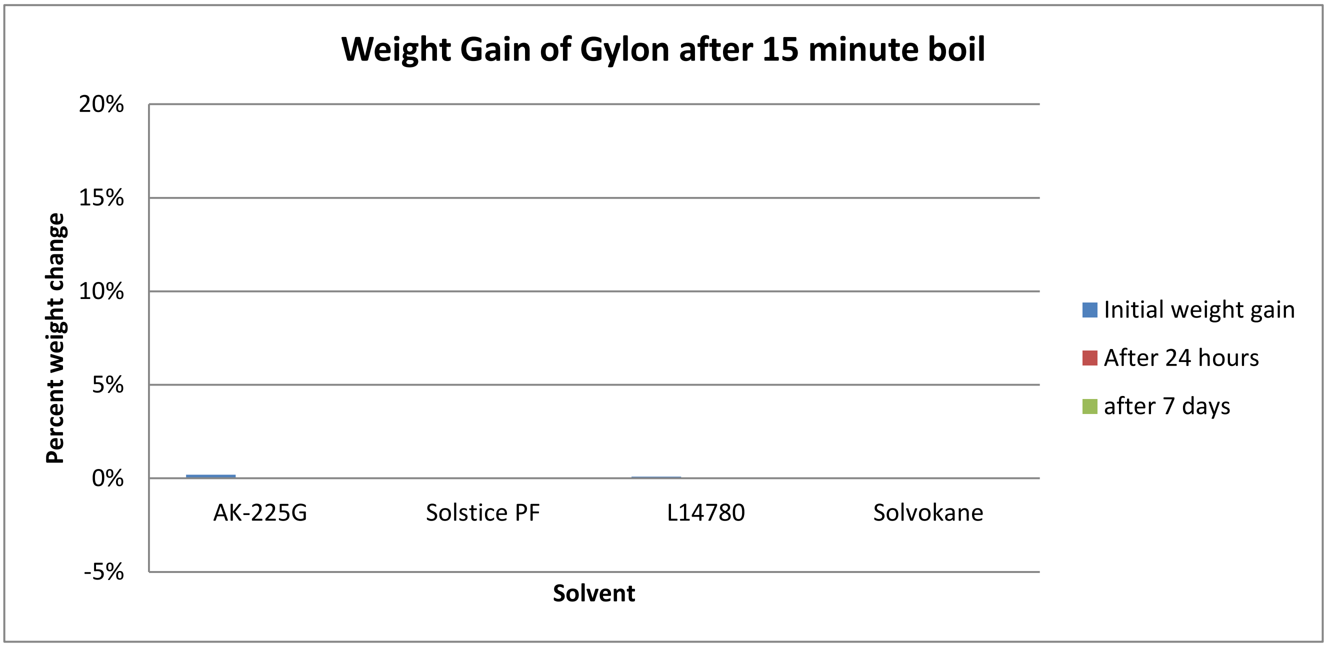
	% weight change	24 hour weight change	7 day weight change
AK-225G	0.0%		
Solstice PF	0.0%		
L14780	0.1%		
Solvokane	0.3%		



	% weight change	24 hour weight change	7 day weight change
AK-225G	0.0%		
Solstice PF	0.0%		
L14780	0.1%		
Solvokane	0.1%		



	% weight change	24 hour weight change	7 day weight change
AK-225G	0.2%		
Solstice PF	0.0%		
L14780	0.1%		
Solvokane	0.0%		



Return to Menu

APPENDIX G—INITIAL OXYGEN COMPATIBILITY TEST REPORTS

Various initial oxygen compatibility test reports can be found in the following sections within appendix G:

Return to App G Pg 22 Pg 23 Pg 24

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

May 8, 2013

Reply to Attn of: EM10 (013-287)
Ref. EM10B (13-005)

EM50/Mark Mitchell

Dear Mr. Mitchell,

The Solstice 1233ZD(E) solvent has been tested for Ambient Pressure Liquid Oxygen Mechanical Impact by the procedures outlined in NASA-STD-6001B, Test 13A. The material has also been tested for Autogenous Ignition Temperature (AIT) by the procedures outlined in ASTM G72-09.

Test parameters and results are provided in the enclosure.

Please direct any comments or questions regarding the quality or timeliness of this test or report to Eddie Davis at 256-544-2490 (eddie.davis@nasa.gov).

Cordially,

Gail H Gordon
Gail H. Gordon
Branch Chief
Materials Test Branch

Enclosures

cc:
EM10/File Copy
EM10/E. Davis
EM03/ERC/K. Baker
EM50/N. Lowrey

The material, test parameters and results are as follows:

Material:	Solstice™ 1233zd(E) Solvent
Manufacturer:	Honeywell, Performance Materials and Technologies
Composition:	Trans-1-Chloro-3,3,3-Trifluoropropene
Submitted by:	EM50/Mark Mitchell
Test Number:	M108709
Material Code:	08965

Mechanical Impact Test 13A Results are as follows:

Sample Thickness:	Approximately 0.05"
Test Conditions:	LOX
Test Temperature:	-297°F
Impact Energy:	72 ft-lbs
Pressure:	14.7 psia
Reactions/Tests:	0/20
Rating:	A
Notes:	Material Evaporated Quickly, Pretest Samples Stored in Laboratory Freezer

Mechanical Impact Bruceton Results are as follows:

Bruceton Testing:	No Reactions, Bruceton Not Performed

AIT Results are as follows:

Sample Weight:	0.50 grams		
Test Pressure:	50 psia		
Ignition Temperature Sample 1:	501 °F		
Ignition Temperature Sample 2:	519 °F		
Ignition Temperature Sample 3:	>800 °F		
Temperature Rise Sample 1:	113 °F	Final Pressure:	199 psig
Temperature Rise Sample 2:	125 °F	Final Pressure:	212 psig
Temperature Rise Sample 3:	No Ignition	Final Pressure:	160 psig
Residue:	Yes, Black		

Sample Weight:	0.50 grams		
Test Pressure:	2,000 psig		
Ignition Temperature Sample 1:	390 °F		
Ignition Temperature Sample 2:	384 °F		
Ignition Temperature Sample 3:	460 °F		
Temperature Rise Sample 1:	213 °F	Final Pressure:	2799 psig*
Temperature Rise Sample 2:	258 °F	Final Pressure:	2790 psig*
Temperature Rise Sample 3:	619 °F	Final Pressure:	3824 psig
Residue:	Yes, Red		

*Sample holder shattered.

Return to App G

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

May 8, 2013

Reply to Attn of: EM10 (013-286)
Ref. EM10B (13-004)

EM50/Mark Mitchell

Dear Mr. Mitchell,

The 3M L-14780 Developmental solvent has been tested for Ambient Pressure Liquid Oxygen Mechanical Impact by the procedures outlined in NASA-STD-6001B, Test 13A. The material has also been tested for Autogenous Ignition Temperature (AIT) by the procedures outlined in ASTM G72-09.

Test parameters and results are provided in the enclosure.

Please direct any comments or questions regarding the quality or timeliness of this test or report to Eddie Davis at 256-544-2490 (eddie.davis@nasa.gov).

Cordially,

Gail H Gordon
Gail H. Gordon
Branch Chief
Materials Test Branch

Enclosures

cc:
EM10/File Copy
EM10/E. Davis
EM03/ERC/K. Baker
EM50/N. Lowrey

The material, test parameters and results are as follows:

Material: L-14780 Developmental Aerosol Solvent
Manufacturer: 3M Corporation
Composition: Methyl Perfluoropropyl Ether and
1, 2-Trans-dichloroethylene with Carbon Dioxide
Submitted by: EM50/Mark Mitchell
Test Number: M108708
Material Code: 08959

Mechanical Impact Test 13A Results are as follows:

Sample Thickness: Approximately 0.05”
Test Conditions: LOX
Test Temperature: -297°F
Impact Energy: 72 ft-lbs
Pressure: 14.7 psia
Reactions/Tests: 0/20
Rating: A
Notes: Material is a Clear Liquid that Evaporated Quickly

Mechanical Impact Bruceton Results are as follows:

Bruceton Testing: No Reactions at 72 ft-lbs

AIT Results are as follows:

Sample Weight: 0.23 grams
Test Pressure: 50 psia
Ignition Temperature Sample 1: 299 °F
Ignition Temperature Sample 2: 300 °F
Ignition Temperature Sample 3: > 800 °F
Temperature Rise Sample 1: 87.1 °F
Final Pressure Sample 1: 128 psi
Residue: Yes, Yellow Color

Sample Weight: 0.20 grams
Test Pressure: 2,000 psia
Ignition Temperature Sample 1: > 800 °F
Ignition Temperature Sample 2: > 800 °F
Ignition Temperature Sample 3: > 800 °F
Temperature Rise: N/A No Ignition
Greatest Final Pressure: 2,386 psi
Residue: Yes, White

Return to App G

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

May 8, 2013

Reply to Attn of: EM10 (013-291)
Ref. EM10B (13-009)

EM50/Mark Mitchell

Dear Mr. Mitchell,

The Solvokane™ has been tested for Ambient Pressure Liquid Oxygen Mechanical Impact by the procedures outlined in NASA-STD-6001B, Test 13A. Autogenous Ignition Temperature (AIT) testing by the procedures outlined in ASTM G72-09 has also been requested. However, AIT tests at 50 psi and 2,000 psi have been delayed and will be sent under a separate memorandum.

Test parameters and results are provided in the enclosure.

Please direct any comments or questions regarding the quality or timeliness of this test or report to Eddie Davis at 256-544-2490 (eddie.davis@nasa.gov).

Cordially,

Gail H Gordon
Gail H. Gordon
Branch Chief
Materials Test Branch

Enclosures

cc:
EM10/File Copy
EM10/E. Davis
EM03/ERC/K. Baker
EM50/N. Lowrey

The material, test parameters and results are as follows:

Material: Solvokane™ Cleaner
Manufacturer: Solvay Chemicals, Solvay Fluorides, LLC
Composition: 1,1,1,3,3-Pentafluorobutane and Trans-Dichloroethylene
Submitted by: EM50/Mark Mitchell
Test Number: M108716
Material Code: 08967

Mechanical Impact Test 13A Results are as follows:

Sample Thickness: Approximately 0.05"
Test Conditions: LOX
Test Temperature: -297°F
Impact Energy: 72 ft-lbs
Pressure: 14.7 psia
Reactions/Tests: 2/6
Rating: X
Note: Reacted Material Produced Strong Odor

Mechanical Impact Bruceton Results are as follows:

Bruceton Testing: 34 Drops, 7 Reactions at 72 ft-lbs, 2 Reactions at 62.9 ft-lbs, No Reactions at 54.6 ft-lbs

AIT Results are as follows:

Note: AIT testing at 50 psi and 2,000 psi has been delayed and will be sent under a separate memorandum once completed.

Return to App G

Return to Pg 23

Return to Pg 24

WSTF Solvent Oxygen Compatibility Testing

Susana Harper
Fred Juarez

Presented 2-19-2014

Summary of Oxygen Compatibility Testing

- AIT Solvent Testing
  - Evaluation of methodology for increased reliability
  - Purpose: Establish solvent ranking based on reaction temperature
  - Test Plan Acceptance Criteria: No ignition preferred. Beyond that, the following criteria have been used. Suitability may require additional testing.

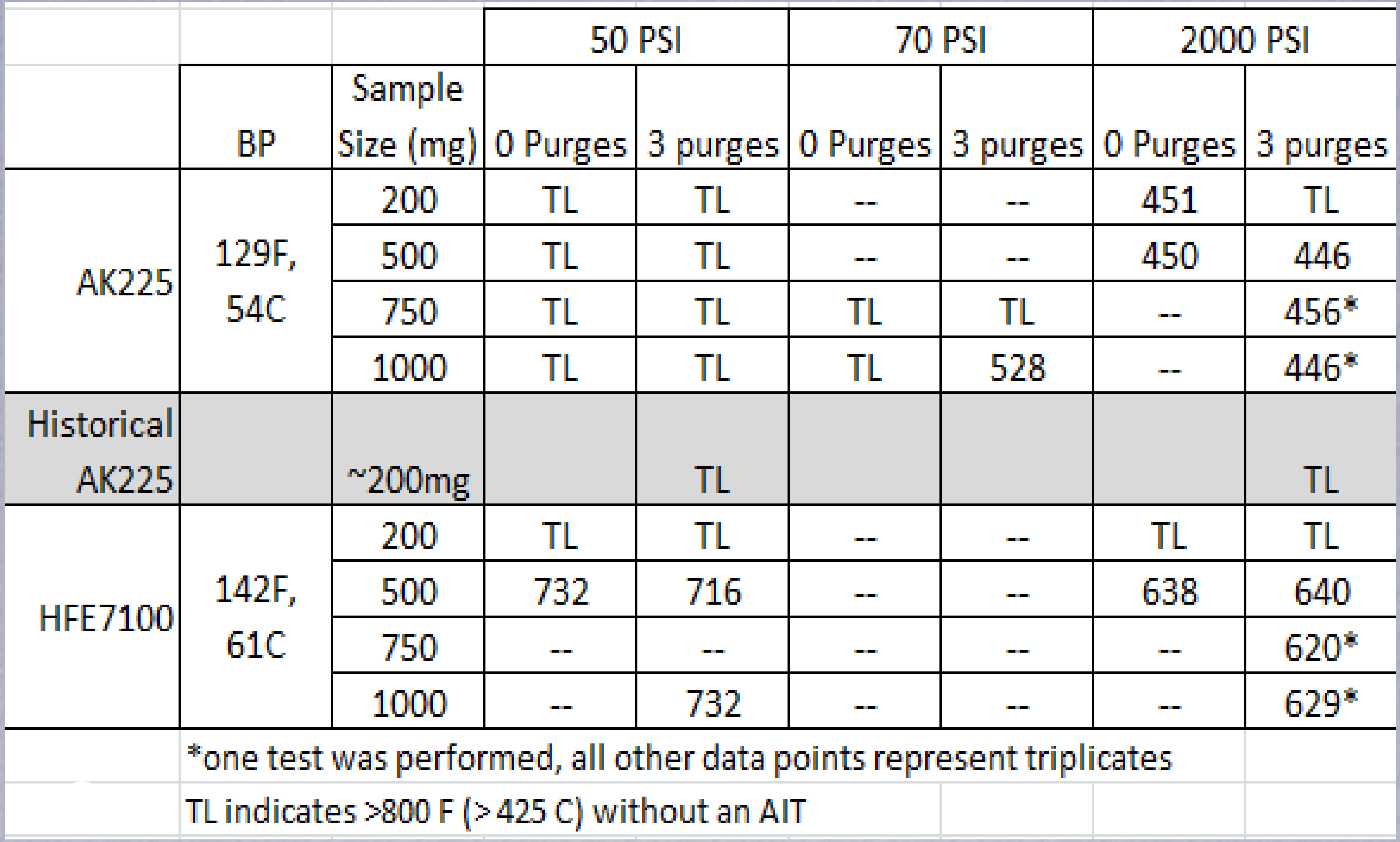
Category A	AIT < 250 ^{o}F	Not recommended for use in oxygen systems
Category B	AIT 250 ^{o}F to 400 ^{o}F	Caution when used in oxygen systems
Category C	AIT > 400 ^{o}F	Recommended for use in oxygen systems

- Test 13 / G86 Mechanical Impact Testing Lox Impact Testing
  - Purpose: To correlate to historical data
  - Test Plan Acceptance Criteria: All solvents that pass testing above 27J (20 ft-lb) or show anomalous results may be considered for further testing or evaluation
- Test 13B/ G86 Pressure Threshold
  - Purpose: Establish solvent ranking based on reaction threshold pressure

AIT Solvent Testing Summary Recommendation

- Historical AK225 showed TL’s @ 50 and 2000psi
- Concern: TL indicates inadequate test conditions for volatile solvents vs. a true >800F AIT
- AIT Test Method
  - Increased fuel size (1000mg)
  - For low pressure testing if TL is obtained consider
  - testing at higher pressures,70psia or higher if necessary
  - Standard Thermocouple placement
  - Chilled Ice Bath for samples, data to follow
  - Standard 3 purges, data to follow
- Propose method for ASTM G72 solvent revision

AIT Evaluation: Purges, Sample Size, Pressure



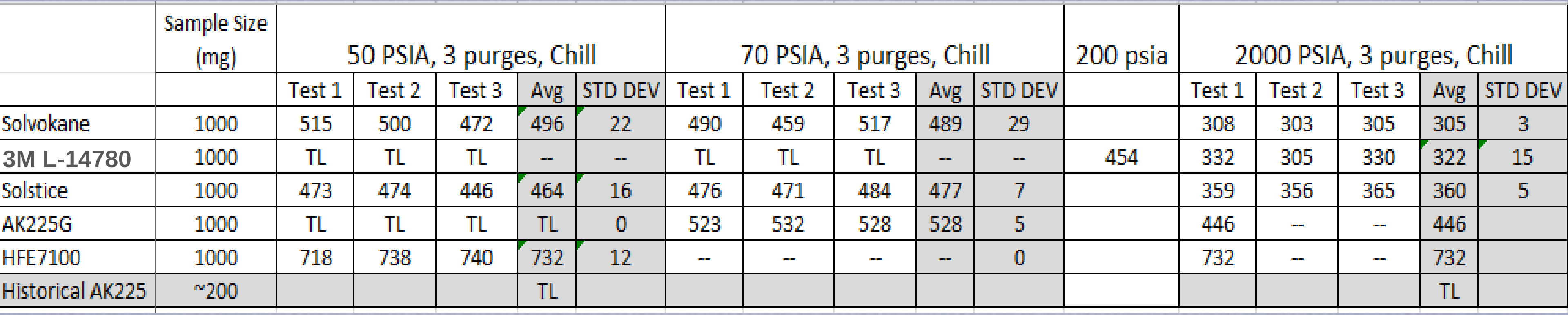
			50 PSI		70 PSI		2000 PSI	
	BP	Sample Size (mg)	0 Purges	3 purges	0 Purges	3 purges	0 Purges	3 purges
AK225	129F, 54C	200	TL	TL	--	--	451	TL
		500	TL	TL	--	--	450	446
		750	TL	TL	TL	TL	--	456*
		1000	TL	TL	TL	528	--	446*
Historical AK225		~200mg		TL				TL
HFE7100	142F, 61C	200	TL	TL	--	--	TL	TL
		500	732	716	--	--	638	640
		750	--	--	--	--	--	620*
		1000	--	732	--	--	--	629*

*one test was performed, all other data points represent triplicates

TL indicates >800 F (> 425 C) without an AIT

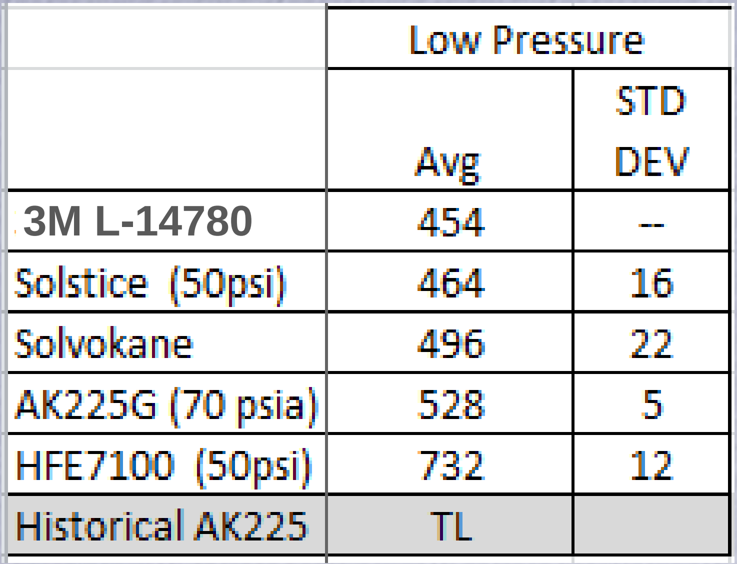
- Historical AK225 showed TL's @ 50 and 2000psi, likely due to inadequate conditions
- Recommendation: 3 purges, No discernable difference in performance
- Recommendation: Increased fuel to 1000mg
- Recommendation: When testing at low Pressures increase Pressure if TL obtained

AIT Data

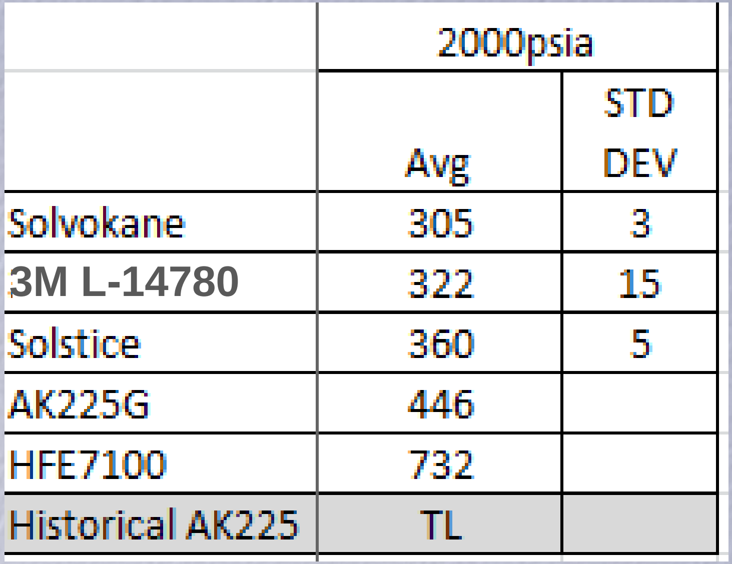


	Sample Size (mg)	50 PSIA, 3 purges, Chill					70 PSIA, 3 purges, Chill					200 psia	2000 PSIA, 3 purges, Chill				
		Test 1	Test 2	Test 3	Avg	STD DEV	Test 1	Test 2	Test 3	Avg	STD DEV		Test 1	Test 2	Test 3	Avg	STD DEV
Solvokane	1000	515	500	472	496	22	490	459	517	489	29		308	303	305	305	3
3M L-14780	1000	TL	TL	TL	--	--	TL	TL	TL	--	--	454	332	305	330	322	15
Solstice	1000	473	474	446	464	16	476	471	484	477	7		359	356	365	360	5
AK225G	1000	TL	TL	TL	TL	0	523	532	528	528	5		446	--	--	446	
HFE7100	1000	718	738	740	732	12	--	--	--	--	0		732	--	--	732	
Historical AK225	~200				TL											TL	

- Solvents Ranks based on AIT, >400 ^{0}F recommended for use with Oxygen

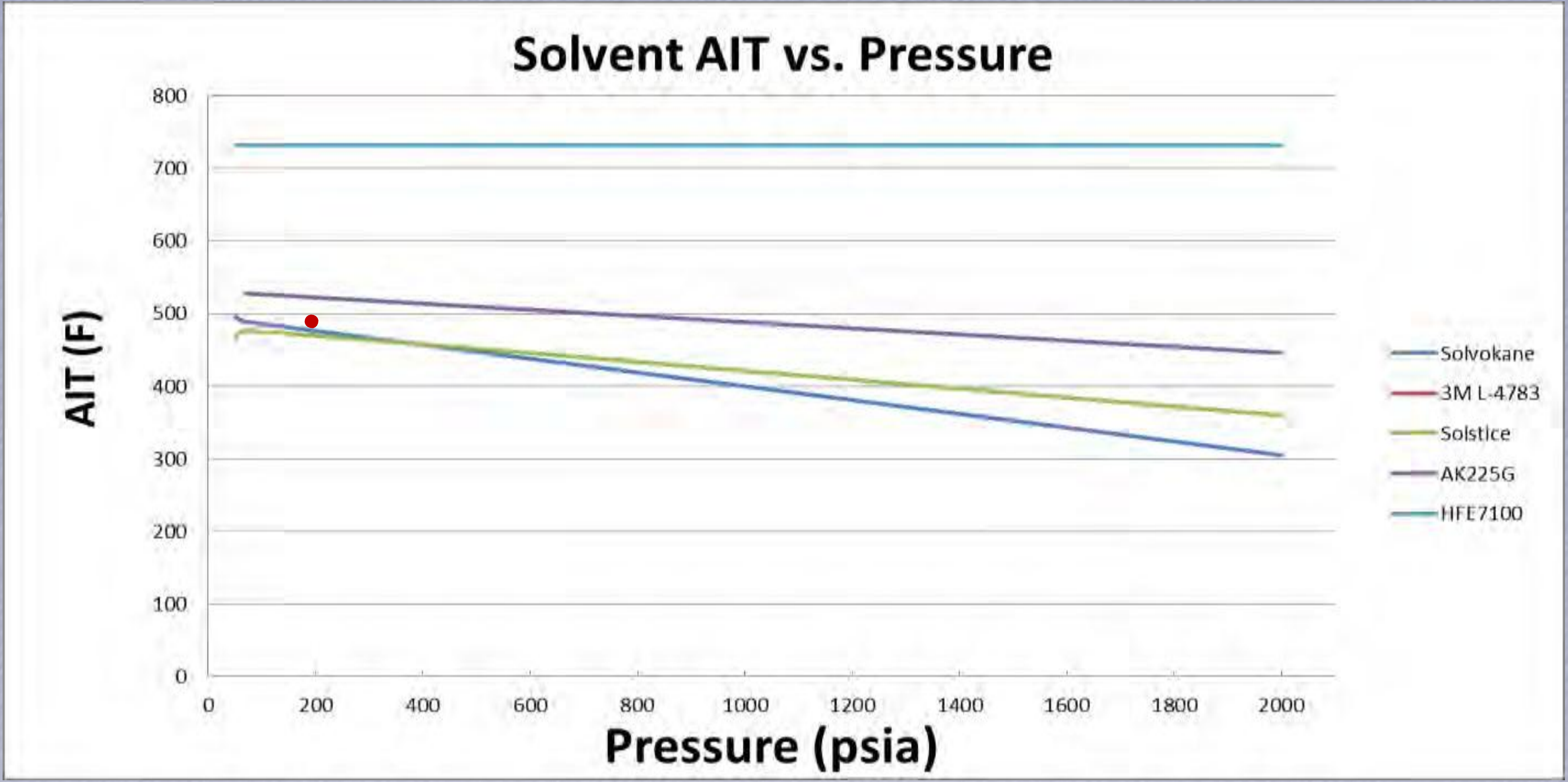


	Low Pressure	
	Avg	STD DEV
3M L-14780	454	--
Solstice (50psi)	464	16
Solvokane	496	22
AK225G (70 psia)	528	5
HFE7100 (50psi)	732	12
Historical AK225	TL	



	2000psia	
	Avg	STD DEV
Solvokane	305	3
3M L-14780	322	15
Solstice	360	5
AK225G	446	
HFE7100	732	
Historical AK225	TL	

Solvent AIT vs. Pressure



Chill vs. No chill: Solvokane Azeotropic Solvent

- 50psia
  - Chill average AIT 496ºF, STDDEV 22
  - No Chill AIT 476ºF
- 70psia
  - Chill average AIT 489ºF, STDDEV 29
  - No Chill AIT 493ºF
- 2000 psia
  - Chill average AIT 305ºF, STDDEV 3
  - No Chill AIT 301ºF
- All No chill data for Solvokane was within standard deviation of chill data
- All final testing was performed with chilling

Test 13 - Mechanical Impact Testing

- Test 13A / ASTM G86:
  - PURPOSE: To correlate to historical data, All solvents that pass testing above 27J (20 ft-lb) or show anomalous results may be considered for further testing or evaluation
  - Impact energy of 97.6 J (72 ft-lb), If fail Energy threshold
  - Ambient LOX
  - If 1 specimen reacts out of 20 continue to test 40 additional samples, discontinue if 2nd failure

- Test 13B / ASTM G86 Pressure Threshold Pressure
  - Purpose: Establish Solvent Ranking based on reaction threshold Pressure
  - Impact energy 97.6J (72 ft-lb)
  - Identify pressure threshold for reaction

3M L-14780

- Material Remained Ice After Pressurization
- Test 13A – Energy Threshold at Ambient Pressure
  - Reacted at 45ft-lbs
  - Passed @ 40 ft-lbs (0/20),
- Test 13BP – Pressure Threshold at 72 ft-lbs
  - Reacted at 8,000psi
  - Passed @ 7500 psi, 72 ft-lbs
- Evaluation was performed to ensure13BP sample retained

WSTF NO. 14-46305
3M Developmental Material L-14780
Pressure Screen LOX
NASA-STD-6001.B Test 13BP
Pre Purge

WSTF NO. 14-46305
3M Developmental Material L-14780
Pressure Screen LOX
NASA-STD-6001.B Test 13BP
Post Purge

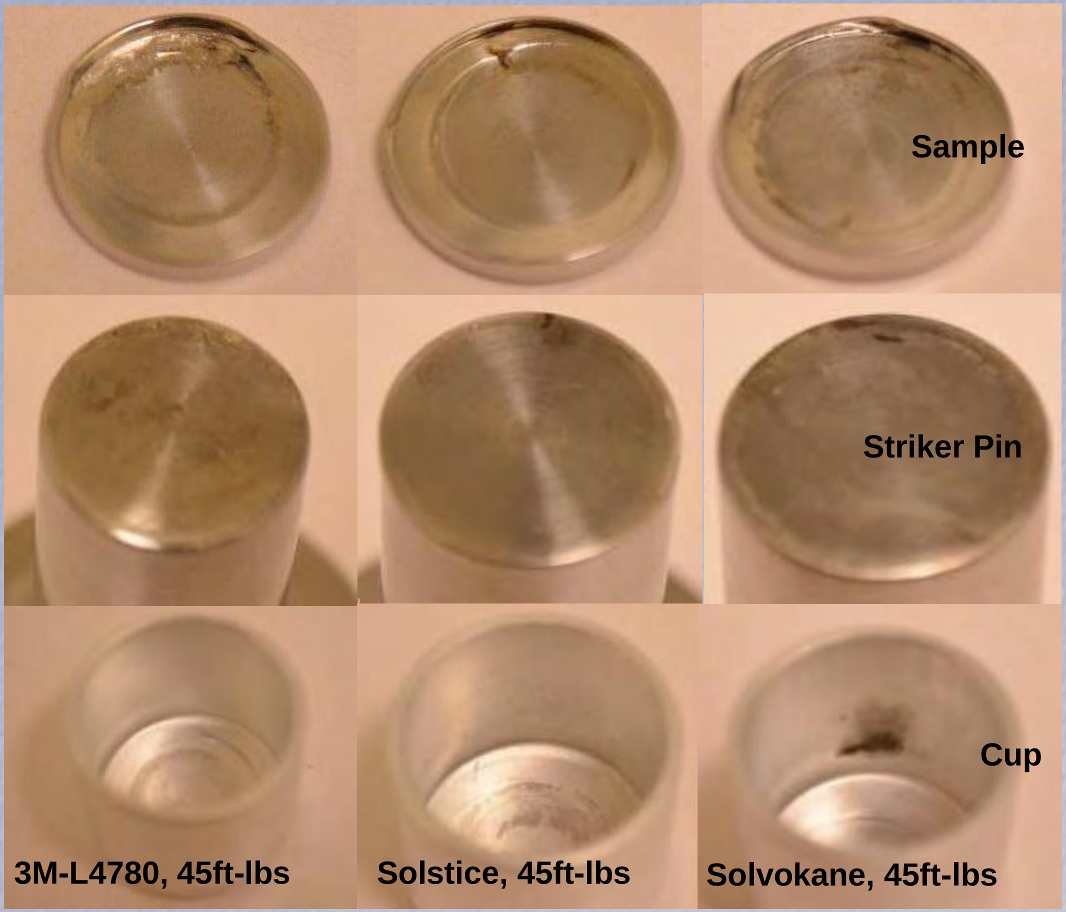
Solstice Performance Fluid

- Material Remained Ice After Pressurization
- Test 13A – Energy Threshold at Ambient Pressure
  - Reacted @ 20 ft-lbs
  - Passed @ 15 ft-lbs,
- Test 13BP –Pressure Threshold at 72 ft-lbs
  - Reacted @ 8,000 psi, 72fts-lbs
  - Passed @ 7500 psi, 72 ft-lbs
- Evaluation was performed to ensure13BP sample retained

Solvokane

- Material Remained Ice After Pressurization
- Test 13A – Energy Threshold at Ambient Pressure
  - Reacted @ 10 ft-lbs (lowest energy level)
  - No threshold defined
- Test 13BP – Pressure Threshold at 72 ft-lbs
  - Reacted @ 500 psi, 72 ft-lbs (lowest pressure level)
  - No threshold defined
- Evaluation was performed to ensure13BP sample retained

Mechanical Impact 13A- Energy Threshold


Lowest Reaction Energy 45ft-lbs

Lowest Reaction Energy 20ft-lbs

Lowest Reaction Energy 10ft-lbs (limit)

3M-L4780, 45ft-lbs

Solstice, 45ft-lbs

Solvokane, 45ft-lbs

All solvents that pass above 27J (20 ft-lb) or show anomalous results may be considered for further testing or evaluation

Test 13BP –Pressure Threshold @ 72 ft-lbs

Lowest Reaction Pressure 8000 psi

3M-L4780, 8000psi

Lowest Reaction Pressure 8000psi

Solstice, 8000psi

Lowest Reaction Pressure 500psi (limit)

Solvokane, 500psi

No recommended acceptance criteria, Establish Solvent Ranking based on reaction threshold Pressure

Return to Menu

Return to Pg 28

APPENDIX H—NONVOLATILE RESIDUE CLEANING EFFECTIVENESS TEST DATA

Nonvolatile residue removal efficiency test data sheets and graphs by contaminant are given in appendix H.

Contaminant: Mineral Oil
Concentration: 2.0884 mg/mL (10.442 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

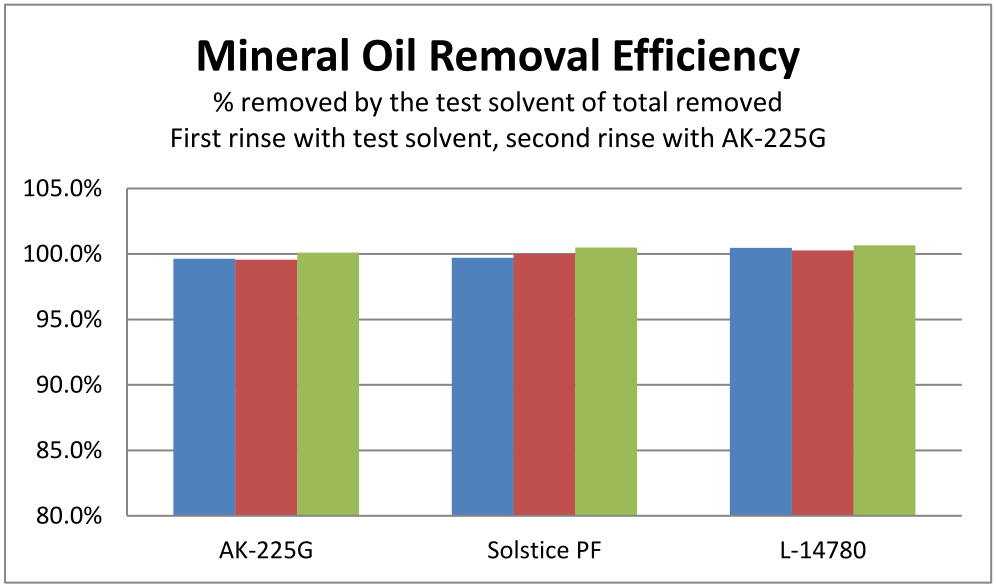
Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	24.03474	24.04039	0.00565	23.75343	23.75359	0.00016	0.00581	99.6%
AK-225G - **A2**	24.10997	24.11677	0.00680	23.88418	23.88435	0.00017	0.00697	99.6%
AK-225G - **A3**	24.10223	24.11188	0.00965	23.96211	23.96224	0.00013	0.00978	100.1%
AK-225G control solvent background	23.91832	23.91846	0.00014					
Panel Control no contamination	23.88720	23.88758	0.00038	23.86872	23.86892	0.00020	0.00058	
Solstice - **S1**	23.85308	23.86370	0.01062	23.90526	23.90543	0.00017	0.01079	99.7%
Solstice - **S2**	24.09097	24.09988	0.00891	23.84934	23.84948	0.00014	0.00905	100.0%
Solstice - **S3**	23.88189	23.89254	0.01065	23.72591	23.72600	0.00009	0.01074	100.5%
Solstice control solvent background	23.77466	23.77483	0.00017					
L-14780 - **L1**	23.84173	23.85245	0.01072	24.03850	24.03859	0.00009	0.01081	100.5%
L-14780 - **L2**	23.89091	23.90242	0.01151	23.90161	23.90172	0.00011	0.01162	100.3%
L-14780 - **L3**	23.68715	23.69940	0.01225	23.73556	23.73562	0.00006	0.01231	100.7%
L-14780 control solvent background	23.75870	23.75885	0.00015					

Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Mineral Oil
Concentration: 2.0884 mg/mL (10.442 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

	AK-225G	Solstice PF	L-14780
1	99.6%	99.7%	100.5%
2	99.6%	100.0%	100.3%
3	100.1%	100.5%	100.7%
Ave	99.8%	100.1%	100.5%



Contaminant: Mobil DTE25 hydraulic fluid
Concentration: 2.0437 mg/ml (10.2125 mg/panel)

Nonvolatile Residue Removal Efficiency Test
Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.09516	2.10662	0.01146	2.12676	2.12682	0.00006	0.01152	99.7%
AK-225G - **A2**	2.13733	2.14946	0.01213	2.08740	2.08746	0.00006	0.01219	99.7%
AK-225G - **A3**	2.19636	2.20768	0.01132	2.17934	2.17937	0.00003	0.01135	99.9%
AK-225G control solvent background	2.09410	2.09412	0.00002					
Panel Control no contamination	2.12337	2.12345	0.00008	2.06650	2.06656	0.00006	0.00014	
Solstice - **S1**	2.23722	2.24826	0.01104	2.07818	2.07824	0.00006	0.01110	99.6%
Solstice - **S2**	2.15522	2.16326	0.00804	2.08608	2.08611	0.00003	0.00807	99.9%
Solstice - **S3**	2.13073	2.13731	0.00658	2.06964	2.06964	0.00000	0.00658	100.3%
Solstice control solvent background	2.08975	2.08980	0.00005					
L-14780 - **L1**	2.09117	2.09834	0.00717	2.05816	2.05819	0.00003	0.00720	99.9%
L-14780 - **L2**	2.23524	2.24206	0.00682	2.08558	2.08567	0.00009	0.00691	99.0%
L-14780 - **L3**	2.23795	2.24802	0.01007	2.12965	2.12973	0.00008	0.01015	99.4%
L-14780 control solvent background	2.00387	2.00397	0.00010					

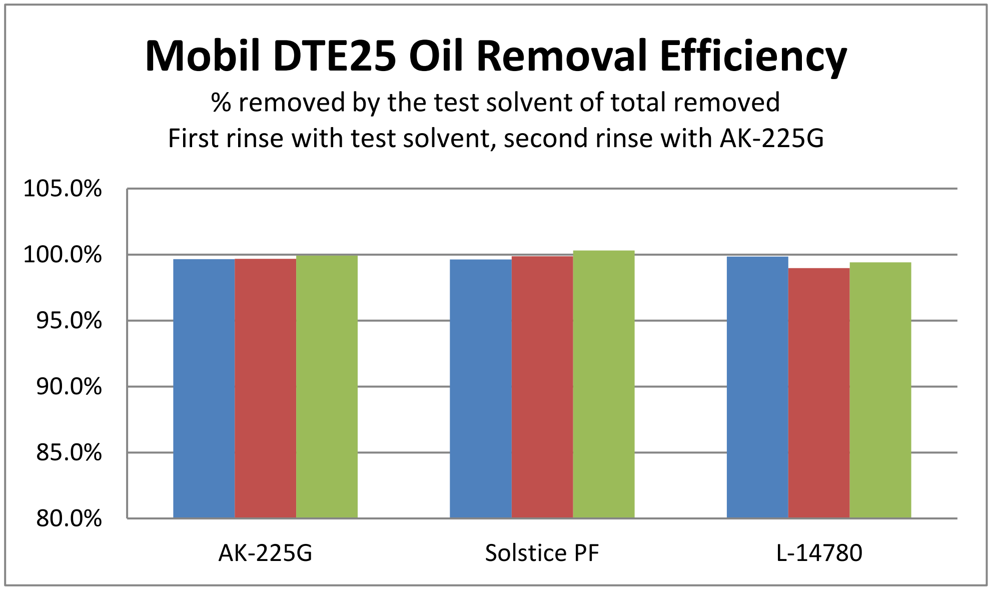
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Mobil DTE25 hydraulic fluid
Concentration: 2.0437 mg/ml (10.2125 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	99.7%	99.6%	99.9%
2	99.7%	99.9%	99.0%
3	99.9%	100.3%	99.4%
Ave	99.74464	99.93986	99.40987



Contaminant: MIL-PRF-83282 Brayco Micronic 882
Concentration: 2.0953 mg/ml (10.4765 mg/panel)

Nonvolatile Residue Removal Efficiency Test
Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.09859	2.10406	0.00547	1.93526	1.93530	0.00004	0.00551	99.3%
AK-225G - **A2**	2.29394	2.30096	0.00702	2.19347	2.19352	0.00005	0.00707	99.3%
AK-225G - **A3**	2.09445	2.10551	0.01106	2.15331	2.15341	0.00010	0.01116	99.1%
AK-225G control solvent background	2.22900	2.22900	0.00000					
Panel Control no contamination	2.22255	2.22271	0.00016	2.17174	2.17189	0.00015	0.00031	
Solstice - **S1**	2.20023	2.20269	0.00246	1.95175	1.95192	0.00017	0.00263	93.4%
Solstice - **S2**	2.04476	2.04666	0.00190	2.23848	2.23866	0.00018	0.00208	91.1%
Solstice - **S3**	2.08768	2.09107	0.00339	2.05307	2.05317	0.00010	0.00349	97.1%
Solstice control solvent background	2.08970	2.08976	0.00006					
L-14780 - **L1**	2.08336	2.08902	0.00566	2.18565	2.18594	0.00029	0.00595	95.0%
L-14780 - **L2**	2.15538	2.15851	0.00313	2.06336	2.06432	0.00096	0.00409	75.7%
L-14780 - **L3**	2.16020	2.16690	0.00670	2.10026	2.10047	0.00021	0.00691	96.9%
L-14780 control solvent background	2.16280	2.16294	0.00014					

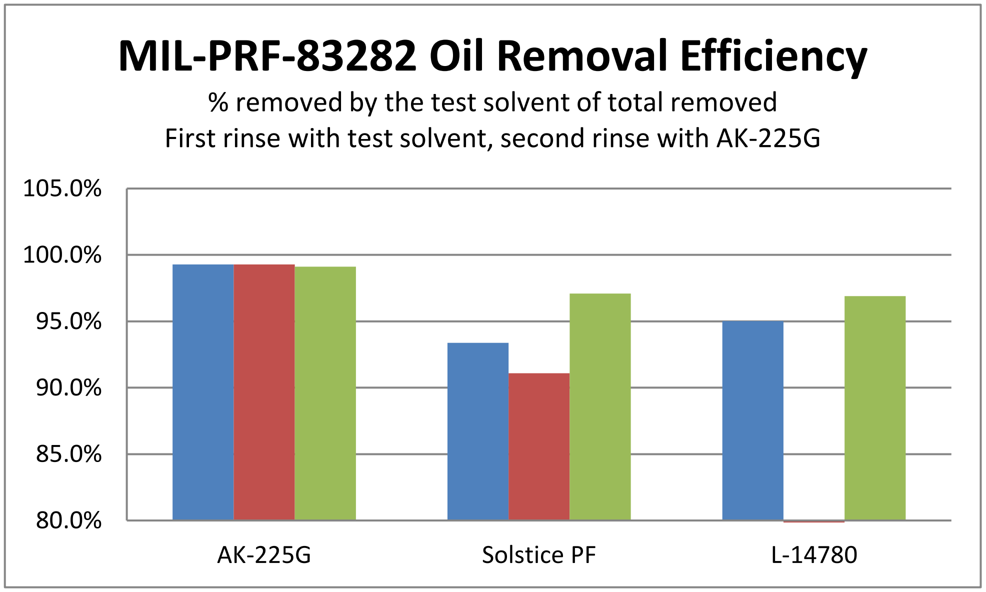
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted.
*Highlighted orange cells: Spilled some sample S3 rinse andto sample L2 pan, results invalid.
Yellow highlighted cells: Only 20-26% of inital target contaminant mass collected. Results suspect. Repeat test.

Contaminant: MIL-PRF-83282 Brayco Micronic 882
Concentration: 2.0953 mg/ml (10.4765 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	99.3%	93.4%	95.0%
2	99.3%	91.1%	75.7%
3	99.1%	97.1%	96.9%
Ave	99.22359	93.85296	89.20096



Contaminant: Di-2-ethylhexyl sebacate
Concentration: 2.0024 mg/ml (10.012 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.13325	2.14054	0.00729	2.12663	2.12693	0.00030	0.00759	99.2%
AK-225G - **A2**	2.13913	2.14923	0.01010	2.14757	2.14793	0.00036	0.01046	98.8%
AK-225G - **A3**	2.12047	2.13269	0.01222	2.09966	2.09999	0.00033	0.01255	99.3%
AK-225G control solvent background	2.16490	2.16514	0.00024					
Panel Control no contamination	2.11294	2.11323	0.00029	2.09938	2.09964	0.00026	0.00055	
Solstice - **S1**	2.05348	2.06216	0.00868	2.10695	2.10720	0.00025	0.00893	99.9%
Solstice - **S2**	2.07035	2.08217	0.01182	2.00609	2.00633	0.00024	0.01206	100.0%
Solstice - **S3**	2.11882	2.12612	0.00730	2.14083	2.14108	0.00025	0.00755	99.9%
Solstice control solvent background	2.12534	2.12538	0.00004					
L-14780 - **L1**	2.02187	2.06216	0.04029	2.26706	2.26727	0.00021	0.04050	100.1%
L-14780 - **L2**	2.14400	2.14427	0.00027	2.30833	2.30856	0.00023	0.00050	110.0%
L-14780 - **L3**	2.23760	2.24636	0.00876	2.13124	2.13150	0.00026	0.00902	99.8%
L-14780 control solvent background	2.11275	2.11291	0.00016					

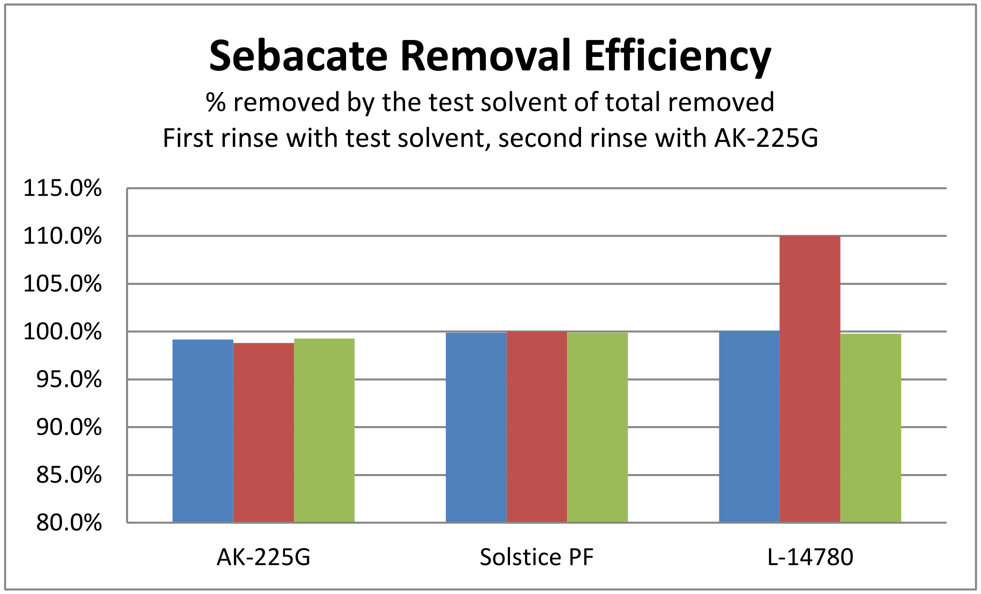
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted.
*Orange color block: Excessive or insufficient total NVR mass collected. Results suspect. Repeat test.

Contaminant: Di-2-ethylhexyl sebacate
Concentration: 2.0024 mg/ml (10.012 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	99.2%	99.9%	100.1%
2	98.8%	100.0%	110.0%
3	99.3%	99.9%	99.8%
Ave	99.06935	99.91561	103.2809



Contaminant: Synthetic Sebum (fingerprint)
Concentration: 2.0036 mg/ml (10.018 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.25918	2.26567	0.00649	2.11363	2.11364	0.00001	0.00650	100.6%
AK-225G - **A2**	2.12524	2.13188	0.00664	2.13601	2.13604	0.00003	0.00667	100.3%
AK-225G - **A3**	2.21280	2.22096	0.00816	2.12693	2.12698	0.00005	0.00821	100.0%
AK-225G control solvent background	2.15760	2.15765	0.00005	2.14500	2.14507			
Panel Control no contamination	2.07784	2.07796	0.00012					
Solstice - **S1**	2.12054	2.12787	0.00733	2.03100	2.03103	0.00003	0.00736	100.3%
Solstice - **S2**	2.13139	2.14283	0.01144	2.07083	2.07086	0.00003	0.01147	100.2%
Solstice - **S3**	2.05551	2.06628	0.01077	2.09393	2.09405	0.00012	0.01089	99.3%
Solstice control solvent background	2.14964	2.14976	0.00012					
L-14780 - **L1**	2.16208	2.16884	0.00676	2.06914	2.06955	0.00041	0.00717	94.9%
L-14780 - **L2**	2.05356	2.05834	0.00478	2.19252	2.19392	0.00140	0.00618	77.7%
L-14780 - **L3**	2.19633	2.20742	0.01109	2.18895	2.18919	0.00024	0.01133	98.3%
L-14780 control solvent background	2.25937	2.25945	0.00008					

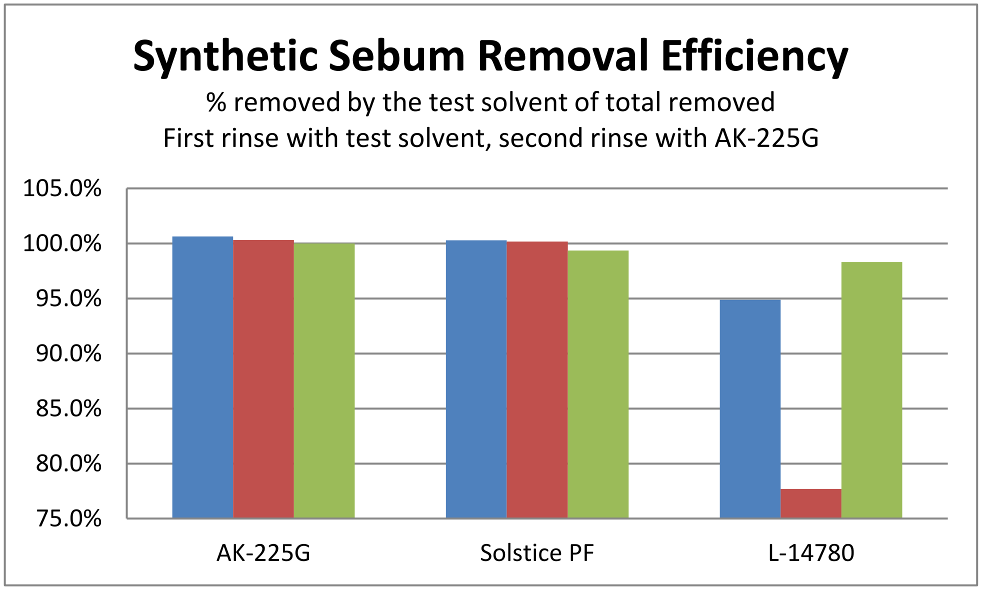
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Results greater than 100% are due to the error tolerance of the analytical balance.
*Yellow highlighted cell: Result significantly out-of-family with other two samples. Repeat test.

Contaminant: Synthetic Sebum (fingerprint)
Concentration: 2.0036 mg/ml (10.018 mg/panel)

Nonvolatile Residue Removal Efficiency Test
Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	100.6%	100.3%	94.9%
2	100.3%	100.2%	77.7%
3	100.0%	99.3%	98.3%
Ave	100.3098	99.93406	90.29196



Contaminant: Krytox 240AC grease
Concentration: 200.61 mg/100mL (10.0305 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	24.03477	24.04514	0.01037	23.75339	23.75344	0.00005	0.01042	99.5%
AK-225G - **A2**	24.11012	24.12024	0.01012	23.88418	23.88417	-0.00001	0.01011	100.1%
AK-225G - **A3**	24.10244	24.11211	0.00967	23.96206	23.96209	0.00003	0.00970	99.7%
AK-225G control solvent background	23.91810	23.91810	0.00000					
Panel Control no contamination	23.88727	23.88724	-0.00003	23.86862	23.86869	0.00007	0.00004	
Solstice - **S1**	23.85303	23.86330	0.01027	23.90516	23.90516	0.00000	0.01027	100.0%
Solstice - **S2**	24.09091	24.10165	0.01074	23.84921	23.84926	0.00005	0.01079	99.5%
Solstice - **S3**	23.88186	23.89164	0.00978	23.72588	23.72585	-0.00003	0.00975	100.3%
Solstice control solvent background	23.77466	23.77473	0.00007					
L-14780 - **L1**	23.84168	23.85205	0.01037	24.03843	24.03855	0.00012	0.01049	98.8%
L-14780 - **L2**	23.89081	23.90067	0.00986	23.90155	23.90158	0.00003	0.00989	99.7%
L-14780 - **L3**	23.68711	23.69691	0.00980	23.73552	23.73554	0.00002	0.00982	99.8%
L-14780 control solvent background	23.75864	23.75946	0.00082					

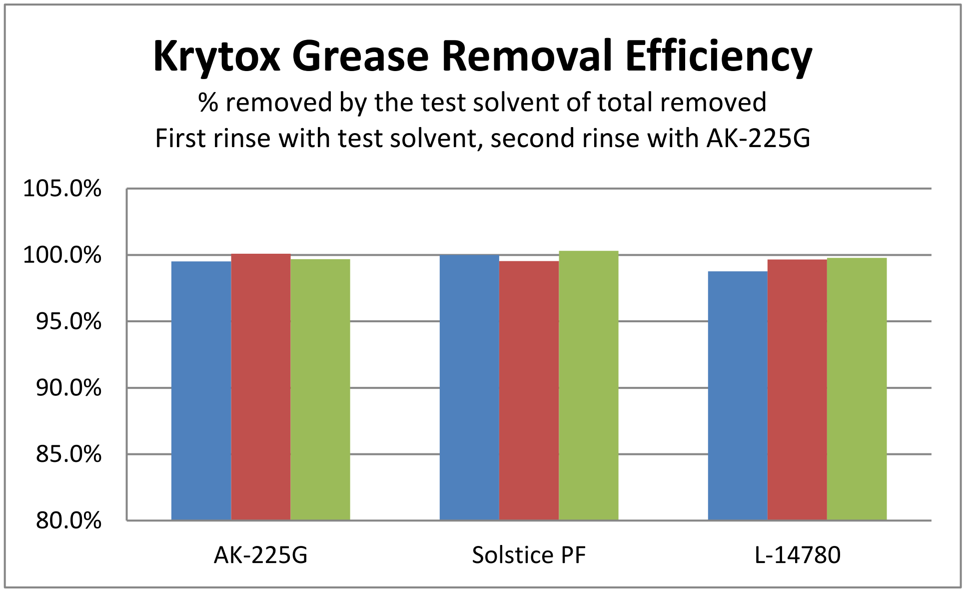
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Krytox 240AC grease
Concentration: 200.61 mg/100mL (10.0305 mg/panel)

Nonvolatile Residue Removal Efficiency Test
Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	99.5%	100.0%	98.8%
2	100.1%	99.5%	99.7%
3	99.7%	100.3%	99.8%
Ave	99.76993	99.94783	99.40202



Contaminant: Big Red (Crane Grease)
Concentration: 2.0092 mg/ml (10.046 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

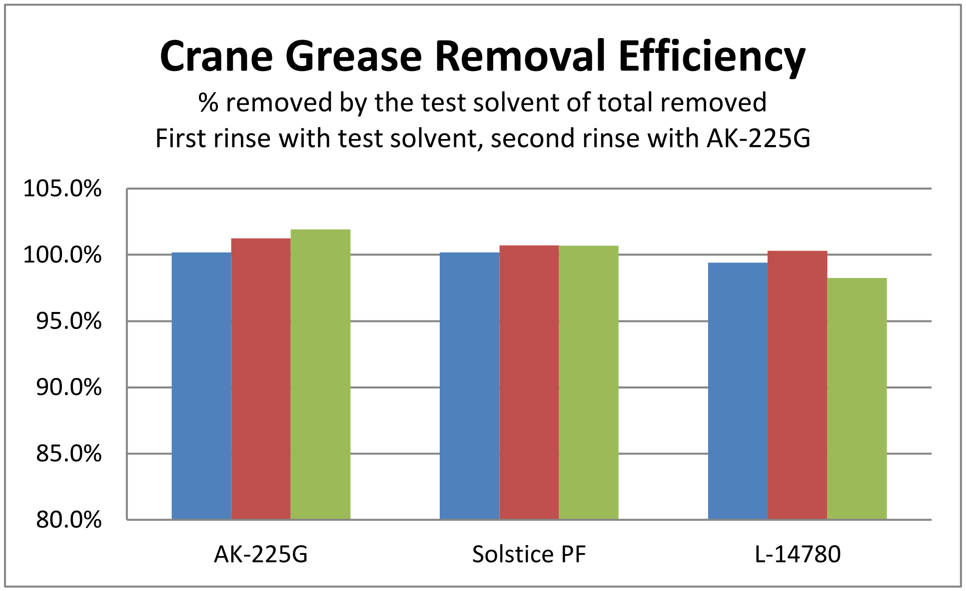
Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	24.03464	24.04525	0.01061	23.75339	23.75339	0.00000	0.01061	100.2%
AK-225G - **A2**	24.10996	24.12066	0.01070	23.88414	23.88403	-0.00011	0.01059	101.2%
AK-225G - **A3**	24.10216	24.11173	0.00957	23.96211	23.96195	-0.00016	0.00941	101.9%
AK-225G control solvent background	23.91816	23.91818	0.00002					
Panel Control no contamination	23.88726	23.88737	0.00011	23.86868	23.86866	-0.00002	0.00009	
Solstice - **S1**	23.85306	23.86389	0.01083	23.90525	23.90525	0.00000	0.01083	100.2%
Solstice - **S2**	24.09098	24.09657	0.00559	23.84934	23.84932	-0.00002	0.00557	100.7%
Solstice - **S3**	23.88194	23.88929	0.00735	23.72594	23.72591	-0.00003	0.00732	100.7%
Solstice control solvent background	23.77470	23.77468	-0.00002					
L-14780 - **L1**	23.84176	23.85173	0.00997	24.03849	24.03857	0.00008	0.01005	99.4%
L-14780 - **L2**	23.89087	23.89757	0.00670	23.90162	23.90162	0.00000	0.00670	100.3%
L-14780 - **L3**	23.68723	23.69284	0.00561	23.73559	23.73571	0.00012	0.00573	98.2%
L-14780 control solvent background	23.75874	23.75876	0.00002					

Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Big Red (Crane Grease)
Concentration: 2.0092 mg/ml (10.046 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

	AK-225G	Solstice PF	L-14780
1	100.2%	100.2%	99.4%
2	101.2%	100.7%	100.3%
3	101.9%	100.7%	98.2%
Ave	101.1142	100.5286	99.31448



Contaminant: WD-40 water displacing oil
Concentration: 2.0092 mg/ml (10.046 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.08520	2.08764	0.00244	2.11896	2.11900	0.00004	0.00248	99.2%
AK-225G - **A2**	2.17867	2.18116	0.00249	2.11343	2.11348	0.00005	0.00254	98.8%
AK-225G - **A3**	2.19427	2.19677	0.00250	2.12739	2.12744	0.00005	0.00255	98.8%
AK-225G control solvent background	2.08856	2.08858	0.00002					
Panel Control no contamination	2.03361	2.03367	0.00006	2.12160	2.12161	0.00001	0.00007	
Solstice - **S1**	2.18524	2.18808	0.00284	2.26753	2.26754	0.00001	0.00285	100.4%
Solstice - **S2**	2.09419	2.09721	0.00302	2.27557	2.27558	0.00001	0.00303	100.3%
Solstice - **S3**	2.07018	2.07300	0.00282	2.04366	2.04368	0.00002	0.00284	100.0%
Solstice control solvent background	2.06507	2.06514	0.00007					
L-14780 - **L1**	2.14996	2.15170	0.00174	2.23583	2.23591	0.00008	0.00182	96.5%
L-14780 - **L2**	2.16084	2.16192	0.00108	2.14286	2.14295	0.00009	0.00117	93.4%
L-14780 - **L3**	2.07347	2.07452	0.00105	2.28748	2.28749	0.00001	0.00106	101.1%
L-14780 control solvent background	2.12420	2.12429	0.00009					

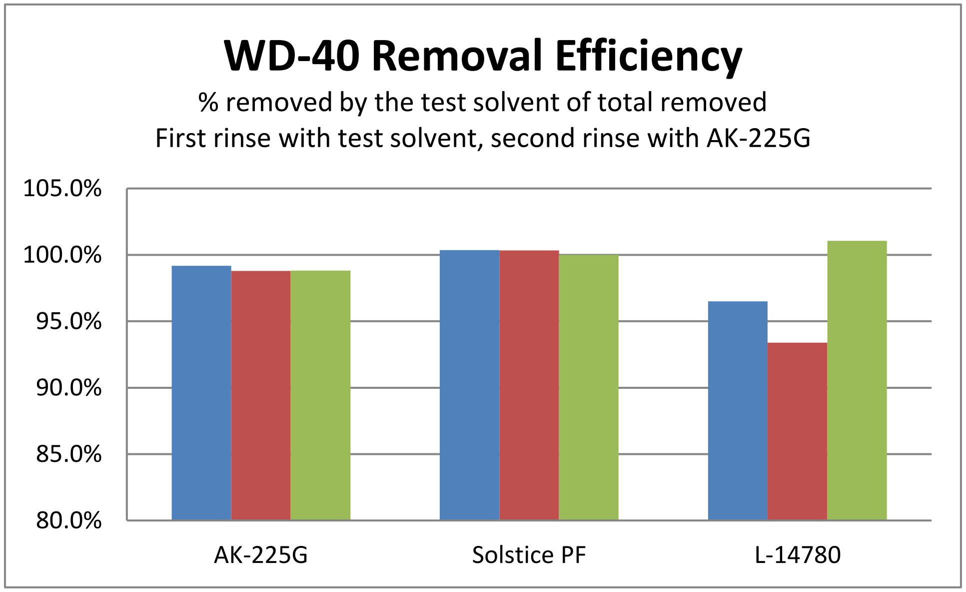
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: WD-40 water displacing oil
Concentration: 2.0092 mg/ml (10.046 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	99.2%	100.4%	96.5%
2	98.8%	100.3%	93.4%
3	98.8%	100.0%	101.1%
Ave	98.92837	100.2342	96.98003



Contaminant: Brayco Micronic 882 Retest
Concentration: 2.0283 mg/ml (10.1415 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.17642	2.18695	0.01053	2.13231	2.13273	0.00042	0.01095	97.5%
AK-225G - **A2**	2.13182	2.14358	0.01176	2.04157	2.04183	0.00026	0.01202	99.1%
AK-225G - **A3**	2.03029	2.04139	0.01110	2.24539	2.24571	0.00032	0.01142	98.5%
AK-225G control solvent background	2.11493	2.11508	0.00015					
Panel Control no contamination	2.27257	2.27274	0.00017	2.17896	2.17913	0.00017	0.00034	
Solstice - **S1**	2.20672	2.21749	0.01077	2.13848	2.13848	0.00000	0.01077	101.4%
Solstice - **S2**	2.17076	2.18190	0.01114	2.15884	2.15901	0.00017	0.01131	99.8%
Solstice - **S3**	2.05896	2.06961	0.01065	2.20407	2.20428	0.00021	0.01086	99.4%
Solstice control solvent background	2.06201	2.06212	0.00011					
L-14780 - **L1**	2.11277	2.12004	0.00727	2.08327	2.08347	0.00020	0.00747	99.3%
L-14780 - **L2**	2.10725	2.11322	0.00597	2.06618	2.06635	0.00017	0.00614	99.7%
L-14780 - **L3**	2.12573	2.13420	0.00847	2.17529	2.17542	0.00013	0.00860	100.2%
L-14780 control solvent background	2.05949	2.05954	0.00005					

Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted.

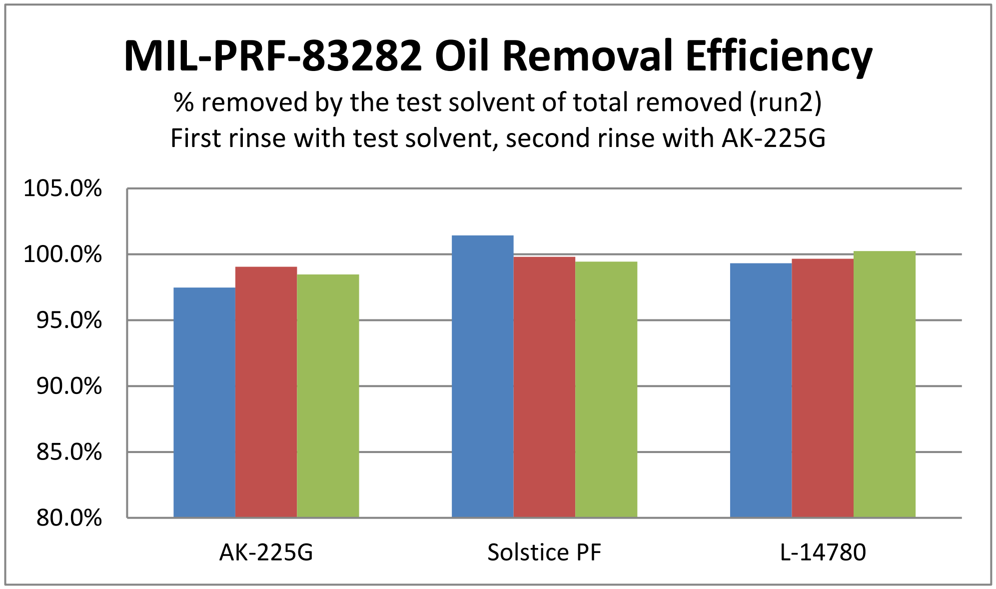
Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Brayco Micronic 882 Retest
Concentration: 2.0283 mg/ml (10.1415 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	97.5%	101.4%	99.3%
2	99.1%	99.8%	99.7%
3	98.5%	99.4%	100.2%
Ave	98.33248	100.2267	99.73788



Contaminant: Di-2-ethylhexyl sebacate Run 2
Concentration: 2.0225 mg/ml (10.115 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.14119	2.14622	0.00503	2.11045	2.11058	0.00013	0.00516	100.2%
AK-225G - **A2**	2.11645	2.12819	0.01174	2.13177	2.13189	0.00012	0.01186	100.2%
AK-225G - **A3**	2.13105	2.13822	0.00717	2.10654	2.10672	0.00018	0.00735	99.4%
AK-225G control solvent background	2.09112	2.09126	0.00014					
Panel Control no contamination	2.12548	2.12568	0.00020	2.06887	2.06901	0.00014	0.00034	100.0%
Solstice - **S1**	2.11505	2.12718	0.01213	2.15723	2.15741	0.00018	0.01231	99.7%
Solstice - **S2**	2.11920	2.12735	0.00815	2.09081	2.09099	0.00018	0.00833	99.5%
Solstice - **S3**	2.13209	2.13384	0.00175	2.14509	2.14528	0.00019	0.00194	96.8%
Solstice control solvent background	2.12245	2.12268	0.00023					
L-14780 - **L1**	2.16937	2.17790	0.00853	2.08487	2.08507	0.00020	0.00873	99.3%
L-14780 - **L2**	2.15868	2.17047	0.01179	2.13559	2.13563	0.00004	0.01183	100.9%
L-14780 - **L3**	2.14933	2.16096	0.01163	2.15043	2.15058	0.00015	0.01178	99.9%
L-14780 control solvent background	2.08958	2.08969	0.00011					

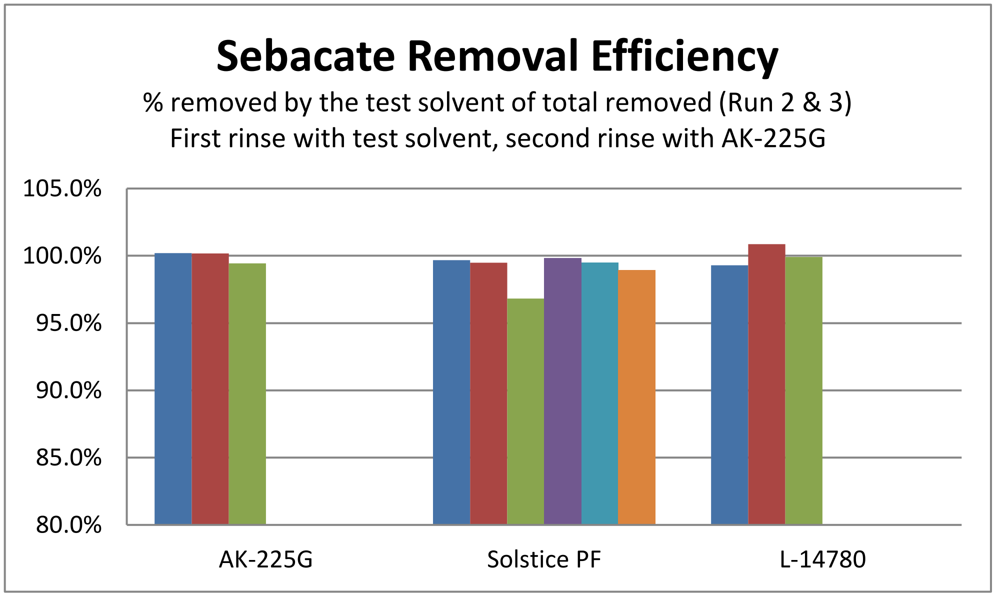
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted.
*Orange highlighted cell: Only 20% of inital target contaminant mass collected. Results suspect. Repeat test.
Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Di-2-ethylhexyl sebacate Run 2
Concentration: 2.0225 mg/ml (10.115 mg/panel)

Nonvolatile Residue Removal Efficiency Test
Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1	100.2%	99.7%	99.3%
2	100.2%	99.5%	100.9%
3	99.4%	96.8%	99.9%
		99.8%	
		99.5%	
		98.9%	
Ave	99.9%	99.0%	100.0%



Contaminant: Di-2-ethylhexyl sebacate Run 3
Concentration: 2.0225 mg/ml (10.115 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**								
AK-225G - **A2**								
AK-225G - **A3**								
AK-225G control solvent background	2.10224	2.10232	0.00008					
Panel Control no contamination	2.12076	2.12099	0.00023	2.14222	2.14237	0.00015	0.00038	
Solstice - **S1**	2.12589	2.13894	0.01305	2.10883	2.10893	0.00010	0.01315	99.8%
Solstice - **S2**	2.13095	2.14436	0.01341	2.07813	2.07828	0.00015	0.01356	99.5%
Solstice - **S3**	2.14430	2.15793	0.01363	2.12842	2.12865	0.00023	0.01386	98.9%
Solstice control solvent background	2.13211	2.13214	0.00003					
L-14780 - **L1**								
L-14780 - **L2**								
L-14780 - **L3**								
L-14780 control solvent background								

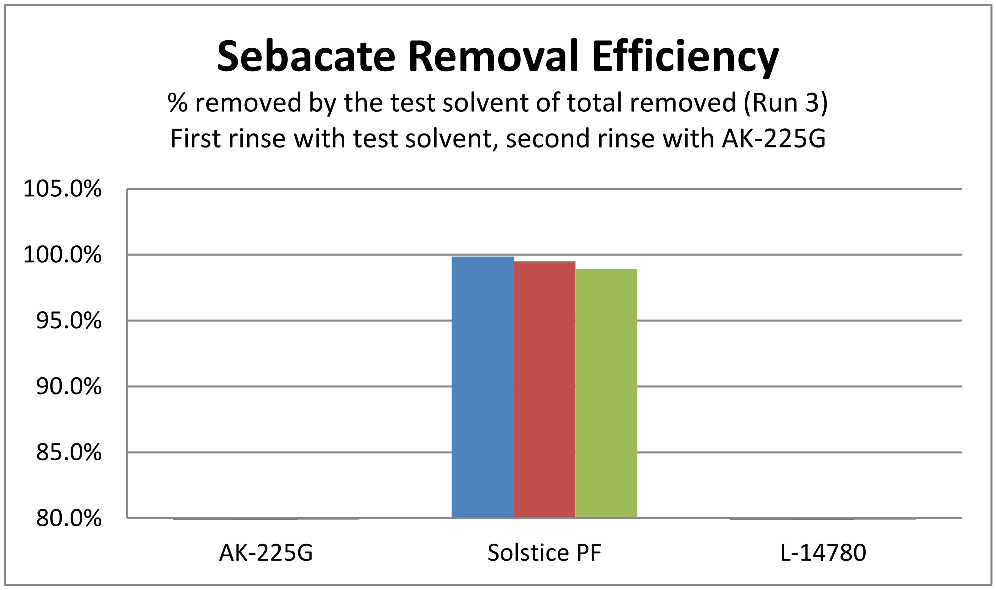
Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Retest of Solstice only. Tests performed parallel to Synthetic Sebum run 2; AK-225G solvent background and Panel Control are same as Sebum run 2.

Contaminant: Di-2-ethylhexyl sebacate Run 3
Concentration: 2.0225 mg/ml (10.115 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

	AK-225G	Solstice PF	L-14780
1		99.8%	
2		99.5%	
3		98.9%	
Ave		99.4%	



Contaminant: Synthetic Sebum Run 2
Concentration: 2.0335 mg/ml (10.1675 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

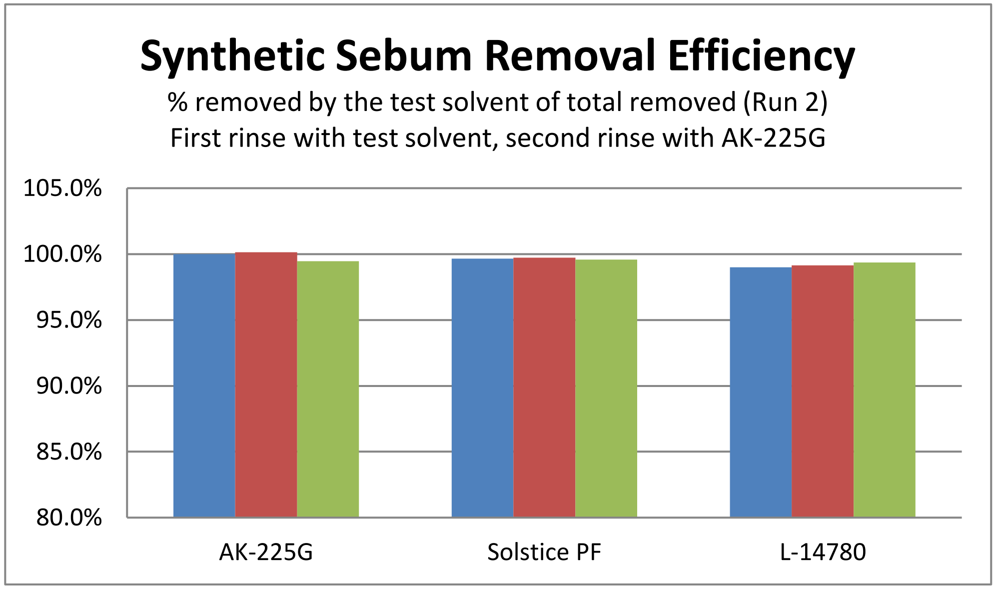
Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.15952	2.16753	0.00801	2.09786	2.09794	0.00008	0.00809	100.0%
AK-225G - **A2**	2.12413	2.13196	0.00783	2.12233	2.12240	0.00007	0.00790	100.1%
AK-225G - **A3**	2.16100	2.16858	0.00758	2.15904	2.15916	0.00012	0.00770	99.5%
AK-225G control solvent background	2.10224	2.10232	0.00008					
Panel Control no contamination	2.12076	2.12099	0.00023	2.14222	2.14237	0.00015	0.00038	
Solstice - **S1**	2.11599	2.12813	0.01214	2.10178	2.10190	0.00012	0.01226	99.7%
Solstice - **S2**	2.14429	2.15639	0.01210	2.07585	2.07596	0.00011	0.01221	99.7%
Solstice - **S3**	2.10507	2.11534	0.01027	2.12308	2.12320	0.00012	0.01039	99.6%
Solstice control solvent background	2.10350	2.10419	0.00069			0.00000	0.00069	
L-14780 - **L1**	2.15497	2.16605	0.01108	2.10466	2.10485	0.00019	0.01127	99.0%
L-14780 - **L2**	2.16588	2.17634	0.01046	2.09490	2.09507	0.00017	0.01063	99.1%
L-14780 - **L3**	2.16420	2.17540	0.01120	2.10578	2.10593	0.00015	0.01135	99.4%
L-14780 control solvent background	2.15489	2.15501	0.00012					

Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted. Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Synthetic Sebum Run 2
Concentration: 2.0335 mg/ml (10.1675 mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

	AK-225G	Solstice PF	L-14780
1	100.0%	99.7%	99.0%
2	100.1%	99.7%	99.1%
3	99.5%	99.6%	99.4%
Ave	99.86623	99.65794	99.17188



Synthetic Sebum Removal Efficiency

% removed by the test solvent of total removed (Run 2)
First rinse with test solvent, second rinse with AK-225G

Contaminant: Christo-Lube
Concentration: 2.0034 mg/ml (10.017mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

HCFC-225cb Replacement
MSFC/SSC joint test plan

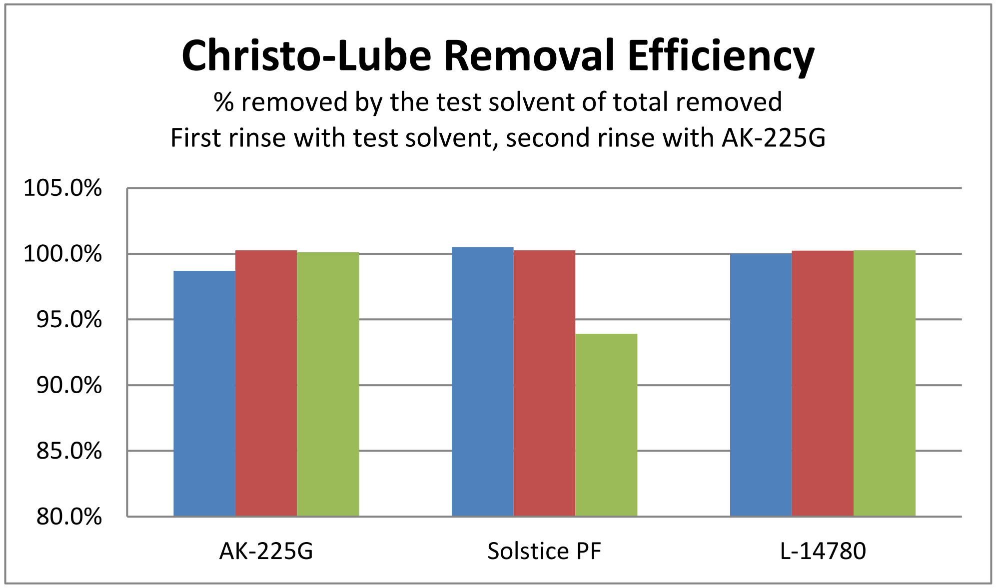
Test Run (all weights in gms)	**Test Rinse Beaker initial weight**	**Test Rinse NVR in Beaker**	**Test Rinse NVR mass**	**AK Rinse Beaker initial weight**	**AK Rinse NVR in Beaker**	**AK Rinse NVR Mass**	**Total NVR Mass collected**	**Test Solvent Removal % Efficiency**
AK-225G - **A1**	2.13014	2.13778	0.00764	2.13306	2.13318	0.00012	0.00776	98.7%
AK-225G - **A2**	2.14893	2.15644	0.00751	2.14809	2.14809	0.00000	0.00751	100.3%
AK-225G - **A3**	2.18831	2.19626	0.00795	2.15092	2.15093	0.00001	0.00796	100.1%
AK-225G control solvent background	2.12489	2.12491	0.00002					
Panel Control no contamination	2.13841	2.13842	0.00001	2.16344	2.16346	0.00002	0.00003	100.0%
Solstice - **S1**	2.13439	2.14280	0.00841	2.16492	2.16490	-0.00002	0.00839	100.5%
Solstice - **S2**	2.05325	2.06132	0.00807	2.12182	2.12182	0.00000	0.00807	100.3%
Solstice - **S3**	2.14631	2.15398	0.00767	2.13411	2.13461	0.00050	0.00817	93.9%
Solstice control solvent background	2.09167	2.09194	0.00027					
L-14780 - **L1**	2.12065	2.12842	0.00777	2.11790	2.11792	0.00002	0.00779	100.0%
L-14780 - **L2**	2.09709	2.10524	0.00815	2.14730	2.14730	0.00000	0.00815	100.2%
L-14780 - **L3**	2.18515	2.19270	0.00755	2.13079	2.13079	0.00000	0.00755	100.3%
L-14780 control solvent background	2.14930	2.14931	0.00001					

Notes: % Removal Efficiency is the NVR mass removed by the test solvent rinse over the total removed by both rinses. Solvent background is subtracted.
*Yellow highlighted cell: High measured solvent NVR background.
Results greater than 100% are due to the error tolerance of the analytical balance.

Contaminant: Christo-Lube
Concentration: 2.0034 mg/ml (10.017mg/panel)

Nonvolatile Residue Removal Efficiency Test Versus AK-225G

	AK-225G	Solstice PF	L-14780
1	98.7%	100.5%	100.0%
2	100.3%	100.3%	100.2%
3	100.1%	93.9%	100.3%


Christo-Lube Removal Efficiency
% removed by the test solvent of total removed
First rinse with test solvent, second rinse with AK-225G

Return to Menu Return to Pg 24

APPENDIX I—INDEPENDENT ASSESSMENT INVESTIGATION, EXTENDED OXYGEN COMPATIBILITY TEST DATA, AND OXYGEN COMPATIBILITY ANALYSIS

Various reports can be found in the following sections within appendix I:

I.1 Independent Assessment Investigation—Ambient Pressure LOX Impact Testing, October 21–22, 2014
I.2 MSFC Final Oxygen Compatibility Data
I.3 WSTF # 15-46483, NASA White Sands Test Facility Solvent Investigation Special Report, January 2014

Return to App I
Return to Pg 32

HCFC225cb (AK225G) Replacement Study

3rd Technical Interchange Meeting

Independent Assessment Investigation

Ambient Pressure LOX Impact Testing

Hosted by
NASA / WSTF

Oct 21-22, 2014

Steven Gentz NASA / NESC
H. Richard Ross - A2R / SSC Gas & Materials Science

Introduction

- The initial mechanical impact testing of candidate solvents performed at WSTF and MSFC exhibited significant differences for reactivity in LOX.
  - WSTF had observed high reaction rates
  - MSFC had not encountered any reactions
  - Both facilities used ASTM G86 for the standard method to determine the impact sensitivity in LOX at ambient pressure
- As a result of the discrepant results, a NASA Independent Assessment Team sponsored by NESC was formed to investigate the test variables and conditions that could affect the reactivity in LOX and to establish a modified test protocol to ASTM G86 for a subsequent set of tests that would provide a reactivity ranking of the candidate solvents.

Initial Reactions Observed by NESC / IA Team at WSTF

June 23, 2014 Solstice PF Lot BA320B-10-118 (1.6 lb)

Energy (ft. lbs)	Temp ^{0}F	% RH	Reaction Frequency	Reaction Rate	SST Disks
72	66	28	2/5	40%	No
65	66	28	2/6	33.3%	No
60	66	28	1/11	9.1%	No
55	66	28	2/6	33.3	No
50	66	28	2/12	16.7%	No
45	66	28	0/3	0%	No

Overall Reaction Rate 20.9%

June 27, 2014 Solstice PF - Same Lot as Above, but from a Different Bottle

Energy (ft. lbs)	Temp ^{0}F	% RH	Reaction Frequency	Reaction Rate	SST Disks
72	67	23	2/3	66.7%	No
65	67	23	2/4	50%	No
60	67	23	2/6	33.3%	No
55	67	23	2/10	20	No
50	67	23	2/7	28.6%	No
45	67	23	2/2	100%	No
40	67	23	2/7	28.6	No
35	67	23	1/9	11.1%	No

Overall Reaction Rate 31.2%

Initial Reactions Observed
by NESC/ IA Team at MSFC

MSFC July 8, 2014 Solstice PF Lot BA320BU-10-119 (1.6 lb)

Energy (ft. lbs)	Temp ^{0}F	% RH	Reaction Freq.	Reaction Rate	SST Disks
72	75	**65**	0/10	**0%**	No
72	75	**65**	0/20	**0%**	Yes
72	75	**65**	0/5 [1]	**0%**	Yes
82.4	75	**65**	0/5	**0%**	Yes

Overall
Reaction
Rate 0%

---

[1] The insert sample (grease) cups were 50% filled with the solvent.

MSFC July 10 , 2014 Solstice PF Lot BA320BU-10-110 (10 lb)

Energy (ft. lbs)	Temp ^{0}F	% RH	Reaction Freq.	Reaction Rate	SST Disks
72	79	**66**	0/22	**0%**	No

Overall
Reaction
Rate 0%

Test Variables / Conditions Observed

- WSTF elevation imposes lower ambient pressure and lower boiling points for LOX and solvent.
- Lower humidity range at WSTF vs MSFC.
- Initially, MSFC and WSTF did not test the same solvent lots.
- Sample preparation differs significantly at each facility.
- MSFC places the SST insert disks inside the aluminum cups and below the SST insert (grease) cups – SST disks are not used for the standard tests at WSTF.
- WSTF reported they used 347SST insert cups. MSFC reported they used 304SST insert cups.
- Different cleanliness level for hardware used at each facility. Level 50A was used at WSTF – MSFC cleans components to MSFC 164 Level IV X "A" (MSFC cleanliness level is comparable to 400A).
- Facility configuration and detection process for observing reactions are different at each facility.

Approach

- Examine and evaluate the test methodology & approaches used at each facility and analyze the test data generated.
- Investigate the test variables and operating conditions that could influence the reactivity rate at MSFC and WSTF and the effect/s solvents may have on test sensitivity.

Process Parameters Evaluated

- Impact Tester Operation & Calibration
- Test Variables (e.g. sample preparation methods, use of the plummet (rebound) catcher, SST disks below the sample insert cups, environmental conditions, etc.)
- Solvent Characteristics
- Type of Impacts vs. Reactivity Rate
- Cleanliness & Contamination Control
- Interpretation of Impact Results by Test Personnel

Process Variability

- No standard reference materials or consensus standards are available for the ASTM G86 test method to assess any measurement bias.
- No prior studies using halogenated solvents were performed to evaluate the variability of mechanical impact testing between MSFC and WSTF.

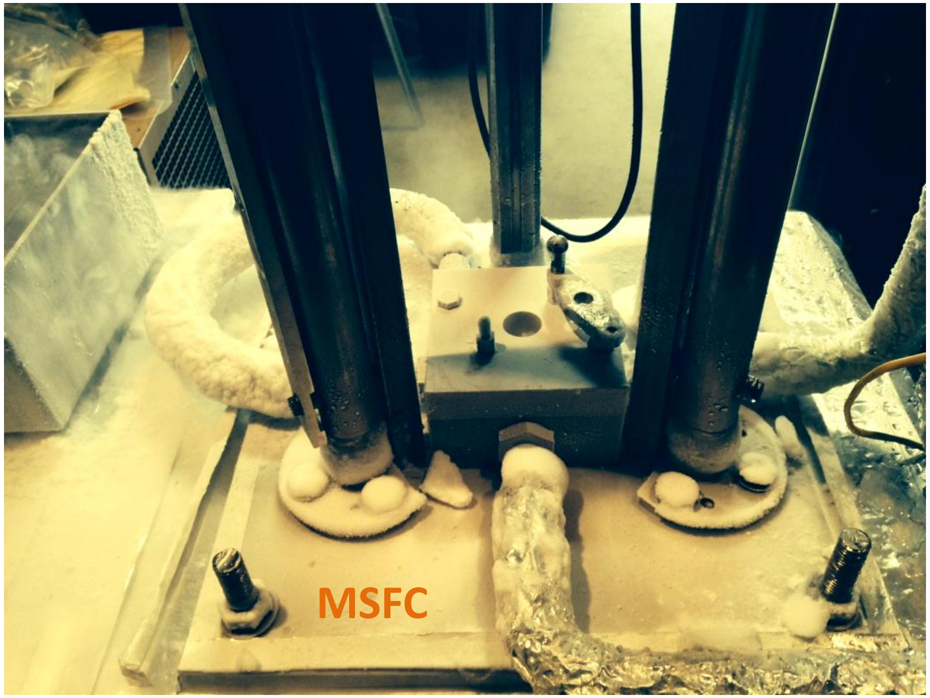
Ambient Pressure Impact Testers

Both testers have a rugged structural frame with the same basic drop tower configuration. Both testers rest on reinforced concrete. The base plate for each tester is anchored to a raised concrete structure with 4 SST bolts.

Process Variation

Cooling the Impact Tester Base

The base is cooled by adding LN2 to the moat. The moat surrounds the sample cup holder in the base. LN2 level in the moat is maintained throughout the testing using multiple Dewar transfers.



The base is cooled by flowing LN2 through the base that surrounds the sample cup holder. Cooling was verified when a LOX filled sample cup holder (blank) was placed into the base. The cup sat for ≈ 4 minutes with no visible boiling or decreased LOX level. Qualitatively, the base is sufficiently cooled. The LN2 flow was maintained during the tests.

Process Variation

- Impact Calibration
  - Dent block testing did not appear to be a scheduled test at MSFC (required to verify impact force is within spec per ASTM G86). Based on IA concerns, dent tests were performed in Aug 2014. WSTF provided the 304 SST dent blocks & the hemispherical striker pins. MSFC performed the dent block drops and reported the plummet mass. WSTF calculated the impact penetration vs. the energy level.
- Sample Preparation
  - Dispensing and obtaining uniform solvent thickness in sample cup is difficult.
  - Solvent testing is infrequent at both facilities. During the initial investigation, both centers were still adjusting their preparation techniques
    - No apparent requirement for frozen test material characteristics (sample uniformity & accumulation limit of frozen material on the sample cup rim)
    - Liquid response to freezing – WSTF evaluated solvent drip directly into the cup on the chill bar vs. filling a cup on SST (cold plate) located outside the chill pan and transferring onto the chill bar.
    - LN2 in MSFC cold box varied and empty sample cup accumulated water frost over extended time
  - Sample preparation techniques have improved; however, the nature of volatile solvents do not allow them to be prepared identically.

MSFC Sample Prep Method

Samples are prepared entirely in a SST cold box filled with LN.

LN level is slightly below the retainer plate / tray.

After each sample cup was pre-chilled on the retainer plate, solvent transfer to the sample cup was done with a rubber bulb - eye dropper from a solvent dispensing bottle.

MSFC Sample Prep Method

Left. SST chill pan is placed near the center of the Cold Box and rests slightly above the retainer plate.

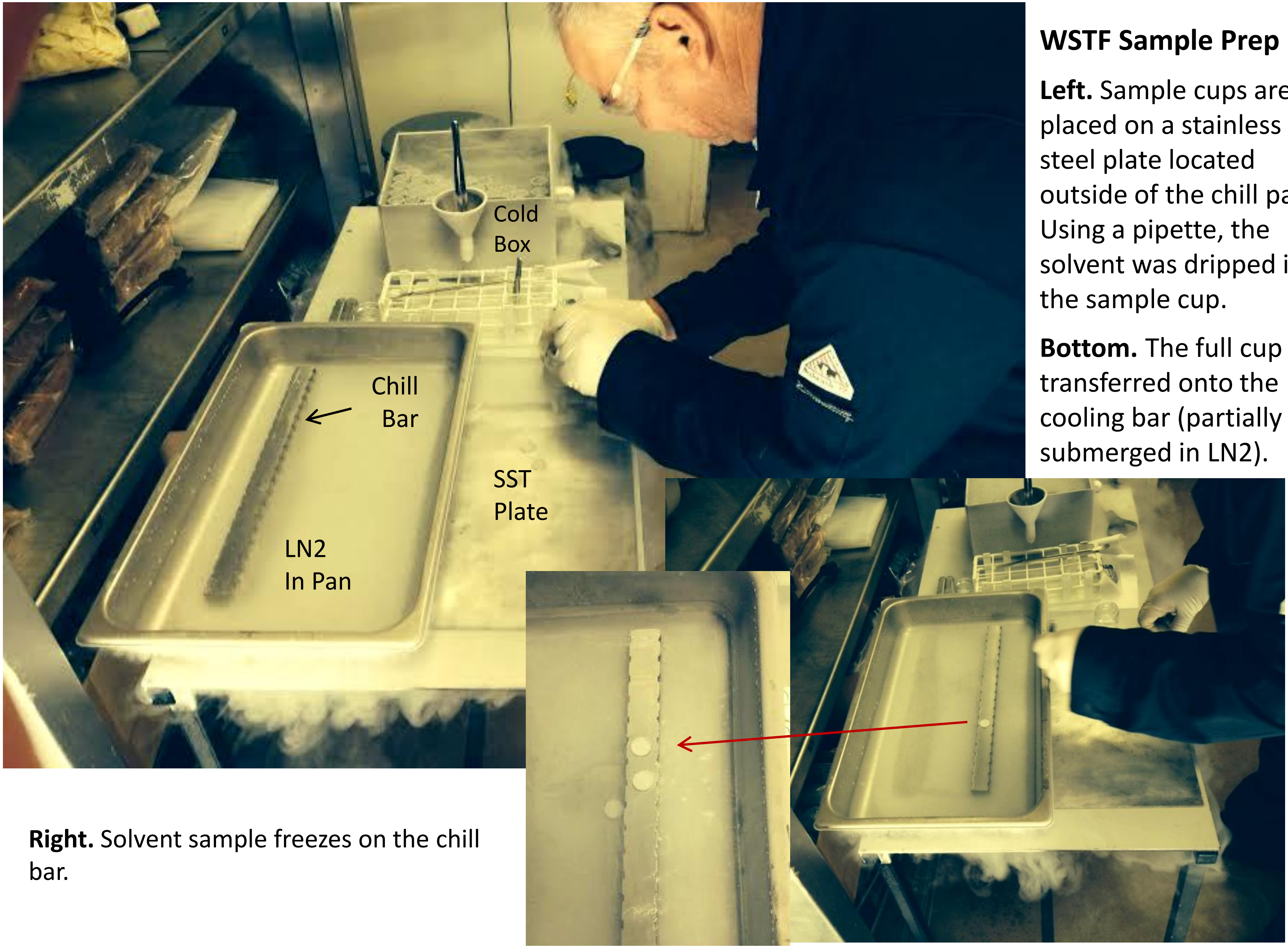
The sample cups are placed in the chill pan. The LN level in the Cold Box was raised slightly above the retainer plate. This allowed the LN to come in contact with the bottom of the chill pan.

Right. A SST ladle was used to slowly add LOX to the SST chill pan. The Al cups w/ the frozen sample and the blanks were submerged in LOX for 30 minutes min.
The LN level in the cold box and the LOX level in the chill pan were maintained until all the impact tests are completed.

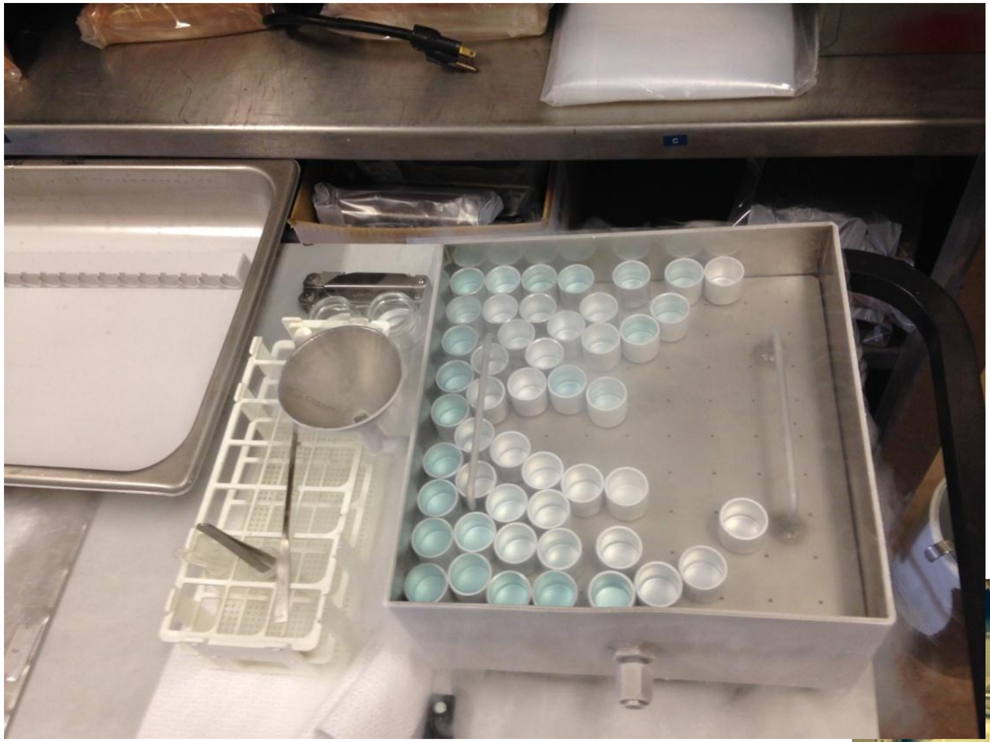
WSTF Sample Prep

Left. Sample cups are placed on a stainless steel plate located outside of the chill pan. Using a pipette, the solvent was dripped into the sample cup.

Bottom. The full cup was transferred onto the cooling bar (partially submerged in LN2).



Right. Solvent sample freezes on the chill bar.



WSTF Sample Prep

Samples are transferred to the pre chilled sample cup holders in the Cold Box.

LOX level is slowly raised until the cups are covered in LOX. Samples are covered in LOX for 30 to 40 min.

Additions of LOX were made, as necessary, while tests were in progress to ensure that each sample cup is completely filled at time of testing.

Sample Preparation & Contamination Control

Isolated sample fragments are present on the frozen samples during the LOX soak.

No cover was placed over the chill box during the LOX soak to prevent a potential buildup from atmospheric contaminants (particulate and condensable matter).

WSTF Preparation

Sep 16, 2014

Sample Preparation & Uniformity

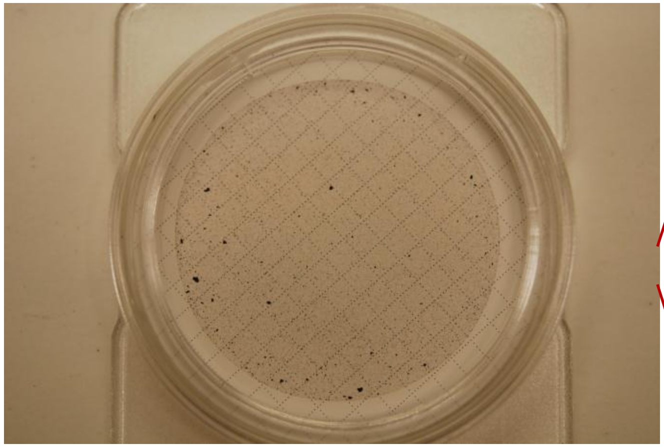
Isolated sample fragments on the frozen samples prior to the LOX soak. Samples show various levels of uniformity and thickness.

Process Variation

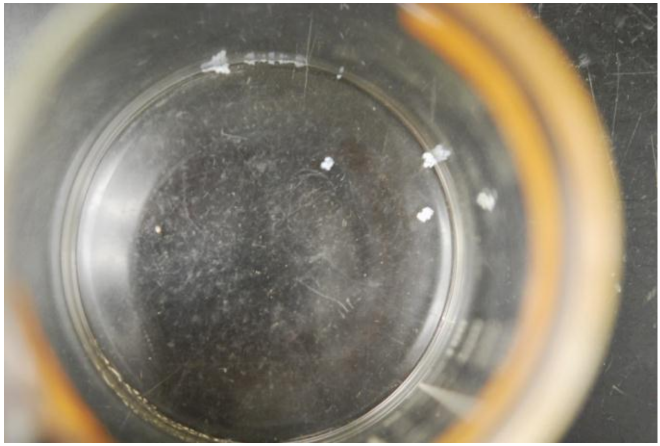
- Solvent Characteristics
  - No NVR testing or solvent filtration is performed - storage container can introduce particulates / contaminants - filtration to remove particulates and reporting the background NVR of is not a requirement in ASTM G86.
  - Solvent NVR and particulates can skew reactivity.
    - Particulates can act as nucleation sites and effect the freezing characteristics / density.
    - The type and amount of NVR can influence the reactivity.
  - Azeotropes
    - Solvent components may not be uniform at cryogenic temperatures (e.g. enriched regions of t-DCE and /or stabilizer/s).

Process Variations

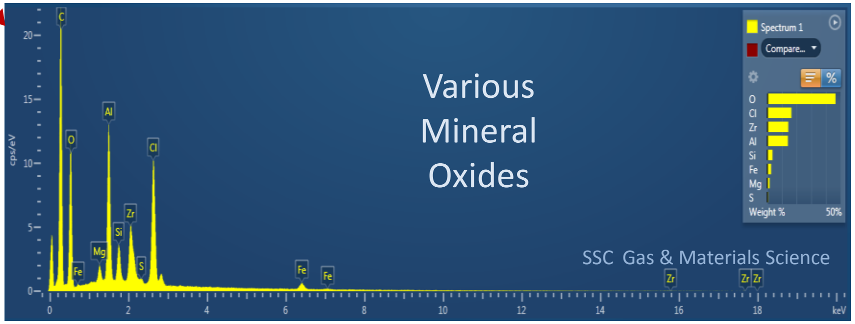
Solvent Particulates



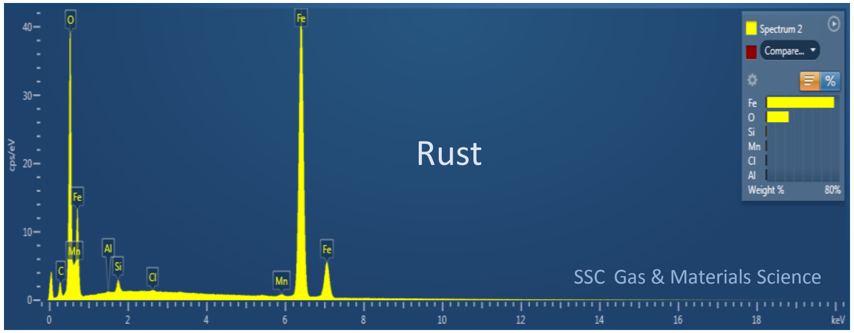
100 ml of Solstice PF in Carbon Steel Cylinder
Lot #: BB337A-10-24



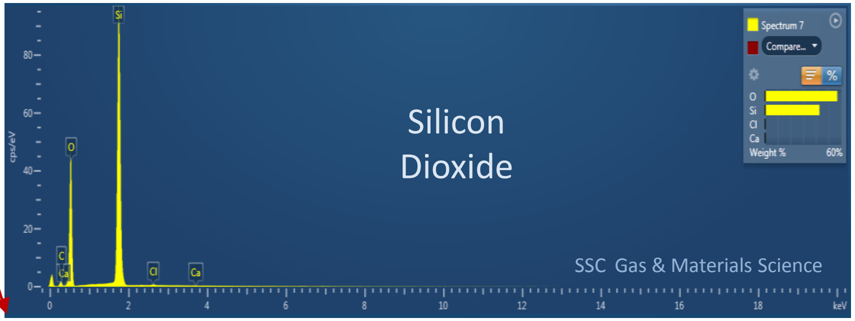
Particulate matter remaining after boiling away
100ml of Solstice PF, Lot # BB-257A-U-10-53



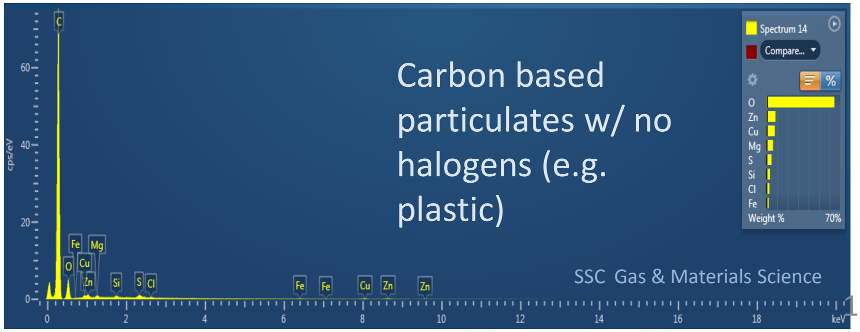
Various Mineral Oxides



Rust



Silicon Dioxide



Carbon based particulates w/ no halogens (e.g. plastic)

Use of SST Insert Disks per ASTM G86-98a

- For ambient pressure LOX impact testing of solvents, MSFC uses SST disks. WSTF does not use SST disks.
  - Tests performed at MSFC and WSTF show this can cause different reaction rates.
- Use of SST Disks are specified in:
  - Par. 7.3, Method 1 - *Leak Check Compounds, Dye Penetrant & Emulsifier* specifies sulfuric acid anodized 6061 T6 aluminum disks or any substrate specified by the manufacturer.
  - Par 7.4 Method 2 -*Preparation of Leak Check Compounds and Emulsifer* specifies to use sample cups or type 316SST disks (see fig. 8). NOTE: Figure 8 is not a disk, but is an "insert sample cup" that specifies 347 SST. Interchanging terms (disks and insert cups that use different alloys) can lead to test variations.
  - Par 7.8, *Coating materials such as Paints, dry film lubricants* shall be applied to 316SST or 347SST disks. "After applying the coatings on the disks and they have dried, they shall be placed in regular sample cups for testing ."
- SST Disks are not specified in:
  - Par. 7.2 Liquid Samples – transfer the samples into special cups (Fig.7). NOTE: Figure 7 shows a special insert cup fabricated from any 3000 or 5000 aluminum alloy. However, there is no reference in this section to use SST disks.

Use of SST Insert Sample Cups in ASTM G86-98a

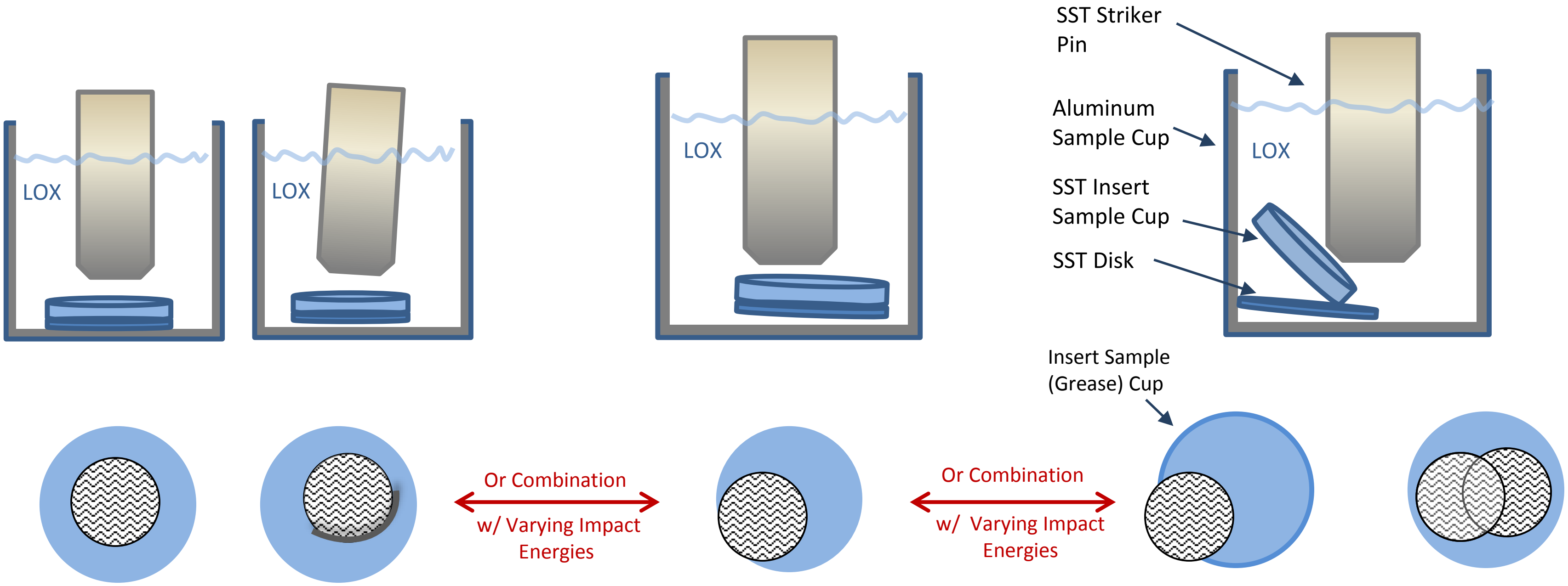
- SST insert cups are not specified in:
  - Par. 7.2 Preparation of Liquid Samples – transfer the samples into special cups (Fig.7). NOTE: Figure 7 shows a special insert cup fabricated from any 3000 or 5000 aluminum alloy. There is no reference to use SST insert cups. Also, a cautionary note should be added – " testing halogenated solvents & compounds with aluminum insert cups can cause a reaction and should not be used."
  - MSFC uses 304SST Insert Cups and WSTF uses 347SST Insert Cups. Neither insert sample cup alloy is specified for liquid samples in par 7.2.
- SST insert cups are specified in:
  - Par 10.1.1.5 Sample Cups (under section 10, Apparatus) specifies a special insert cup made of any dead soft 3000 or 5000 series aluminum alloy (see Fig. 7).
  - Par 10.1.1.7 Auxiliary Equipment (under section 10, Apparatus), includes a grease cup holder, see Fig. 8. Fig. 8 is an in inset sample cup that lists SST 347. This detail should be included in 7.5, for the Preparation of Greases that specifies to use an aluminum cup inset. Sections 7.5 and 10.1.1.7 specify different insert cup requirements for preparing greases & semi solid samples.

    and again in

  - Par 10.2.1.7 Sample Cups (also under section 10, Apparatus) specifies a one piece cup used for liquid or non-solid materials (See Fig.14). Fig 14 shows a 316SST sample cup and is not identified as an insert sample cup, yet it has the same dimensions as an insert sample cup specified in Fig. 7 for the preparation of liquid samples that is constructed from any 3000 or 5000 series aluminum alloy. To eliminate the potential for process variability, the insert sample cups with their respective alloys should be fully specified in their applicable sections (e.g. 7.2 for Liquids, 7.5 for Greases, etc.)

- The ASTM standard does not use consistent terminology and is easy to misread since applicable details are not always addressed in the appropriate sections. This condition appears to have caused an interpretation issue on the use the SST disks and the alloys used for the sample cup inserts .

LOX Mechanical Impact Events Observed



LOX

SST Striker Pin

Aluminum Sample Cup

SST Insert Sample Cup

SST Disk

Insert Sample (Grease) Cup

Or Combination w/ Varying Impact Energies

Or Combination w/ Varying Impact Energies

Centered or Near Centered Impact

Non Uniform (Off Angle) Impact

Edge of the Striker Pin Impacts the Inner Rim Wall

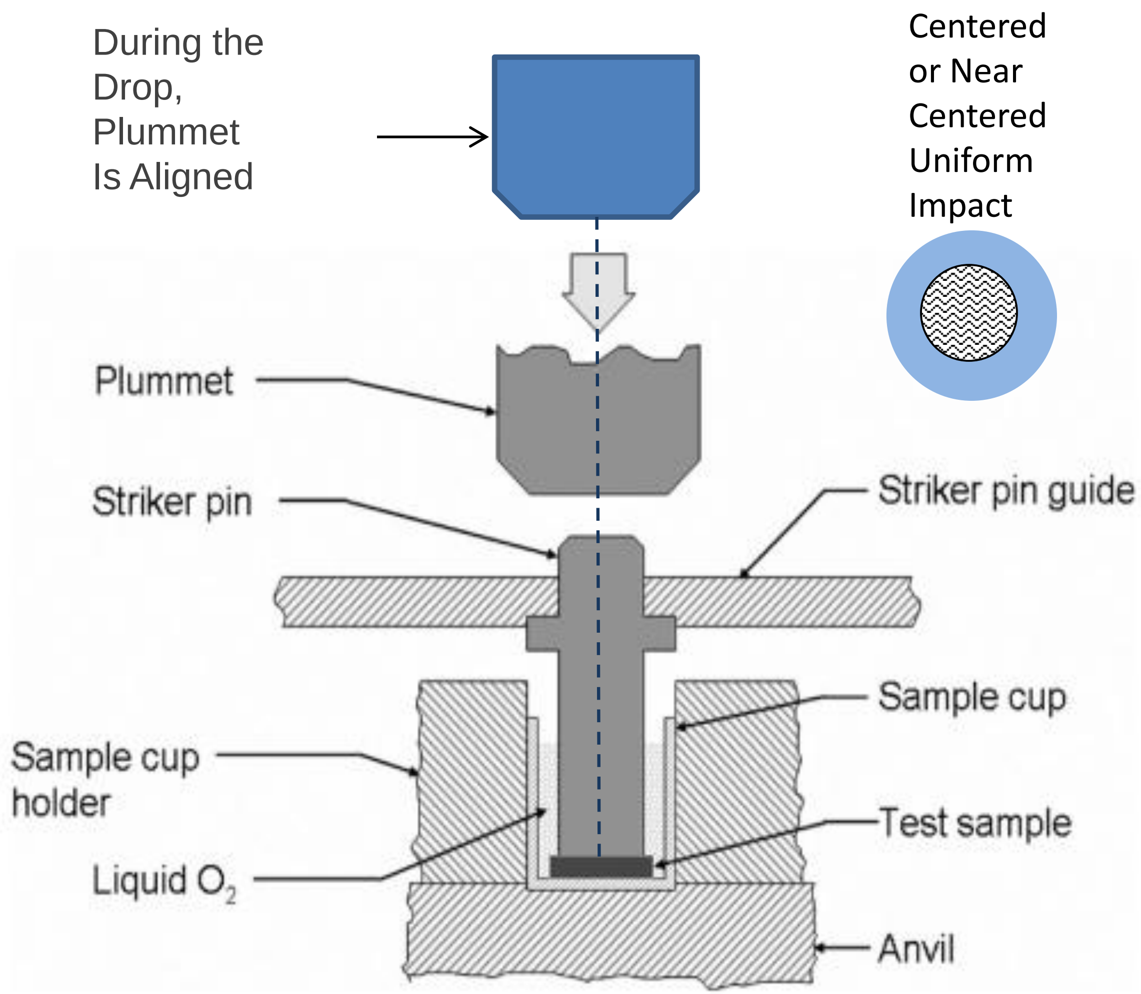
Rim Impact (Edge Effect)

Intersecting Impacts Can Produce an Edge Effect

The majority of the reactions were from non-uniform impacts & were due to:

- Rebound impacts occurred after relocation and a change of orientation of the sample cup following the initial impact. Also, the rebound energy imparted to the sample is unknown.
- Off Angle Impact from random non-perpendicular contacts between the plunger & the striker pin
- Edge of striker pin Impacts the inner rim wall and/or the striker pin hits the rim of the insert cup
- How well the sample cup insert and the insert disc were centered in the aluminum sample holder.

Plummet Perpendicular to the Vertical Drop Axis at Impact with the Striker Pin



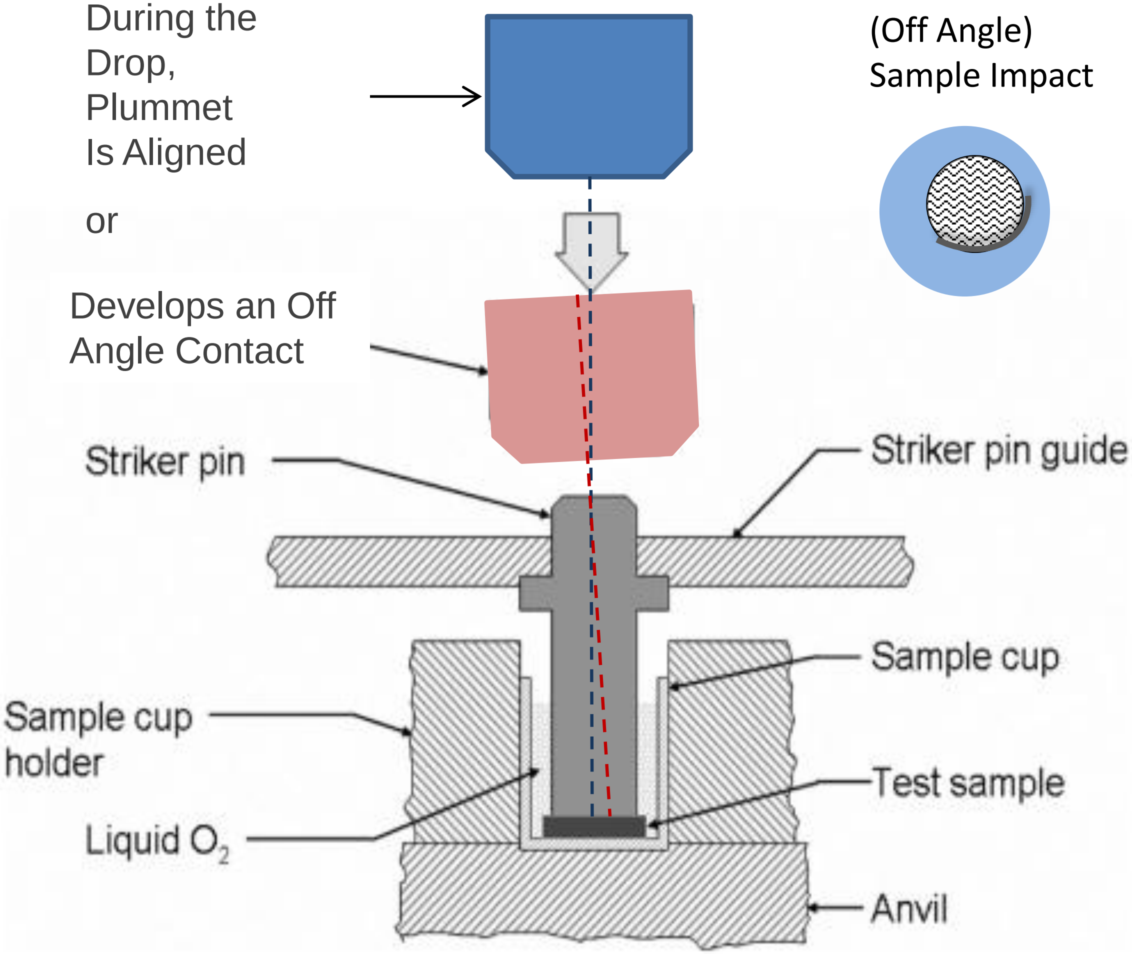
During the
Drop,
Plummet
Is Aligned

Centered
or Near
Centered
Uniform
Impact

WSTF
Sep
2014

Spider plummet bearings are in the guide tract.

Plummet Off Perpendicular to the Vertical Drop Axis at Impact



During the Drop, Plummet Is Aligned

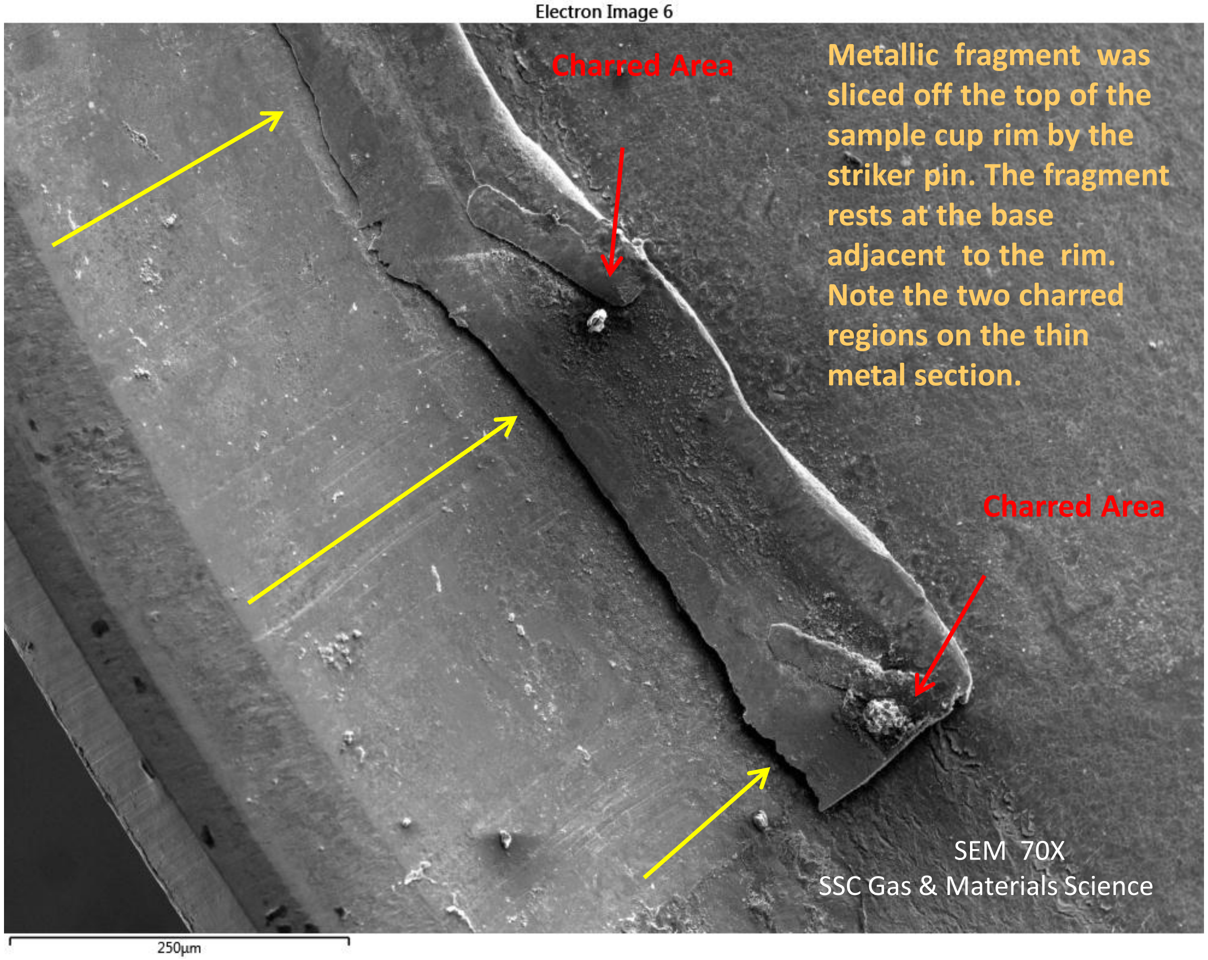
or

Develops an Off Angle Contact

Non Uniform (Off Angle) Sample Impact

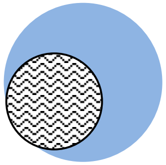
Mechanical play between the spider bearings and the guide track and the wear clearance in the striker pin guide can cause a non-uniform impact. The yellow arrows shows the upper & lower spider bearings are out of the guide track.

Striker Pin Impact On the Inner Rim Wall of the Sample Cup



Metallic fragment was sliced off the top of the sample cup rim by the striker pin. The fragment rests at the base adjacent to the rim. Note the two charred regions on the thin metal section.

SEM 70X
SSC Gas & Materials Science

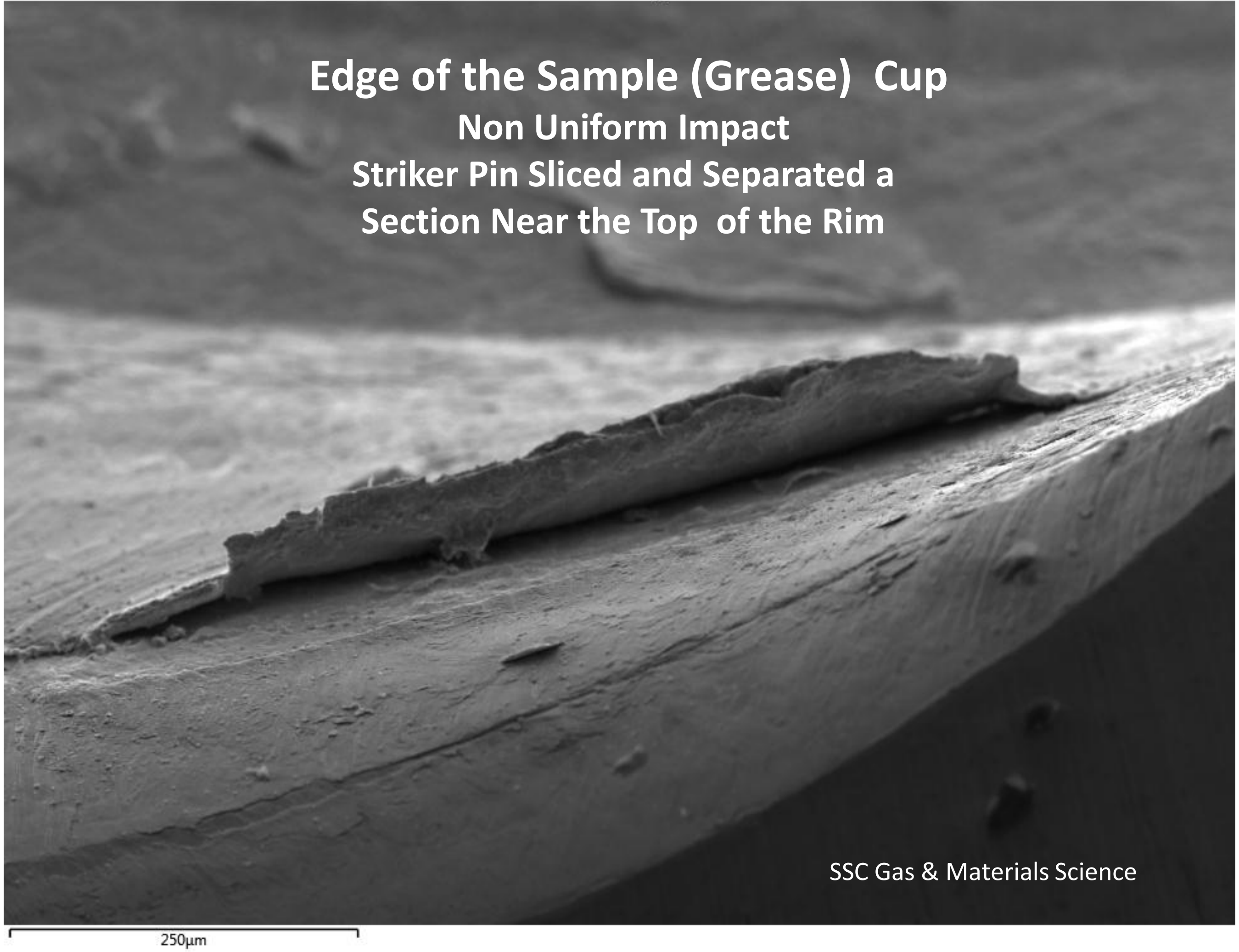


Edge of Striker Pin Impacted the Inner Rim Wall

The impacted area is subjected to a high strain rate from eccentric loading.

Striker Pin Impact On the Inner Rim Wall of the Sample Cup

Electron Image 8

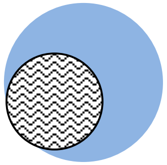


Edge of the Sample (Grease) Cup

Non Uniform Impact

Striker Pin Sliced and Separated a Section Near the Top of the Rim

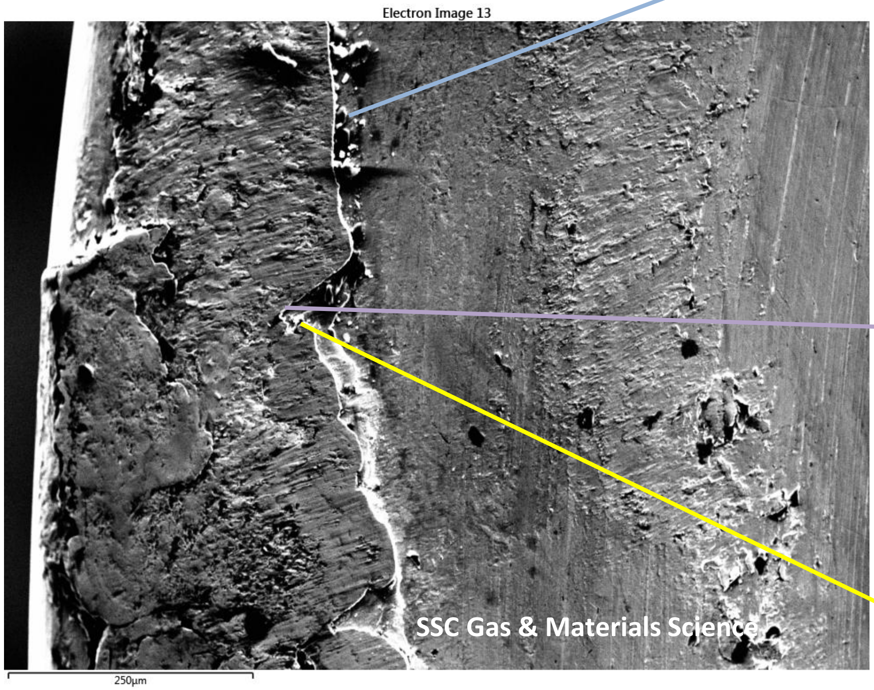
SSC Gas & Materials Science



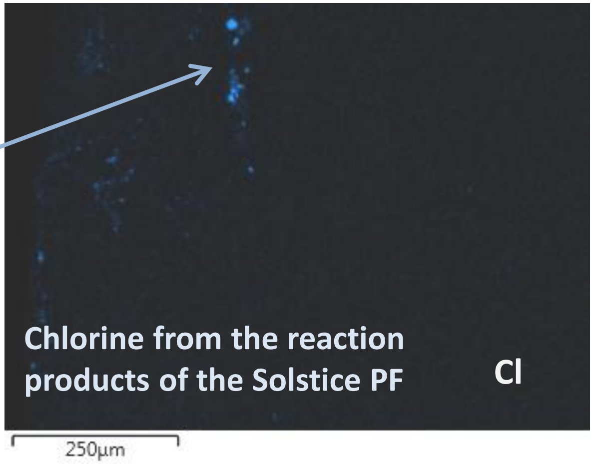
Edge of the
Striker Pin
Impacted the
Inner Rim Wall

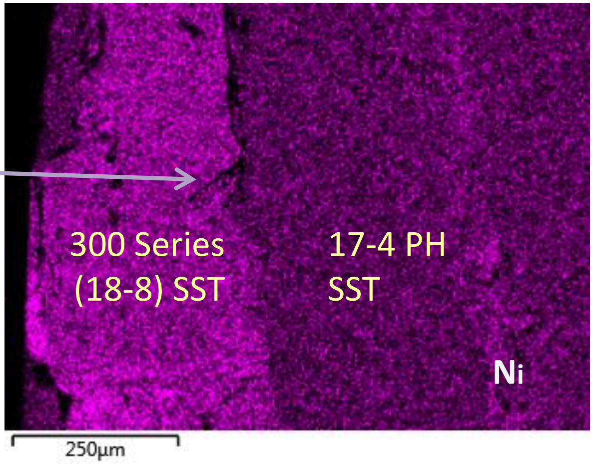
Striker Pin Edge Impact On the Inner Rim Wall of the Insert Sample Cup

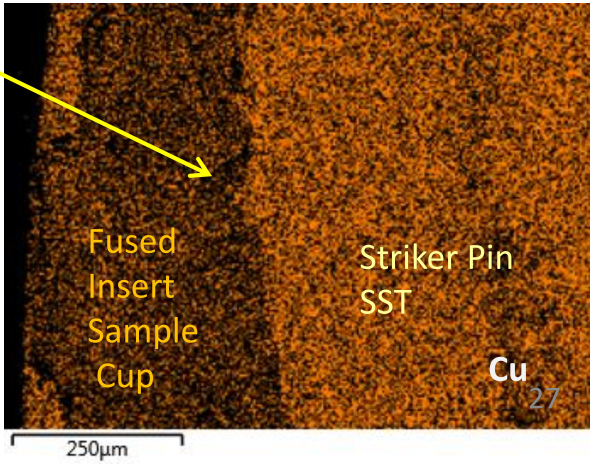
Corresponding X-Ray Maps of the bottom edge of the striker pin.



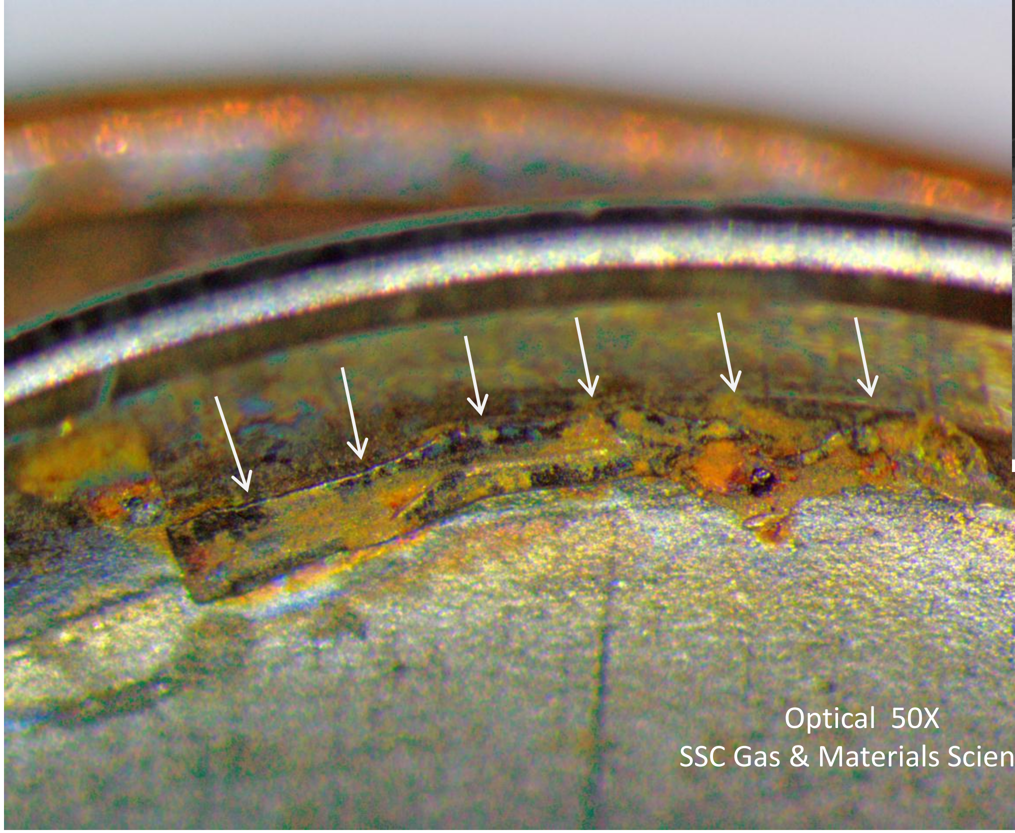
SEM Image. A thin section of the SST grease cup is fused to the bottom outer edge of the striker pin following an impact reaction at MSFC. The elevated Ni and reduced Cu intensities in the corresponding X-ray maps are due to a thin section of the 300 series SST from the sample cup adhering to the striker pin SST 17-4 PH.

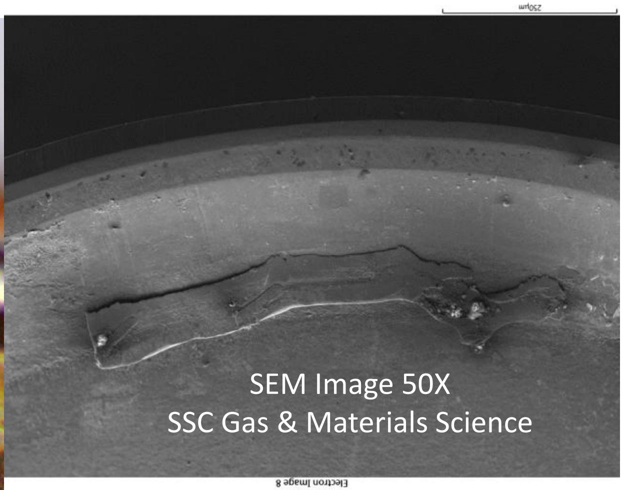




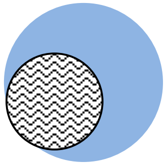


Striker Pin Edge Impact On the Inner Rim Wall Area of the Sample Cup

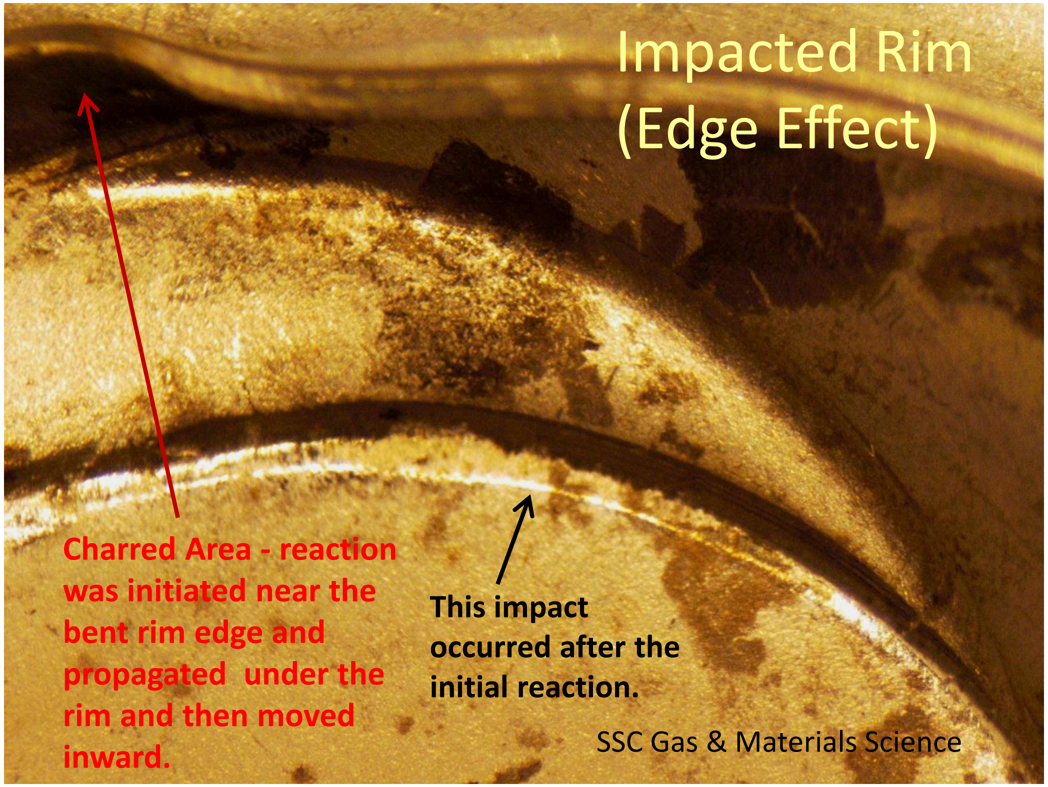

Optical 50X
SSC Gas & Materials Science



SEM Image 50X
SSC Gas & Materials Science


Edge Impact Occurred On the Inner Rim Wall

The white arrows show the metal section was peeled away from the rim area. A thermal oxide film adjacent to the metallic section is also present. The orange colors on the separated metal section represent the Solstice PF reaction products. Macro-reactions appear to be assisted by frictional forces that serve both to remove the oxide layers and to contribute to localized heating.

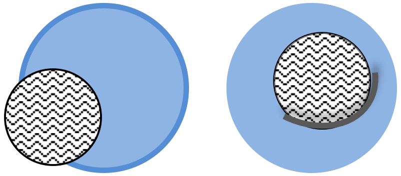


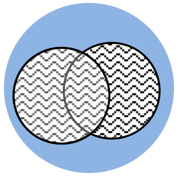
Charred Area - reaction was initiated near the bent rim edge and propagated under the rim and then moved inward.

This impact occurred after the initial reaction.

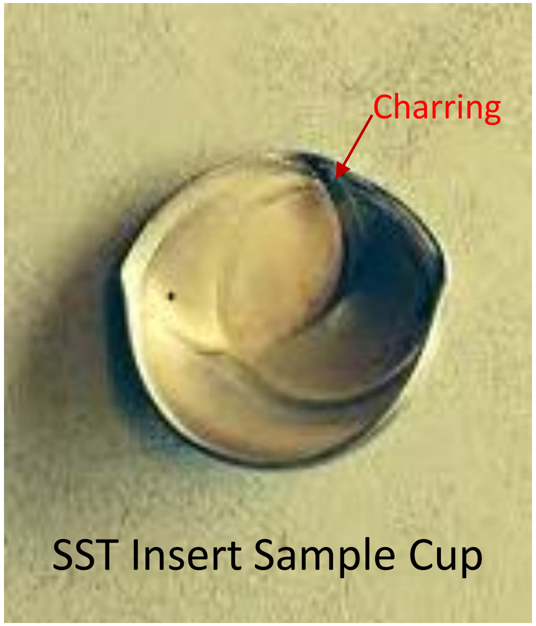
SSC Gas & Materials Science

Left. Rim impact followed by a non uniform rebound impact.



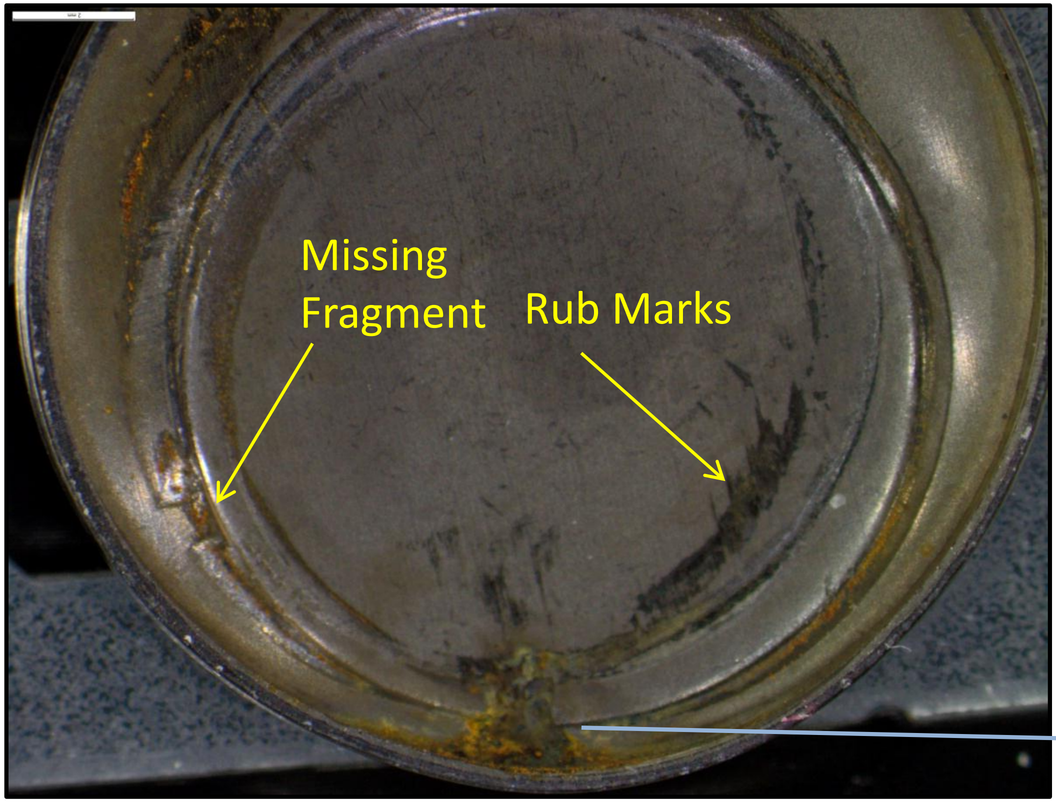


Rebounds can result in intersecting impacts and deformation of the insert sample cup

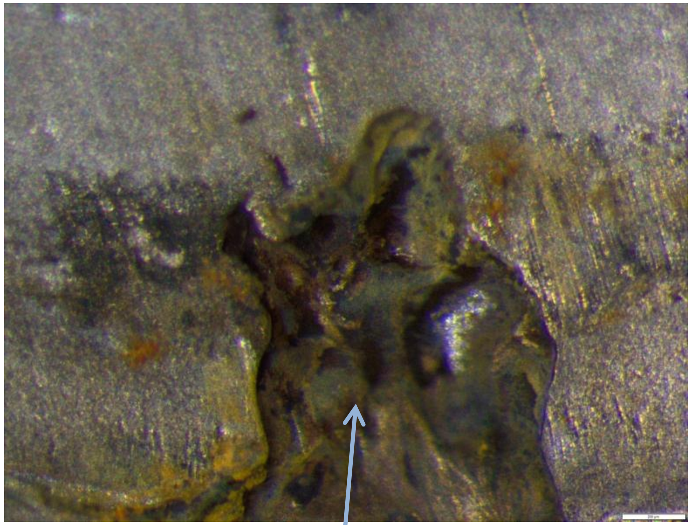


SST Insert Sample Cup

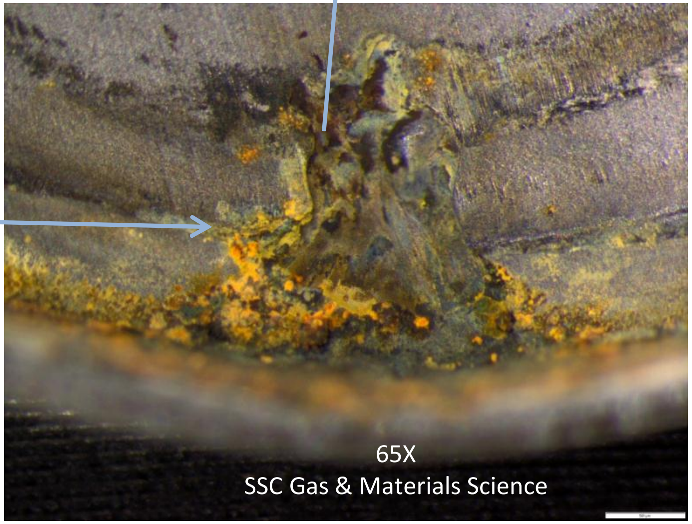
Multiple Overlapping Impacts



Insert Sample Cup With Multiple Overlapping Impacts On and Adjacent to the Rim Wall. Note the ridge that formed near the base of the rim wall from the overlapping impacts.



Spalling



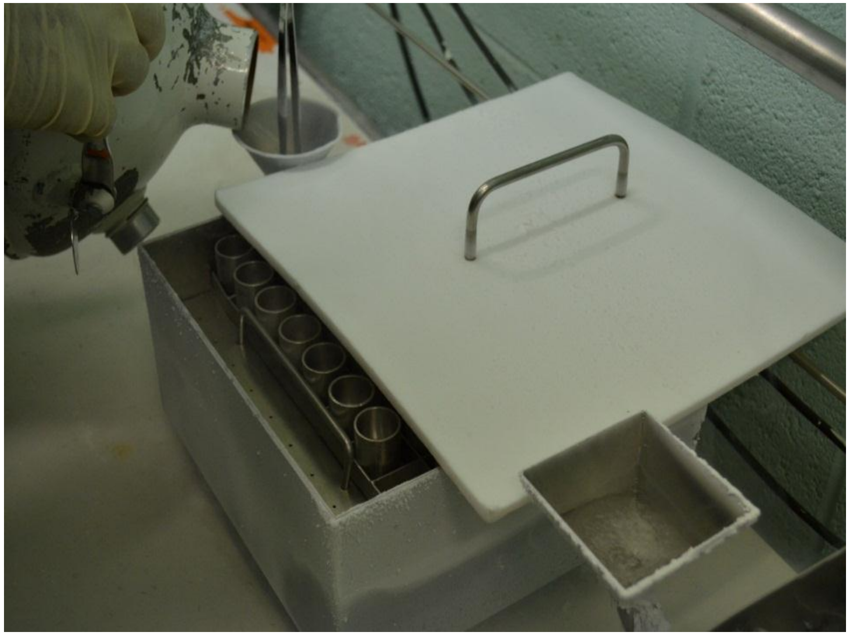
Solstice PF Reaction Products

Process Variation

- External Contamination
  - Handling hardware with non clean Latex gloves is not recommended, these gloves could introduce NVR and particulates. Lint free clean Nylon or Polyester gloves or SST tongs should be used for handling hardware with critical surfaces.
  - For handling non critical hardware, Polyethylene or Buna N gloves are preferred. Polyethylene gloves perform the best in both contact transfer handling and whenever solvent contact is a possibility.
  - Because of ergonomics and work operations, test personnel usually do not replace their gloves after handling non cleaned hardware and equipment -this condition leads to a buildup of contamination on the gloves that can be transferred to clean items . Consequently, the cleanliness level of the sampling / test hardware cannot be maintained.
  - Aqueous cleaned items used at WSTF (e.g. Chill Box) that receive a final rinse using potable water can introduce particulates, dissolved solids and NVR. Water used for the final rinse should have a specific resistance greater than 1 meg-ohm-cm or a conductivity of less than 1 micro-Siemen/cm, meet or exceed particulate cleanliness Level 400 and have a total carbon content 1ppm (max).

Process Variation

- External Contamination



Placing a SST cover on the Chill Box during the MSFC LOX soak should be required to minimize any potential buildup from atmospheric contaminants (particulates and condensable matter).

  - Rubber bulb from the eye dropper used by MSFC could introduce NVR and particulates - a glass micropipette is recommended.

Several Samples Remained Intact Following Impact Testing at WSTF

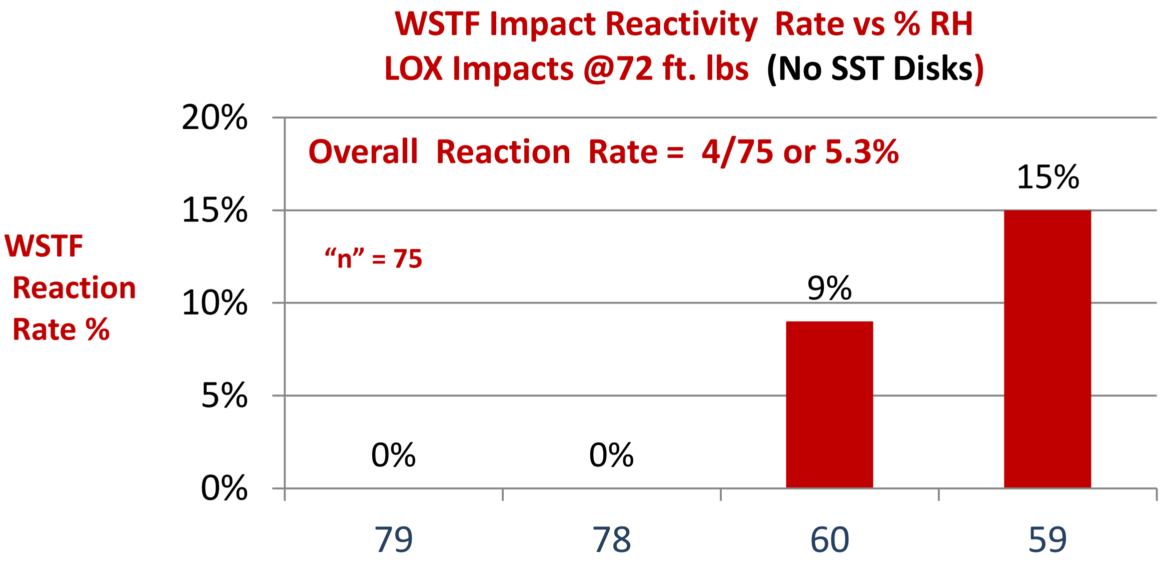
- Several post impact samples remained intact (had the same frozen appearance as freshly prepared samples)
  - With Rebound Catcher & No SST Disks
    - MSFC prepared samples - “16” samples remained intact at $\geq$ 72 ft lbs
    - WSTF prepared samples - “9” samples remained intact at 72 ft lbs.
  - With No Rebound Catcher & Using SST Disks
    - MSFC prepared samples - “3” samples remained intact at 60 ft lbs. & “1” sample tested on Sep 10 remained intact @ 72 ft lbs and exhibited charring on the striker pin. After the Solstice PF sample melted, charring was also observed at the interior bottom of the insert sample (grease) cup.
- After the post test intact samples evaporated, the insert sample (grease) cups showed uniform circular depressions from the striker pin. All the impacts struck near dead center.
- Data shows the plummet catcher reduces rebound (non-uniform) impacts.
- None of the MSFC or WSTF prepared samples remained intact following the impact tests that were done at MSFC (Aug 8-11, 2014).

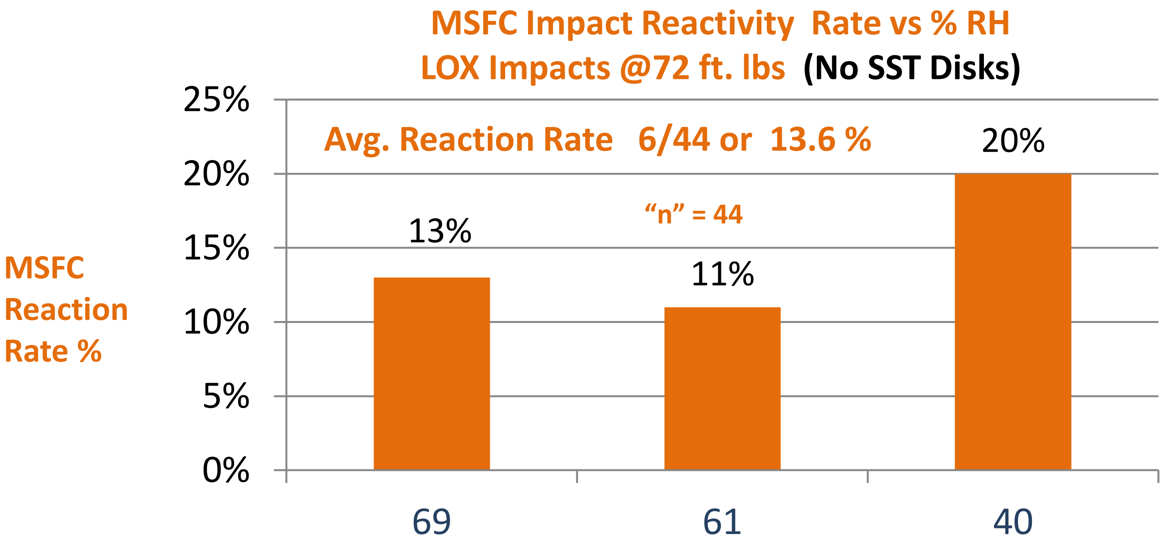
Several Solstice PF Samples Remained Intact (Uniform Solid) Following Impact Testing at WSTF

The bold orange and red entries show the intact samples after impact

Date	Prep By	Rebound Catcher	SST Disc	Intact Samples	Reaction Frequency	Energy Level
9/8/2014	WSTF	No	No	None	0/20	72
9/8/2014	WSTF	No	Yes	None	**2/9**	72
9/9/2014	MSFC	No	Yes	None	**2/23**	72
9/9/2014	**MSFC**	**No**	**Yes**	**3 /20**	0/20	60
9/10/2014	**MSFC**	**No**	**Yes**	**1/28 (Charring)**	**2/28**	72
9/11/2014	MSFC	No	No	None	**2/13**	72
9/11/2014	**MSFC**	**Yes**	**No**	**11/20**	0/20	72
9/11/2014	**MSFC**	**Yes**	**No**	**5/5**	0/5	82
9/12/2014	WSTF	No	No	None	**2/22**	72
9/15/2014	**WSTF**	**Yes**	**No**	**3/20**	**1/20**	72
9/16/2014	WSTF	No	No	None	0/20	72
9/16/2014	**WSTF**	**Yes**	**No**	**6/20**	0/20	72
9/16/2014	WSTF	No	Yes	None	**2/23**	72

Influence of the SST Disks on the LOX Impact Reaction Rates





MSFC and WSTF Reaction Rates

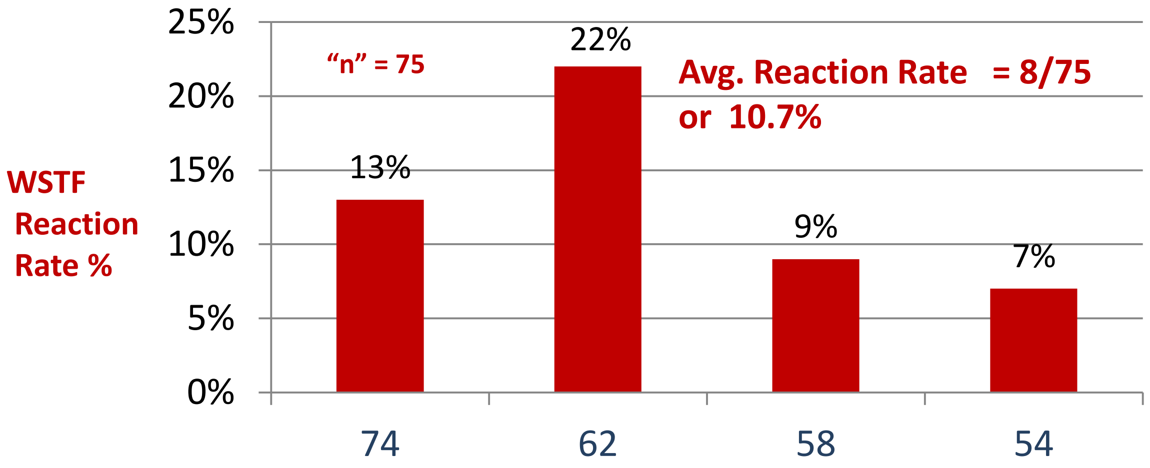
"No Plummet Catcher"

Solstice PF Lot # BR13019-50-42 (Unfiltered)

← **% RH (Mode) During Sample Prep**

← **% RH (Mode) During Sample Prep**

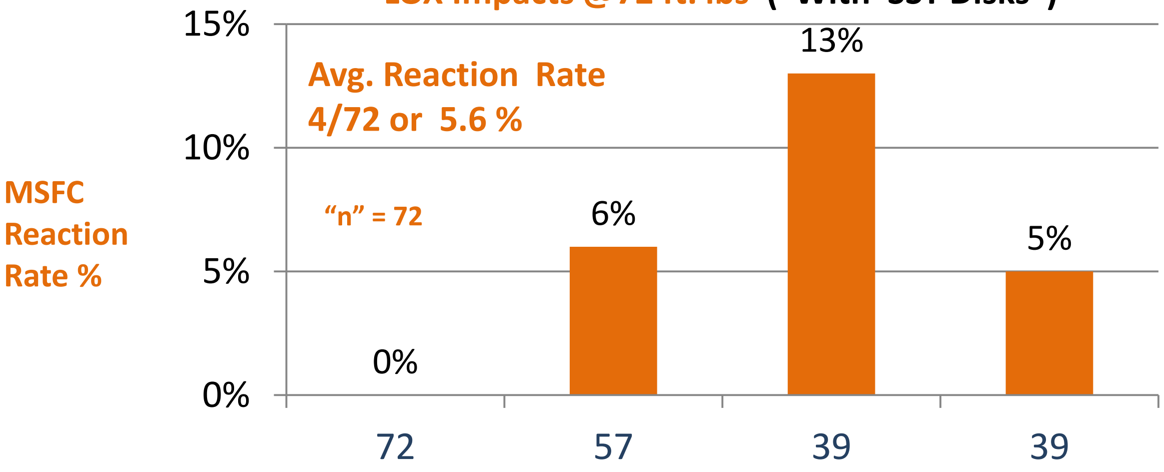
WSTF Impact Reactivity Rate vs % RH
LOX Impacts @72 ft. lbs ("With SST Disks")



"n" = 75

Avg. Reaction Rate = 8/75 or 10.7%

MSFC Impact Reactivity Rate vs % RH
LOX Impacts @72 ft. lbs ("With SST Disks")



Avg. Reaction Rate 4/72 or 5.6 %

"n" = 72

MSFC and WSTF Reaction Rates

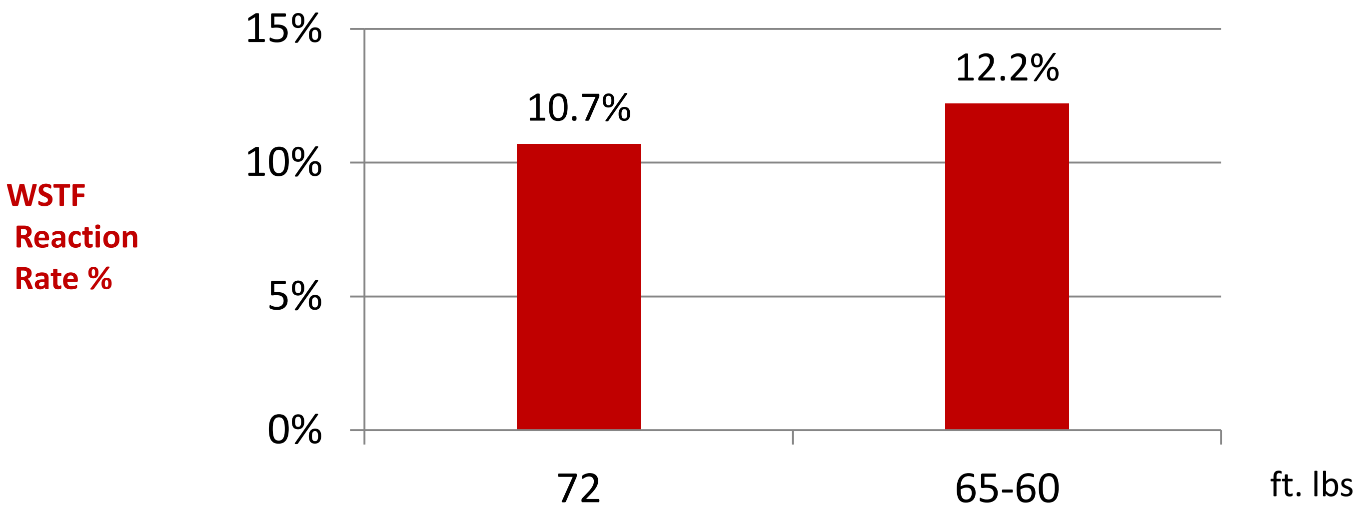
"No Plummet Catcher"

Solstice PF Lot # BR13019-50-42 (Unfiltered)

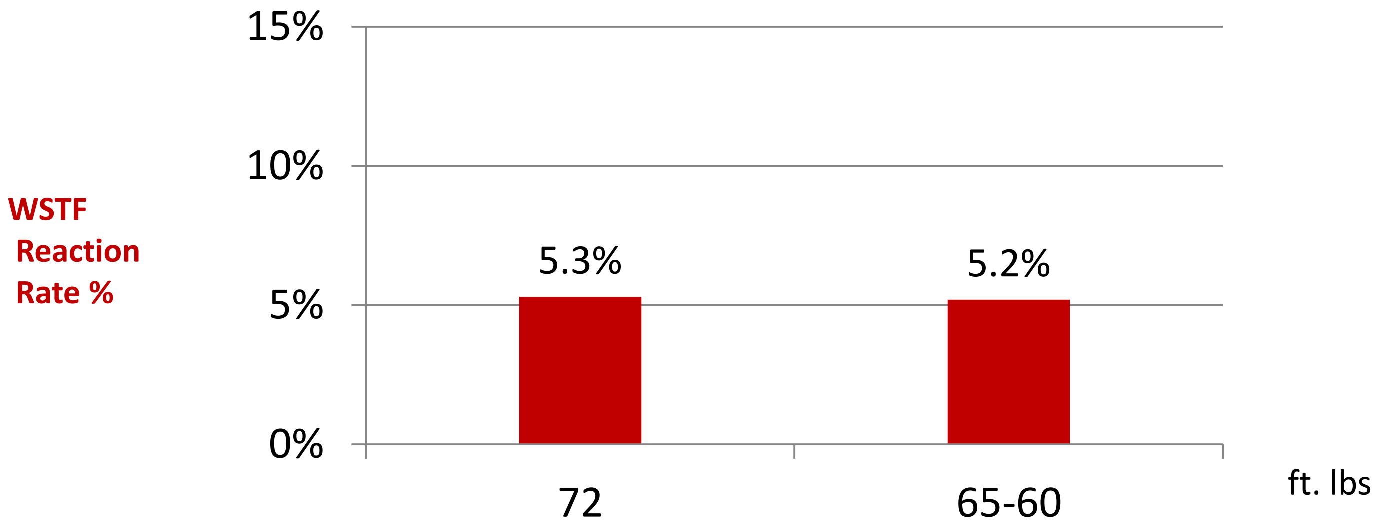
← % RH (Mode) During Sample Prep

← % RH (Mode) During Sample Prep

WSTF LOX Impact Reaction Rates for the Solstice PF Samples Tested
Sep 8 – 16, 2014

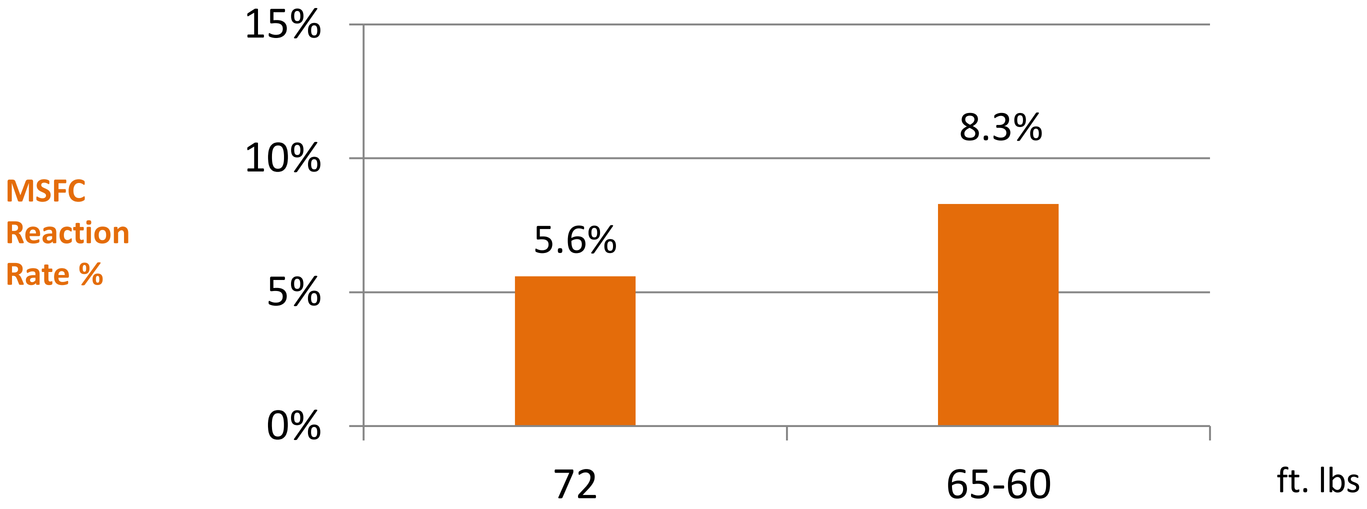


With SST Disks
No Rebound Catcher

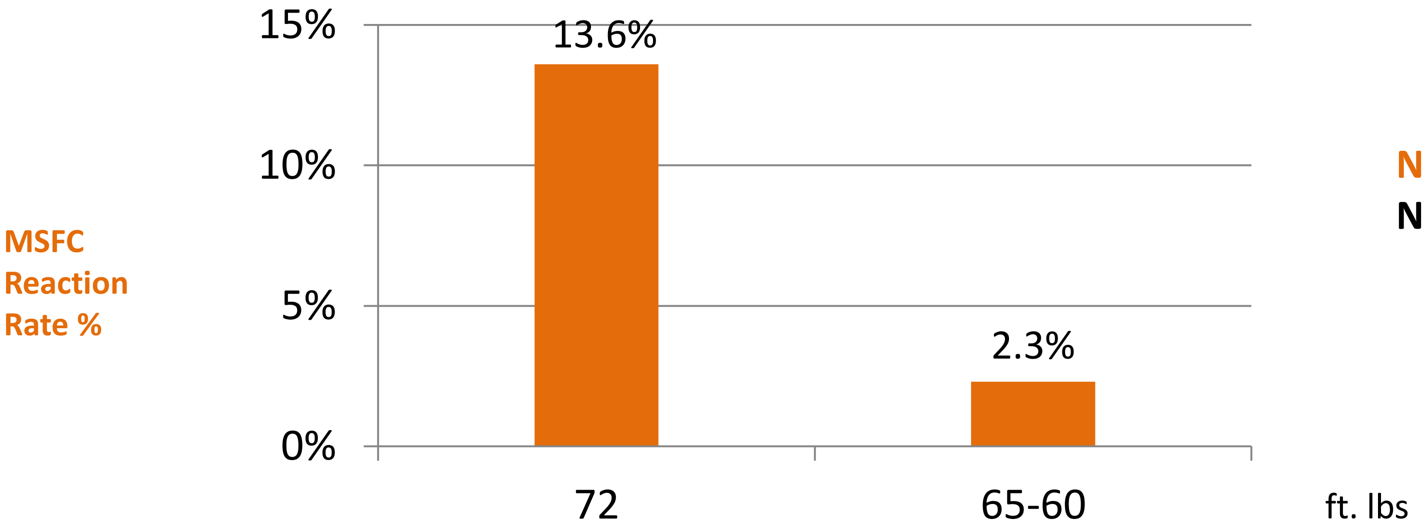


No SST Disks
No Rebound Catcher

MSFC LOX Impact Reaction Rates for the Solstice PF Samples Tested Aug 11-18, 2014

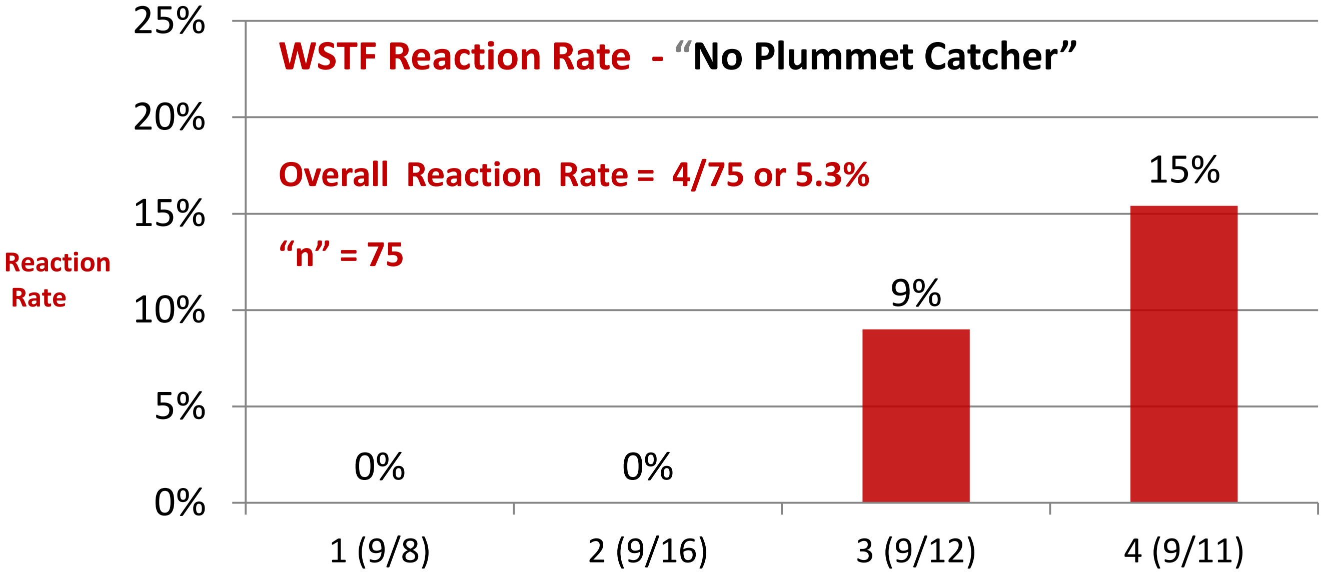


With SST Disks
No Rebound Catcher



No SST Disks
No Rebound Catcher

Reactivity Rates With and W/O the Plummet Catcher



WSTF Reaction Rate - “No Plummet Catcher”

Overall Reaction Rate = 4/75 or 5.3%

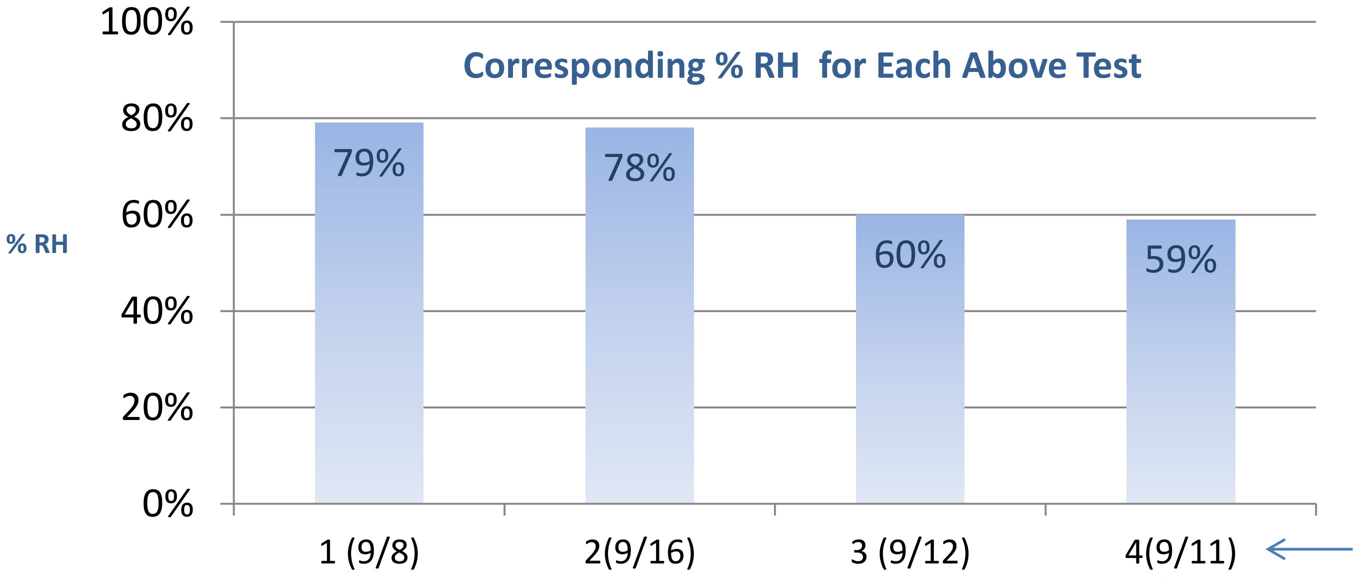
“n” = 75

Test Solvent:
Unfiltered Solstice PF
Lot # BR13019-50-42
LOX Impacts @72 ft. lbs.

“No SST Disks”

2014 Test Dates

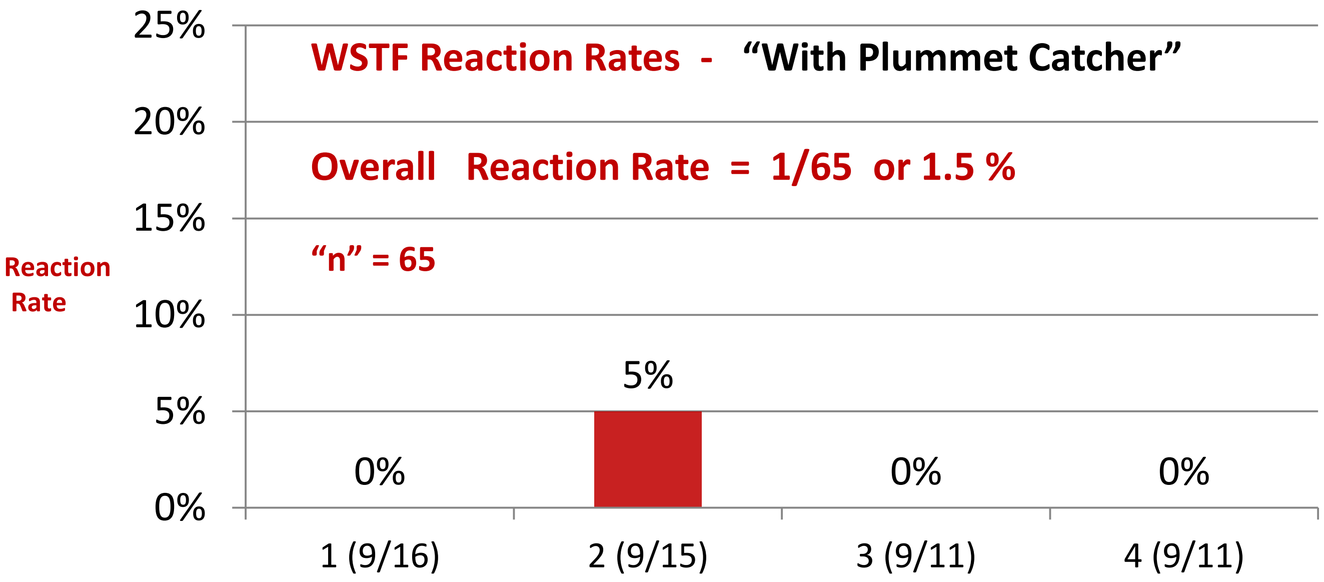
1 (9/8)	2 (9/16)	3 (9/12)	4 (9/11)	
0/20	**0/20**	**2/22**	**2/13**	**WSTF Reaction Frequency “Reactions/Drops”**



Corresponding % RH for Each Above Test

% RH (Mode) During the Sample Prep Session

Dates Arranged from High RH to Low RH.



WSTF Reaction Rates - "With Plummet Catcher"

Overall Reaction Rate = 1/65 or 1.5 %

"n" = 65

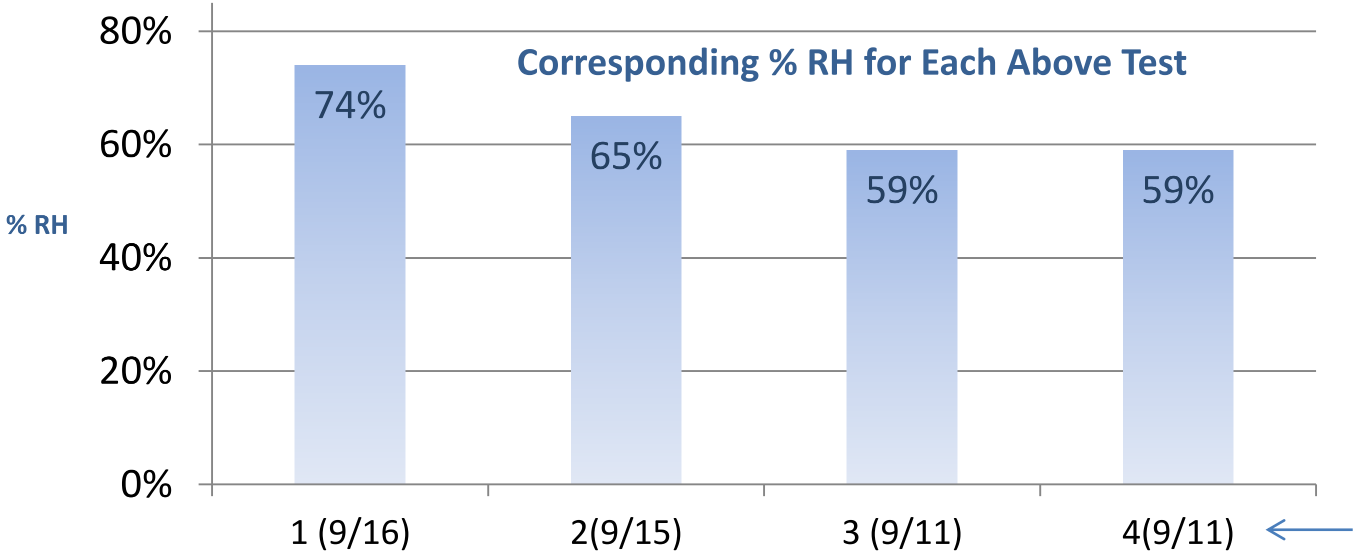
1 (9/16)	2 (9/15)	3 (9/11)	4 (9/11)
0/20	**1/20**	**0/20**	**0/5**

Test Solvent :
Unfiltered Solstice PF
Lot # BR13019-50-42
LOX Impacts @72 ft. lbs. & 5 drops @ 82 ft lbs. on 9/11.

"No SST Disks"

2014 Test Dates

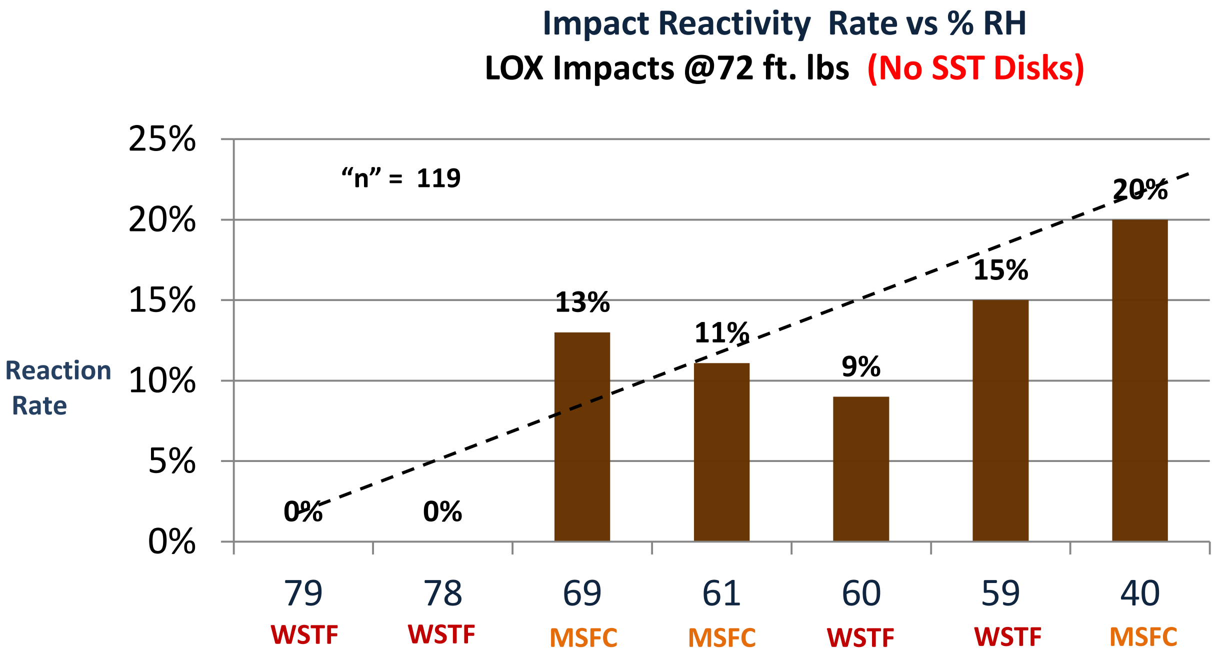
WSTF Reaction Frequency "Reactions/Drops"



Corresponding % RH for Each Above Test

% RH (Mode) During the Sample Prep Session

← **Dates Arranged from High RH to Low RH.**


Impact Reactivity Rate vs % RH
LOX Impacts @72 ft. lbs (No SST Disks)

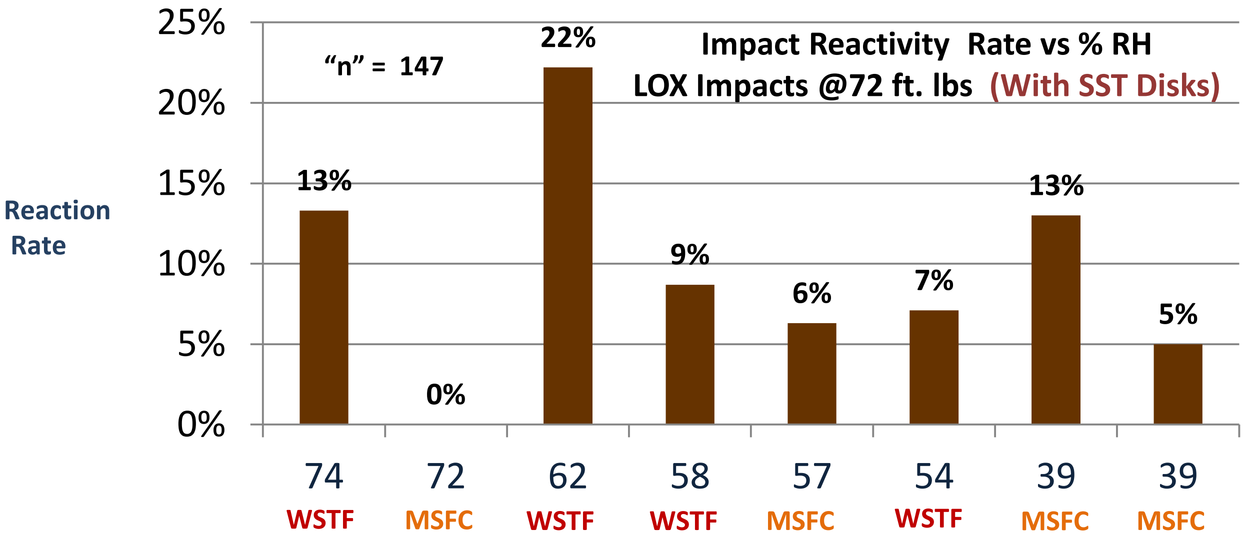
% RH (Mode) During Sample Prep

Reaction Rate vs % RH During Sample Preparation

Tests Performed at MSFC and WSTF

"No Plummet Catcher "

Solstice PF Lot #
BR13019-50-42


Impact Reactivity Rate vs % RH
LOX Impacts @72 ft. lbs (With SST Disks)

% RH (Mode) During Sample Prep

Influence of Sample Preparation Methods on the Reaction Rate for all Impact Energies Tested at WSTF

Sept 8-16, 2014

Sample Prep Method	**SST Disks (Inserts)**	**Reaction Freq.[1]**	**Reaction Rate**
MSFC	Yes	7/99	7.1%
WSTF	Yes	6/53	11.76%
MSFC	No	2/58	3.4%
WSTF	No	8/124	6.4%

There were not enough tests performed at MSFC to provide a similar comparison.

[1] Solstice PF Lot # BR13019-50-42

Reactivity Summary for Solstice PF, Lot # BR13019-50-42

Aug thru Sep 2014

WSTF Summary		
"n" Reactions	"n" Drops	% Reactivity
26	398	**6.5%**

MSFC Summary		
"n" Reactions	"n" Drops	% Reactivity
15	218	**6.9%**

WSTF Rebound vs Initial Reactions Flash & Audible Type		
Rebounds	Initial	Total
13	4	17
% Reactions Occurred on a Rebound	**76.5%**	

MSFC Rebound vs Initial Reactions Flash & Audible Type		
Rebounds	Initial	Total
8	1	9
% Reactions Occurred on a Rebound	**89%**	

Flash and Audible Rebound Reactions / Total of All Reactions 13/26 = 50%

Flash and Audible Rebound Reactions / Total of All Reactions 8 / 15 = 53%

- 20 impact tests for a new material is insufficient to statistically verify the material will not have a reaction.
- As the solvents become more reactive in LOX, the variability in the test parameters become more critical.
- AIT and Heat of Combustion should be included with the LOX impact data to rate the reactivity risks in oxygen systems.

Reactivity Rate vs. Test Variables

- Impact tests performed at MSFC and WSTF from Aug thru Sep 2014, were done from the same cylinder of Solstice PF
  - W/O SST Disks
    - The reactivity rate is more dependent on the % RH during sample preparation. Lower % RH increases the reactivity rate significantly.
    - Previous impact tests performed at WSTF also show the reactivity rates for Solstice PF increased when the samples were prepared and tested at lower % RH.
  - With SST Disks
    - The % RH during sample preparation has little or no influence on the reaction rate.
  - Affect of SST Disks on the Reactivity Rate at WSTF vs MSFC
    - The insert disks doubled the reaction rates for the WSTF ABMA
    - The use of insert disks on the MSFC ABMA reduced the reaction rate ≈ 50% at 72 ft lbs and increased the reactivity rate by 360% for the lower impact energies (60 -65 ft. lbs)
  - Plummet Catcher
    - Utilizing the plummet catcher at WSTF reduced the reaction rates from 5.3% to 1.5%.
    - After the initial impact, sample morphology has changed and the rebound energy imparted to the sample is unknown.
    - Eccentric impacts of the samples were reduced, but were not eliminated by the plummet catcher. Impact testers have a fair amount of mechanical slop that will create non-uniform impacts (energy imparted to the sample / surface area).

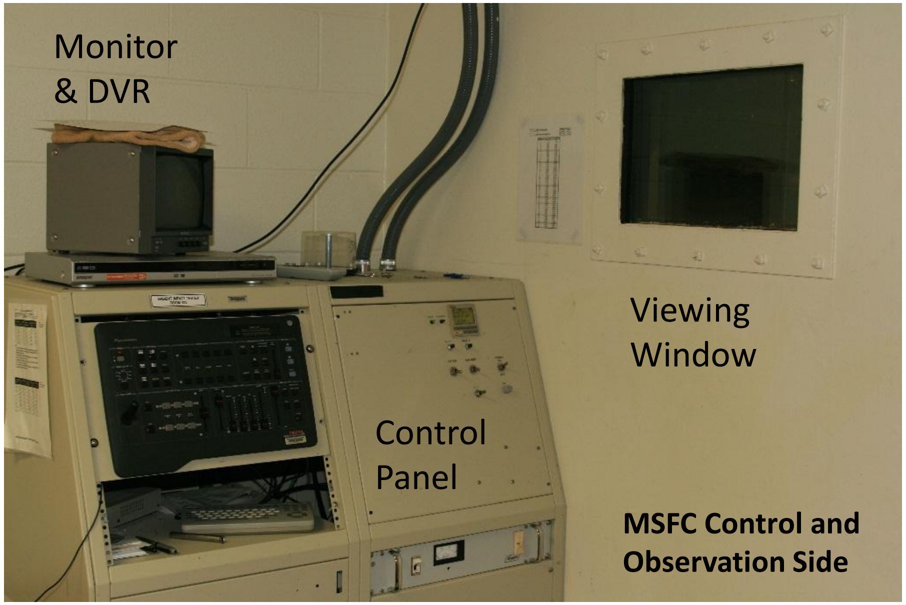
IA Test Protocol

- A proposed test procedure was prepared to evaluate the relative reactivity of the candidate cleaning solvents by minimizing the influence of test variables not addressed in the standard test method, ASTM G86.
  - Verify all components and handling hardware are cleaned to Level 400A. Pins and sample cups be handled by cleaned forceps or tongs, or with cleaned nylon or polyester gloves. Items that do not come in direct contact with LOX can be handled with powder free gloves (Buna N).
  - Aqueous cleaned components (e.g., cold box) should receive a final DI water rinse. The DI water shall:
    - Have a specific resistance greater than 1 meg-ohm-cm or a conductivity of less than 1 micro-Siemen/cm
    - Meet or exceed particulate cleanliness Level 400
    - Total carbon content 1ppm (max).
  - Filter Samples
    - Particulates from solvent storage container can bias test results
  - Prepare Samples @ < 60% RH
    - Do not prepare the samples if there is an approaching weather front, storms or other significant weather events.

IA Test Protocol(Continuation)

- During the LOX soak, place a SST cover over the aluminum cup holders containing the sample cups and discs to maintain a positive pressure and to minimize interference from humidity and atmospheric contaminants.
- Use SST Disks and Rebound Catcher
  - The impact test should be considered invalid if an impact occurs on the raised rim or edge. Non-uniform or edge impacts can produce elevated or irregular impact energies and can produce metallic particles.
- Prior to placing the striker pin into the striker pin guide (yoke), visually verify the frozen sample and disc are in the center of the aluminum cup holder.
- Repeat the sample preparation and impact tests over sequential days until 100 drop tests for each candidate solvent are performed.
- Data from the IA Protocol to be reported by WSTF and/or by MSFC

Flash and Audible Detection at MSFC



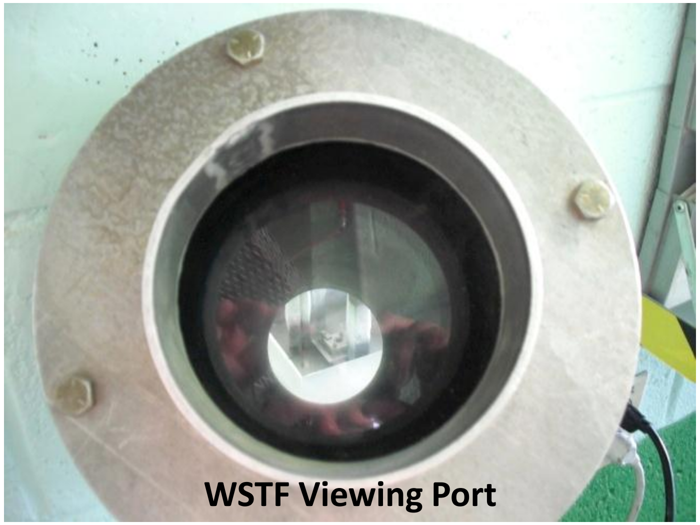
MSFC does not use backup personnel (buddy arrangement) for observing flash and audible reactions.

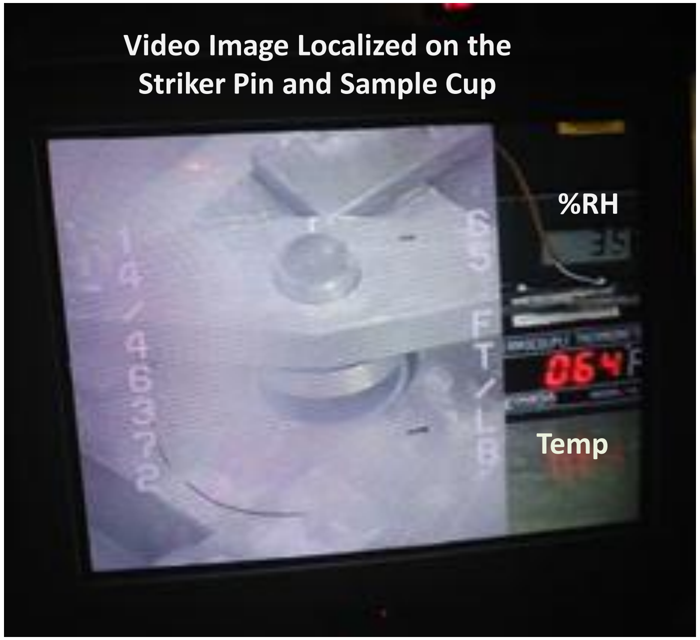
Viewing window opens to the entire cell which limits flash detection.

Video detection utilizes an IR camera that has a high background continuum when the lights are off. Provides a white background for all frost near the base plate which limits flash detection, also the monitor is small and the camera view is not localized to the sample cup .

Audible detection is difficult, test cell is isolated (closed) during tests.

Flash and Audible Detection at WSTF





WSTF uses two personnel for observing reactions. During a drop, one tech observes for flashes through the viewing port while the 2nd tech views the video monitor.

The viewing port and monitor are localized and are directed to the sample cup holder in the base plate where the flash events would occur – this configuration optimizes flash detection.

Video camera is a low lux camera that operates in the visible spectrum. When lights are off for testing, monitor is black and provides a high contrast for detecting any flashes.

Flash and Audible Detection at WSTF (Continuation)


WSTF Control and Observation Side


WSTF Test Side

For detecting audible events – min of two personnel participate, one tech is located near the end of barrier wall on the control side (has an open enclosure - no door).

Plummet drop & rebound sound level could limit low audible detection.


WSTF Control Panel, Viewing Port and Data Recording

Control panel also measures the drop time of the plummet to verify the desired drop time is ± 3% for the appropriate energy level.

Ergonomics

- Sample preparation process is not ergonomic.

Left. Repetitive bending and kneeling. Difficult to observe grease cup fill level.

Right. The installation of a short LN2 transfer and connection to offload LN2 from the trailer to the WSTF Impact Tester would significantly improve the safety and functional features. The transfer line would eliminate the need to have to repeatedly refill dewars from outside and transport them back to the test cell to maintain the LN2 level in the open moat. Repetitive bending while pouring LN2 from several dewars at multiple times during testing represents an ergonomic challenge.

Test Personnel at MSFC and WSTF

- Personnel proficient on coordinated and efficient test operations.
- Very professional and conscientious.
- Mgmt personnel were interested in the test challenges and ways to improve the testing protocols.

Summary

- Most of the flash and audible reactions occurred on a rebound. Rebound impacts are more likely to produce edge effects that can produce non-uniform and elevated impact energies - edge effects can result in metal on metal contact that produces heat. Rebound impacts can change sample morphology and occur after a change of position and orientation of the insert components (cup and disk) following the initial impact.
- The plummet catcher reduced the reaction rates at WSTF by 350%. Tests with the plummet catcher at MSFC were not performed.
- Humidity (limited testing suggests elevated humidity during sample preparation quenches the reactivity). Further testing using the plummet catcher for samples prepared at different humidity conditions are required. The correlation between moisture and reactivity were significantly higher when the tests were performed w/o the insert disks.
- Insert disks provide a more rigid surface which delivers more energy to the sample during impact. Insert disks resulted in an increased reaction rate for all energies tested at WSTF, however the test results at MSFC were reduced significantly at 72 ft. lbs.
- Tests were performed at WSTF comparing the reactivity rate of the WSTF and MSFC sample preparation methods. The overall reaction rate for the WSTF sample preparation method was 9.1% and 5.25% for the samples prepared by MSFC. There were not enough tests conducted at MSFC to provide a similar comparison.
- No reactions were observed for the blank sample component tests performed at WSTF and MSFC. Cleanliness of the components does not appear to be an issue.

Recommendations

- Continue to perform impact testing with the same cylinder of Solstice PF to assess the effects of Low RH on the reactivity
  - Continue to use IA protocol and conduct tests with and w/o disks
  - Determine if other test organizations are using disks when testing liquids
  - The reactivity rate of other solvents may be influenced by the %RH
- Minimum of two set of eyes and ears should be used for detecting visual and audible reactions
- Support revisions to ASTM standards to eliminate test variables that can bias test results (use the plummet catcher, filter the solvent, perform and document solvent NVR, clarify the use of insert disks and insert sample cup alloy/s required for testing halogenated solvents, document environmental conditions during sample prep and testing, develop more organized and less fragmented sections and references in the standard for sample preparation).
- Develop and test an unibody solvent cup to eliminate multiple sample components that can shift position and promote reactions due to edge affects. Halogenated liquids / greases are more reactive from shear forces with newly exposed aluminum (as those found in the aluminum sample cups).
- Audio characteristics should be studied to assess reactions.
- Investigate methods that will provide more uniform samples and sample volume to the insert sample cups.
- Evaluate ways to reduce mechanical slop in the impact tester - determine if specifying a clearance tolerance to the yoke for the striker pin would reduce the frequency of non-perpendicular impacts.
- Implement IA contamination control recommendations.
- Periodic round robin testing between MSFC and WSTF and /or with other facilities are strongly recommended to develop technical data and information to minimize test variability.

Closing

- Selection of a replacement solvent/s for AK225G is based on their relative performance in various oxygen compatibility studies (autogenous ignition temperature (AIT), heat of combustion, mechanical impact, and other performance features (cleaning efficiency, material compatibility, environmental considerations, toxicity, stability, reuse, operational handling, field cleaning performance and human factors).

Return to App I Return to Pg 32

HCFC-225 Replacement Solvent Study
Marshall Space Flight Center (MSFC)

Jennifer McMillian, Gary Glass, and Mark Griffin

January 9, 2015

The Defense Logistics Agency – Hazardous Minimization and Green Products Branch funded MSFC to perform a solvent study to find a replacement for HCFC-225. MSFC's Materials Combustion Research Facility (MCRF) was assigned the responsibility of oxygen compatibility testing (mechanical impact and autogenous ignition tests).

MSFC MCRF was provided the Test Plan, and any deviations from the test plan have been captured below.

Test 13A: Mechanical (Ambient) Impact

Cleaning and Preparation

All of the MSFC components for mechanical impact testing were cleaned to MSFC-164D IV X A in the precision cleaning lab, in building 4705. This includes striker pins, grease cups, samples cups, and insert disks. The components were batch tested before any of the newly cleaned components were used. Before the testing began, the test cell was inspected for cleanliness, and the floors were swept and mopped, if necessary. The top plate, well, ladle, hand dewars, forceps, glass eye dropper, chill box, and stainless steel pans were cleaned by wiping them down with Freon-113. The impact pin surfaces were inspected for cleanliness and surface finish, before use.

Sample Preparation

Sample preparation was performed in accordance with ASTM D 2512-95, with few adjustments. First, the LN_2 was turned on to chill down the base. (At MSFC, the anvil plate has been machined so that the LN2 flows inside the anvil plate instead of flowing into a moat.) Because of the volatility of the solvents, additional steps were taken to ensure there was little to no sample lost before testing. This included pre-chilling the solvents. The solvent containers were placed in the freezer and allowed to chill, at least overnight. L-14780, Solvokane, Solkane, Honeywell Solstice PF, and Capstone 4-I were all prepared as follows: 25 Aluminum cups were placed into a clean stainless steel pan. Then, stainless steel inserts were placed into each of the aluminum cups, and the pan was placed into a plastic zip lock bag. To determine the sample weight of the solvent, a stainless steel grease cup was placed onto the balance, and the solvent was inserted into the grease cup with an eye dropper. The grease cup was considered full when the solvent level was flush (flat) with the rim of the grease cup at eye level. (The depth, width, and weight of the grease cups had been pre-measured when received from cleaning.) The chill box (located in the Ambient Impact test cell) was cleaned with Freon and filled with LN2. The pre-chilled sample was removed from the freezer and taken to the Ambient Impact test cell, along with the pan of cups, the glass eye dropper, and 20 stainless steel grease cups. When the LN2 level in the chill box was below the top plate, 5 stainless steel grease cups were placed onto the top plate of the chill box, and the solvent was inserted into them with the eye dropper. This process was done until all 20 grease cups were prepared. The 25 aluminum cups were taken out of the stainless steel pan and placed onto the top plate of the chill box to chill. Once all of the aluminum cups had time to chill, the 20 grease cups were placed into them, on top of the stainless steel inserts. When all of the grease cups were inside of the aluminum cups, the LN2 level was brought up to surround the aluminum cups. The stainless steel pan was placed into the chill box and allowed to chill. Once chilled, the aluminum cups were placed into the stainless steel pan. LOX was slowly ladled into the stainless steel pan until the samples were submerged. The samples were allowed to soak for a minimum of 30 minutes.

It should be noted that the Vertrel MCA was not pre-chilled due to the separation of its components.

Procedure

At MSFC, a 13A test includes 5 blanks and 20 samples. Two of the blanks are used to verify the cleanliness of the base before a sample is dropped on. The other 3 blanks are used throughout the test (after sample drops 5, 10, and 15) to ensure the cleanliness of the base during testing. When a reaction is detected, the well is blown out with GN2 and an additional 2 blanks are dropped on. This is to ensure there are no contaminants in the system.

Testing was performed in accordance with ASTM G 86-98a, with few adjustments. One of the five blanks was placed into the well. A striker pin was then placed into the cup, and the pin holder was secured into place. The plummet was manually lowered onto the pin and a measuring rod was placed on the top side of the plummet, with the other end placed against the plummet holder, to verify that the drop height is correct. The plummet was raised back to position. The technician moved to the control room, and dropped the plummet onto the pin. The plummet was then raised and the blank was inspected for a reaction. If there was no reaction, a second blank was loaded and dropped on. If a reaction occurred, testing was halted; the base was thawed and re-cleaned. It should be noted that zero blanks reacted during these sets of testing. If there are no reactions with the two blanks, 5 samples are dropped on with each sample being inspected after each drop. This is continued until a complete test set has been reached, with one blank after every 5 drops.

Bruceton Method

During the initial set of impact testing at MSFC, the Bruceton Method was used to determine a threshold. Bruceton Method is also referred to as the ‘Up and Down’ testing method. It consists of dropping on 34 samples, beginning at 72.0 ft•lbs (43.3 inches). Each drop results in either a non-reaction or a reaction, which determines the following drop. A non-reaction will result in the latter drop staying at the same energy level (72 ft•lbs) until 34 drops are achieved. A reaction will result in the latter drop being reduced to a specified lower energy level. If there is no reactions on the lowered energy level drop, the next drop in the series will step up to the higher energy level until 34 drops are achieved. A sample data sheet is attached as Attachment 1. However, it was later determined that the thresholding method defined in ASTM G 86 should be used in future testing of these solvents. The table below shows the results of the initial mechanical impact and/or Bruceton thresholding results.

Table 1.1 Initial Mechanical Impact and/or Bruceton Threshold Testing Results

	Rxns	Energy (ft•lbs)	Inserts?	Initial, R1, or R2?	Bruceton?	Bruceton Reactions?
L-14780	0/20	72	Yes	N/A	Yes	0/34
Vertrel MCA[1]	2/16	72	Yes	R1, R1	Yes	4/34 @ 72ft•lbs
Solvokane	2/6	72	Yes	Initial, R1	Yes	7/34 @ 72 ft•lbs and 2/34 @ 62.9 ft•lbs
Solkane	2/4	72	Yes	R1, R1	Yes	4/34 @ 72 ft•lbs and 1/34 @ 62.9 ft•lbs
Vertrel MCA[2]	2/5	72	Yes	R1, R1	Yes	2/34 @ 72 ft•lbs
Solstice PF	2/28	72	Yes	R1, R1	Yes	2/34 @ 72 ft•lbs
Capstone 4-1	0/20	72	Yes	N/A	No	N/A
AK-225G Distilled	0/20	72	Yes	N/A	No	N/A

[1] The original Vetrel MCA provided to MSFC had no documentation of the stabilizer.
[2] Denotes the Vertrel MCA with the known stabilizer.

As can be seen in Table 1.1, the energy threshold for all eight of the solvents was found to be 72 ft•lbs.

Independent Assessment Team (IAT)

While MSFC was working on the DLA Solvent Study, White Sands Test Facility (WSTF) was working on a solvent study funded by NASA Rocket Propulsion Test Program, both looking for a replacement for HCFC-225. Some of the testing overlapped and allowed project leads to compare data. However, when the data was compared, there were significant discrepancies in the observed reaction thresholds. Because of differences in test results between MSFC and WSTF, an independent assessment team (IAT) was developed to work alongside both centers while testing was performed. This was to document any differences that may have resulted in differing data between the two centers. When the IA team first visited MSFC, the concentration was on AK-225G, L-14780, and Solstice PF (from two different containers). It should be noted that the samples were pre-chilled only during the first round of testing. When the IA team got involved, the solvents were kept at room temperature prior to testing. The results can be seen in the table below.

Table 1.2 Mechanical Impact Testing Results (IA Team’s First Visit)

Solvent	Rxns	Energy (ft•lbs)	Inserts?	RH	Temp. (F)
AK-225G Distilled	0/20	72	Yes	-	-
AK-225G	0/20	72	Yes	-	-
L-14780 MSFC Prep – MSFC Hardware	0/20	72	Yes	-	-
Solstice 1.6 lb container (blue)	0/10	72	No	-	-
Solstice 1.6 lb container (blue)	0/20	72	Yes	64%	75
Solstice 1.6 lb container (blue)	0/5	72[1]	-	-	-
Solstice 1.6 lb container (blue)	0/5	82.5[1]	-	-	-
Solstice 10 lb container (white)	0/22	72	-	66%	79

[1] Denotes that the grease cups were only filled ½.

It can be seen in Table 1.2, that MSFC was not seeing any reactions with the AK-225G, L-14780, or Solstice PF. Because additional testing was being performed on the Solstice PF, there were a couple of different containers at MSFC. It was decided that both containers should be tested to rule out any batch differences that could possibly be present. However, there were no reactions detected at 72 ft•lbs. Because of this, the project lead and IA team member asked the technician to raise the plummet to an energy of 82.5 ft•lbs, to see if any reactions occurred. Again, none were seen.

These cups were full unless noted otherwise. In the testing of the 10-lb container, particulate contaminants were noted in samples 20, 21, and 22, but still no reactions occurred.

There were still discrepancies between the two centers, so the IA team decided to conduct another set of impact tests on Solstice PF. The last round of testing was performed to control variables such as use of inserts, technicians preparing the samples, and hardware used. It was also determined that relative humidity (RH) and temperature should be recorded during sample preparation. If no RH or temperature is listed, it was simply not recorded. Due to the fact that the 13A Test does implement a rebound catcher, the plummet is allowed to impact the pin and sample multiple times. Reactions are recorded as initial, R1, R2, or char only. Initial is defined as a reaction occurring during the initial impact, R1 is the first rebound, and R2 is the second. When the reactions are reported as char only, it could not be determined if the reaction took place on the initial impact or a rebound. In the tables below, threshold testing was performed, per G86-98a.

Table 1.3 – Solstice PF Mechanical Impact without Inserts at MSFC (IA Team's Second Visit)

	Rxns	Energy (ft•lbs)	Inserts?	RH	Temp. (F)	Initial, R1, or R2?
MSFC prep – MSFC Hardware	2/16	72	No	-	-	R2, R2
	0/20	65	No	70%	73	N/A
WSTF prep - WSTF Hardware	2/18	72	No	61%	79	Char only
	0/20	65	No	-	-	N/A
*make-ups for bad drop time	0/5	65	No	-	-	N/A
WSTF Prep – MSFC Hardware	2/10	72	No	38%	79	Char only
	1/3	65	No	41%	79	Char only
	0/20	60	No	41%	79	N/A
*make-ups for bad drop time	0/2	60	No	41%	79	N/A

It can be seen in the Table 1.3 that when both centers were prepping with their own hardware, the results were very similar. Both saw two reactions at 72 ft•lbs and zero reactions at 65 ft•lbs, resulting in a threshold value of 65 ft•lbs. The humidity and temperature were also comparable during the first two sets of tests. The final set of testing, without inserts, took place with WSTF prepping using MSFC's hardware. Similar results were seen with two reactions at 72 ft•lbs, one at 65 ft•lbs, and zero at 60 ft•lbs. Therefore, the threshold was determined to be 60 ft•lbs. While the temperature remained consistent, there was a significant decrease in RH. It should also be noted that no flash or audible reactions occurred on initial impact.

Table 1.4 – Solstice PF Mechanical Impact with Inserts (IA Team's Second Visit)

	Rxns	Energy (ft•lbs)	Inserts?	RH	Temp. (F)	Initial, R1, or R2?
MSFC prep – WSTF Hardware	2/16	72	Yes	40%	79	R1, R1
	0/4	65	Yes	41%	77	N/A
	1/1	65	Yes	37%	81	R1
	1/1	60	Yes	-	-	R1
	1/3	55	Yes	-	-	Char only
	0/20	50	Yes	39%	79	N/A
MSFC prep – MSFC Hardware	1/20	72	Yes	39%	77	R1
	1/16	72	Yes	57%	79	Char only
	1/10	65	Yes	57%	79	R1
	0/20	60	Yes	57%	79	N/A

Table 1.4 shows the same type of threshold testing as Table 1.3, but inserts were used. MSFC prepared samples using WSTF's hardware, and a threshold was determined to be 50 ft•lbs. Because this testing took place over a number of days, it

can be seen that the RH and temperature varied from set to set, but only by a small amount. When MSFC prepared the samples with their own hardware, the threshold was determined to be 60 ft•lbs. It should be noted again that none of the reactions seen in Table 1.4 were on the initial impact.

During the testing, the IA team questioned if MSFC was using SS 347 grease cups and inserts. After XRF and Carbon/Sulfur analysis, it was determined that MSFC's grease cups and inserts disks were SS 304. Marshall analyzed WSTF's insert disks to be SS 304 as well.

While WSTF employees were at MSFC for this last set of testing, it was requested that WSTF assist MSFC in performing a calibration (dent) test. WSTF took the data from the test and analyzed it. A report was given to MSFC. The results stated that MSFC's actual energy values were slightly higher than the ideal energy values, due to a slightly higher plummet weight than suggested. Table 1.5 shows the ideal and actuals. Marshall is looking into correcting the plummet discrepancy.

Table 1.5 – Calibration (Dent) Test Results

Drop Height		Ideal Energy (20-lb Plummet)		Calculated Energy (20.4-lb Plummet)	
(in)	(m)	(ft-lbf)	(J)	(ft-lbf)	(J)
43.3	1.1	72	98	74.1	100.4
30.0	0.76	50	69	51.5	69.8
18.0	0.46	30	41	30.6	41.5
9.0	0.23	15	20	15.3	20.7

Autogenous Ignition Temperature Testing

Cleaning and Preparation

System cleanliness is also extremely important in the AIT Testing. The test chamber was placed in the ultrasonic cleaner with oxygen compatible detergent and DI water, followed by a Freon-113 wipe down. The thermocouple and brass sample tube holder were wiped down with Freon as well. The glass sample tubes were soaked in chromic acid and then rinsed with DI water.

Sample Preparation

The solvent container, brass holder, and 3 sample tubes were placed in the freezer and allowed to chill, at least overnight. The day of the testing, the solvent container and a sample tube were removed from the freezer. A glass beaker, with enough ice water to fill the bottom of it, was placed on the balance. The sample tube was placed in the beaker, and the balance was zeroed. A clean eye-dropper was used to transfer the appropriate amount of solvent from its container to the sample tube. (In instances where an eye-dropper could not be used to retrieve the solvent, a small amount of solvent was poured into a beaker, as a secondary container). The sample tube was then transferred into a hand dewar that was filled with ice. The brass sample tube holder was removed from the freezer and placed in the hand dewar as well. The lid was placed on the dewar, and transported to the AIT test cell. When needed, the additional two sample tubes were removed from the freezer and filled with the appropriate amount of solvent. Additionally, the test chamber was pre-chilled in an ice bath before each individual test sample was tested.

The idea of pre-chilling the solvents came from data that was provided by Honeywell. They had previously contracted Wendell Hull to perform AIT testing on Solstice, and the pre-chilling method was used. MSFC found it easiest to follow similar steps to ensure the appropriate amount of solvent remained. It should be noted that the Vertrel MCA was not pre-chilled due to the separation of its components.

Procedure

The Autogenous Ignition Testing was performed in accordance with ASTM G72-09, with few adjustments. Because of the volatility of the solvents, the traditional 3 purge that MSFC does, was not performed. A test sample was purged to 50 psi to verify that the liquid sample was still in the sample holder. This technique was used on all solvents that were tested. Due to previous (non-solvent) violent reactions destroying the borosilicate glass test tube inner reaction vessel and stopper, MSFC used a machined brass holder to hold the sample tube.

The results of the AIT Testing of each of the solvents can be seen in the table below.

Table 2.1 Autogenous Ignition Temperature Test Data

Solvent	Sample Weights (g)	Ignition Temp (F)	Temp Rise on Ignition (F)	P_i (psig)	P Rise on Ignition (psig)
L-14780	0.23	299.23	87.07	58	70
	0.23	299.96	39.34	58	92
	0.23	>800	-	56	N/A
	0.20	>800	-	2040	N/A
	0.20	>800	-	2065	N/A
	0.20	>800	-	2055	N/A
	1.0	318	973	2002	1967
Vertrel MCA	0.23	>800	-	2068	N/A
	0.23	>800	-	2077	N/A
	0.23	>800	-	2049	N/A
Solvokane	0.51	400	65	52	8
	0.51	320	50	51	6
	0.51	340	50	53	14

	0.51	327	104	2001	559
	0.51	317	180	2009	910
	0.51	331	158	2028	813
Solstice PF	0.50	501.26	112.96	54	72.5
	0.50	519.16	125.28	54	72.5
*Oil	0.50	>800	-	52	-
	0.50	390	213	2057	230
	0.50	384	258	2044	-
*Oil	0.50	460	619	2053	607
	0.23	378	65	54	21
	0.23	>800	-	50	-
	0.23	>800	-	52	-
*No Oil	0.23	>800	-	52	-
	0.23	>800	-	2038	-
	0.23	>749	-	2049	-
*No Oil	0.23	>800	-	2073	-
Capstone 4-I	0.23	>800	-	2061	N/A
	0.23	>800	-	2038	N/A
	0.23	>800	-	2033	N/A
AK-225G	0.23	>800	-	54	N/A
	0.23	>800	-	51	N/A
	0.23	>800	-	54	N/A
	0.23	>800	-	2067	N/A
	0.23	>800	-	2082	N/A
	0.23	>800	-	2072	N/A

During the L-14780 test at 50 psi, it was noted that there was no pressure rise on the temperature event on samples 1 and 2.

When Solvokane was tested at 50 psi, it was documented that all three samples had a pressure drop of 20-30 degrees during the test. During each of the tests at 2000 psi, the glass sample holders were destroyed when the ignition occurred.

It was determined that the vendor may have contaminated the first batch of Solstice PF that was tested. This is referred to as Solstice PF *Oil, while the other is referred to as Solstice PF *No oil. Between samples 2 and 3, of the 2000 psi test (*Oil), the 2000 psi pressure transducer was replaced with a 5000 psi pressure transducer. This is because during sample 2, no pressure rise was seen. The pressure was at 2790 psi at the 380F ignition. It can be seen that during the *no oil tests at 50 psi, 4 samples were tested. The first sample ignited, however it was thought that this could have been an error. Three additional samples were tested and resulted in an ignition temperature greater than 800F.

During the AK-225g test at 50 psi, sample number 3, a temperature drop was reported at 381F. A 75 degree drop was recorded. No pressure decrease or increase was seen during this event.

The following is a table of the residue reported from each of the solvent AIT tests at different pressures and mass.

Table 2.2 Residue Reporting During AIT Tests

Solvent	Residue reported
L-14780 at 50 psi	Goldish color
L-14780 at 2000 psi (0.20 g)	White
L-14780 at 2000 psi (1.0 g)	Green
Vertrel MCA	Reddish
Solvokane	Green and white
Solstice at 50 psi *Oil	Black soot
Solstice at 2000 psi *Oil	Reddish
Solstice at 50 psi *No oil	Reddish-brown
Capstone at 50 psi	White
Capstone at 2000 psi	White
AK-225g	White

BRUCETON "UP and DOWN" TECHNICIAN WORKSHEET

Test No.: 108720 Date: 4-26-13 Project:

Requestor: M.Mitchell Manufacturer Designation: Vertrel MCA Manufacturer:

Composition: Sample: Avg. Diam./Wdth. (in): 0.657 Inserts Used: [✓ Y] / N

Subst. Matrl.: N/A Subst. Thick. (in.): N/A Avg. Wgt.(g): 0.508

Insert Matrl.: SS Cup Matrl.: Aluminum Length (in.): N/A

Insert Thick. (in.): 0.0611 Thickness (in.): 0.0495

Test:

Ft-lbf	Ht. (in.)	DROP NUMBER																																	
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34
72.0	43.3	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	+		O	O	O	O	O	O	O	O	O	+		O
62.9	37.7																						O											O	
54.6	32.7																																		
47.6	28.5																																		
40.9	24.5																																		
35.9	21.5																																		
31.2	18.7																																		
27.2	16.3																																		
23.5	14.1																																		
20.5	12.3																																		
17.9	10.7																																		
15.5	9.3																																		
13.5	8.1																																		
11.7	7.0																																		
10.2	6.1																																		
8.8	5.3																																		
7.7	4.6																																		
6.7	4.0																																		
5.8	3.5																																		
5.0	3.0																																		
4.3	2.6																																		
	Drop Time	.449	.446	.445	.448	.447	.445	.448	.450	.448	.453	.445	.446	.442	.453	.449	.450	.442	.449	.449	.448	.450	.423	.450	.447	.448	.445	.450	.450	.448	.454	.450	.448	.420	.450
Legend	S1																																		
Audible (A)	R1																																		
Flash (F)	R2																																		
Charring (PIC)	R3																					A,F,C											AC		
	R4																																		

Pressure (psia): 14.7 Temperature (F): -297.0 Technician: Gary W. Wilson

Environment: (LOX/GOX): LOX Date: 4-26-13

Return to App I   Pg 32   Pg 33   Pg 34

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

1.0 INTRODUCTION

NASA Marshall Space Flight Center (MSFC) requested NASA White Sands Test Facility (WSTF) to perform testing in support of the down-selection for the solvent to replace AK-225G.[1] A method for assessing the compatibility of materials for use in oxygen systems at a system level has been established and maintains a worthy track record. A similar approach, at a broader level, was implemented to analyze the oxygen compatibility of candidate replacement solvents.

2.0 TEST DOCUMENTATION

- ASTM D240[2]
- ASTM G72[3]
- ASTM G86[4]
- ASTM G63[5]
- ASTM G94[6]
- WSTF Forms 514, *Test Request and Special Instructions* (Appendix A)
- TPS 103379[7]
- TPS 105062[8]
- NASA-STD-6001B[9]
- NASA/TM-2007-213740[10]

---

[1] Asahiklin AK-225G®, a hydrochlorofluorocarbon solvent, is a registered trademark of Asahi Glass Company (Tokyo, Japan).

[2] ASTM. *Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter.* ASTM D240. American Society for Testing and Materials, West Conshohocken, PA, 2014.

[3] ASTM. *Standard Test Method for Autogenous Ignition Temperature of Liquids and Solids in a High-Pressure Oxygen-Enriched Environment.* ASTM G72. American Society for Testing and Materials, West Conshohocken, PA, 2009.

[4] ASTM. *Standard Testing for Determining Ignition Sensitivity of Materials to Mechanical Impact in Ambient Liquid Oxygen and Pressurized Liquid and Gaseous Oxygen Environments.* ASTM G86. American Society for Testing and Materials, West Conshohocken, PA, 1998.

[5] ASTM. *Standard Guide for Evaluating Nonmetallic Materials for Oxygen Service.* ASTM G63. American Society for Testing and Materials, West Conshohocken, PA, 1999 (R 2007).

[6] ASTM. *Standard Guide for Evaluating Metals for Oxygen Service.* ASTM G94. American Society for Testing and Materials, West Conshohocken, PA, 2005 (R 2014).

[7] In-house document. TPS 103379, *Standard Test Solvent Evaluation (AIT).* Opened: July 22, 2013.

[8] In-house document. TPS 105062, *Solvent Study 13A.* Opened: September 5, 2014.

[9] NASA. *Flammability, Odor, Offgassing, and Compatibility Requirements and Test Procedures.* NASA-STD-6001B. National Aeronautics and Space Administration, Washington, DC, 2011.

[10] NASA Technical Memorandum. *Guide for Oxygen Compatibility Assessments and Components and Systems. TM-2007-213740. March 2007.*

3.0 OBJECTIVES

The data from this test series will be used with other performance features to evaluate the down-selection requirements for replacing AK-225G, a class II Ozone Depleting Chemical (ODC). The evaluation of materials for oxygen compatibility is the main concern of WSTF. Some refinement of the test methods proposed will be required to account for the specific material properties. The objective of WSTF is also to make recommendations as to the method of obtaining the appropriate test data to aid in the ranking of the proposed solvents with respect to oxygen compatibility.

Documentation of the logic process used to analyze the test data with respect to the oxygen compatibility of the candidate solvents will link this process to the developed method for assessment outlined in ASTM G63, G94, and NASA-STD-6001.B.

4.0 TESTING APPROACH

In support of the investigation for down-selection for a solvent to replace AK-225G, the following solvent materials were evaluated:

Solvent	WSTF Material Number
HFE-7100	13-46204
AK-225G	13-46203, 13-46221, 14-46373
Solstice PF®[1]	14-46309, 14-46371, 14-46375
L-14780[2]	14-46305, 14-46372
Solvokane®[3]	14-46299

Testing included NASA-STD-6001B: Test 13 ASTM G86-98 (Appendix A.2.5) *Mechanical Impact for Materials in Ambient Pressure LOX* (Test 13A) and *Mechanical Impact for Materials in Variable Pressure GOX and LOX* (Test 13B); ASTM G72-01 (Appendix A.2.8) *Autogenous Ignition Temperature* (AIT); and ASTM D240-14 (Appendix A.2.9) *Heat of Combustion* (HOC) testing. Each of these tests were performed on selected solvents. Any deviations from the standards that were required to obtain reliable data were documented. Test data sheets are provided in Appendix B.

Marshall Space Flight Center participated in several aspects of this investigation, including testing in parallel of AIT, and Tests 13A and 13B.

---

[1] Solstice PF® is a registered trademark of Honeywell International, Inc., Morristown, NJ.

[2] L-14780 is manufactured by the 3M Company, St. Paul, MN.

[3] Solvokane® is a registered trademark of Solvay Corporation, Bruxelles, Belgium.

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

Testing was required to quantify which potential replacement solvent was less reactive in oxygen systems. Legacy testing provided a starting point for this evaluation. Testing used to analyze oxygen compatibility during previous evaluations relied on ignition tests, NASA-STD-6001 Test 13, and AIT. A flammability test (HOC) was included during this evaluation to quantify the amount of energy released by each solvent. Testing of legacy solvents aided in the direct comparison between potential replacement solvents and historically used solvents.

The test methods used for past selection criteria are well established and yield good results for most materials; however, solvents proved to be challenging materials to test due to the volatility of the chemicals. The relatively low boiling points of the solvents resulted in time-dependent test requirements. Special accommodations were required for each of the test methods to obtain reliable results.

Liquids were extracted from shipping containers and poured directly into glassware used for sample preparation. All glassware was triple-rinsed prior to sample preparation. No filtration of test liquids was performed prior to testing unless otherwise noted.

5.0 MECHANICAL IMPACT TESTING – NASA STANDARD TEST 13

Mechanical impact testing was performed based on previous solvent evaluations that relied on mechanical impact results to make oxygen compatibility assessment. Testing outlined in ASTM G-86 included the three candidate materials undergoing evaluation (Solstice PF, L-14780, Solvokane) as well as control tests on AK-225G. All tests were performed in liquid oxygen (LOX). Samples were frozen.

5.1 PREPARATION METHOD – NASA STANDARD TEST 13

Preparation of samples was performed by test technicians in the test cell. Preparation of samples for Tests 13A and 13B were identical. Preparation temperatures and humidity were not recorded during initial testing. Preparation conditions were recorded only after ambient preparation conditions were suspected of influencing the CONSISTENCY of the test samples. Chilling of the aluminum cups was accomplished by placing them within a chill box partially filled with LOX.

Two methods of preparation were identified during the testing of solvents the week of June 23, 2014. The two methods of sample preparation yielded different sample consistencies. The different methods of sample preparation are as follows:

1. The stainless steel insert (grease) cups were placed on a stainless steel plate located outside of the chill pan. The solvent was dripped into the cup and then the cup was transferred onto the cold block (partially submerged in LN_2) cooling bar in the chill pan. This method of sample preparation was called Sample Preparation Method 1 (Figure 1).
2. The stainless steel insert (grease) cups were placed directly on the cold block (partially submerged in LN_2) chill bar, or in the case of MSFC testing, directly on the chill box pan, and solvent was dripped into the cup. This method of sample preparation was called Sample Preparation Method 2 (Figure 2).

Once frozen, the samples were transferred, one each, into the aluminum cups located within the chill box. Samples were allowed to chill within the aluminum cups for 10 min followed by a minimum of 45 min submersion in LOX.

Both preparation methods were used during Tests G and H (AK-225G).[1] Obtaining samples using Sample Preparation Method 1 was difficult due to inconsistencies in sample homogeneity. Sample homogeneity was observed to be dependent on the amount of solvent within the grease cup; if the cup was not full enough the sample would freeze partially white and partially clear. Clear portions of the samples tended to crack. All samples tested were homogenous and white in color. Sample Preparation Method 2 consistently yielded homogenously white samples. The grease cups were purposely overfilled; excess frozen solvent was scraped off the grease cups prior to transferring samples to the aluminum cups.

Preparation of Solstice PF was done entirely using Sample Preparation Method 1. Obtaining samples using Sample Preparation Method 1 was difficult due to inconsistencies in sample homogeneity, as previously described. Samples prepared using Sample Preparation Method 2 tended to freeze in the cup in a less dense solid. The samples resembled loose-pack snow and were slightly concave. Visibility was obscured when filling the grease cups using this method because the volatilized solvent created a volcano-like vapor cloud.

---

[1] See Table 1 (Section 5.2.1) for a listing of all LOX mechanical impact tests at ambient pressure.

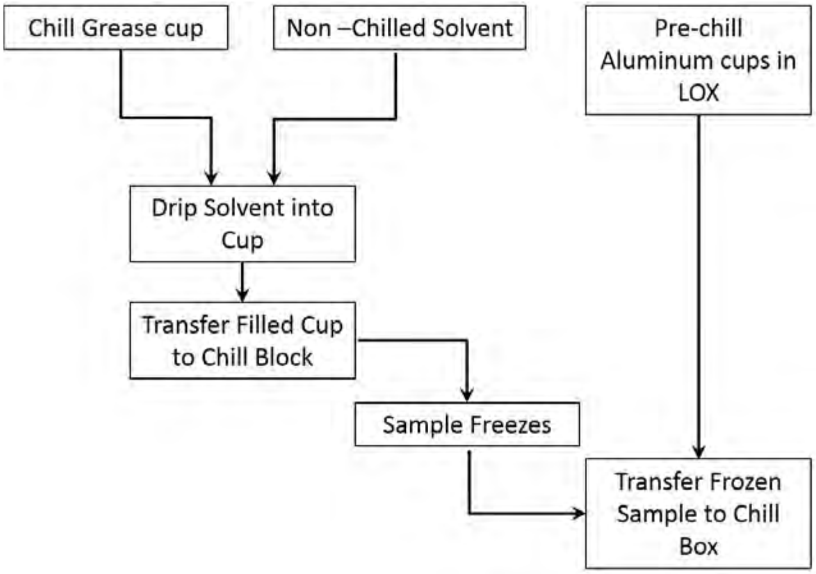


Figure 1
Sample Preparation Method 1 – Flow Chart

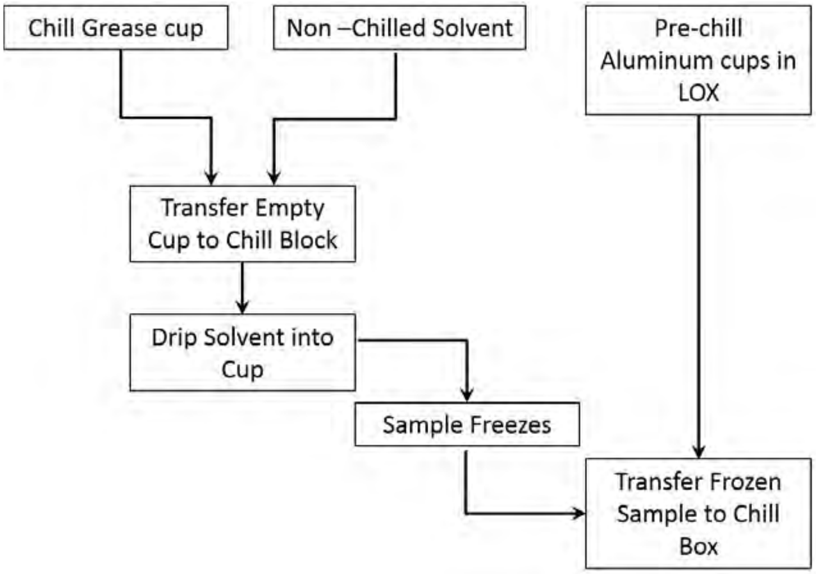


Figure 2
Sample Preparation Method 2 – Flow Chart

5.1.1 PREPARATION VARIABLE INVESTIGATION – NASA STANDARD TEST 13

During testing of G and H (AK-225G), no reactions were observed using either Sample Preparation Method 1 or Method 2. Five additional tests were performed using each of the sample preparation methods at the highest energy level attainable in test fixture, ~ 108.4 J (80 ft-lbf). No reactions were observed. Samples were prepared at 17 °C (63 °F) and 46 percent relative humidity (RH).

5.1.2 FINAL PREPARATION METHOD – NASA STANDARD TEST 13

Sample Preparation Method 1, displayed in Figure 1, was chosen as the preferred method. This method resulted in a more consistent homogenous surface texture. Formation of ice crystals on the surface of the test samples was observed when preparing samples using Sample Preparation Method 2 (Figure 2). These ice crystals offered a non-uniform sample, and therefore Method 2 was not the preferred method of sample preparation.

5.1.3 ACCEPTANCE CRITERIA – NASA STANDARD TEST 13

Any samples displaying inconsistencies were discarded. Samples that were cracked, broken or were non-homogenous were not tested. Prior to test, all aluminum cups were examined for ice fragments, and all fragments were picked out of the cups. These final inspections were performed to ensure consistent test samples.

5.2 AMBIENT PRESSURE LOX MECHANICAL IMPACT TESTING - STANDARD TEST METHOD TEST 13A

Standard WSTF protocol for the ASTM G86 test began with the above-described Sample Preparation Method 1. No insert discs were placed inside the aluminum cups below the sample (grease) cups. A rebound catcher, which is a mechanical apparatus that limits the number of impacts to each sample to just one, was not used during standard testing.

Standard WSTF protocol involves performing an energy threshold. Testing began at the highest energy level of 97.6 J (72 ft-lbf). Two reactions at the 97.6 J (72 ft-lbf) energy level were required prior to decreasing the amount of energy imparted onto the sample. Subsequent decreases in energy levels required only one reaction per energy level. If the intensity of the reactions was such that the test team suspected reactions to occur at the next lower energy level, skipping energy levels was acceptable to expedite the bracketing of the reaction/energy threshold. Twenty consecutive tests in which no reactions were witnessed by any means, audible or visual (charring or flashing), with at least one reaction at the next higher energy level, were required to affirm a passing threshold energy level. One reaction in sixty samples at 97.6 J (72 ft-lbf) was also considered a passing energy threshold test.

The data for all ambient pressure LOX mechanical impact tests performed are summarized in Table 1.

5.2.1 STANDARD TEST METHOD – 13A TEST RESULTS

All four solvents were tested using the standard WSTF test protocol. Initial test results for ambient pressure LOX mechanical impact testing are shown in Table 2.

Test A was performed August 21, 2013 with AK-225G. Tests B, C, and D were performed in February 2014, a period of low relative humidity. The humidity during preparation for Tests A through D was not recorded. (Note: At WSTF, near Las Cruces, New Mexico, relative humidity tends to fall between 12 and 22 percent in the month of February.) All tests were performed with no stainless steel inserts below the stainless steel (grease) cups. All stainless steel cups were placed directly in the larger aluminum cup submerged in LOX. Stainless steel (grease) cups met ASTM G86 (1995) drawing cup dimensions (16.8 mm diameter, 1.3 mm depth). Stainless steel insert (grease) cups were machined and provided 90-degree angle internal edges between base and sidewall. Tests A (AK-225G), C (L-14780), and D (Solstice PF) passed at 97.6, 54.2, and 20.3 J (72, 40, and 15 ft-lbf), respectively. Test B (Solvokane) failed at the lowest energy level. For Test D (Solstice PF), average reaction frequency for 94.6 and 88.1 J (72 and 65 ft-lbf) was 64 percent.

Test F (L-14780) was prepared using Sample Preparation Method 1. The material passed at 88.1 J (65 ft-lbf) with 0/20 reactions. Samples were prepared at 17 °C (63 °F) and 47 percent RH.

Test E (Solstice PF) was performed June 23, 2014 using Sample Preparation Method 1. Testing was stopped due to depletion of the test material. Two reactions were observed at 68 J (50 ft-lbf), and three tests were performed at 61 J (45 ft-lbf) with no reactions. Average reaction frequency for 97.6 and 88 J (72 and 65 ft-lbf) was 37 percent. Samples were prepared at 19 °C (66 °F) and 28 percent RH.

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

Table 1
All Ambient Pressure LOX Mechanical Impact Test Results

Date	Test	Material	Description of Test	RH (%)	Temp °C (°F)	Reactions/Test @ Energy Level J (ft-lbf)													
						108.4 (80)	97.6 (72)	88.1 (65)	81.3 (60)	74.6 (55)	68 (50)	61 (45)	54.2 (40)	47.5 (35)	40.7 (30)	33.9 (25)	27 (20)	20.3 (15)	13.5 (10)
8/21/13	A	AK-225G	STD	--	--	--	0/20	--	--	--	--	--	--	--	--	--	--	--	--
2/5/14	B	Solvokane	STD	--	--	--	2/7	1/1	--	--	--	--	1/2	1/1	1/8	1/12	1/12	1/2	1/11
2/6/14	C	L-14780	STD	--	--	--	2/6	1/13	1/15	1/8	1/3	1/4	0/20	--	--	--	--	--	--
2/11/14	D	Solstice PF	STD	--	--	--	2/7	1/3	--	1/5	1/2	1/14	1/11	1/15	1/6	1/2	1/9	0/20	--
6/23/14	E	Solstice PF	STD	28	19 (66)	--	2/5	2/6	1/11	2/6	2/12	0/3	--	--	--	--	--	--	--
6/24/14	F	L-14780	STD	28	18 (65)	0/3	0/72	0/20	--	--	--	--	--	--	--	--	--	--	--
6/25/14	G	AK 225G	STD, Prep 1	46	17 (63)	0/5	0/20	--	--	--	--	--	--	--	--	--	--	--	--
6/25/14	H	AK 225G	STD, Prep 2	46	17 (63)	0/5	0/20	--	--	--	--	--	--	--	--	--	--	--	--
6/27/14	I	Solstice PF	STD	23	19 (67)	--	2/3	2/4	2/6	2/10	2/7	2/2	2/7	1/9	--	--	--	--	--
7/15/14	J	Solstice PF	w/insert	67	20 (68)	--	2/9	1/14		--	--	--	--	--	--	--	--	--	--
7/17/14	K	Solstice PF	STD	72	17 (63)	--	2/14	2/7	0/3	--	--	--	--	--	--	--	--	--	--
9/8/14	L	Solstice PF	STD WS P/HW	80	18 (65)	--	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/8/14	M	Solstice PF	WS P/HW w/insert	61	21 (69)	--	2/9	1/7	1/4	1/2	1/11	0/20	--	--	--	--	--	--	--
9/9/14	N	Solstice PF	M P/WS HW w/insert	64	17 (62)	--	2/23	1/5	0/20		--	--	--	--	--	--	--	--	--
9/10/14	O	Solstice PF	M P/HW w/insert	50	18 (65)	--	2/28	1/2	1/1	0/20	--	--	--	--	--	--	--	--	--
9/11/14	P	Solstice PF	STD M P/HW	55	18 (65)	--	2/13	0/20	--	--	--	--	--	--	--	--	--	--	--
9/11/14	Q	Solstice PF	STD M P/HW RC	55	18 (65)	0/6	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/12/14	R	Solstice PF	STD WS P/M HW	72	19 (67)	--	2/22	1/3	1/15	1/8	1/7	1/8	0/20	--	--	--	--	--	--
9/15/14	S	Solstice PF	STD WS P/M HW	67	17 (63)	--	1/20	--	--	--	--	--	--	--	--	--	--	--	--
9/16/14	T	Solstice PF	STD WS P/HW F	75	16 (61)	--	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/16/14	U	Solstice PF	STD WS P/HW RC	75	18 (65)	--	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/16/14	V	Solstice PF	WS P/HW w/insert	75	18 (65)	--	2/15	1/10	--	--	--	--	--	--	--	--	--	--	--
9/25/14	W	Solstice PF	WS P/HW w/insert F RC	57	19 (67)	--	1/61	--	--	--	--	--	--	--	--	--	--	--	--
9/25/14	X	L-14780	WS P/HW w/insert F RC	55	19 (67)	--	0/70	--	--	--	--	--	--	--	--	--	--	--	--

STD = WSTF Standard Test Method; WS = WSTF; M = MSFC; P = Prep; HW = Hardware; F = Filtered solvent; RC = Rebound catcher used

Table 2

Ambient Pressure LOX Mechanical Impact Test Results for Specific Investigations

Date	Test	Material	Description of Test	RH (%)	Temp °C (°F)	Reactions/Test @ Energy Level J (ft-lbf)													
						108.4 (80)	97.6 (72)	88.1 (65)	81.3 (60)	74.6 (55)	68 (50)	61 (45)	54.2 (40)	47.5 (35)	40.7 (30)	33.9 (25)	27 (20)	20.3 (15)	13.5 (10)
WSTF Standard Method																			
8/21/13	A	AK-225G	STD	--	--	--	0/20	--	--	--	--	--	--	--	--	--	--	--	--
2/5/14	B	Solvokane	STD	--	--	--	2/7	1/1	--	--	--	--	1/2	1/1	1/8	1/12	1/12	1/2	1/11
2/6/14	C	L-14780	STD	--	--	--	2/6	1/13	1/15	1/8	1/3	1/4	0/20	--	--	--	--	--	--
2/11/14	D	Solstice PF	STD	--	--	--	2/7	1/3	--	1/5	1/2	1/14	1/11	1/15	1/6	1/2	1/9	0/20	--
Using a Rebound Catcher																			
9/11/14	Q	Solstice PF	STD M P/HW RC	55	18 (65)	0/6	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/16/14	U	Solstice PF	STD WS P/HW RC	75	18 (65)	--	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/25/14	W	Solstice PF	WS P/HW w/insert F RC	57	19 (67)	--	1/61	--	--	--	--	--	--	--	--	--	--	--	--
Using Filtered Solvents																			
9/16/14	T	Solstice PF	STD WS P/HW F	75	16 (61)	--	0/20												
9/25/14	W	Solstice PF	WS P/HW w/insert F RC	57	19 (67)	--	1/61												
						--													

STD = WSTF Standard Test Method; WS = WSTF; M = MSFC; P = Prep; HW = Hardware; F = Filtered solvent; RC = Rebound catcher used

Test I (Solstice PF) was performed June 27, 2014 with a new container of the solvent from the same lot and batch as the previously provided container. Testing was restarted due the new solvent container. The samples were prepped using Sample Preparation Method 1. Two reactions were observed at 54 J (40 ft-lbf), and 1/9 samples tested reacted at 47.5 J (35 ft-lbf). Testing was stopped when no clean grease cups were available. Average reaction frequency for 97.6 and 88 J (72 and 65 ft-lbf) was 58 percent. Samples were prepared at 17 °C (63 °F) and 22 percent RH.

Test J (Solstice PF) was performed July 15, 2014 and was prepared using Sample Preparation Method 1. Although it was known that insufficient grease cups were available to complete a test series, it was decided to divide available cups into two groups in order to assess material reactivity in the higher humidity environment that was currently available and to examine the effect of using stainless steel disks. In this test stainless steel discs were placed below the grease cups. Two reactions were observed at 97.6 J (72 ft-lbf) and 1/9 samples tested reacted at 88.1 J (65 ft-lbf). Charring during test was light gray or of a discolored nature when reactions were observed. Average reaction frequency for 97.6 and 88.1 J (72 and 65 ft-lbf) was 15 percent. Samples were prepared at 17 °C (63 °F) and 67 percent RH.

Test K (Solstice PF) was performed July 17, 2014 and was prepared using Sample Preparation Method 1. Again, it was known that not enough grease cups were available to complete a test series, and it was decided to divide available cups into two groups in order to assess material reactivity in the higher humidity environment that was currently available and to examine the effect of using stainless steel disks. In this test, stainless steel discs were placed below the grease cups. This test provided a sample set to compare with tests performed without stainless steel discs below the grease cups. Two reactions were observed at 88.1 J (65 ft-lbf), and 0/3 samples tested reacted at 81.3 J (60 ft-lbf). Reactions observed during test J were less noticeable than reactions observed during Test K. Charring observed during this test were black and more abundant than in Test J when reactions were observed. Average reaction frequency for 88.1 and 81.3 J (65 and 60 ft-lbf) was 21percent. Samples were prepared at 17°C (63°F) and 72 percent RH.

5.2.2 STANDARD TEST METHOD – 13A DISCUSSION

Results obtained during the initial testing of each of the solvents showed the AK-225G to be the superior solvent with respect to oxygen compatibility. Candidate solvents were tested between February 5 and 11, 2014. The L-14780 and Solstice PF solvents performed better than the Solvokane, which failed at the lowest obtainable energy level. All potential replacement solvents resulted in reactions at some energy level.

Tests performed at MSFC on the Solstice PF solvent resulted in an energy threshold of 97.6 J (72 ft-lbs). With such a large variance in results, several variables were considered as possible sources for these inconsistencies.

Further testing in June and July of 2014 at WSTF was performed using Standard WSTF protocol, exploiting the higher humidity during spring/summer months. Tests performed using the standard WSTF method are displayed in Table 1.

5.3 TEST VARIABLE INVESTIGATION – NASA STANDARD TEST 13A

Cooperative WSTF and MSFC testing took place at WSTF during the weeks of September 8 and 15, 2014 after conflicting results were reported by the two test centers. The purpose of the cooperative testing was to help establish a standardized test method that could be used to move forward in the solvent evaluation. Variables considered during this portion of the evaluation included the following:

1. Sample preparation method
2. The use of a rebound catcher
3. Filtering solvents before test
4. Hardware cleanliness (hardware provided by MSFC and WSTF)
5. Preparation humidity
6. Use of insert disks below sample (grease) cups

All testing to evaluate the effect of the described variables were performed using the Solstice PF solvent.

5.3.1 VARIABLE – SAMPLE PREPARATION

The sample preparation investigation consisted of comparing the test results obtained when technicians from each Center (MSFC and WSTF) prepared samples for testing. WSTF technicians prepared samples using Sample Preparation Method 1 described in Section 5.1. MSFC technicians prepared samples similar to Sample Preparation Method 2, also described in Section 5.1. WSTF chose to use Sample Preparation Method 1 due to the consistency in obtaining homogenous sample surface textures. This variable investigation was not concerned with the sample acceptance criteria but rather the effect on energy threshold results.

5.3.2 VARIABLE – REBOUND CATCHER

During the variable evaluation, an elevated number of the reactions were observed during sample cup edge/striker pin edge interaction. Two modes of edge-to-edge interaction were identified. Figure 3 shows an example of each of the edge-to-edge orientations during impact.

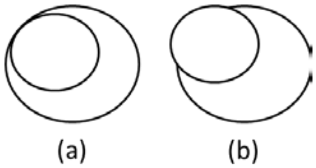


Figure 3
Examples of Edge Impact: (a) Edge (Uniform), (b) Non-Uniform

Non-uniform impacts were determined to be especially severe. Two methods of obtaining non-uniform impacts were identified:

1. Uncentered sample cup and striker pin
2. Secondary impacts resulting in non-uniform impacts

Non-uniform impacts result in the potential for higher energy imparted onto the test sample. Scraping caused by the metal-to-metal contact can produce heat and induce reactions. When testing solid samples, the primary impact often shatters the material and subsequent impacts result in higher surface area to mass ratios, which encourage reactions. Heat caused by metal-to-metal contact coupled with shattered test samples promote ignition of the material.

Rebound catchers are mechanical devices used to prevent secondary impacts. The effects of secondary impacts were eliminated by the use of a rebound catcher.

5.3.3 VARIABLE – FILTERED SOLVENTS

Impurities within the solvents have the potential to react in LOX, as they have the potential to kindle to the solvent and cause reactions when impacted. Removing these impurities would prevent this potential for reactions. Prior to use in the field, all solvents are filtered; therefore, filtering of solvents prior to test would ensure testing reflects in-use configuration of the materials. Filtering of solvents prior to testing is also in accordance with good lab practices.

5.3.4 VARIABLE – HARDWARE CLEANLINESS

The impact on cleanliness was considered as a possible reason for the disparity in test results between the two test centers. Hardware at WSTF is typically cleaned using a Brulin®[1]/tap water solution followed by sonication (WSTF Job Instruction (WJI-) 800-0121.A. [2] All stainless steel discs and grease cups are cleaned to oxygen service 50A. Marshall Space Flight Center cleans hardware for oxygen service in accordance with MSFC-SPEC-164D.[3]

To obtain comparable results, WSTF cleaned all hardware (aluminum cups, stainless steel cups and discs, and striker pins) to oxygen service 50A.

5.3.5 VARIABLE – PREPARATION HUMIDITY

Relative humidity at the time of sample preparation was considered a potential cause of the disparity in results between testing centers. Relative humidity in Las Cruces, New Mexico during the month of February usually falls between 12 and 20 percent, whereas typical humidity in Huntsville, Alabama is much higher. Initial testing did not include the recording of humidity in the prep area.

It was believed that the formation and entrapment of water ice within or on the surface of samples might take place when preparing samples in humid environments. This entrapment of moisture was thought to decrease the reactivity of the test samples.

For this evaluation, the humidity was monitored and recorded near the preparation area. The evaluation took place during the month of September, a more humid time of the year at WSTF.

---

[1] Brulin® is a registered trademark of Brulin & Company, Inc., Indianapolis, IN.

[2] In-house document. WJI-800-0121.A. *Sample Preparation Manual for NASA-STD-6001, Navy Protocol, and Selected ASTM Procedures.* July 15, 2009.

[3] Marshall Space Flight Center. MSFC-SPEC-164D, *Cleanliness of Components for Use in Oxygen, Fuel, and Pneumatic Systems Specification.* January 30, 2014.

5.3.6 VARIABLE – INSERT DISCS

Insert discs are typically placed between the soft aluminum LOX cup and the smaller stainless steel grease cup. Insert discs provide a more solid impact surface as opposed to the soft aluminum cups, which deform and absorb energy when impacted. The use of insert discs should result in a more severe test, but MSFC results indicated otherwise. WSTF does not typically use these components; however, tests repeatedly resulted in lower energy thresholds than MSFC. Controlled tests were performed to analyze the significance of this variable.

5.4 TEST VARIABLE INVESTIGATION RESULTS – NASA STANDARD TEST 13A

The following are the results obtained from the variable investigation performed using the Solstice PF solvent. Tests were performed at WSTF by WSTF and MSFC personnel, and results are displayed in Table 3.

Two test series (L and M) were performed September 8, 2014. The first test performed (Test L) was a standard WSTF test (Sample Preparation Method 1, WSTF hardware, no insert disk, no rebound catcher, and unfiltered solvent). Preparation conditions were 80 percent humidity at 18 °C (65 °F). No reactions were observed at 97.6 J (72 ft-lbf). A second batch of samples was prepared with insert disks below the stainless steel grease cups. Test M yielded an energy threshold of 54.23 J (40 ft-lbf). Preparation conditions were 61 percent RH at 20.6 °C (69 °F).

MSFC personnel prepared the test samples September 9, 2014 with WSTF hardware, unfiltered solvent, and insert disks. No rebound catcher was used for this test (Test N). The threshold was achieved at 81.3 J (60 ft-lbf). The following day, the same test (Test O) performed with MSFC hardware at lower humidity resulted in a slightly lower threshold of 74.6 J (55 ft-lbf).

Two tests were performed September 11, 2014, prepared by MSFC personnel. Tests were executed without stainless steel inserts. Samples used for both tests were prepared simultaneously with identical environmental conditions. The first of the tests (Test P) resulted in an energy threshold of 88.2 J (65 ft-lbf). The rebound catcher was installed and a second test (Test Q) was performed, which resulted in an energy threshold of 98.6 J (72 ft-lbf). Six additional tests at approximately 108.4 J (80 ft-lbf) were performed, with no reactions.

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

Table 3
Ambient Pressure LOX Mechanical Impact Test Variable Investigation Results

Date	Test	Material	Description of Test	RH (%)	Temp °C (°F)	Reactions/Test @ Energy Level J (ft-lbf)													
						108.4 (80)	97.6 (72)	88.1 (65)	81.3 (60)	74.6 (55)	68 (50)	61 (45)	54.2 (40)	47.5 (35)	40.7 (30)	33.9 (25)	27 (20)	20.3 (15)	13.5 (10)
2/11/14	D	Solstice PF	STD	--	--	--	2/7	1/3	--	1/5	1/2	1/14	1/11	1/15	1/6	1/2	1/9	0/20	--
9/8/14	L	Solstice PF	STD WS P/HW	80	18 (65)	--	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/11/14	P	Solstice PF	STD M P/HW	55	18 (65)	--	2/13	0/20	--	--	--	--	--	--	--	--	--	--	--
9/11/14	Q	Solstice PF	STD M P/HW RC	55	18 (65)	0/6	0/20	--	--	--	--	--	--	--	--	--	--	--	--
9/12/14	R	Solstice PF	STD WS P/M HW	72	19 (67)	--	2/22	1/3	1/15	1/8	1/7	1/8	0/20	--	--	--	--	--	--
9/8/14	M	Solstice PF	WS P/HW w/insert	61	21 (69)	--	2/9	1/7	1/4	1/2	1/11	0/20	--	--	--	--	--	--	--
9/9/14	N	Solstice PF	M P/WS HW w/insert	64	17 (62)	--	2/23	1/5	0/20		--	--	--	--	--	--	--	--	--
9/10/14	O	Solstice PF	M P/HW w/insert	50	18 (65)	--	2/28	1/2	1/1	0/20	--	--	--	--	--	--	--	--	--

STD = WSTF Standard Test Method; WS = WSTF; M = MSFC; P = Prep; HW = Hardware; F = Filtered solvent; RC = Rebound catcher used

Marshall Space Flight Center supplied clean hardware for the investigation. While testing on September 12, 2014, one batch of the striker pins was grossly contaminated with aluminum-oxide flakes thought to have been generated by the anodized pin racks. Two extremely energetic reactions were observed when using striker pins from this batch. The suspect striker pins were analyzed for particulate, and gross metallic particulate contamination was confirmed within this batch. In subsequent tests performed by WSTF personnel using MSFC hardware, the hardware was cleaned using dry gaseous nitrogen to blow off/remove particulate.

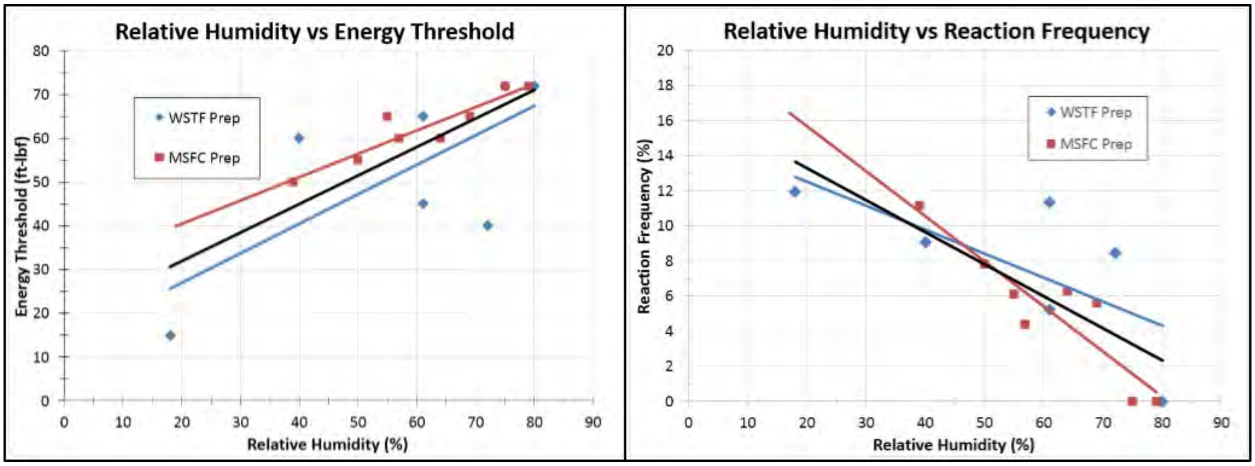
A high humidity (72 percent RH) test was performed using MSFC hardware prepared by WSTF. No inserts were used. Tests resulted in an energy threshold of 54.2 J (40 ft-lbf).

Three tests were performed September 16, 2014. All samples were prepared under the same environmental conditions by WSTF personnel with WSTF hardware. Tests using filtered solvent (Test T) and unfiltered solvent with the rebound catcher installed (Test U) resulted in energy thresholds of 98.6 J (72 ft-lbf) each. The third test performed that day was performed with insert disks. This test was an incomplete data set resulting in 1/10 reactions at 88.1 J (65 ft-lbf).

In an attempt to further analyze the effects of variables, tests were separated into the three different types of data plots: for sample preparation method, hardware cleanliness, and use of insert discs. Since no reactions were observed using either filtered solvents or rebound catchers, tests utilizing these variables were not considered during this portion of the evaluation. All tests presented in Figures 4 through 6 (Sections 5.4.1, 5.4.4, and 5.4.6, respectively) represent completed tests. Energy thresholds and reaction frequencies were plotted as functions of relative humidity. Black trend lines represent linear regression models of all data included in the plot while colored trend lines correspond to the data of the particular Center (WSTF blue; MSFC red).

5.4.1 TEST RESULTS – SAMPLE PREPARATION

Sample preparation performed by WSTF and MSFC was compared using Sample Preparation Method 1 (Section 5.1). Energy threshold results for both MSFC and WSTF preparation trended very closely to the combined linear regression. The tightly grouped regressions indicate little to no dependence of sample preparation on energy threshold (Figure 4 a). When analyzing the differences between WSTF and MSFC prepped samples with respect to reaction frequency (Figure 4 b), some differences were observed.



(a) (b)

Figure 4

NASA Standard Test 13A – Sample Preparation Investigation

5.4.2 TEST RESULTS – REBOUND CATCHER

Table 2 displays the results of these tests. All tests performed with the rebound catcher resulted in 97.6 J (72 ft-lbf) energy thresholds. Tests performed using the rebound catcher were executed between 55 and 75 percent RH. No reactions were observed when using the rebound catcher.

5.4.3 TEST RESULTS – FILTERED SOLVENTS

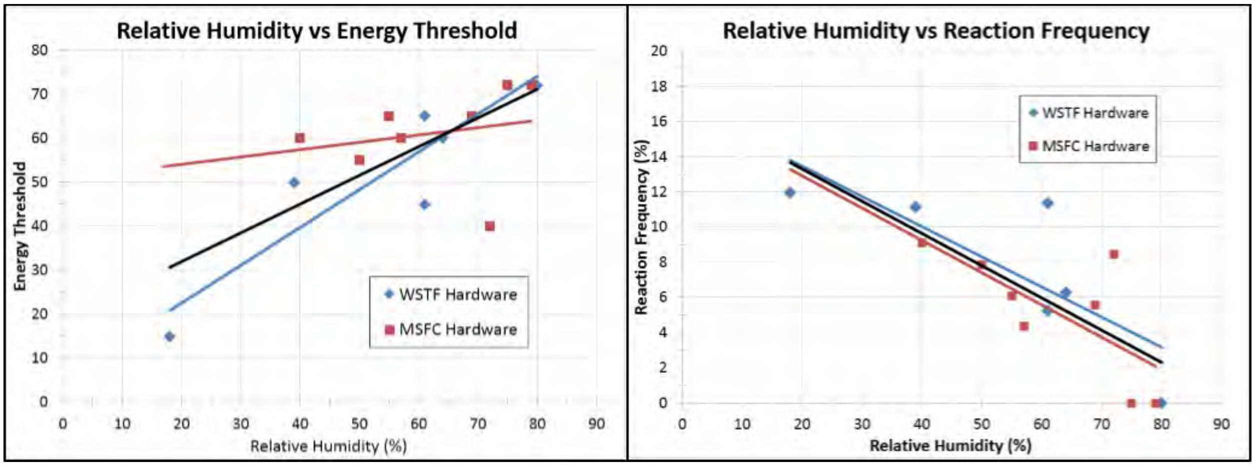
Two tests were performed using filtered Solstice PF solvent (Table 2). All tests on filtered Solstice PF solvents resulted in energy thresholds of 97.6 J (72 ft-lbf); however, results were inconclusive due to the coupling with other variables. It is difficult to discern if the decrease in reactivity was a result of the filtering of the solvent or due to the use of the rebound catcher or high prep humidity. Due to the end use configuration and good lab practices, WSTF suggests the filtering of all solvents for testing. No reactions were observed when using the filtered solvents.

5.4.4 TEST RESULTS – HARDWARE CLEANLINESS

As part of standard procedures, a minimum of one blank test after five sample tests is required to ensure cleanliness of hardware. A blank test sample consists of a stainless steel insert placed within an aluminum cup submerged in LOX. The blank test sample is impacted in just the same way as an actual test sample. At no time during testing were reactions observed during blank impacts. Reactions during a blank test would indicate contamination of hardware.

Further analysis of the differences in test center cleanliness was performed by analyzing the plotted data sets (Figure 5). As before, all complete data sets (excluding tests utilizing filtered solvents and rebound catchers) were used to compare the impact of where the hardware was cleaned on reaction frequency and energy threshold as a function of relative humidity. Reaction frequency as a function of relative humidity for both preparation methods correlated well with the combined linear regression (Figure 5 b). This relationship indicates no significant dependence of reaction frequency on the method of hardware cleaning.

The relationship between energy threshold and relative humidity (Figure 5 a) is more inconclusive. Performing testing with MSFC-cleaned hardware at lower relative humidity would fill in gaps within the data set and possibly correlate more closely to the overall linear regression.



(a) **(b)**

Figure 5

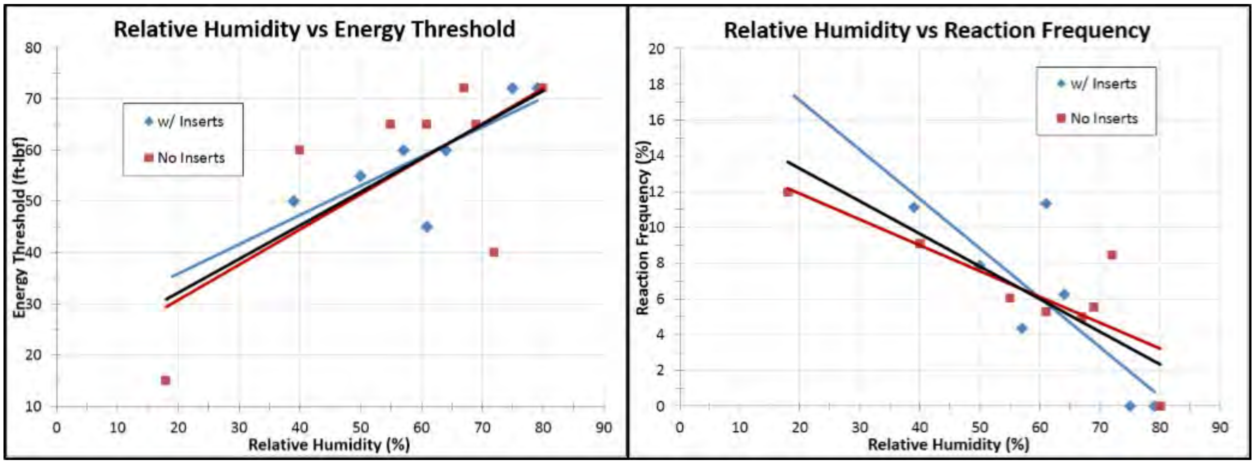
NASA Standard Test 13A – Hardware Cleanliness Investigation

5.4.5 TEST RESULTS – PREPARATION HUMIDITY

Decoupling variables was difficult due to the uncontrollable influence of sample preparation relative humidity. When analyzing the data plots described in Sections 5.4.1, 5.4.4, and 5.4.6 (Figures 4 through 6), the dependence of reaction frequency and energy threshold to relative humidity is obvious. All testing indicates lower preparation humidity results in a more severe and reactive test.

5.4.6 TEST RESULTS– INSERT DISCS

When examining data with respect to the use of inserts, energy threshold data (Figure 6 a) trended nearly identically to average energy threshold data. Energy thresholds tended to decrease as a result of an increase in relative humidity. Tests performed with inserts with respect to reaction frequency (Figure 6 b) resulted in data that appeared to be more dependent on relative humidity; however, the data set was not very tightly grouped. More data would be required to perform a more reliable analysis of the reaction frequency dependence on relative humidity as a result of insert comparisons.



(a) (b)

Figure 6

NASA Standard Test 13A – Insert Investigation

5.5 MODIFIED TEST METHOD

Because of the differing results at WSTF and MSFC from the LOX mechanical impact testing, a NASA Independent Assessment (IA) Team, sponsored by NASA Engineering & Safety Center (NESC), was formed to investigate the test variables and conditions that could affect the reactivity in LOX and to establish a modified test protocol to ASTM G86 for subsequent testing. Conclusions from the test variable investigation resulted in changes in the test method described in the following section.[1]

5.5.1 MODIFIED TEST METHOD – DESCRIPTION

The exclusion of non-uniform and secondary impacts increases the reproducibility of the test method; therefore, it was agreed that a rebound catcher be used to mitigate each of the variables. An acceptance criteria excluding non-uniform impacts ensures that variability associated with such impacts would not affect the reproducibility of the test method.

The sample preparation method did not appear to have a significant impact in the reactivity of test samples; however, the pre-chilling of the sample (grease) cups prior to the introduction of solvent was thought to limit the chance of introducing moisture into test samples. Although the method of sample preparation did not have a significant impact, relative humidity at the time of sample preparation was shown to play a significant role in the impact sensitivity.

Limited data were gathered evaluating the impact of solvent filtration on impact sensitivity. Nonetheless, filtration should be performed prior to performing mechanical impact testing due to the following:

1. Filtration is a good lab practice
2. Current storage containers of the size used for some solvents are plain, unlined carbon steel susceptible to oxidation
3. Filtration will be performed prior to use in the field

---

[1] NESC Independent Assessment. HCFC225cg (AK225G) Replacement Study, 3rd Technical Interchange Meeting, Independent Assessment Investigation/Ambient Pressure LOX Impact Testing. Steven Gentz (NASA/NESC) and H. Richard Ross (A2R/SSC Gas & Materials Science). Hosted by NASA/WSTF October 21-22, 2014.

Following the variable investigation, it was decided that the final tests would incorporate the following:

1. Pre-chilling of the sample (grease) cups prior to the introduction of solvent
2. Use of insert disks below sample (grease) cups
3. Use of a rebound catcher
4. Filtered solvents
5. Preparation humidity below 60 percent RH
6. Invalidation of non-uniform impacts resulting in reactions

5.5.2 MODIFIED TEST METHOD - TEST RESULTS

Both L-14780 and Solstice PF were filtered using the same filter and filtration flask used by the independent assessor. The Solstice PF was filtered first. A triple rinse of the equipment with non-filtered L-14780 was performed on all the glassware prior to filtration. A triple rinse of the filtration flask was performed prior to final filtration. A second filter was installed prior to final filtration of the 3M solvent L-14780. Both solvents were filtered at approximately 10:00 AM on September 25, 2014.

Sample preparation began at approximately 11:00 AM. Eighty samples of each of the solvents were prepped the day of testing. Table 4 shows the variation in prep temperature and humidity in ~ 15 min intervals for each of the solvent preps. Solstice PF was prepped, immediately followed by the preparation of the L-14780 solvent. The solvents were not pre-chilled. All grease cups were pre-chilled prior to sample preparation.

Table 4
Modified Test Method Prep Conditions

Solvents	Time	Humidity (%)	Temperature (F)
Solstice PF	11:05	56	68
	11:20	57	67
	11:35	57	67
	11:48	58	65
L-14780	12:00	53	64
	12:15	55	62
	12:30	58	66
	12:42	58	65

Testing commenced at 1:00 PM. Two blank impacts were performed at the start of the first test. Forty tests of the Solstice solvent were performed prior to changing over to the L-14780 solvent. Five blank impacts were performed between testing of different solvents. Testing of L-14780 began at approximately 2:30 PM. Seventy tests were performed with L-14780 before switching back to the Solstice PF. At approximately 6:20 PM five blank impacts were performed prior to switching back to Solstice PF. An additional 22 tests were performed with the Solstice PF.

No reactions for 70 impacts occurred during the testing of L-14780. Two reactions were observed during the testing of the Solstice solvent. One of these reactions occurred during a non-uniform impact; the second occurred on an edge strike.

5.6 SUMMARY STANDARD TEST 13 METHOD CONCLUSIONS

All candidate materials were shown to react when mechanically impacted while submerged in LOX. The L-14780 performed better than either of the other two candidate materials (Solstice PF and Solvokane); however, variables were shown to greatly affect the reactivity of samples. These variables were characterized through testing; and test parameters such as secondary rebounds, non-uniform impacts, particulate within the solvent and test hardware, as well as preparation humidity, were shown to affect test results. Further testing would be required to fully characterize the variables discussed in this paper; in particular, more replicates of each test performed should be tested during periods of lower relative humidity.

Recommendations to ensure control of parameters were employed with positive results. Tests performed with these controls in place allowed for an evaluation of the materials rather than on external variables. Preparing adequate samples for a complete test series of each of the final two candidate materials, in approximately the same conditions, at the same time, and employing proper filtration and the use of the rebound catcher to mitigate uncontrolled impacts, resulted in a passing energy threshold evaluation for each of the materials.

All tests performed during this evaluation are displayed in Table 1.

6.0 HIGH-PRESSURE LOX MECHANICAL IMPACT TESTING

Limited tests were performed to analyze the effect of testing in a high-pressure oxygen environment.

6.1 PREPARATION METHOD

The preparation of test samples for high-pressure LOX tests and ambient LOX tests were identical. The method of preparation is documented in Section 5.1 of this report.

6.2 HIGH-PRESSURE LOX MECHANICAL IMPACT TESTING - STANDARD TEST METHOD TEST 13B

Standard WSTF protocol involves performing a pressure threshold. Testing began at the highest energy level of 97.6 J (72 ft-lbf) at some pressure above ambient conditions. One reaction at the 97.6 J (72 ft-lbf) energy level was required prior to decreasing the test pressure. Pressure was varied by 3.45 MPa (500 psi) increments. If the intensity of the reactions was such that the test team suspected reactions to occur at the next lower energy level, skipping test pressures was acceptable to expedite the bracketing of the reaction/energy threshold. Twenty consecutive tests in which no reactions were witnessed by any means, audible or visual (charring or flashing), with at least one reaction at the next higher energy level, were required to affirm a passing threshold pressure level. One reaction in 60 samples at 97.6-J (72 ft-lbf) was also considered a passing energy threshold test.

Unlike testing at ambient pressures, high-pressure tests require the use of a rebound catcher.

6.3 STANDARD TEST METHOD – 13B TEST RESULTS/DISCUSSION

Solstice PF and L-14780 performed similarly, each passing at 51.7 MPa (7500 psia). Solvokane failed the pressure threshold evaluation, reacting at 3.45 MPa (500 psia). Solstice PF and L-14780 could be considered equivalent with respect to oxygen compatibility, with Solvokane performing much more poorly.

7.0 AUTOGENOUS IGNITION TEMPERATURE TESTING

Autogenous ignition temperature (AIT) tests are performed as a means of determining the temperature at which liquids and solids spontaneously ignite. The standard is detailed in ASTM G72.

7.1 PREPARATION METHOD – AIT TESTING

The only deviation from the standard preparation method described in ASTM G72 was the pre-chilling of sample material in an attempt to limit the evaporation of the test material. Glass beakers of the solvent were chilled inside a refrigerator. Maintaining pre-weighed amounts of a chilled solvent was achieved by nesting the test vial within a 20 mL glass vial partially submerged in a saltwater ice bath.

7.2 STANDARD TEST METHOD – AIT TESTING

The standard test method is performed as follows. A sample holding assembly, contained within a reaction vessel pressurized with 100 percent oxygen to 10.3 MPa (1500 psi), is heated in an electric furnace at a rate of 5 ± 1 °C (9 ± 1 °F)/min from 60 to 260 °C (140 to 500 °F). Heating of the vessel is continued at an uncontrolled rate to a maximum temperature of 450 °C (842 °F). Temperatures are monitored as a function of time by means of a thermocouple and data acquisition system. During testing, pressure is monitored but not maintained. Ignition of the test sample is indicated by a rapid temperature rise of at least 20 °C (36 °F) and confirmed posttest by the destruction of the sample.

Standard testing is performed at 10.3 MPa (1500 psia) with a sample size of 0.20 ± 0.03 g. A sample size of 200 ± 30 mg is selected to prevent damage to the test apparatus as a result of an overpressure caused by a reaction of test material. Samples are typically maintained at room temperature. Three purges of 100 percent oxygen at a minimum of 345 kPa (50 psia) are performed prior to each test to ensure the venting of residual air.

Deviation from ASTM G72 pressures was requested of WSTF to coincide with testing performed during the last solvent evaluation. During that solvent evaluation, AIT tests were performed at two pressures: 345 kPa (50 psia) and 13.8 MPa (2000 psia). Tests with the control solvents (AK-225G and HFE-7100) performed at the requisite test pressures of 345 kPa (50 psia) and 13.8 MPa (2000 psia) yielded no AITs. These results correspond to legacy testing.

Test results using standard methods proved to be inconclusive. Evidence of combustion was noted; however, potential reactions were not rapid enough to register AITs. Prior to testing the potential replacement solvents, an investigation into the test variables was performed to optimize the test method.

7.3 TEST VARIABLE INVESTIGATION – AIT TESTING

Control tests performed using HFE-7100 and AK-225G aided in the development of an improved test method. These solvents are considered oxygen compatible solvents and exhibit volatility similar to the potential replacement solvents. The absence of an AIT was indicative of a lack of adequate sample material within the reaction vessel during tests. Forced evaporation during purging and inadequate sample size were identified as two potential causes for inadequate available sample material during testing.

Initially, three variables were considered during this investigation: sample size, pretest purges, and thermocouple placement. After obtaining inconclusive results, a fourth variable, test pressure, was examined resulting in AITs. To correlate with historical data, tests were performed at the requested test pressures of 345 kPa (50 psia) and 13.8 MPa (2000 psia).

7.3.1 VARIABLE – TEST SAMPLE SIZE

Any combustion event requires three components: fuel, oxidizer, and an ignition source. Autogenous ignition temperature tests are typically performed in 100 percent oxygen environments (oxidizer), and the test method provides the heat/ignition source; therefore, ensuring adequate combustible material is key to combustion.

Volatile solvents present a challenge prior to testing; however, once the reaction chamber is sealed, the volatility should not present a problem igniting the material. These materials all burn in the gas phase; therefore, evaporation of the material is required for combustion. Standard sized samples may be an insufficient amount of material to react violently enough to register as an AIT.

Addressing inadequate sample size involved gradually increasing the sample size until a reaction was observed. This gradual increase in sample size allowed for a controlled method of varying the test protocol while ensuring safe operation of the test system.

7.3.2 VARIABLE – PRETEST PURGES

The potential for forced evaporation due to pretest purging was considered and evaluated for both control solvents. Two types of tests were performed to evaluate the effect of purging on AIT test results.

The first test used to evaluate purging consisted of pressurizing test samples immediately followed by removal of the test samples. No heat was induced on samples during these simulation tests. Samples were pre-chilled and weighed prior to loading into a reaction vessel. Three consecutive pressurization/vent cycles were completed prior to removal of the test sample. Posttest weights were recorded and compared to pretest weights. These tests were conducted at 345 kPa (50 psia), 6.9 MPa (1000 psia), and 13.8 MPa (2000 psia).

The second evaluation conducted to investigate this variable involved performing AIT tests with and without purges. AIT tests were performed at each of the requested pressures with three or zero test pressure purges.

7.3.3 VARIABLE – THERMOCOUPLE PLACEMENT

The position of the thermocouple was examined to determine its influence on AIT results. Figure 7 is a schematic of the AIT assembly. The standard depth of the tip of the thermocouple relative to the sample holder is 15 mm below the upper edge of the test vial. The ‘X’ dimension located on Figure 7 represents the length that was varied for this evaluation. Tests were performed to examine this variable at the standard depth of 15, 30, and 60 mm (0.59, 1.18, and 2.36 in.). All tests were performed using three purges and 500 mg samples.

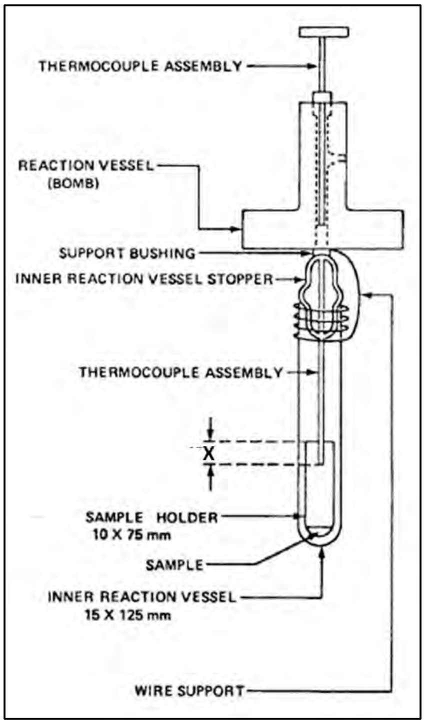


Figure 7
AIT Assembly

7.4 TEST RESULTS – AIT TESTING

The results of each of the variables described in Section 7.3 are discussed in this section. Triplicates of each test were performed unless otherwise noted.

7.4.1 TEST RESULTS – TEST SAMPLE SIZE

The results of the sample size study are presented in Tables 5 and 6. Sample sizes were incrementally increased at each pressure until AITs were observed. Temperature Limit (TL) indicates tests in which the TLs of the test system were reached with no reactions. In the past, tests with these results were potentially erroneously reported as AITs > 800 °F.

No noticeable effect is observed when increasing the sample size beyond the mass at which an AIT is observed. An increase in pressure tends to lower the AIT as expected.

Tests of 500 mg of each of the two control solvents resulted in AITs when tested at 13.8 MPa (2000 psia). AITs were observed when testing HFE 7100 at 345 kPa (50 psia); however, no AITs were observed when testing AK-225G at that pressure. A lack of oxygen was suspected as a cause for the absence of any reactions when testing AK-225G. AK-225G was tested at 483 kPa (70 psia), yielding an AIT higher than recorded at higher pressures. Findings from the sample size investigation concluded 1000 mg as an optimal level for solvent evaluation testing. Averages and standard deviations are reported in Tables 5 and 6 for all triplicates performed.

Table 5

AIT Method Improvement Test Matrix

		345 kPa (50 psia)										13.8 MPa (2000 psia)									
		0 Purges					3 purges					0 Purges					3 purges				
	Mass (mg)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)
AK-225G	200	TL	TL	TL	TL	--	TL	TL	TL	TL	--	237 (458)	228 (443)	233 (452)	233 (451)	4.4 (8)	TL	TL	TL	TL	--
	500	TL	TL	TL	TL	--	TL	TL	TL	TL	--	230 (446)	229 (444)	237 (459)	232 (450)	4.4 (8)	226 (438)	230 (446)	234 (454)	230 (446)	4.4 (8)
	750	TL	TL	TL	TL	--	TL	TL	TL	TL	--	--	--	--	--	--	235 (456)	--	--	235 (456)	--
	1000	TL	TL	TL	TL	--	TL	TL	TL	TL	--	--	--	--	--	--	230 (446)	--	--	230 (446)*	--
Historical AK-225G	200	--	--	--	--	--	TL	--	--	TL	--	--	--	--	--	--	TL	--	--	TL	--
HFE-7100	200	TL	TL	TL	TL	--	TL	TL	TL	TL	--	TL	TL	TL	TL	--	TL	TL	TL	TL	--
	500	388 (731)	389 (732)	391 (735)	389 (733)	1.1 (2)	TL	374 (705)	380 (716)	377 (711)	4.4 (8)	341 (645)	336 (636)	334 (634)	337 (638)	3.3 (6)	337 (639)	341 (646)	3376 (636)	338 (640)	2.8 (5)
	750	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	327 (620)	--	--	327 (620)	--
	1000	--	--	--	--	--	381 (718)	392 (738)	393 (740)	389 (732)	6.7 (12)	--	--	--	--	--	332 (629)	--	--	332 (629)*	--

TL = Temperature Limit, no AIT detected, heater went to full limit; SD =Standard Deviation; Avg = average; * only one test performed

Table 6

AIT Method Improvement Test Matrix, 483 kPa (70 psia)

		0 Purges					3 Purges				
	Mass (mg)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)
AK-225G	200	--	--	--	--	--	--	--	--	--	--
	500	--	--	--	--	--	--	--	--	--	--
	750	TL	TL	TL	TL	--	TL	TL	TL	TL	--
	1000	TL	TL	TL	TL	--	273 523	278 532	276 528	276 528	2.8 (5)

TL = Temperature Limit, no AIT detected, heater went to full limit; SD = Standard Deviation; Avg = average

7.4.2 TEST RESULTS – PRETEST PURGES

Results of the purge mass loss evaluation indicate more mass was lost to purging at higher pressure for each of the solvents than at lower pressure. Approximately the same amount of solvent was lost during purging regardless of the initial sample size. Table 7 presents the results of the purge mass loss evaluation for AK-225G and HFE-7100.

An inherent outcome of the purging process is the loss of some of the sample, and at higher test pressures this loss becomes a significant factor when compared to initial sample mass. The evaluation of pretest purging is presented in Table 7. Historical tests for AK-225G are also presented in Table 5 for direct comparison. An increase in sample size results in AIT for both control solvents.

The influence of residual air within the reaction vessel is most notable at lower pressures due the ratio of air to oxygen. Table 7 shows what kind of influence the lack of 100 percent oxygen pretest purges has on test results. An inadequate amount of oxygen within the reaction vessel at the beginning of the test results in a test with no AIT, as can be seen in Table 6 (483 kPa (70 psi)). Pretest purging with oxygen yields results during testing of 1000 mg AK-225G at 483 kPa (70 psia), while the lack of purging results in tests that reach the Temperature Limit (TL) of the test system (Table 6). As a result of these findings, three purges at test pressure are preferred for final solvent evaluation testing.

Table 7
AK-225G and HFE-7100 Purge Mass Loss Evaluation*

Purge Pressure MPa (psia)	Pretest Temperature °C (°F)	Pretest Mass (mg)	Posttest Mass (mg)	Mass Loss (mg)	Mass Loss (%)	Avg. Mass Loss (mg)	Avg. Mass Loss (%)	Standard Deviation (mg)
AK-225G								
0.3 (50)	2.2 (36)	169	149	20	11.8	18.3	10.8	2.1
	2.2 (36)	167	151	16	9.6			
	2.2 (36)	171	152	19	11.1			
	2.2 (36)	515	497	18	3.5	15	2.9	8.9
	1.7 (35)	506	484	22	4.3			
	1.7 (35)	505	500	5	1.0			
6.9 (1000)	2.2 (36)	185	151	34	18.4	31.3	15.2	4.6
	3.3 (38)	230	196	34	14.8			
	-1.1 (30)	210	184	26	12.4			
	3.9 (39)	519	488	31	6.0	34	6.8	4.4
	-1.1 (37)	496	457	39	7.9			
	3.9 (39)	485	453	32	6.6			
13.8 (2000)	0.6 (33)	227	146	103	35.7	58.7	26.4	22.5
	2.2 (36)	219	183	81	16.4			
	3.3 (38)	217	158	36	27.2			
	-1.1 (30)	482	446	59	7.5	40.7	8.3	4.0
	-0.6 (31)	488	445	36	8.8			
	0.6 (33)	502	459	43	8.6			
HFE-7100								
0.3 (50)	0.6 (33)	204	189	15	7.4	13.3	6.2	3.8
	-1.1 (30)	214	198	16	7.5			
	-1.1 (30)	235	226	9	3.8			
	-1.1 (30)	489	477	12	2.5	13	2.6	2.6
	-1.1 (30)	516	505	11	2.1			
	-1.1 (30)	516	500	16	3.1			
6.9 (1000)	-1.1 (30)	230	198	32	13.9	27	12.5	4.6
	-1.1 (30)	190	167	23	12.1			
	-1.1 (30)	226	200	26	11.5			
	-1.1 (30)	520	507	13	2.5	25	4.9	10.4
	-1.1 (30)	514	483	31	6.0			
	-1.7 (29)	514	483	31	6.0			
13.8 (2000)	-0.6 (31)	219	203	16	7.3	28.3	12.8	11.6
	0.0 (32)	223	184	39	17.5			
	0.6 (33)	223	193	30	13.5			
	2.8 (37)	507	438	69	13.6	58.3	11.5	18.5
	1.7 (35)	513	476	37	7.2			
	1.1 (34)	509	440	69	13.6			

[*] 100 % oxygen w/ 3 purges

7.4.3 TEST RESULTS – THERMOCOUPLE PLACEMENT

For thermocouple placement, no effect was observed between 15 and 30 mm, and minimal effect was observed at 60 mm. Because changes in thermocouple placement did not appreciably influence test results (Table 8), the final AIT test configuration would utilize standard thermocouple placement.

Table 8
Thermocouple Placement Evaluation*

Placement from Top (mm)	Pretest Mass (mg)	AIT °C (°F)	Average °C (°F)	Standard Deviation °C (°F)
15	505	226 (438)	230 (446)	4.4 (8)
15	525	230 (446)		
15	514	234 (454)		
30	521	233 (451)	229 (445)	4.7 (8.5)
30	516	226 (439)		
60	509	238 (461)	241 (466)	2.6 (4.6)
60	524	243 (470)		
60	519	242 (467)		

* AK-225G tests in 100% oxygen @ 13.8 MPa (2000 psia) w/ 3 purges

7.5 MODIFIED TEST METHOD – AIT TESTING

Incorporating the test variable conclusions into a final test method that would adequately evaluate the candidate solvents was the ultimate goal of the test variable investigation. As a result of the testing described, the final test configuration for each of the solvents undergoing evaluation consisted of the following:

1. Pre-chill the solvent prior to test (record temperature)
2. Use minimum test sample size of 1000 mg, larger if required
3. Maintain standard thermocouple placement
4. Perform three purges at test pressure prior to pressurization
5. Perform tests at requisite pressures of 345 kPa (50 psia) and 13.8 MPa (2000 psia), as well as 483 kPa (70 psia)

While performing tests at low pressures, anomalous temperature surges were observed at ~ 121 °C (250 °F). These exotherms, first thought to be combustion events, proved to be decomposition caused by heating. Proof that these temperature surges were not oxidation events but rather solvent decomposition was provided by tests in 100 percent nitrogen, in which similar temperature surges were observed. Results comparing tests performed in oxygen and nitrogen are presented in Figure 8. Figure 8 also demonstrates the effectiveness of purges at low pressure. No AITs were recorded for tests in 100 percent oxygen without purges.

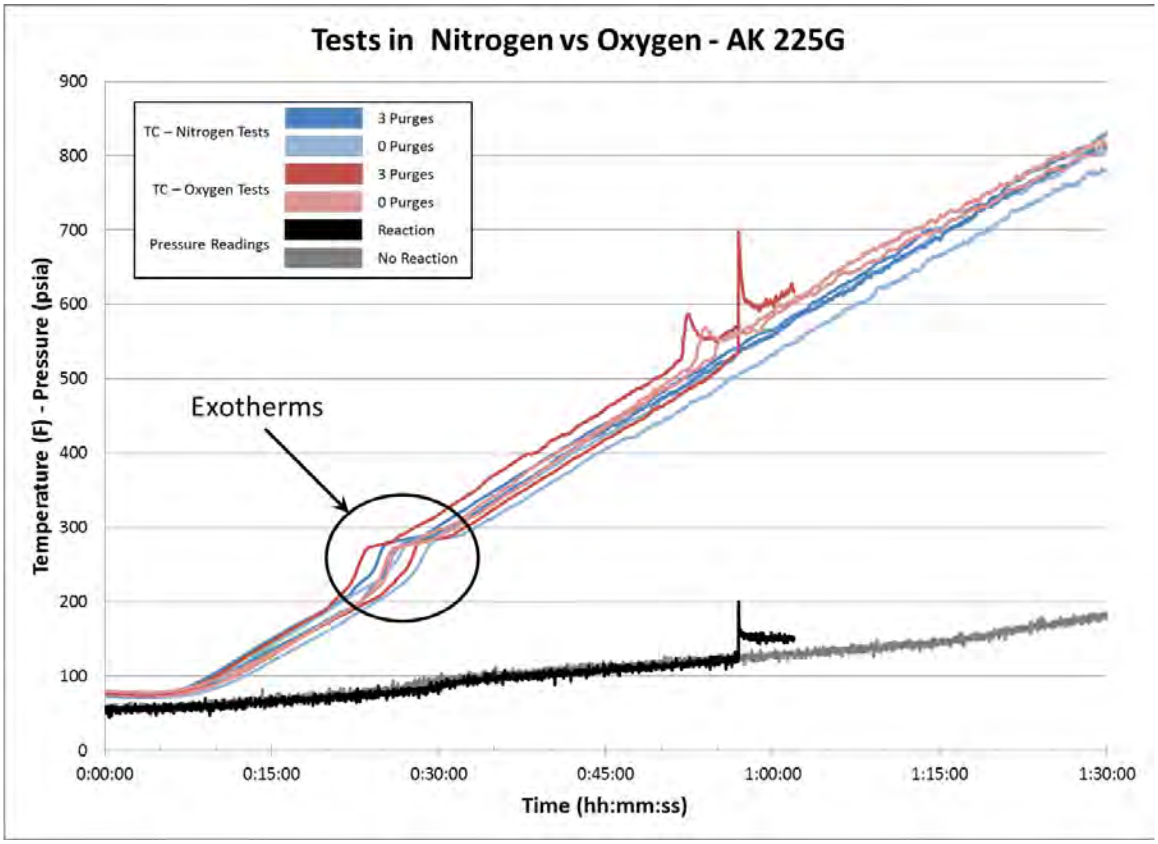


Figure 8
Exotherm Evaluation

7.5.1 MODIFIED TEST METHOD – AIT TESTING RESULTS

The results of the modified test method are displayed in Table 9. Tests were successfully performed

Table 9

Modified Test Method Final AIT Data

		345 kPa (50 psia)						483 kPa (70 psia)						1.38 MPa (200 psia)						13.8 MPa (2000 psia)					
		Chilled					NC	Chilled					NC	Chilled					NC	Chilled					NC
	Mass (mg)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F	Test °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °F	SD °F	Test °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Test °C (°F)	Avg °C (°F)	SD °C (°F)	Test °C (°F)
Solvokane	200	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	TL	--	--	--	--	--
	500	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	147 (296)	--	--	--	--	--
	750	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	143 (290)	--	--	--	--	--
	1000	268 (515)	260 (500)	244 (472)	258 (496)	12.2 (22)	247 (476)	254 490	238 459	269 517	254 489	16.1 29	256 493	--	--	--	--	--	--	153 (308)	151 (303)	152 (305)	152 (305)	1.7 (3)	149 (301)
Solstice PF	200	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	TL	--	--	--	--	--
	500	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	192 (378)	--	--	--	--	--
	750	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	187 (369)	--	--	--	--	--
	1000	245 (473)	246 (474)	230 (446)	240 (464)	8.9 (16)	--	247 (476)	244 (471)	251 (484)	247 (477)	3.9 (7)	--	--	--	--	--	--	--	192 (359)	180 (356)	185 (365)	182 (360)	2.8 (5)	--
L-14780	200	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	TL	--	--	--	--	--
	500	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	167 (333)	--	--	--	--	--
	750	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	168 (334)	--	--	--	--	--
	1000	TL	TL	TL	TL	--	--	TL	TL	TL	TL	--	--	234 (454)	--	--	234 (454)*	--	--	167 (332)	152 (305)	166 (330)	161 (322)	8.3 (15)	--
AK225G	200	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	TL	TL	TL	TL	--	--
	500	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	226 (438)	230 (446)	235 (454)	230 (446)	4.4 (8)	--
	750	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	236 (456)	--	--	236 (456)	--	--
	1000	TL	TL	TL	TL	--	--	273 (523)	278 (532)	276 (528)	276 (528)	2.8 (5)	--	--	--	--	--	--	--	230 (446)	--	--	230 (446)*	--	
HFE-7100	200	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	TL	TL	TL	TL	--	--
	500	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	337 (639)	341 (646)	336 (636)	338 (640)	2.8 (5)	--
	750	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	327 (620)	--	--	327 (620)	--	--
	1000	TL	TL	TL	TL	--	--	381 (718)	392 (738)	393 (740)	389 (732)	6.7 (12)	--	--	--	--	--	--	--	332 (629)	--	--	332 (629)*	--	

TL = Temperature Limit, no AIT detected, heater went to full limit (>800°F,>425°C); SD = Standard Deviation; Avg = average; NC = Not Chilled * Only one test performed

7.5.2 MODIFIED TEST METHOD – DISCUSSION OF AIT TEST RESULTS

Three candidate solvents were tested using the modified AIT test protocol. Results are displayed in Table 9. Average AITs and standard deviations are displayed in the grey boxes for each of the triplicates performed. To ensure safe operation of the test system, sample sizes were incrementally increased to 1000 mg when testing at the highest pressure, 13.8 MPa (2000 psia). By increasing the sample size while testing at the highest pressure, safe operation at lower pressures was ensured. No appreciable differences were found between results obtained at 500 and 1000 mg for any of the materials tested. Triplicates were performed with 1000 mg of material at each pressure.

Results for AIT tests conducted with L-14780 were not obtained at 345 kPa (50 psia) or 483 kPa (70 psia); therefore, pressures were increased to 1.38 MPa (200 psia), at which point an AIT was observed. Only one test was conducted at these conditions. As before, lower pressures resulted in higher AITs, indicating more energy is required to ignite materials.

Higher AITs indicate less reactive materials. All three candidate materials performed more poorly than the two established materials (AK-225G and HFE-7100). The three candidate materials ranked similarly, with Solstice PF performing slightly better than either Solvokane or L-14780.

Three extra tests were performed with Solvokane to further investigate the effects of pre-chilling the solvents prior to test. One test using 1000 mg at each pressure was performed with no appreciable change in results, indicating AITs are more dependent on initial sample size than pre-chilling of sample material.

Combustion by-products for each of the solvents resulted in acidic environments that degraded the components within the reactions vessel. Some etching of the reaction vessels was noted, however not enough to compromise the integrity of the vessel. Thermocouples were the most affected components; 1.59 mm (0.0625 in.) sheathed thermocouples were used during testing and were often corroded. This corrosion did not adversely affect test results, and the thermocouples were routinely replaced. Table 10 documents the different types of posttest residue observed when testing at various conditions.

Table 10
AIT Observed Posttest Residue

	Sample Size	345 kPa (50 psia)		480 kPa (70 psia)		1.38 MPa (200 psia)		13.79 MPa (2000 psia)	
Material	(mg)	O_2	N_2	O_2	N_2	O_2	N_2	O_2	N_2
Solvokane	200	--	--	--	--	--	--	W	--
	500	--	--	--	--	--	--	G	--
	750	--	--	--	--	--	--	G	--
	1000	B	--	Y,B,W,G	--	--	--	W,Y,G	--
Solstice PF	200	--	--	--	--	--	--	W	--
	500	--	--	--	--	--	--	W	--
	750	--	--	--	--	--	--	W,G	--
	1000	B.Y	--	Y,G	--	--	--	W,G,B	--
L-14780	200	--	--	--	--	--	--	W	--
	500	--	--	--	--	--	--	W	--
	750	--	--	--	--	--	--	W	--
	1000	W	--	W,R,BR	--	W	--	W,G	--
AK-225G	200	Y,R,W	--	Y,R,W	--	--	--	W	--
	500	BR,R,Y	--	BR,R,Y	--	--	--	W,Y	--
	750	Y,BR,B	--	--	--	--	--	W,Y	--
	1000	Y,R	B	Y,R	--	--	--	W,Y	--
HFE-7100	200	W	--	W	--	--	--	W	--
	500	W	--	W	--	--	--	W	--
	750	W	--	--	--	--	--	W,B	--
	1000	W,B	--	W	--		--	W,B	--

G-Green, Y-Yellow, R-Red, B-Black, W-White, G-Grey, BR -Brown

7.6 SUMMARY TEST METHOD CONCLUSIONS – AIT TESTING

Successful application of the AIT test method was performed due to some modifications to the method. Legacy testing has shown to be incomplete, and a method of mitigating shortcomings of the test method has been developed.

Purging and adequate sample size have shown to play a significant role in the acquisition of reliable data. Both practices ensure abundant amounts of either fuel or oxidizer required for combustion are available within the reaction vessel at the time of testing. A method for obtaining an adequate sample size for testing without compromising system or personnel safety was developed and proven successful.

Pre-chilling of the sample material was shown to be unnecessary for the solvents tested; however, this practice is the preferred method as a means of due diligence in obtaining quality data.

The final method for obtaining reliable AIT data when evaluating volatile solvents should follow the steps outlined in Section 7.5 of this report. High pressures should suffice as an evaluation tool due to the more severe conditions. If lower pressures are required, some thought and effort should be directed to ensure adequate oxygen is available for combustion by incrementally increasing the pressure until reactions are observed.

All results are displayed in Table 9. Results highlighted in yellow are the final results for each of the two pressure regimes. In terms of AIT results, Solvokane and L-14780 performed similarly, with Solstice PF outperforming the two at higher pressures (13.8 MPa (2000 psia)). At lower pressures Solstice PF and L-14780 performed similarly, with Solvokane performing better. None of the candidate solvents performed as well as AK-225G.

8.0 HEAT OF COMBUSTION TESTING

Heat of combustion is determined in this test method by burning a weighed sample in an oxygen bomb calorimeter under controlled conditions. The HOC is computed from temperature observations before, during, and after combustion, with proper allowance for thermochemical and heat transfer corrections.

The sample size required was determined by running several tests, varying mass quantities to obtain an increase in temperature of approximately 2.6 °C (4.7 °F). This increase is approximately equal to the temperature increase observed when testing a 1-g capsule of benzoic acid (calibration material). Test samples were not chilled.

8.1 PREPARATION METHOD

Sample preparation proved difficult due to the volatility of the solvents. The use of pressure sensitive tape was employed, as recommended in both ASTM D240 and D4809,[1] with poor results. Test material was not maintained when testing either candidate solvent (Solstice PF or L-14780). As solvent evaporated, the pressure increase broke the tape/test cup seal. Instead, gelatin capsules were used to perform tests. Sample preparation was performed in accordance with ASTM D240 and utilized gelatin capsules to maintain test material prior to performing the test.

---

[1] ASTM. *Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method)*. ASTM D4809. American Society for Testing and Materials, West Conshohocken, PA, 2013.

Tests were run using various mass quantities of gelatin capsules to establish a heat of combustion of the capsules. A predetermined number of empty gelatin capsules, dependent on the solvent, were individually weighed and masses recorded. Identity of each of the capsules was maintained. The number of capsules required for each solvent is shown in Table 11.

Table 11

Solvent Sample Preparation Requirements – Heat of Combustion

Solvent	No. of Capsules	Target Solvent Mass (g)
AK-225G	3	4.0
Solstice PF	2	2.0
L-14780	3	3.2

Gelatin capsules were filled with solvent and the cap of each was then compressed tightly until an audible click was heard; compression of the capsule was continued until the edges of the inner portion of the capsule were visibly deformed. Filled capsules were weighed and the mass of the solvent was determined. Filled capsules were kept on the balance during preparation of the test system. If mass loss of the samples was observed, the discrepant capsule was replaced. The mass of the solvent was recorded just prior to installing the capsule into the test system, as described in ASTM D240.

8.2 STANDARD TEST METHOD – HEAT OF COMBUSTION

Testing is outlined in NASA-STD-6001B *Heat of Combustion (HOC) Testing (ASTM D240)*. No significant deviation from the standard was required to acquire reliable data.

8.3 HEAT OF COMBUSTION TEST RESULTS

Testing was performed between August 4 and 22, 2014. The results are presented in Table 12. Each of the solvents exhibited tightly grouped results. AK-225G resulted in lower HOC than either of the prospective replacement solvents.

A target increase in temperature was ~ 2.6 °C (4.7 °F). Heat of combustion results are recorded per unit mass, and therefore are independent of masses tested. The standard deviation of each of the solvents is indicative of the volatility of the solvent. The standard deviation is inversely proportional to volatility of the solvent. Despite this relationship between standard deviation and volatility, results conform to the repeatability required within D240.

Table 12
Heat of Combustion Test Results

		Mass	Temperature Rise		HOC	Average HOC	SD HOC
Material	Test	(g)	(°C)	(°F)	(cal/g)	(cal/g)	(cal/g)
AK-225G	768	4.00	2.58	4.65	1158.2	1151.7	8.6
	769	3.75	2.45	4.41	1154.3		
	770	3.94	2.54	4.57	1153.9		
	771	3.97	2.52	4.54	1136.7		
	772	3.84	2.45	4.41	1155.5		
Solstice PF	750	2.03	2.51	4.51	2450.6	2447.7	22.4
	751	1.99	2.48	4.46	2475.1		
	754	2.10	2.59	4.67	2461.6		
	756	2.08	2.52	4.54	2431.5		
	757	2.06	2.52	4.53	2419.5		
L-14780	728	3.73	3.11	5.60	1926.6	1925.3	19.8
	729	3.22	2.68	4.82	1910.9		
	730	3.14	2.63	4.74	1905.8		
	734	3.17	2.66	4.79	1926.3		
	763	2.28	2.27	4.09	1956.7		

SD – Standard Deviation

8.4 HEAT OF COMBUSTION TEST CONCLUSIONS

Modifications to the test method were not required to obtain repeatable HOC test results. Care was required when preparing test samples due to the volatility of the test material. Methods for preparing samples as described in ASTM D240 were adequate to maintain material during testing. Mass measurements were taken immediately before testing to obtain as accurate a measurement as possible. The grouping of test results for each of the test materials adhered to statistical spread described in the standard.

Both of the final candidate solvents exhibited higher heats of combustion than AK-225G, indicating more potential energy when compared to AK-225G.

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

9.0 OXYGEN COMPATABILITY ANALYSIS APPROACH

Fires occur in oxygen systems when oxygen, fuel, and heat combine to create a self-sustaining chemical reaction Although oxygen, fuel, and ignition sources are present in almost all oxygen systems, fire hazards can be mitigated by limiting the propensity for a chemical reaction to occur. Controlling the risk factors associated with the oxygen, fuel, or heat will, in turn, prevent the chemical reaction.

9.1 HISTORICAL APPROACH

As a result of the 1987 Montreal Protocol to phase out CFC-113, Asahi AK-225G was selected as a replacement. AK-225G had been a preferred solvent with regard to oxygen compatibility for its perceived non-reactivity in the tests used to evaluate potential solvents.

Historically, solvents were found acceptable (non-ignitable) according to the tests used to assess the oxygen compatibility, mechanical impact (D2512/G86), and AIT (G72). In the past this approach was effective, as some solvents such as AK-225G showed non-ignitions during tests. Differentiation between solvents was made simple due to the clear distinction between reactive solvents and non-reactive solvents such as AK-225G.

In this oxygen compatibility analysis (OCA), LOX mechanical impact testing acceptance criteria during the last round of solvent evaluations gave preference to solvents exhibiting no reactions at 97.6 J (72 ft-lbf). Any solvent passing above 27 J (20 ft-lbf) or showing anomalous results was also considered for further testing or evaluation.

The second portion of the OCA involved AIT testing. Solvents exhibiting non-ignitions during standard test configurations were given preference; however, the following categories had been laid out for evaluation of solvents that resulted in non-ignitions during testing:

Category A	AIT < 121 °C (< 250 °F)	Not recommended for use in oxygen systems
Category B	AIT 121 to 204 °C (250 to 400 °F)	Caution when used in oxygen systems
Category C	AIT > 204 °C (> 400 °F)	Recommended for use in oxygen systems

During this round of assessment all candidate solvents, as well as past proven solvents, were found to be flammable in oxygen enriched environments. A clear, distinct differentiation between acceptable and non-acceptable solvents was impossible to make; therefore, a more rigorous approach was needed.

9.2 OCA/NASA STD-6001.B/G63 & G94 APPROACH

As for analysis of oxygen systems, the NASA STD-6001.B/ASTM G63 and ASTM G94 approach was used as a basis for evaluating the candidate solvents Solstice PF and L-14780. This process is a systematic approach that can be used as both a design guide and an approval process for materials components and systems. NASA STD-6001.B Section 6.3.1 states that materials, components, and systems used in LOX and GOX environments shall be evaluated using the approach shown in Figure 9.

Performing the tests required by NASA STD-6001.B was not possible due to the physical properties of the solvents, and Tests 1 and 17 are meant for the evaluation of solid materials. The purpose of the required tests is to establish the flammability of the material and to ascertain the conditions at which the candidate materials are flammable. All candidate solvents were determined to be flammable during initial testing. NASA STD-6001.B 6.3.2 gives further guidance per the following: “If the materials are determined to be flammable, an OCA shall be performed in accordance to TM-2007-213740, Guide for Oxygen Compatibility Assessments and Components and Systems.”

NASA/TM-2007-213740 uses a system level approach for the determination of compatibility in LOX and GOX environments (Figure 9). The solvents will be used in a wide variety of systems and this type of assessment would be impossible to capture every possible scenario. The essence of NASA/TM-2007-213740 was used to develop the following logic:

1. Is the material flammable?
2. Will it ignite?
3. What is the damage potential?
4. How does this compare to reference materials that have been successfully used for historically effective guidelines?

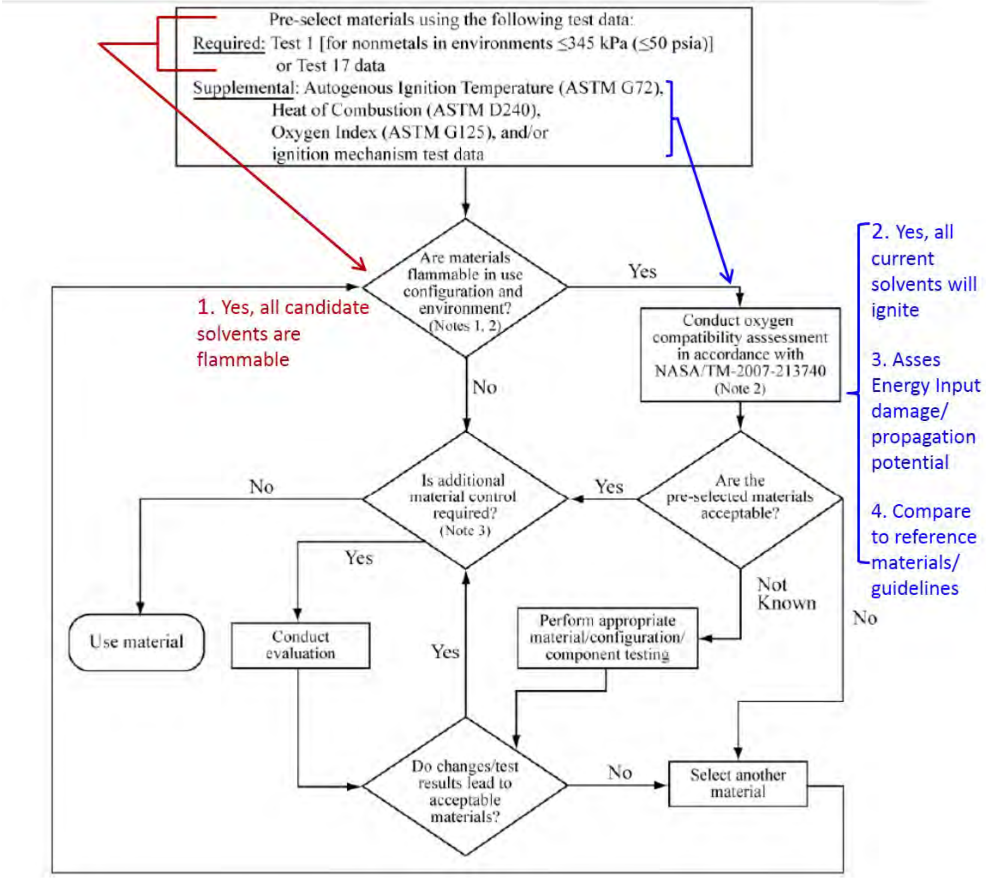


Figure 9
NASA-STD-6001.B Oxygen Compatibility Assessment Decision Tree

9.2.1 FLAMMABILITY/ IGNITABILITY

NASA Standard Tests 13A and AIT tests established that the candidate solvents are flammable and will ignite under certain circumstances.

Reactions were observed during mechanical impact testing. Variables were shown to affect the reaction frequency; however, despite the final test method, reactions were observed when samples were impacted. While evaluating the candidate materials using the AIT test method, reactions were observed only after provisions were made to ensure that an adequate amount of test material was present during heating. Despite the deviation from the conventional standard, ignition of both candidate materials was observed and characterized, establishing the potential ignition through external heating (Table 13).

Table 13

Solvent Assessment and Comparison Materials

	GOX Impact Pressure Threshold		LOX Impact Energy Thresh Modified Method Standard Method		LOX Impact Results @ 97.6 J (72 ft-lbf)	AIT @ 345-1379 kPa (50-200 psi) Modified Method Standard Method		AIT @ 13.79 MPa (2000 psi) Modified Method Standard Method @ 1500psi		HOC
Solvent	MPa	psi	J	ft-lbf		°C (SD)	°F (SD)	°C (SD)	°F (SD)	cal/g (SD)
FlrGr		>4415[b]				N/P		479[a]	248[a]	2400[a]
PTFE		>7415[c]			(1/60)	N/P		434[a]	223[a]	1700[a]
PCTFE		6615[d]				N/P		377[a]	192[a]	2557[a]
IPA						N/P				7165[a]
Solstice PF	52	7500	97.6 (0/20)(1/61)	72	N/P	464 (29) @ 345 kPa (50 psi) 477 (13) @ 480 kPa (70 psi)	240 (16) 247 (7)	360 (9)	182 (5)	2448 (22)
L-14780	52	7500	97.6 (0/20)(0/70)	72	0/20	454* @ 1.38 MPa (200 psi)	234*	322 (27)	161 (15)	1925 (20)
AK-225G			97.6 (0/20)	72	9-0/20 1-2/43 1-6/60 1-3/60 2-0/60	528* @ 480 kPa (70 psi)	276*	446	230	1153 (11)

[a] ASTM Manual 36, 2nd edition

[b] WSTF No. 05-39250, FlrGr E600

[c] WSTF No. 01-56989, PTFE

[d] WSTF No. 12-45752, PCTFE P/N 16041-1000, no reactions @ 100% GOX and 20ft-lbf

*single data point

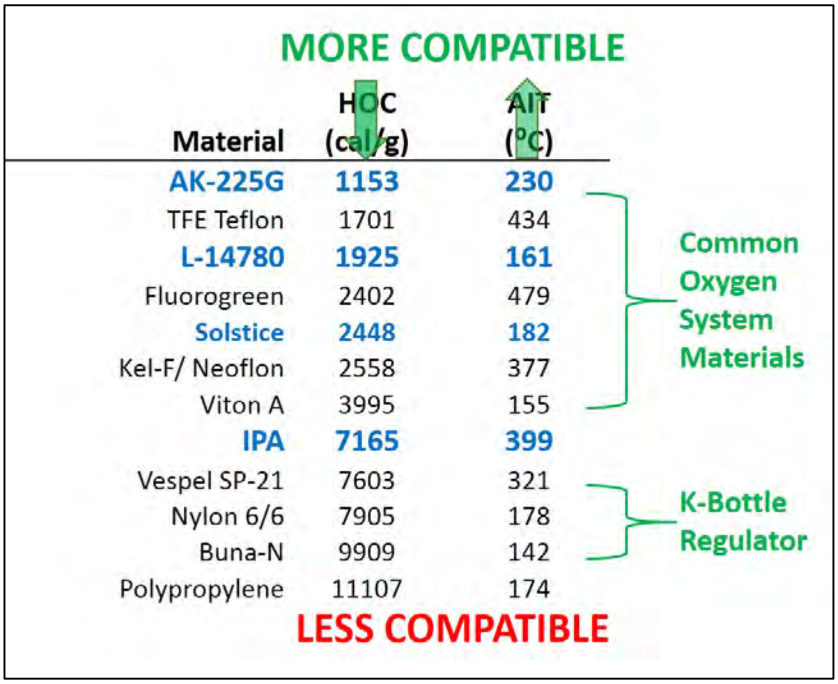
9.2.2 DAMAGE POTENTIAL

A quantitative comparison of the damage potential of each of the candidate solvents to AK-225G was achieved by performing HOC testing (ASTM D240). The average HOC and standard deviations (SDs) are presented in Table 13. Heat of combustion data are presented as the energy output per unit mass. Solvents are anticipated to reside within a system’s crevices in low mass quantities, making the damage potential small relative to most soft goods that have a long track record of safe use in oxygen systems.

9.2.3 COMPARISON TO REFERENCE MATERIALS

Results from HOC testing convey the damage potential per unit mass of any material. For illustrative purposes, the candidate solvents, AK-225G, and ‘good compatible’ materials have been placed in Figure 10 for direct comparison. Figure 10 contains values for HOC and AIT as a means of assessing damage potential and ignitability simultaneously. Larger values for AIT and smaller values for HOC are preferred.

Note: AIT data shown in Figure 10 for isopropyl alcohol (IPA) were obtained using ASTM E659, not ASTM G72. Tests in adherence to ASTM G72 are performed in 100 percent oxygen at 10.3 MPa (1500 psia), while tests in adherence to ASTM E659 are performed in ambient conditions (20.9 percent oxygen and 101.4 kPa (14.7 psia)). Isopropyl alcohol AIT test results performed in adherence to ASTM G72 generally tend to be much lower. Therefore, no direct comparison can be made between the IPA data presented in Figure 10 and the rest of the results presented.



MORE COMPATIBLE

Material	HOC (cal/g)	AIT (°C)
AK-225G	**1153**	**230**
TFE Teflon	1701	434
L-14780	**1925**	**161**
Fluorogreen	2402	479
Solstice	**2448**	**182**
Kel-F/ Neoflon	2558	377
Viton A	3995	155
IPA	**7165**	**399**
Vespel SP-21	7603	321
Nylon 6/6	7905	178
Buna-N	9909	142
Polypropylene	11107	174

Common Oxygen System Materials

K-Bottle Regulator

LESS COMPATIBLE

Figure 10
HOC and AIT for Common Oxygen System Materials

No direct correlation between energy potential and ignition susceptibility can be inferred from Figure 10, and testing should be conducted to verify unknown material properties. Both L-14780 and Solstice PF were poorer performers relative to AK-225G and ranked well compared to preferred soft good materials such as Teflon®[1] and Fluorogreen. Poorer performance compared to AK-225G coincides with both AIT and mechanical impact testing. Both candidate solvents are comparable to each other and are much better performers (lower HOC) than IPA. Isopropyl alcohol is considered a good solvent; however, post rinse procedures are required after use.

Procedural controls have been used to mitigate the oxygen compatibility hazards associated with IPA. The successful use of IPA as a solvent in oxygen systems, despite its high HOC relative to the two candidate solvents, is proof of the significance of thoughtful use of materials as means of risk management as opposed to the pursuit of a non-flammable alternative. Proper evaluation of materials and consideration before use should be required to prevent a false security of use provided by incomplete data.

---

[1] Teflon® is a registered trademark of E. I. Du Pont de Nemours and Company, Wilmington, DE.

Another example of energetic and ignitable materials being used with wide success in oxygen systems is Nylon 6/6. Nylon 6/6 is relatively energetic, readily burns in air and is commonly used in oxygen regulators. The reason for its success is its end use configuration. Despite its flammability characteristics, it is not flammable in its configuration due to the lack of ignition hazards. Both candidate solvents are less energetic per unit mass than Nylon 6/6.

9.3 OXYGEN COMPATABILITY ANALYSIS CONCLUSION

Materials can be used safely and successfully when proper attention is given to how and when they are used. The Solstice PF and L-14780 solvents were found to be flammable in enriched oxygen environments, and plausible ignition mechanisms have been demonstrated; however, that does not preclude the safe use of these solvents in oxygen systems.

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 to soft goods.

10.0 SUMMARY OF SOLVENT INVESTIGATION

Historic methods for assessing the compatibility of solvents for use in oxygen systems utilized test methods for evaluating ignition susceptibility. While both AIT and mechanical impact testing have a long track record of use within NASA, these test methods alone do not fully characterize the safe use of solvent materials within oxygen systems. A more systematic approach, which takes into consideration the damage potential of such materials (i.e., heat of combustion data), was required in conjunction with ignition susceptibility tests to perform a more complete evaluation.

Favorable results in both AIT and mechanical impact testing indicated inadequate test conditions rather than superior materials. The volatility of the materials as well as the interaction with environmental variables presented a challenge to the evaluation of the materials. Care should be exercised when testing these types of materials to ensure proper test conditions as outlined in this report. All materials will react in the right conditions. To perform a proper assessment of materials, effort should be focused on achieving reactions in tests such as AIT to gain an understanding of the reactivity of materials relative to one another.

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

The methods described in this report outline the logic used to assess and compare the solvent materials for safe use in oxygen systems. All materials were shown to be flammable in oxygen enriched environments and therefore pose some risk when being used in oxygen systems. No material is completely benign, and caution should be exercised when using these materials for cleaning. With proper checks and controls these materials, in the configurations tested, can be used safely in oxygen systems.

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

Prepared by: 2/20/15
Alfredo Juarez
NASA Test and Evaluation Contract

Reviewed by: 2-20-15
Jerry Ledesma
NASA Test and Evaluation Contract

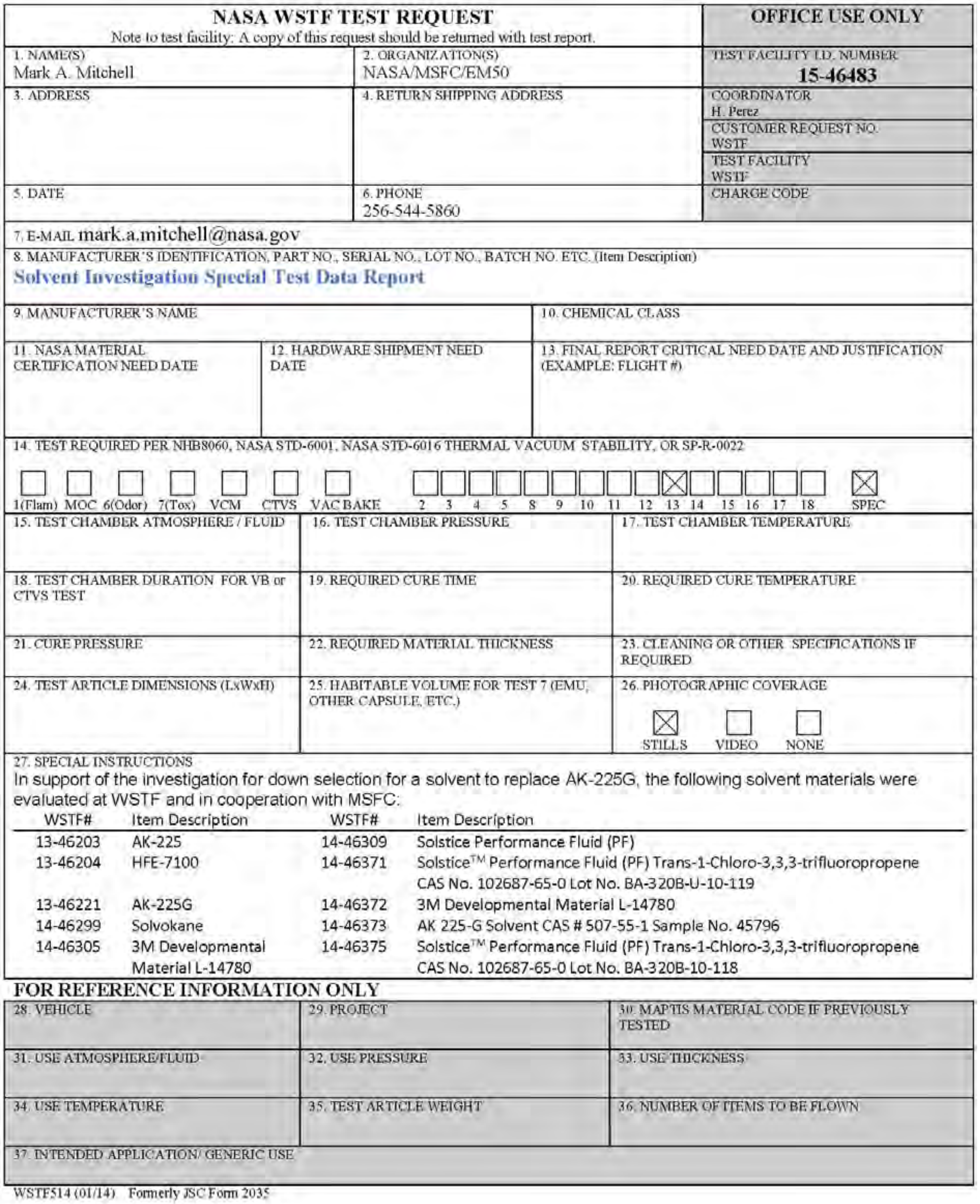
Approved by: 2/23/15
Stephen Peralta for Susana A. Harper
NASA Materials and Components Laboratories Office

NASA WHITE SANDS TEST FACILITY

SOLVENT INVESTIGATION
SPECIAL TEST DATA REPORT

WSTF # 15-46483
February 20, 2015

APPENDIX A



NASA WSTF TEST REQUEST

Note to test facility: A copy of this request should be returned with test report.

OFFICE USE ONLY

TEST FACILITY I.D. NUMBER **15-46483**

COORDINATOR H. Perez

CUSTOMER REQUEST NO. WSTF

TEST FACILITY WSTF

CHARGE CODE

1. NAME(S) Mark A. Mitchell

2. ORGANIZATION(S) NASA/MSFC/EM50

3. ADDRESS

4. RETURN SHIPPING ADDRESS

5. DATE

6. PHONE 256-544-5860

7. E-MAIL mark.a.mitchell@nasa.gov

8. MANUFACTURER'S IDENTIFICATION, PART NO., SERIAL NO., LOT NO., BATCH NO. ETC. (Item Description)
Solvent Investigation Special Test Data Report

9. MANUFACTURER'S NAME

10. CHEMICAL CLASS

11. NASA MATERIAL CERTIFICATION NEED DATE

12. HARDWARE SHIPMENT NEED DATE

13. FINAL REPORT CRITICAL NEED DATE AND JUSTIFICATION (EXAMPLE: FLIGHT #)

14. TEST REQUIRED PER NHB8060, NASA STD-6001, NASA STD-6016 THERMAL VACUUM STABILITY, OR SP-R-0022

☐ 1(Flam) ☐ MOC ☐ 6(Odor) ☐ 7(Tox) ☐ VCM ☐ CTVS ☐ VAC BAKE ☐ 2 ☐ 3 ☐ 4 ☐ 5 ☐ 8 ☐ 9 ☐ 10 ☐ 11 ☐ 12 ☒ 13 ☐ 14 ☐ 15 ☐ 16 ☐ 17 ☐ 18 ☒ SPEC

15. TEST CHAMBER ATMOSPHERE / FLUID

16. TEST CHAMBER PRESSURE

17. TEST CHAMBER TEMPERATURE

18. TEST CHAMBER DURATION FOR VB or CTVS TEST

19. REQUIRED CURE TIME

20. REQUIRED CURE TEMPERATURE

21. CURE PRESSURE

22. REQUIRED MATERIAL THICKNESS

23. CLEANING OR OTHER SPECIFICATIONS IF REQUIRED

24. TEST ARTICLE DIMENSIONS (LxWxH)

25. HABITABLE VOLUME FOR TEST 7 (EMU, OTHER CAPSULE, ETC.)

26. PHOTOGRAPHIC COVERAGE

☒ STILLS ☐ VIDEO ☐ NONE

27. SPECIAL INSTRUCTIONS

In support of the investigation for down selection for a solvent to replace AK-225G, the following solvent materials were evaluated at WSTF and in cooperation with MSFC:

WSTF#	Item Description	WSTF#	Item Description
13-46203	AK-225	14-46309	Solstice Performance Fluid (PF)
13-46204	HFE-7100	14-46371	Solstice^{TM} Performance Fluid (PF) Trans-1-Chloro-3,3,3-trifluoropropene CAS No. 102687-65-0 Lot No. BA-320B-U-10-119
13-46221	AK-225G	14-46372	3M Developmental Material L-14780
14-46299	Solvokane	14-46373	AK 225-G Solvent CAS # 507-55-1 Sample No. 45796
14-46305	3M Developmental Material L-14780	14-46375	Solstice^{TM} Performance Fluid (PF) Trans-1-Chloro-3,3,3-trifluoropropene CAS No. 102687-65-0 Lot No. BA-320B-10-118

FOR REFERENCE INFORMATION ONLY

28. VEHICLE

29. PROJECT

30. MAPTIS MATERIAL CODE IF PREVIOUSLY TESTED

31. USE ATMOSPHERE/FLUID

32. USE PRESSURE

33. USE THICKNESS

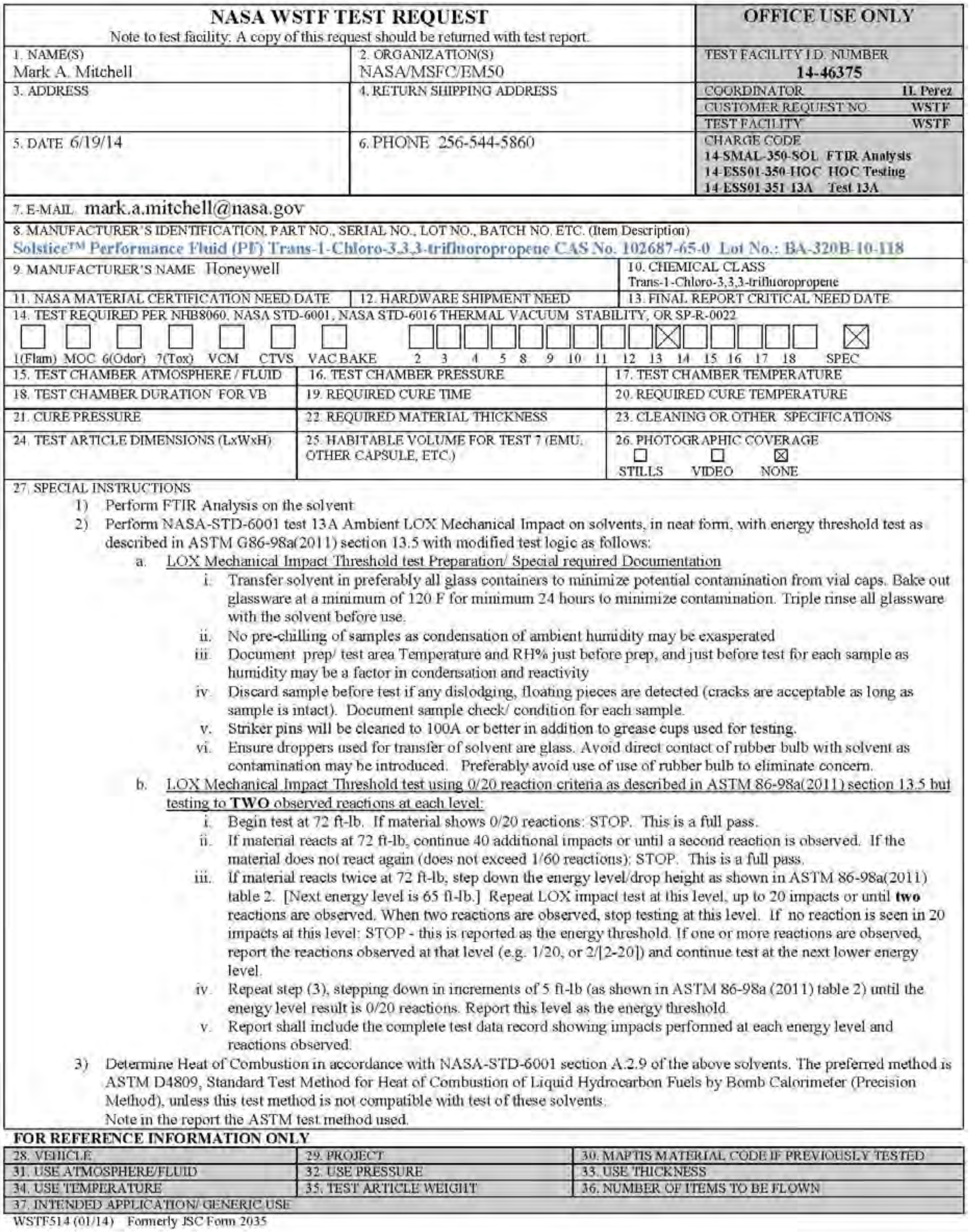
34. USE TEMPERATURE

35. TEST ARTICLE WEIGHT

36. NUMBER OF ITEMS TO BE FLOWN

37. INTENDED APPLICATION/ GENERIC USE

WSTF514 (01/14) Formerly JSC Form 2035



NASA WSTF TEST REQUEST

Note to test facility: A copy of this request should be returned with test report.

OFFICE USE ONLY

TEST FACILITY I.D. NUMBER **14-46375**
COORDINATOR **H. Perez**
CUSTOMER REQUEST NO. **WSTF**
TEST FACILITY **WSTF**
CHARGE CODE
14-SMAL-350-SOL FTIR Analysis
14-ESS01-350-HOC HOC Testing
14-ESS01-351-13A Test 13A

1. NAME(S) Mark A. Mitchell
2. ORGANIZATION(S) NASA/MSFC/EM50
3. ADDRESS
4. RETURN SHIPPING ADDRESS
5. DATE 6/19/14
6. PHONE 256-544-5860
7. E-MAIL mark.a.mitchell@nasa.gov
8. MANUFACTURER'S IDENTIFICATION, PART NO., SERIAL NO., LOT NO., BATCH NO. ETC. (Item Description)
Solstice™ Performance Fluid (PF) Trans-1-Chloro-3,3,3-trifluoropropene CAS No. 102687-65-0 Lot No.: BA-320B-10-118
9. MANUFACTURER'S NAME Honeywell
10. CHEMICAL CLASS Trans-1-Chloro-3,3,3-trifluoropropene
11. NASA MATERIAL CERTIFICATION NEED DATE
12. HARDWARE SHIPMENT NEED
13. FINAL REPORT CRITICAL NEED DATE
14. TEST REQUIRED PER NHB8060, NASA STD-6001, NASA STD-6016 THERMAL VACUUM STABILITY, OR SP-R-0022
☐ 1(Flam) ☐ MOC ☐ 6(Odor) ☐ 7(Tox) ☐ VCM ☐ CTVS ☐ VAC BAKE ☐ 2 ☐ 3 ☐ 4 ☐ 5 ☐ 8 ☐ 9 ☐ 10 ☐ 11 ☐ 12 ☒ 13 ☐ 14 ☐ 15 ☐ 16 ☐ 17 ☐ 18 ☒ SPEC
15. TEST CHAMBER ATMOSPHERE / FLUID
16. TEST CHAMBER PRESSURE
17. TEST CHAMBER TEMPERATURE
18. TEST CHAMBER DURATION FOR VB
19. REQUIRED CURE TIME
20. REQUIRED CURE TEMPERATURE
21. CURE PRESSURE
22. REQUIRED MATERIAL THICKNESS
23. CLEANING OR OTHER SPECIFICATIONS
24. TEST ARTICLE DIMENSIONS (LxWxH)
25. HABITABLE VOLUME FOR TEST 7 (EMU, OTHER CAPSULE, ETC.)
26. PHOTOGRAPHIC COVERAGE ☐ STILLS ☐ VIDEO ☒ NONE
27. SPECIAL INSTRUCTIONS

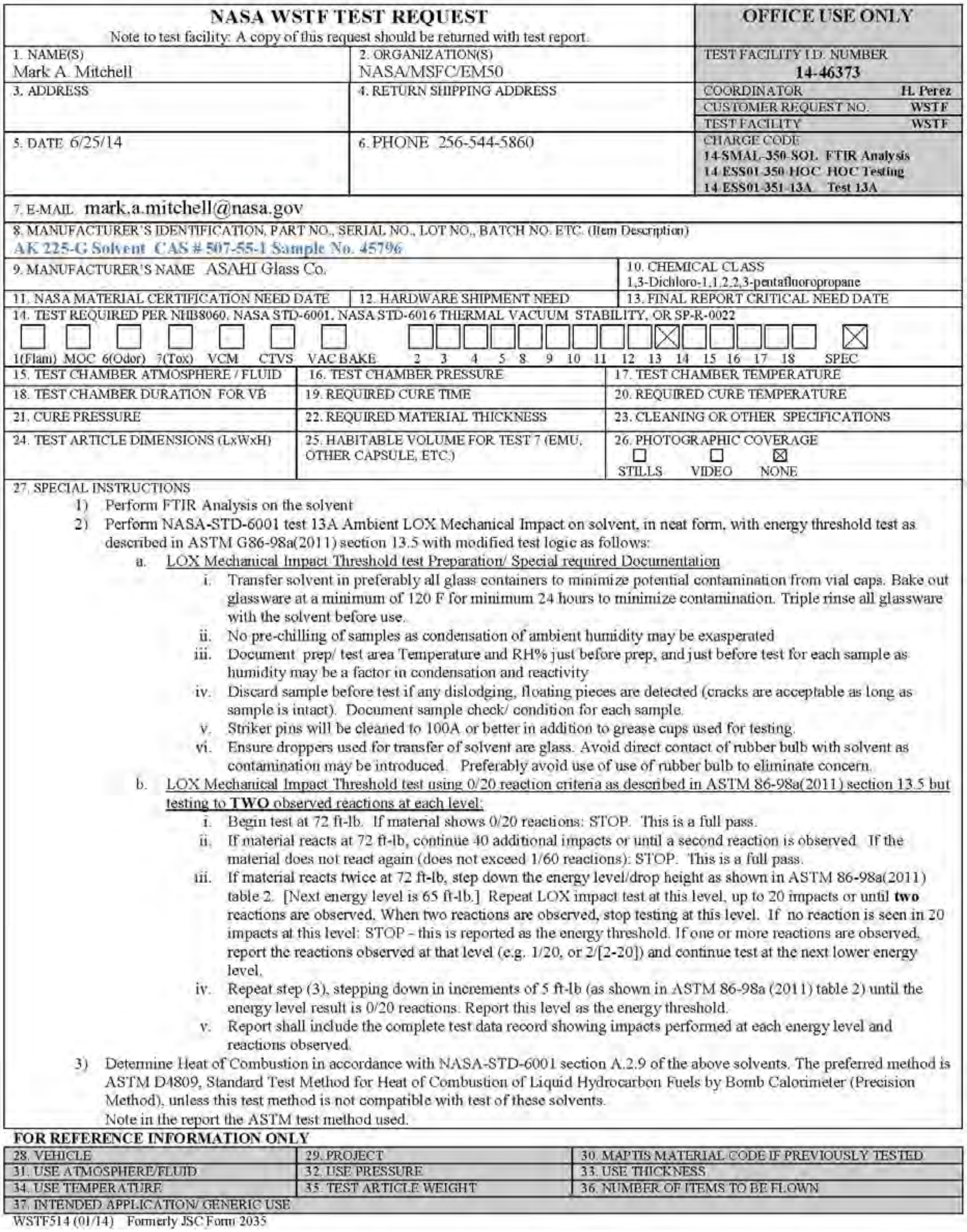
1) Perform FTIR Analysis on the solvent
2) Perform NASA-STD-6001 test 13A Ambient LOX Mechanical Impact on solvents, in neat form, with energy threshold test as described in ASTM G86-98a(2011) section 13.5 with modified test logic as follows:
    a. LOX Mechanical Impact Threshold test Preparation/ Special required Documentation
        i. Transfer solvent in preferably all glass containers to minimize potential contamination from vial caps. Bake out glassware at a minimum of 120 F for minimum 24 hours to minimize contamination. Triple rinse all glassware with the solvent before use.
        ii. No pre-chilling of samples as condensation of ambient humidity may be exasperated
        iii. Document prep/ test area Temperature and RH% just before prep, and just before test for each sample as humidity may be a factor in condensation and reactivity
        iv. Discard sample before test if any dislodging, floating pieces are detected (cracks are acceptable as long as sample is intact). Document sample check/ condition for each sample.
        v. Striker pins will be cleaned to 100A or better in addition to grease cups used for testing.
        vi. Ensure droppers used for transfer of solvent are glass. Avoid direct contact of rubber bulb with solvent as contamination may be introduced. Preferably avoid use of use of rubber bulb to eliminate concern.
    b. LOX Mechanical Impact Threshold test using 0/20 reaction criteria as described in ASTM 86-98a(2011) section 13.5 but testing to **TWO** observed reactions at each level:
        i. Begin test at 72 ft-lb. If material shows 0/20 reactions: STOP. This is a full pass.
        ii. If material reacts at 72 ft-lb, continue 40 additional impacts or until a second reaction is observed. If the material does not react again (does not exceed 1/60 reactions); STOP. This is a full pass.
        iii. If material reacts twice at 72 ft-lb, step down the energy level/drop height as shown in ASTM 86-98a(2011) table 2. [Next energy level is 65 ft-lb.] Repeat LOX impact test at this level, up to 20 impacts or until **two** reactions are observed. When two reactions are observed, stop testing at this level. If no reaction is seen in 20 impacts at this level: STOP - this is reported as the energy threshold. If one or more reactions are observed, report the reactions observed at that level (e.g. 1/20, or 2/[2-20]) and continue test at the next lower energy level.
        iv. Repeat step (3), stepping down in increments of 5 ft-lb (as shown in ASTM 86-98a (2011) table 2) until the energy level result is 0/20 reactions. Report this level as the energy threshold.
        v. Report shall include the complete test data record showing impacts performed at each energy level and reactions observed.
3) Determine Heat of Combustion in accordance with NASA-STD-6001 section A.2.9 of the above solvents. The preferred method is ASTM D4809, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method), unless this test method is not compatible with test of these solvents.
Note in the report the ASTM test method used.

FOR REFERENCE INFORMATION ONLY

28. VEHICLE
29. PROJECT
30. MAPTIS MATERIAL CODE IF PREVIOUSLY TESTED
31. USE ATMOSPHERE/FLUID
32. USE PRESSURE
33. USE THICKNESS
34. USE TEMPERATURE
35. TEST ARTICLE WEIGHT
36. NUMBER OF ITEMS TO BE FLOWN
37. INTENDED APPLICATION/ GENERIC USE

WSTF514 (01/14) Formerly JSC Form 2035

S



NASA WSTF TEST REQUEST

Note to test facility: A copy of this request should be returned with test report.

OFFICE USE ONLY

1. NAME(S) Mark A. Mitchell
2. ORGANIZATION(S) NASA/MSFC/EM50
3. ADDRESS
4. RETURN SHIPPING ADDRESS
5. DATE 6/25/14
6. PHONE 256-544-5860

TEST FACILITY I.D. NUMBER	**14-46373**
COORDINATOR	**H. Perez**
CUSTOMER REQUEST NO.	**WSTF**
TEST FACILITY	**WSTF**
CHARGE CODE	**14-SMAL-350-SOL FTIR Analysis** **14-ESS01-350-HOC HOC Testing** **14-ESS01-351-13A Test 13A**

7. E-MAIL mark.a.mitchell@nasa.gov
8. MANUFACTURER'S IDENTIFICATION, PART NO., SERIAL NO., LOT NO., BATCH NO. ETC. (Item Description)
AK 225-G Solvent CAS # 507-55-1 Sample No. 45796
9. MANUFACTURER'S NAME ASAHI Glass Co.
10. CHEMICAL CLASS 1,3-Dichloro-1,1,2,2,3-pentafluoropropane
11. NASA MATERIAL CERTIFICATION NEED DATE
12. HARDWARE SHIPMENT NEED
13. FINAL REPORT CRITICAL NEED DATE
14. TEST REQUIRED PER NHB8060, NASA STD-6001, NASA STD-6016 THERMAL VACUUM STABILITY, OR SP-R-0022

☐ 1(Flam) ☐ MOC ☐ 6(Odor) ☐ 7(Tox) ☐ VCM ☐ CTVS ☐ VAC BAKE ☐ 2 ☐ 3 ☐ 4 ☐ 5 ☐ 8 ☐ 9 ☐ 10 ☐ 11 ☐ 12 ☒ 13 ☐ 14 ☐ 15 ☐ 16 ☐ 17 ☐ 18 ☒ SPEC

15. TEST CHAMBER ATMOSPHERE / FLUID
16. TEST CHAMBER PRESSURE
17. TEST CHAMBER TEMPERATURE
18. TEST CHAMBER DURATION FOR VB
19. REQUIRED CURE TIME
20. REQUIRED CURE TEMPERATURE
21. CURE PRESSURE
22. REQUIRED MATERIAL THICKNESS
23. CLEANING OR OTHER SPECIFICATIONS
24. TEST ARTICLE DIMENSIONS (LxWxH)
25. HABITABLE VOLUME FOR TEST 7 (EMU, OTHER CAPSULE, ETC.)
26. PHOTOGRAPHIC COVERAGE ☐ STILLS ☐ VIDEO ☒ NONE

27. SPECIAL INSTRUCTIONS

1) Perform FTIR Analysis on the solvent
2) Perform NASA-STD-6001 test 13A Ambient LOX Mechanical Impact on solvent, in neat form, with energy threshold test as described in ASTM G86-98a(2011) section 13.5 with modified test logic as follows:
    a. LOX Mechanical Impact Threshold test Preparation/ Special required Documentation
        i. Transfer solvent in preferably all glass containers to minimize potential contamination from vial caps. Bake out glassware at a minimum of 120 F for minimum 24 hours to minimize contamination. Triple rinse all glassware with the solvent before use.
        ii. No pre-chilling of samples as condensation of ambient humidity may be exasperated
        iii. Document prep/ test area Temperature and RH% just before prep, and just before test for each sample as humidity may be a factor in condensation and reactivity
        iv. Discard sample before test if any dislodging, floating pieces are detected (cracks are acceptable as long as sample is intact). Document sample check/ condition for each sample.
        v. Striker pins will be cleaned to 100A or better in addition to grease cups used for testing.
        vi. Ensure droppers used for transfer of solvent are glass. Avoid direct contact of rubber bulb with solvent as contamination may be introduced. Preferably avoid use of use of rubber bulb to eliminate concern.
    b. LOX Mechanical Impact Threshold test using 0/20 reaction criteria as described in ASTM 86-98a(2011) section 13.5 but testing to **TWO** observed reactions at each level:
        i. Begin test at 72 ft-lb. If material shows 0/20 reactions: STOP. This is a full pass.
        ii. If material reacts at 72 ft-lb, continue 40 additional impacts or until a second reaction is observed. If the material does not react again (does not exceed 1/60 reactions): STOP. This is a full pass.
        iii. If material reacts twice at 72 ft-lb, step down the energy level/drop height as shown in ASTM 86-98a(2011) table 2. [Next energy level is 65 ft-lb.] Repeat LOX impact test at this level, up to 20 impacts or until **two** reactions are observed. When two reactions are observed, stop testing at this level. If no reaction is seen in 20 impacts at this level: STOP - this is reported as the energy threshold. If one or more reactions are observed, report the reactions observed at that level (e.g. 1/20, or 2/[2-20]) and continue test at the next lower energy level.
        iv. Repeat step (3), stepping down in increments of 5 ft-lb (as shown in ASTM 86-98a (2011) table 2) until the energy level result is 0/20 reactions. Report this level as the energy threshold.
        v. Report shall include the complete test data record showing impacts performed at each energy level and reactions observed.
3) Determine Heat of Combustion in accordance with NASA-STD-6001 section A.2.9 of the above solvents. The preferred method is ASTM D4809, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method), unless this test method is not compatible with test of these solvents.
Note in the report the ASTM test method used.

FOR REFERENCE INFORMATION ONLY

28. VEHICLE
29. PROJECT
30. MAPTIS MATERIAL CODE IF PREVIOUSLY TESTED
31. USE ATMOSPHERE/FLUID
32. USE PRESSURE
33. USE THICKNESS
34. USE TEMPERATURE
35. TEST ARTICLE WEIGHT
36. NUMBER OF ITEMS TO BE FLOWN
37. INTENDED APPLICATION/ GENERIC USE

WSTF514 (01/14) Formerly JSC Form 2035

NASA WSTF TEST REQUEST

Note to test facility: A copy of this request should be returned with test report.

OFFICE USE ONLY

TEST FACILITY I.D. NUMBER **14-46372**
COORDINATOR **H. Perez**
CUSTOMER REQUEST NO. **WSTF**
TEST FACILITY **WSTF**
CHARGE CODE
14-SMAL-350-SOL FTIR Analysis
14-ESS01-350-HOC HOC Testing
14-ESS01-351-13A Test 13A

1. NAME(S) Mark A. Mitchell
2. ORGANIZATION(S) NASA/MSFC/EM50
3. ADDRESS
4. RETURN SHIPPING ADDRESS
5. DATE 6/19/14
6. PHONE 256-544-5860
7. E-MAIL mark.a.mitchell@nasa.gov
8. MANUFACTURER'S IDENTIFICATION, PART NO., SERIAL NO., LOT NO., BATCH NO. ETC. (Item Description)
3M Developmental Material L-14780
9. MANUFACTURER'S NAME 3M
10. CHEMICAL CLASS
11. NASA MATERIAL CERTIFICATION NEED DATE
12. HARDWARE SHIPMENT NEED
13. FINAL REPORT CRITICAL NEED DATE
14. TEST REQUIRED PER NHB8060, NASA STD-6001, NASA STD-6016 THERMAL VACUUM STABILITY, OR SP-R-0022

☐ 1(Flam) ☐ MOC ☐ 6(Odor) ☐ 7(Tox) ☐ VCM ☐ CTVS ☐ VAC BAKE ☐ 2 ☐ 3 ☐ 4 ☐ 5 ☐ 8 ☐ 9 ☐ 10 ☐ 11 ☐ 12 ☒ 13 ☐ 14 ☐ 15 ☐ 16 ☐ 17 ☐ 18 ☒ SPEC

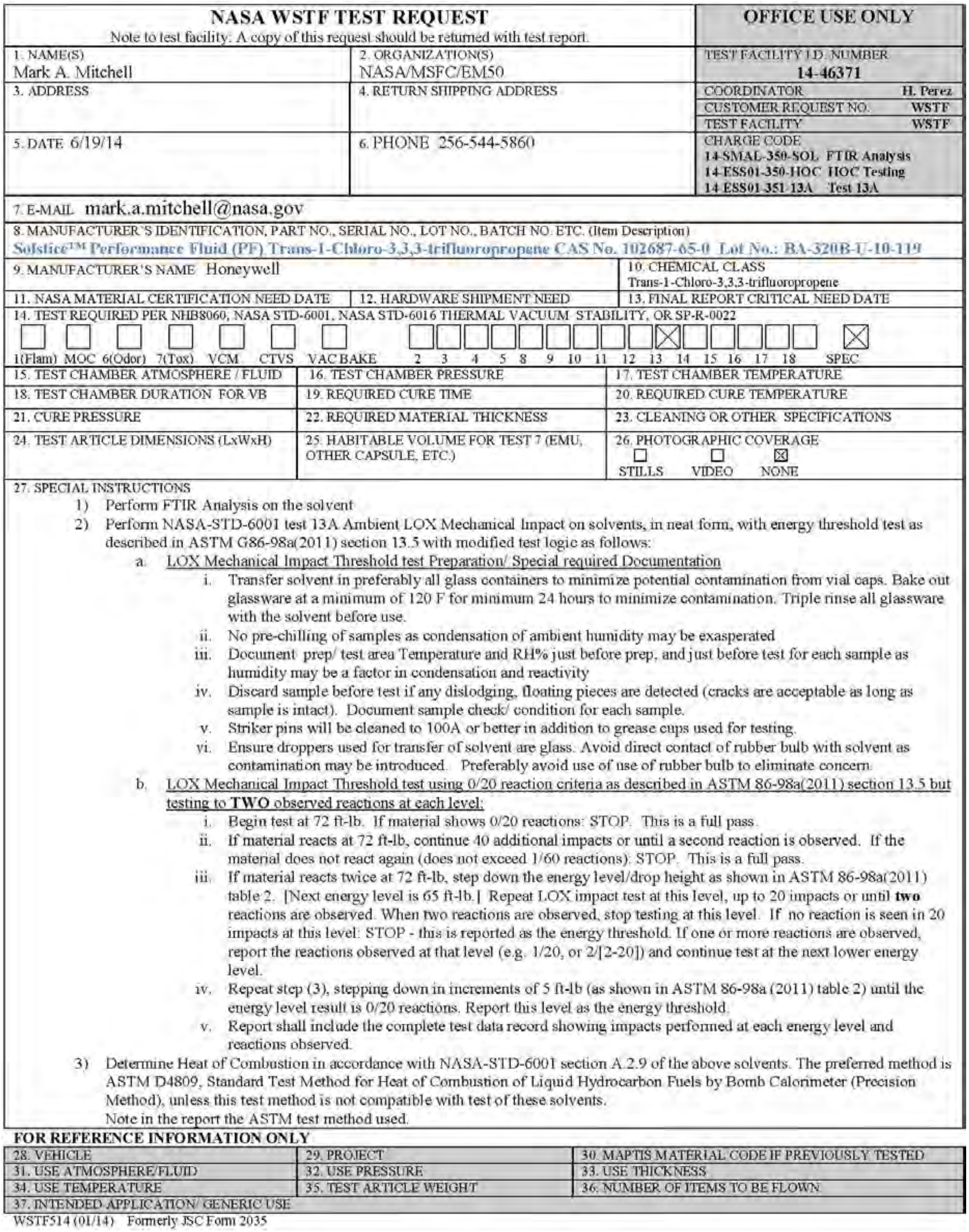
15. TEST CHAMBER ATMOSPHERE / FLUID
16. TEST CHAMBER PRESSURE
17. TEST CHAMBER TEMPERATURE
18. TEST CHAMBER DURATION FOR VB
19. REQUIRED CURE TIME
20. REQUIRED CURE TEMPERATURE
21. CURE PRESSURE
22. REQUIRED MATERIAL THICKNESS
23. CLEANING OR OTHER SPECIFICATIONS
24. TEST ARTICLE DIMENSIONS (LxWxH)
25. HABITABLE VOLUME FOR TEST 7 (EMU, OTHER CAPSULE, ETC.)
26. PHOTOGRAPHIC COVERAGE ☐ STILLS ☐ VIDEO ☒ NONE
27. SPECIAL INSTRUCTIONS

1) Perform FTIR Analysis on the solvent
2) Perform NASA-STD-6001 test 13A Ambient LOX Mechanical Impact on solvent, in neat form, with energy threshold test as described in ASTM G86-98a(2011) section 13.5 with modified test logic as follows:
    a. <u>LOX Mechanical Impact Threshold test Preparation/ Special required Documentation</u>
        i. Transfer solvent in preferably all glass containers to minimize potential contamination from vial caps. Bake out glassware at a minimum of 120 F for minimum 24 hours to minimize contamination. Triple rinse all glassware with the solvent before use.
        ii. No pre-chilling of samples as condensation of ambient humidity may be exasperated
        iii. Document prep/ test area Temperature and RH% just before prep, and just before test for each sample as humidity may be a factor in condensation and reactivity
        iv. Discard sample before test if any dislodging, floating pieces are detected (cracks are acceptable as long as sample is intact). Document sample check/ condition for each sample.
        v. Striker pins will be cleaned to 100A or better in addition to grease cups used for testing.
        vi. Ensure droppers used for transfer of solvent are glass. Avoid direct contact of rubber bulb with solvent as contamination may be introduced. Preferably avoid use of use of rubber bulb to eliminate concern.
    b. <u>LOX Mechanical Impact Threshold test using 0/20 reaction criteria as described in ASTM 86-98a(2011) section 13.5 but testing to **TWO** observed reactions at each level:</u>
        i. Begin test at 72 ft-lb. If material shows 0/20 reactions: STOP. This is a full pass.
        ii. If material reacts at 72 ft-lb, continue 40 additional impacts or until a second reaction is observed. If the material does not react again (does not exceed 1/60 reactions): STOP. This is a full pass.
        iii. If material reacts twice at 72 ft-lb, step down the energy level/drop height as shown in ASTM 86-98a(2011) table 2. [Next energy level is 65 ft-lb.] Repeat LOX impact test at this level, up to 20 impacts or until **two** reactions are observed. When two reactions are observed, stop testing at this level. If no reaction is seen in 20 impacts at this level: STOP - this is reported as the energy threshold. If one or more reactions are observed, report the reactions observed at that level (e.g. 1/20, or 2/[2-20]) and continue test at the next lower energy level.
        iv. Repeat step (3), stepping down in increments of 5 ft-lb (as shown in ASTM 86-98a (2011) table 2) until the energy level result is 0/20 reactions. Report this level as the energy threshold.
        v. Report shall include the complete test data record showing impacts performed at each energy level and reactions observed.
3) Determine Heat of Combustion in accordance with NASA-STD-6001 section A.2.9 of the above solvents. The preferred method is ASTM D4809, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method), unless this test method is not compatible with test of these solvents.
   Note in the report the ASTM test method used.

FOR REFERENCE INFORMATION ONLY

28. VEHICLE
29. PROJECT
30. MAPTIS MATERIAL CODE IF PREVIOUSLY TESTED
31. USE ATMOSPHERE/FLUID
32. USE PRESSURE
33. USE THICKNESS
34. USE TEMPERATURE
35. TEST ARTICLE WEIGHT
36. NUMBER OF ITEMS TO BE FLOWN
37. INTENDED APPLICATION/ GENERIC USE

WSTF514 (01/14) Formerly JSC Form 2035



NASA WSTF TEST REQUEST

Note to test facility: A copy of this request should be returned with test report.

OFFICE USE ONLY

TEST FACILITY I.D. NUMBER **14-46371**
COORDINATOR **H. Perez**
CUSTOMER REQUEST NO. **WSTF**
TEST FACILITY **WSTF**
CHARGE CODE
14-SMAL-350-SOL FTIR Analysis
14-ESS01-350-HOC HOC Testing
14-ESS01-351-13A Test 13A

1. NAME(S) Mark A. Mitchell
2. ORGANIZATION(S) NASA/MSFC/EM50
3. ADDRESS
4. RETURN SHIPPING ADDRESS
5. DATE 6/19/14
6. PHONE 256-544-5860
7. E-MAIL mark.a.mitchell@nasa.gov
8. MANUFACTURER'S IDENTIFICATION, PART NO., SERIAL NO., LOT NO., BATCH NO. ETC. (Item Description)
Solstice™ Performance Fluid (PF) Trans-1-Chloro-3,3,3-trifluoropropene CAS No. 102687-65-0 Lot No.: BA-320B-U-10-119
9. MANUFACTURER'S NAME Honeywell
10. CHEMICAL CLASS Trans-1-Chloro-3,3,3-trifluoropropene
11. NASA MATERIAL CERTIFICATION NEED DATE
12. HARDWARE SHIPMENT NEED
13. FINAL REPORT CRITICAL NEED DATE
14. TEST REQUIRED PER NHB8060, NASA STD-6001, NASA STD-6016 THERMAL VACUUM STABILITY, OR SP-R-0022
☐ 1(Flam) ☐ MOC ☐ 6(Odor) ☐ 7(Tox) ☐ VCM ☐ CTVS ☐ VAC BAKE ☐ 2 ☐ 3 ☐ 4 ☐ 5 ☐ 8 ☐ 9 ☐ 10 ☐ 11 ☐ 12 ☒ 13 ☐ 14 ☐ 15 ☐ 16 ☐ 17 ☐ 18 ☒ SPEC
15. TEST CHAMBER ATMOSPHERE / FLUID
16. TEST CHAMBER PRESSURE
17. TEST CHAMBER TEMPERATURE
18. TEST CHAMBER DURATION FOR VB
19. REQUIRED CURE TIME
20. REQUIRED CURE TEMPERATURE
21. CURE PRESSURE
22. REQUIRED MATERIAL THICKNESS
23. CLEANING OR OTHER SPECIFICATIONS
24. TEST ARTICLE DIMENSIONS (LxWxH)
25. HABITABLE VOLUME FOR TEST 7 (EMU, OTHER CAPSULE, ETC.)
26. PHOTOGRAPHIC COVERAGE ☐ STILLS ☐ VIDEO ☒ NONE
27. SPECIAL INSTRUCTIONS

1) Perform FTIR Analysis on the solvent
2) Perform NASA-STD-6001 test 13A Ambient LOX Mechanical Impact on solvents, in neat form, with energy threshold test as described in ASTM G86-98a(2011) section 13.5 with modified test logic as follows:
    a. <u>LOX Mechanical Impact Threshold test Preparation/ Special required Documentation</u>
        i. Transfer solvent in preferably all glass containers to minimize potential contamination from vial caps. Bake out glassware at a minimum of 120 F for minimum 24 hours to minimize contamination. Triple rinse all glassware with the solvent before use.
        ii. No pre-chilling of samples as condensation of ambient humidity may be exasperated
        iii. Document prep/ test area Temperature and RH% just before prep, and just before test for each sample as humidity may be a factor in condensation and reactivity
        iv. Discard sample before test if any dislodging, floating pieces are detected (cracks are acceptable as long as sample is intact). Document sample check/ condition for each sample.
        v. Striker pins will be cleaned to 100A or better in addition to grease cups used for testing.
        vi. Ensure droppers used for transfer of solvent are glass. Avoid direct contact of rubber bulb with solvent as contamination may be introduced. Preferably avoid use of use of rubber bulb to eliminate concern.
    b. <u>LOX Mechanical Impact Threshold test using 0/20 reaction criteria as described in ASTM 86-98a(2011) section 13.5 but testing to **TWO** observed reactions at each level:</u>
        i. Begin test at 72 ft-lb. If material shows 0/20 reactions: STOP. This is a full pass.
        ii. If material reacts at 72 ft-lb, continue 40 additional impacts or until a second reaction is observed. If the material does not react again (does not exceed 1/60 reactions): STOP. This is a full pass.
        iii. If material reacts twice at 72 ft-lb, step down the energy level/drop height as shown in ASTM 86-98a(2011) table 2. [Next energy level is 65 ft-lb.] Repeat LOX impact test at this level, up to 20 impacts or until **two** reactions are observed. When two reactions are observed, stop testing at this level. If no reaction is seen in 20 impacts at this level: STOP - this is reported as the energy threshold. If one or more reactions are observed, report the reactions observed at that level (e.g. 1/20, or 2/[2-20]) and continue test at the next lower energy level.
        iv. Repeat step (3), stepping down in increments of 5 ft-lb (as shown in ASTM 86-98a (2011) table 2) until the energy level result is 0/20 reactions. Report this level as the energy threshold.
        v. Report shall include the complete test data record showing impacts performed at each energy level and reactions observed.
3) Determine Heat of Combustion in accordance with NASA-STD-6001 section A.2.9 of the above solvents. The preferred method is ASTM D4809, Standard Test Method for Heat of Combustion of Liquid Hydrocarbon Fuels by Bomb Calorimeter (Precision Method), unless this test method is not compatible with test of these solvents.
   Note in the report the ASTM test method used.

FOR REFERENCE INFORMATION ONLY

28. VEHICLE
29. PROJECT
30. MAPTIS MATERIAL CODE IF PREVIOUSLY TESTED
31. USE ATMOSPHERE/FLUID
32. USE PRESSURE
33. USE THICKNESS
34. USE TEMPERATURE
35. TEST ARTICLE WEIGHT
36. NUMBER OF ITEMS TO BE FLOWN
37. INTENDED APPLICATION/ GENERIC USE

WSTF514 (01/14) Formerly JSC Form 2035

Return to Menu

APPENDIX J—REPORTS FROM ON-SITE VENDOR DEMONSTRATIONS

The following on-site vendor demonstration reports can be found in sections J.1 and J.2:

J.1 Honeywell's Precision Cleaning Demonstration at the SSC Component Facility Using Solstice PF, September 19, 2013

J.2 Summary of an on-site demonstration of Honeywell Solstice PF solvent at Marshall Space Flight Center Propulsion Test Laboratories, November 4–6, 2013

Return to App J

Return to Pg 35

Honeywell's Precision Cleaning Demonstration at the SSC Component Facility Using Solstice PF

Sampling Assistance Provided
by the Component Processing Facility, B2204

Sept. 19, 2013

System Setup and Configuration for Performing the Field Cleaning Tests w/ Solstice PF

Cleaning Demonstration at the Component Processing Facility Using Solstice PF

Two SST braided, 6’ x ¼” Teflon-lined hoses were doped with mineral oil (mixed aliphatics) as the representative NVR. Hose 1 and Hose 2 were doped with 2.4mg and 4 mg of mineral oil respectively.

The demonstration was performed in the clean line area near an open high bay door. At the time of the test, the temperature was 86 deg F and the relative humidity was 72%.

Honeywell also demonstrated rough cleaning of the exterior surface of an A/N Fitting that was grossly contaminated with a fluorocarbon grease.

Honeywell Chemist

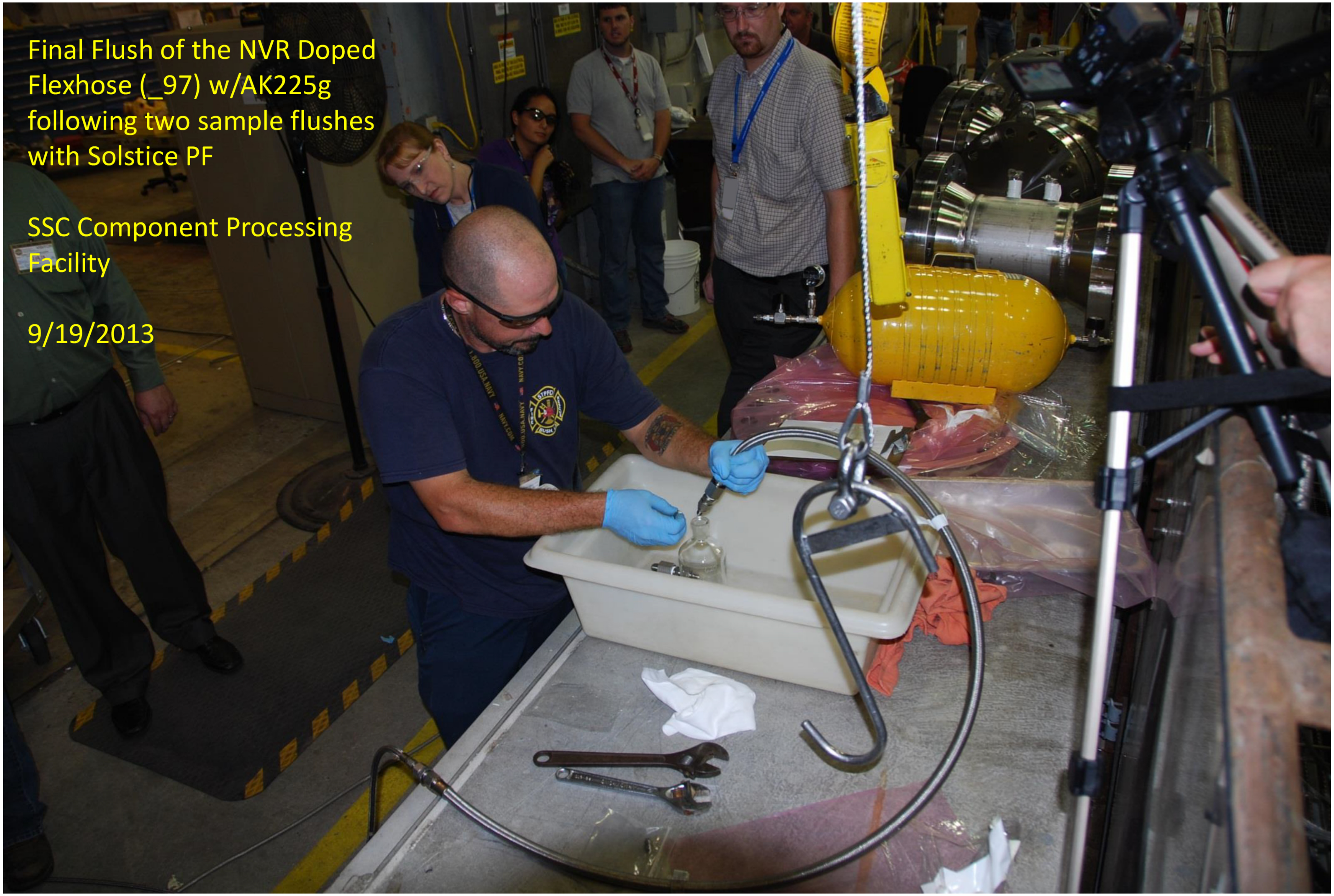
Solstice PF flush & sample collection from Flexhose "1" that was doped with 2.4mgs of NVR. SSC uses 200ml solvent per sq. ft.

CPF Tech

Pressure & flow rates could be adjusted (reduced) to minimize the cooling effects from cleaning and sampling.

Frost formed on the bottle due to the cooling from rapid solvent expansion - the warm temperature combined w/ the high humidity acerbated this condition.

Solstice PF Flowing thru the Flex Hose and Collected in a Catch Pan



Final Flush of the NVR Doped Flexhose (_97) w/AK225g following two sample flushes with Solstice PF

SSC Component Processing Facility

9/19/2013

Quick Summary of the Field Cleaning (Flexhose) Results Using Solstice PF

Sample 1: Flexhose doped with 2.4 mgs of NVR (Mineral Oil)

1st Solstice Sample, NVR, 2.27 mgs
2nd Solstice Flush (Catch Pan - No NVR Sample)
3rd Solstice Sample Flush - NVR 0.46 mgs
Background (Solvent) NVR / 200ml = 0.4mgs.
Final AK225g Flush = 0.01mgs
Solstice PF Removal Efficiency > 99.9%

Sample 2: Flexhose doped with 4 mgs of NVR (Mineral Oil)

1st Solstice Sample, 2.6 mgs
2nd Solstice Flush (Catch Pan - No NVR Sample)
3rd Solstice Sample Flush, 0.5 mgs
Background NVR /200ml = 0.4 mgs.
Final AK225g Flush 0.2mgs
3.8 mgs of NVR removed with Solstice PF Flushes or a 95% Removal Efficiency

Pre Solstice PF Flush - A/N Fitting Contaminated with Fluorocarbon Grease

Threaded areas were contaminated with Christo lube MCG 111 Grease. Solstice PF was flowed across the exterior surface and collected in a SST pan.

Fitting from the SSC CPF
9/19/2013

Post Flush (Rough) Cleaning of an A/N Fitting that was grossly coated with Christo lube MCG111 grease (similar to Krytox 240 AC)

Appearance of the fitting following rough cleaning with Solstice PF.

The vapor pressure developed in the head space of the Solstice PF cylinder produced a solvent flow rate that resulted in a moderate impingement on the surface of the fitting.

Performed at the SSC CPF on 9/19/2013

Return to App J   Return to Pg 36

Summary of an on-site demonstration of Honeywell Solstice PF solvent at Marshall Space Flight Center Propulsion Test Laboratories, November 4-6, 2013.

Representatives of Honeywell visited MSFC on November 4-6, 2013 to demonstrate use of their new cleaning solvent Solstice PF, trans-1-chloro-3,3,3-trifluoropropene or 1233zd(E), CAS No. 102687-65-0, for precision cleaning and field cleaning of propulsion oxygen system components and associated ground test systems as a potential replacement for Asahiklin AK-225G.

This was a follow-up to the Honeywell demonstration of Solstice PF at Stennis Space Center (SSC) on September 18-19, 2013. Representatives from SSC and White Sands Test Facility were invited to this demonstration but were unable to attend.

Visitors from Honeywell were:
Jon Herdlein – Solstice PF chemist
Diana Mercier – Marketing manager

Monday, November 4:

Jon and Diana arrived in the afternoon to set up a portable Crest Ultrasonics degreaser that was shipped by Honeywell to MSFC the previous week for this demonstration. Ten pound and 50 pound pressure cylinders of Solstice PF, and supporting equipment, were also supplied by Honeywell for this demonstration. The degreaser was set up in the building 4653 Valve and Component Lab alongside the existing AK-225G degreaser system. Richard Joye (ET02/METTS), Mark Mitchell (EM50), and Nikki Lowrey (EM50/Jacobs ESSSA) were present during set up.

The Crest Ultrasonics degreaser was a portable unit on wheels, custom designed by Honeywell and Crest for use with Solstice PF solvent. The degreaser included a heated boil sump; a chilled 132 kHz ultrasonic immersion sump; a vapor phase chamber with dual layer cooling coils, tall freeboard, and sliding cover for solvent containment; an automated basket to lower and raise parts from the immersion sump; a filtration system; a water separation system; and touch-screen electronic controls. To fill the system, 50 pound cylinders with a dip tubes were connected, one at a time, via a Teflon or braided hose and a small pump to fill the sumps. (The same system was also used later to drain the degreaser, with the cylinder vented back into the chilled immersion sump for recapture of the vent vapors.) A simple heating blanket was wrapped around the cylinder to generate pressure to help drive the solvent into the sump. The cylinder was placed on an electronic scale to weigh the solvent during fill and drain to gauge solvent quantity. A 50 pound cylinder filled with Solstice PF weighs approximately 80 pounds.

Honeywell noted that they have also worked with Branson and Ultracool to design vapor degreaser systems to use Solstice PF. [Later contact indicated that Baron Blakeslee could also supply a vapor degreaser designed to use Solstice PF.]

Tuesday November 5 – Vapor degreaser demonstration in Building 4653:

MSFC Participants:
Mark Mitchell (EM50)
Nikki Lowrey (EM50/Jacobs ESSSA)
DeWitt Burns (EM50)
Buford Moore (EM50/METTS)
Steve Gentz (NESC)
Richard Joye (ET02/METTS)
Stefanie Wallburg (ES23/METTS)
Wayne Ellenburg (ES23/METTS)

Garry McGuire (ES43)
Adam Gowan (ES43)

MSFC Audio Visual Information Services (AVIS) filmed the Tuesday demonstrations.

Demonstration Series 1: Valve and Component Lab Parts supplied by R. Joye

Unless otherwise noted, all test items were cleaned in the immersion sump with ultrasonics for 5 minutes, drained in the vapor chamber, removed and air dried. It was noted that 132 kHz is a high frequency for ultrasonics and is less aggressive than more typical 40 kHz or 47kHz ultrasonic baths used to clean metal parts. Parts quantitatively tested for contamination were analyzed using standard Valve Lab procedures with AK-225 as the verification flush solvent.

- Test 1 – Three Poppets from a relief valve were cleaned with Solstice and tested by S. Wallburg for particulate and NVR. They passed MSFC criteria for oxygen or hydrogen systems for particulate and NVR.
- Test 2 – A spindle was cleaned with Solstice and tested by S. Wallburg for particulate and NVR. It passed MSFC criteria for oxygen or hydrogen systems for particulate and NVR.
- Test 3 – A half valve body, 300 series stainless steel, contaminated with gross environmental soils, was obtained from an exterior storage rack behind 4653. Items such as this would normally be precleaned in the Valve Lab to remove gross soils and fibers. For a severe test, this item was cleaned in the Solstice PF degreaser as-is and then tested by S. Wallburg for particulate and NVR. The item passed MSFC NVR criteria for oxygen systems. It nearly passed the particulate criteria for hydrogen system. Two particles were found in the rinse sample that exceeded the 400 micron limit. It was noted that these were fibers that would have been removed in the standard precleaning operation.

Demonstration Series 2: Removal of miscellaneous Valve and Component Lab soils from 2.5 inch x 6 inch 2219 aluminum panels with a smooth surface finish. The test panels were provided by EM50. A variety of spray cans and grease tubes were gathered from around the lab and applied by spraying or smearing the contents on individual test panels. Contaminated test panels were cleaned one at a time in the degreaser. Cleanliness verification was to Visually Clean Sensitive plus ultraviolet. Contaminants tested were:

- Fingerprints
- LPS KB 88 Penetrant – NASA Stock Number 12584
- Mineral Oil – NASA Stock Number 152
- SiliKroil Penetrating Oil – NASA Stock Number 276
- M1 All Purpose Lubricant – NASA Stock Number 1711
- Mil-G-47219A Halocarbon 25-5S grease – NASA Stock Number 105
- Tri-Flow Industrial Lubricant – NASA Stock Number 8616
- Slic Tite lubricant – NASA Stock Number 9954
- Loctite silicone – NASA Stock Number 11348
- Fabulous PB Blaster penetrant – NASA Stock Number 12811
- CRC Duster (1,1,1,2 tetrafluoroethane) 14085 – NASA Stock Number 9065

All of these panels passed VC Sensitive + UV inspection criteria after cleaning.

Demonstration Series 3: Removal of difficult soils identified in the MSFC/SSC AK-225 solvent replacement test plan. These soils were provided by the EM50 Contamination Lab. Test panels, application methods, and inspection criteria were the same as demonstration series 2. Contaminants tested were:

- Di (2-ethyl hexyl) sebacate (gauge calibration oil) – NASA Stock Number 3293
- Big Red crane grease – NASA Stock Number 8712
- Krytox 240AC oxygen compatible grease – NASA Stock Number 2527

All of these panels passed VC Sensitive + UV inspection criteria after cleaning.

Demonstration Series 4: Removal of mixed contaminant as required by ADS-61A-PRF, *Performance Specification for Army Aircraft Cleaners, Aqueous and Solvent,* section 4.5.1 Standard contaminant. This contaminant mix was used for a previous study of vapor degreaser solvent alternatives performed for the Army Research Lab in 2011-2012. The contaminant was:

(1) MIL-PRF-83282 Hydraulic Fluid – Radcolube FR282, Radco Industries, Inc. (two parts by weight)

(2) MIL-PRF-81322G Grease- Aeroshell Grease 22, Shell Aviation Ltd. (one part by weight) [a highly fluorescent grease]

(3) Powdered carbon black ASTM D1765 grade N990, sieved just prior to use (one tenth part by weight) [a visual indicator]

The contaminant mix was applied by brush onto eight 2.5 in x 6 in 2219 aluminum panels, two with smooth surfaces and six with grit blasted surfaces. The contaminated panels were based for two hours at 130^{o}F (55^{o}C). All eight panels were hung in parallel and spaced on a cleaning rack and cleaned together. The smooth panels appeared clean under visual and UV inspection. All of the grit blasted panels appeared clean under UV inspection but some appeared to have trace carbon black contamination. One grit blasted panel with apparent trace carbon residual and one smooth, visually clean panel were analyzed quantitatively by S. Wallburg for contamination. These passed the particulate and NVR criteria for oxygen or hydrogen systems (Table 1).

Wednesday Morning, November 6 – Field cleaning demonstrations in the West Test Area

MSFC Participants:

Mark Mitchell (EM50)
Nikki Lowrey (EM50/Jacobs ESSSA)
Tim Gautney (ET10) [present for initial introductions and demonstration kick-off]
DeWitt Burns (EM50)
Buford Moore (EM50/METTS)
Daniel Clanton (ET10/METTS)
Chris Smith (ET10/METTS)
Kevin Thompson (ET10/METTS)

Tim Gautney escorted us into the West Test Area and tasked Daniel Clanton to lead the cleaning trials. Daniel was asked to select a variety of cleaning challenges where AK-225G is currently being used. Solstice PF in 10 pound cylinders was used with a variety of high and low pressure spray nozzles provided by Honeywell, in combination with wipers (Kimwipes or equivalent) supplied by ET10. AK-225G in a pressurized Sure Shot spray can was used during these trials for comparison. The weather was moderate with no precipitation.

NOTE: We were unable to perform field cleaning tests on propulsion test stand 4670 due to asbestos remediation activity in progress.

The Solstice PF cylinders used in this trial included a cylinder containing only Solstice PF, modestly pressurized by evaporation, and a cylinder containing Solstice PF Spray Cleaner pressurized with Honeywell Solstice 1234ze propellant. Solstice 1234ze, trans-1,3,3,3-tetrafluoroprop-1-ene, CAS 29188-24-9, is a related hydrofluoro-olefin (HFO) that is also non-ozone depleting, VOC exempt, very low global warming, and also has a time weighted average exposure guideline of 800 ppm. It has a boiling point of -19°C (-2.2°F)

NOTE: Solstice 1234ze has not been tested for compatibility with liquid or gaseous oxygen. It has no flash point.

For these field cleaning trials, an ultraviolet LED flashlight provided by EM50 was used in addition to standard visual inspection to assess performance. No quantitative analyses were performed.

First Trial Set – at the 4699 Cryogenic Structural Test stand.

1. We went inside an adjacent shed containing a hydraulic pump system. The aged MIL-PRF-5606 hydraulic fluid found here was reported to be a difficult contaminant to remove. Daniel used a low pressure spray of Solstice PF and Kimwipes to clean hydraulic fluid drips and coated surfaces. The Solstice PF appeared to perform well. Only slight cooling of surfaces was noted, with no condensation.
   NOTE: During this cleaning process, some Solstice PF dripped on a painted structure supporting the hydraulic system. When wiped off, it was noted that the Solstice PF appeared to remove some of the paint. The technicians remarked that AK-225G is not normally used to clean painted surfaces so this should not be held against the Solstice PF. A cautionary note might be in order that Solstice PF may remove paint.
2. A high pressure spray nozzle, similar to those found on aerosol cans, was used with the Solstice PF Spray Cleaner cylinder (containing 1234ze propellant) to clean hydraulic system external surfaces. AK-225G in a Sure Shot sprayer, pressurized with High Purity Air (HPA) to approximately 90 psi, was tried for comparison. Cleaning performance was comparable. It was also noted that both cleaning systems cooled the part being cleaned, with the resulting temperatures subjectively the same. Condensation was not observed.
3. Kevin filled a small cap fitting, about 2 inches in diameter, with Solstice PF to observe evaporation. He commented that parts are sometimes soaked in AK-225G for a few minutes to loosen heavy soils. The solvent stayed in the liquid form and technicians agreed that it would be suitable for part soaking for the amount of time they usually require.
4. We went outside to the test stand where Daniel wiped several fittings and a mounting panel with Solstice PF and Kimwipes. These parts had gross accumulated environmental contamination – dirt, pollen, etc. This cleaning performance was judged to be comparable to AK-225G.

Second Trial Set – Field Shop cleaning in West Test Area building 4671. In this facility, a wide variety of test fixtures are manually cleaned and assembled. Clean aluminum foil is placed on a table for a temporary clean surface on which to place parts.

1. A union fitting contaminated with Christo-lube III (a fluorocarbon grease equivalent to Krytox) was selected. One side of the union was cleaned by flushing and wiping with Solstice PF and the other side was cleaned with AK-225G. Cleaning was judged to be comparable.
2. A cap contaminated with halocarbon grease was cleaned with Solstice PF. The solvent removed this contaminant well.
3. Three o-rings were placed in a Ziploc bag with Solstice PF and soaked for about three minutes to evaluate whether the solvent would dry or crack the o-ring materials. O-rings are frequently cleaned in this manner in 4671. The materials tested were Viton, Buna rubber, and Teflon. Each o-ring was flexed and stretched after the soak. No stiffness or cracking was observed.
4. A crush washer was sprayed and wiped to remove an unknown contaminant. The contaminant was effectively removed.

5. Back-to-back compression fittings of three different sizes were effectively cleaned.
6. Another union fitting with Christo-lube and environmental contamination was effectively cleaned.
7. A flange with a Gylon gasket was disassembled and wiped clean. The Gylon appeared to be unharmed.
8. Anodized 6061 aluminum back plates are used in the test area for mounting of fittings and instrumentation. One of these was sprayed and wiped with Solstice PF to observe any effect on the aluminum. The back plate appeared to be unaffected.

Note: The o-ring and gasket materials mentioned here, as well as 6061 aluminum and other metals used for construction of the parts tested here, are being tested in the lab for compatibility with Solstice PF as a part of MSFC/SSC joint AK-225G replacement test plan. The compatibility of Solstice PF with Ziploc bags is not a part of this test plan.

The general feedback from the West Test Area technicians was that Solstice PF cleaned just as well as AK-225G. No objections were raised with regard to odor, condensation, or other handling issues. The technicians commented that they would be willing to use the Solstice directly from the 10 pound cylinders in lieu of the Sure Shot sprayers.

During these trials, Jon also demonstrated a configuration for a spray catch basin system, based on a PIG Corporation Poly Drum Funnel, which could be used to recapture and recycle solvent during part flushing operations. He also discussed recommended filling and handling practices for Solstice PF in 10 pound, 50 pound, and 500 pound cylinders.

Conclusions:

Workers in the Valve and Component Lab and in the West Test Area judged Solstice PF to be comparable to (or better-for Krytox/Christo-lube) AK-225G for their cleaning applications for propulsion test systems. The handling requirements for Solstice PF pressure cylinders appeared to be quite manageable. There were no objections regarding odor, condensation, or other handling issues.

Solstice PF attacked the paint on a support stand. This may require a usage precaution to avoid exposure to painted surfaces. It might be useful to investigate Solstice PF for paint removal.

Nikki M. Lowrey
Senior Contamination Control Engineer
Jacobs Technology, Inc./ Jacobs ESSSA Group
Materials & Processes, Environmental Effects Branch
NASA-Marshall Space Flight Center

Table 1. Results from testing of Solstice PF during the Honeywell Demonstration on 11/5/2013 in the valve lab. All results are comparable to what would be expected from cleaning with AK-225G

Sample Description	Contaminants & Treatment	**Clean time immersion	Particulate (microns)		NVR (mg)
Poppets	Contaminants from routine process in valve lab, Pre-cleaned	10 minutes	100-175	2	0.029
			175-400	0	
			>400	0	
Spindle	Contaminants from routine process in valve lab, no Pre-clean	10 minutes	100-175	6	0.049
			175-400	1	
			>400	1	
Valve outer body part	Excessive contamination from outdoor exposure, no pre-clean	10 minutes	100-175	6	0.035
			175-400	4	
			>400	2	
			Fibers	7	
SP Alum. 2219 coupon	*2 parts hydraulic fluid, 1 part lubricating grease, 1 tenth carbon black, baked 2 hours 55°C	5 minutes	N/A	N/A	0.156
GB Alum. 2219 coupon	*2 parts hydraulic fluid, 1 part lubricating grease, 1 tenth carbon black, baked 2 hours 55°C	5 minutes	N/A	N/A	0.206

* Contaminant mix as specified in ADS-61A-PRF, Performance Specification for Army Aircraft Cleaners, Aqueous and Solvent, February 4, 2002

** Ultrasonic vapor degreaser, operated at 132 kHz.

Test results by Stefanie Wallburg - Chemist, InfoPro Corporation - MSFC, METTS

Return to Menu

Return to Pg 42

APPENDIX K—COMPONENT LEVEL CLEANING ASSESSMENT REPORT

Appendix K contains the Efficiency of Solstice PF and L-14780 to Remove NVR from Components, AK225G Replacement Effort, NASA Stennis Space Center SSC Gas & Materials Science, October 24, 2014.

Efficiency of Solstice PF & L-14780 to Remove NVR from Components

AK225G Replacement Effort

NASA Stennis Space Center

SSC Gas & Materials Science

October 24, 2014

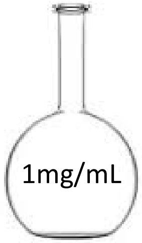
Purpose

Determine the cleaning efficiency of Solstice PF and 3M-L14780 to remove various NVR materials from components. The test results will be compared to AK225G (the baseline solvent).

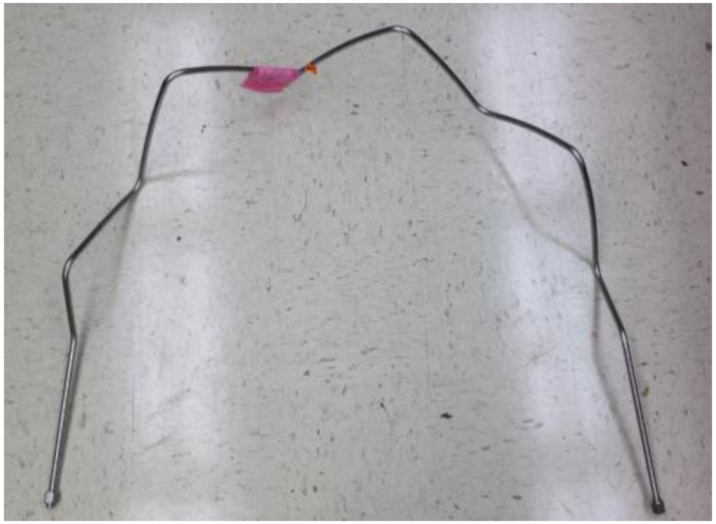
Doping Components with Known Amounts of NVR

Components - Flex hoses and rigid tubing w/ multiple 90^0 bends

- Prepare the following non-volatile residue (NVR) solutions
  - 100mg of mineral oil in 100mL of AK225G.
  - 100mg of 85W-140 gear oil in 100ml of AK225G
- Label the NVR solutions “1mg /ml” for each NVR type
- Clean two SST flex hoses and two SST tubing sections that have ten 90 degree bends (zig-zag tubing) to level 400A.



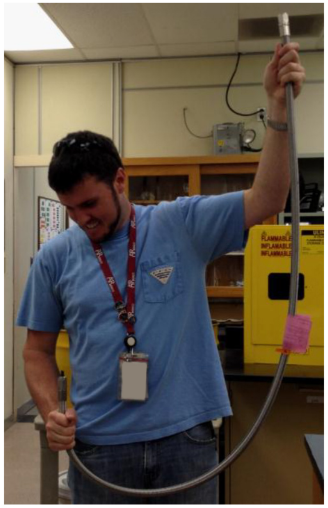
1/2” x 4’ Flex Hose



3/8” x 92” Zig Zag Tubing w/ “ten” 90^0 bends

Experiment and Test Details
Doping Components with NVR

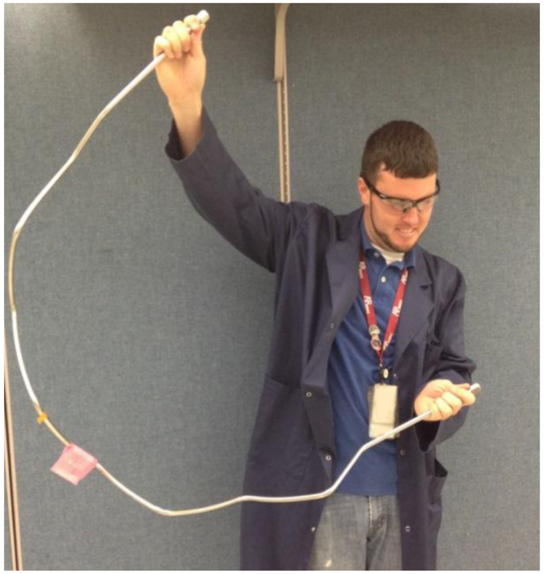
- Using a pipette, place 2mL (2mg) of the NVR mineral oil solution (1mg/1ml of AK225g) into the flex hose and zig zag tubing.



- Fasten the "AN" caps on both ends and move the hose / tubing from side to side to ensure uniform coverage throughout the internal surfaces.
- Bend the flexhose to form a "slight "C" shape. The ends should be near vertical to prevent lost of the NVR solution. Use lab clamps to secure.
- Remove the A/N caps and slowly apply a tickle N2 purge (50 - 100 ml/min) through the hose & zig zag tubing for 8 hrs to remove the carrier solvent (AK225G).
- Place 100 mL of the candidate cleaning solvent into a clean beaker. Remove the N2 purge from the components and slowly add 30 -40 ml from the 100ml solvent beaker to the flex hose / tubing.

Experiment and Test Details

- Cap the ends with clean A/N fittings and move the hose/tubing back and forth to ensure the solvent is wetting all the internal surfaces. Collect the flush in an empty beaker. Repeat the flushes until all 100 mL has been used to flush the flex hose / tubing.



- Analyze a 50 mL aliquot from the 100 mL flush with the FTIR to determine the amount of NVR removed.
- Perform a 2nd flush w/ AK225G and analyze the NVR to determine NVR removal efficiency of the candidate solvent.
- Re-clean all components and dope the flex hose and zig-zag tubing w/ 4mg of NVR for each NVR type and repeat the analytical process.

NVR (Mineral Oil) Removed w/ 3M L-14780

NVR Added (mg) to Component	Component	NVR Removed (mgs) with 100 mL flush of L14780	NVR removed (mgs) from 2 ^{0} Flush (100mL AK225G)	% NVR Removal Efficiency L14780
1.87 mg	Flex hose	1.853	0.019	98.98
3.78 mg	Flex hose	3.746	0.036	99.04
1.83 mg	Zig zag tubing	1.834	-0.002	100
3.77 mg	Zig zag tubing	3.770	-0.003	100

NVR (Mineral Oil) Removed w/ Solstice PF

NVR Added (mg) to Component	Component	NVR Removed (mgs) with 100 mL flush of Solstice PF	NVR removed (mgs) from 2 ^{0} Flush (100mL AK225G)	% NVR Removal Efficiency Solstice PF
1.81 mg	Flex hose	1.772	0.037	97.95
3.58 mg	Flex hose	3.558	0.024	99.33
1.75 mg	Zig zag tubing	1.749	-0.003	100
4.0 mg	Zig zag tubing	3.990	0.006	99.85

NVR (85W-140 Gear Oil) Removed w/ 3M L-14780

NVR Added (mg) to Component	Component	NVR Removed (mgs) with 100 mL flush of L14780	NVR removed (mgs) from 2 ^{0} Flush (100mL AK225G)	% NVR Removal Efficiency L14780
2.04 mg	Flex hose	2.004	0.034	98.3
3.79 mg	Flex hose	3.762	0.029	99.24
1.94 mg	Zig zag tubing	1.940	0.002	99.9
3.74 mg	Zig zag tubing	3.732	0.004	100

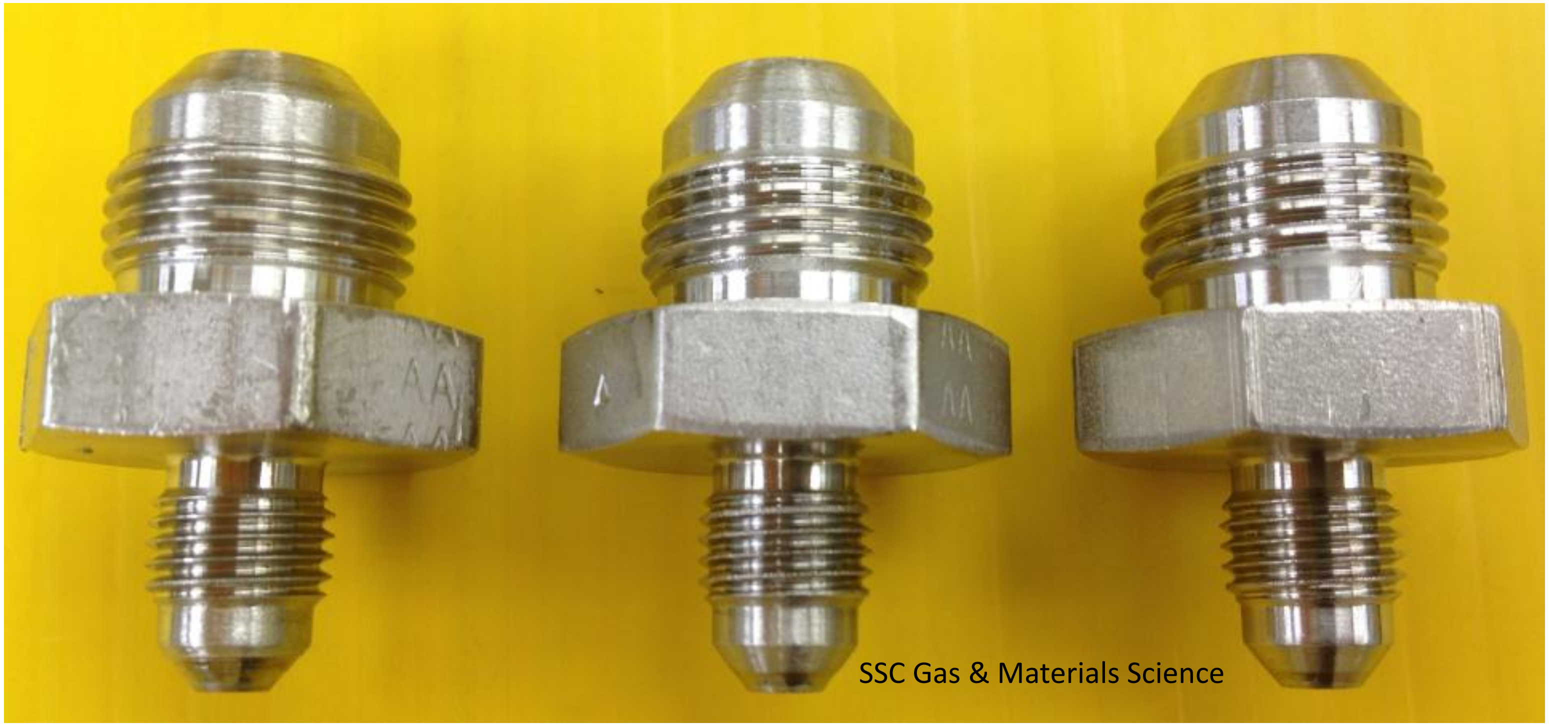
NVR (85W-140 Gear Oil) Removed w/ Solstice PF

NVR Added (mg) to Component	Component	NVR Removed (mgs) with 100 mL flush of Solstice PF	NVR removed (mgs) from 2 ^{0} Flush (100mL AK225G)	% NVR Removal Efficiency Solstice PF
1.95 mg	Flex hose	1.877	0.069	96.4
3.78 mg	Flex hose	3.760	0.024	99.4
1.97 mg	Zig zag tubing	1.960	0.009	99.5
4.16 mg	Zig zag tubing	4.150	0.010	99.8

Experiment and Test Details

Dope A/N fittings w/ Krytox Grease:

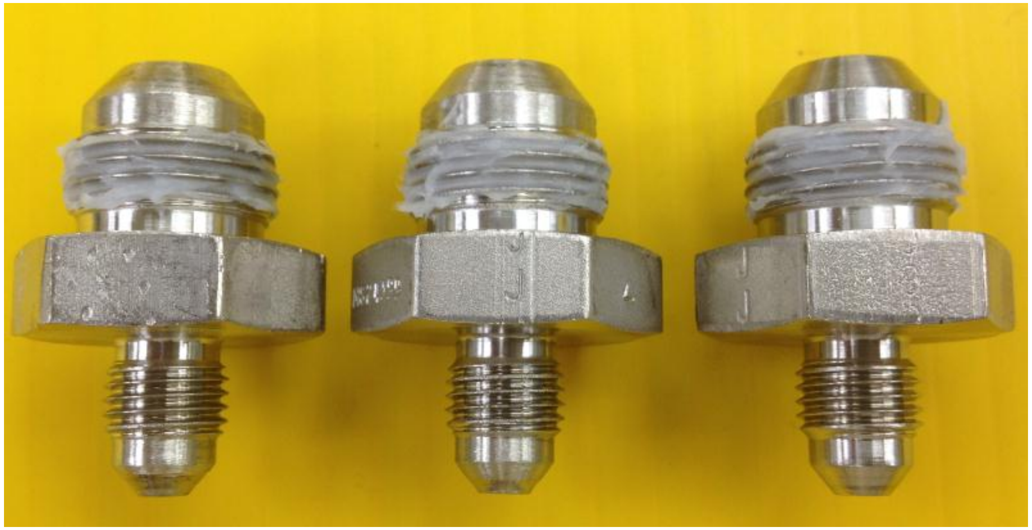
- Obtain three 1/4” to 1/2” AN fittings.
- Clean the fittings to level “400A”.



SSC Gas & Materials Science

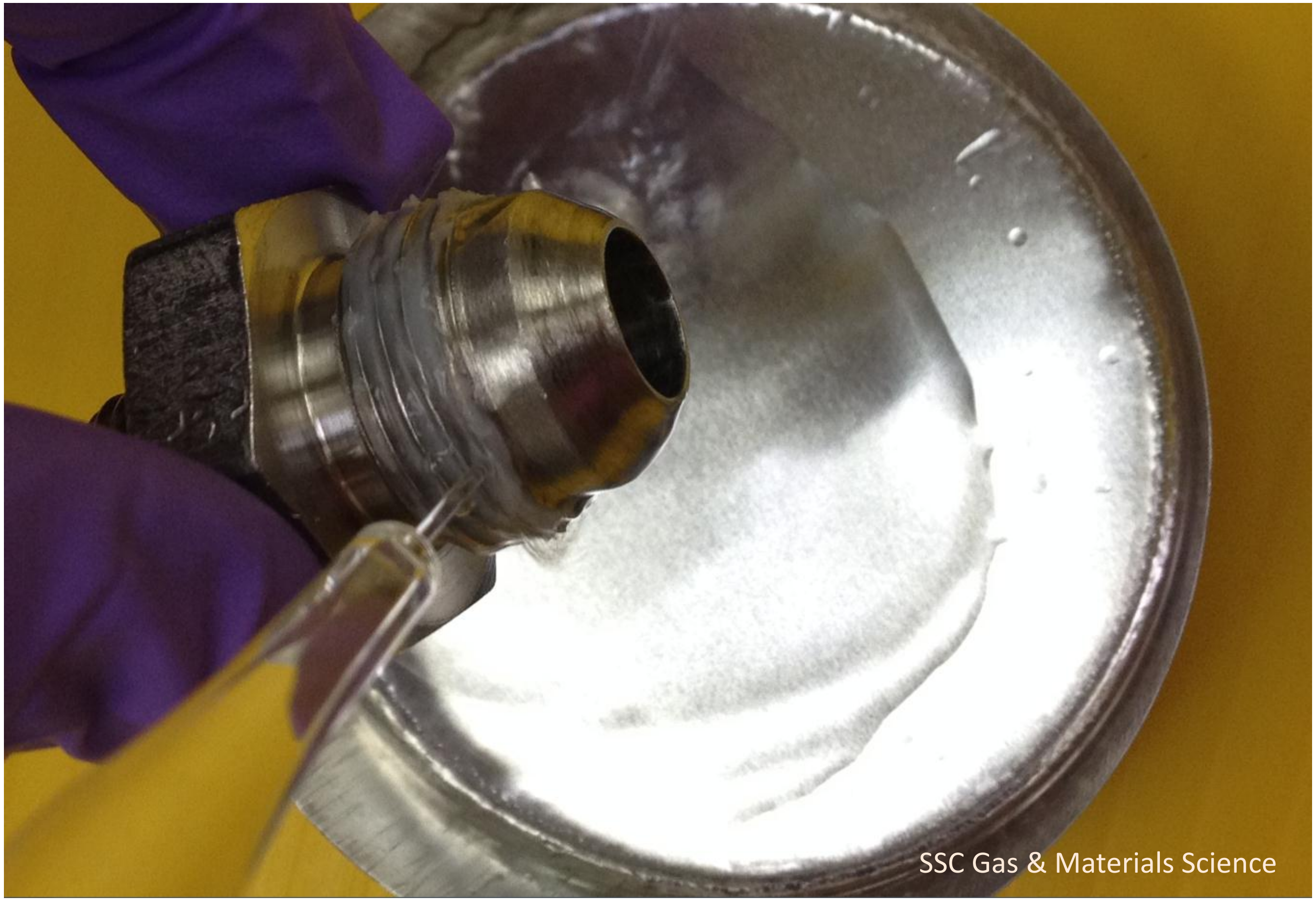
Experiment and Test Details

- Apply 500mg of Krytox grease to each fitting.
- Coat grease evenly through out the 1/2” threads.



Experiment and Test Details

- Gravity rinse one side of each fitting with 50mL, rotate the fitting 180 degrees and perform a 2nd 50 ml rinse. Measure the NVR removed from each fitting .

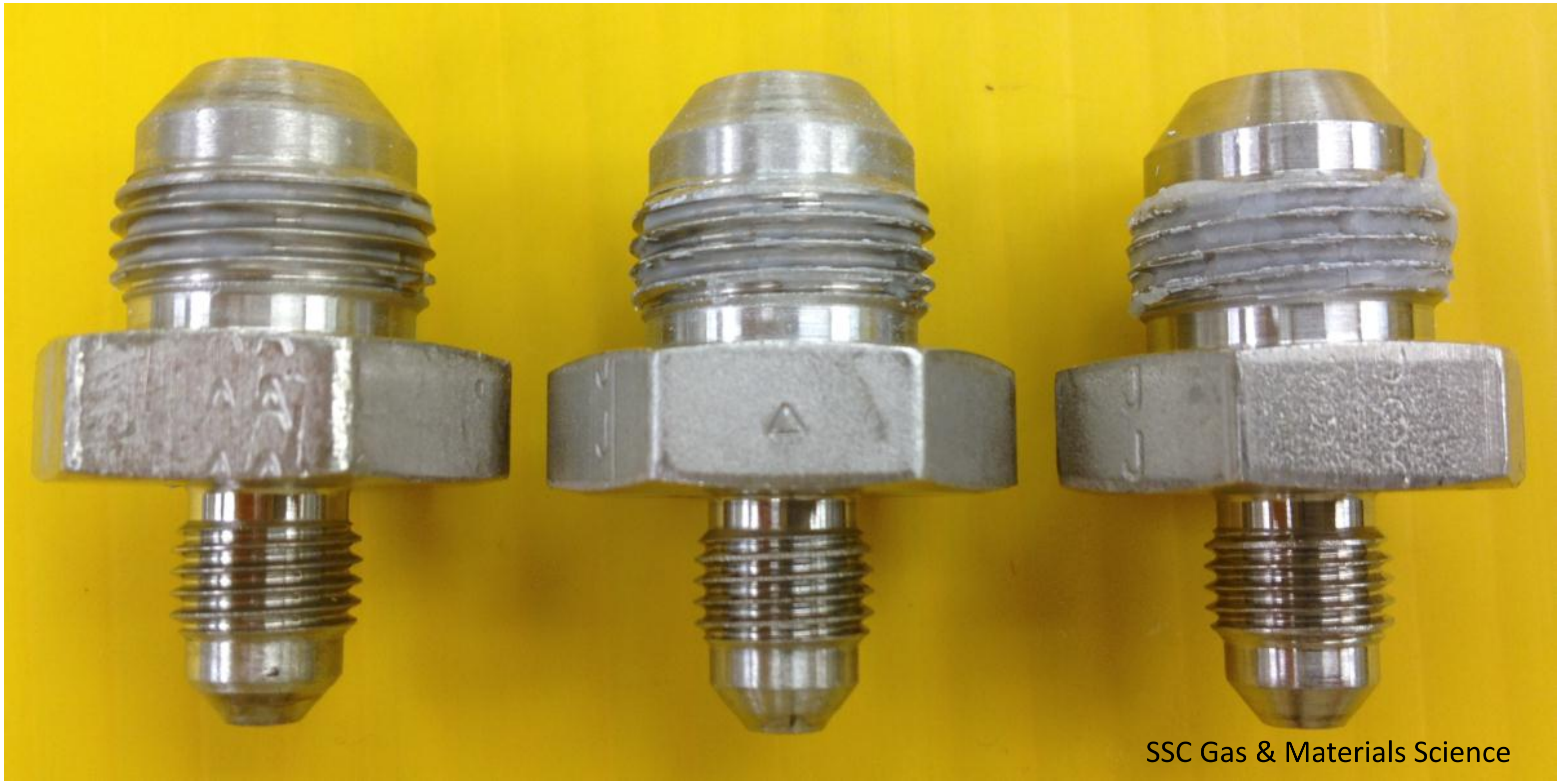


SSC Gas & Materials Science

Test Results

- AK225G is tested as the control.

AK225G Control
3M L-14780
Solstice PF



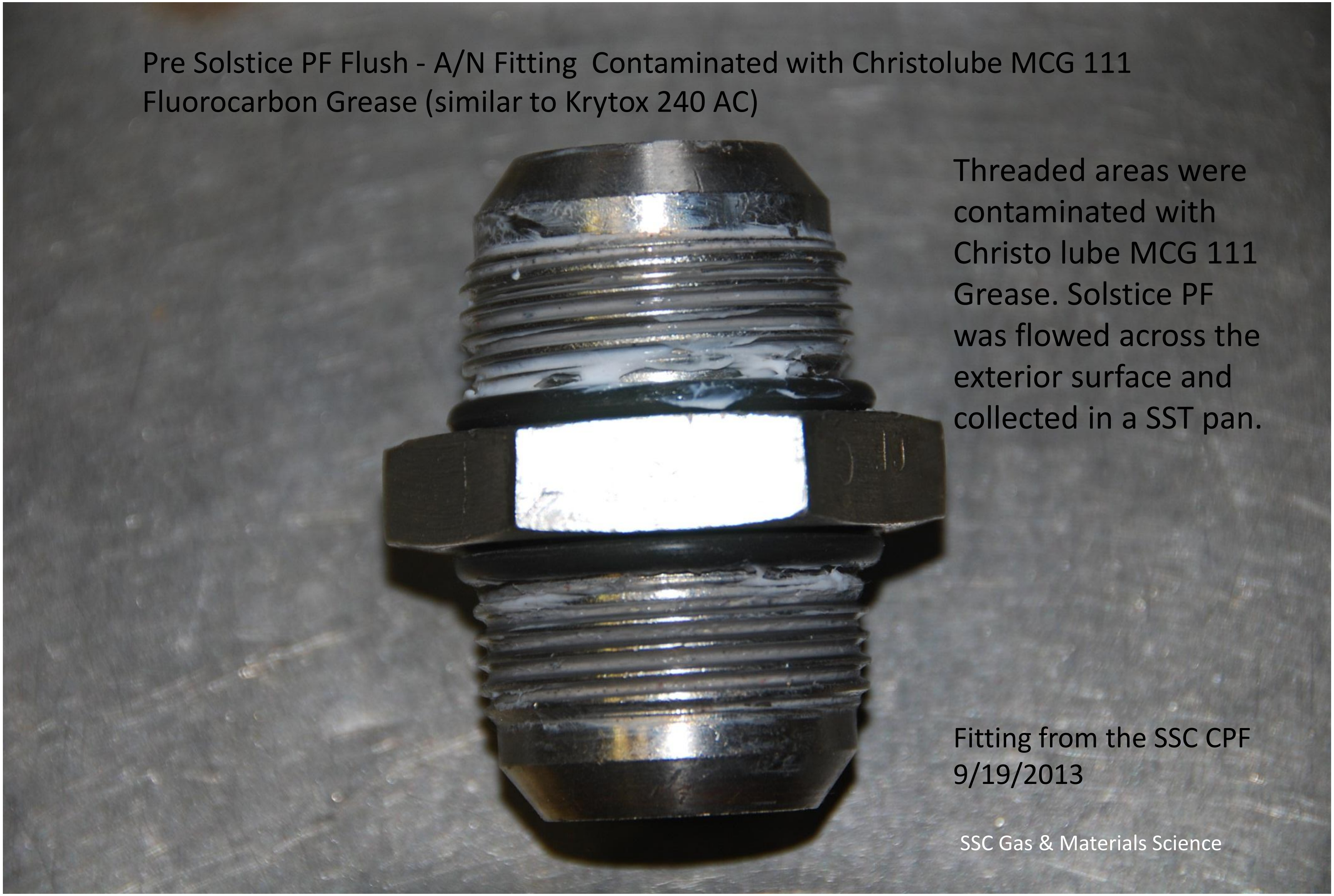
SSC Gas & Materials Science

58.5%
56.9%
51.2%

NVR Removal Efficiency

Mechanical Energy e.g. impingement, ultrasonics, etc. must be used in conjunction with the solvents to remove Krytox or other fluorocarbon-based greases.

Pre Solstice PF Flush - A/N Fitting Contaminated with Christolube MCG 111 Fluorocarbon Grease (similar to Krytox 240 AC)



Threaded areas were contaminated with Christo lube MCG 111 Grease. Solstice PF was flowed across the exterior surface and collected in a SST pan.

Fitting from the SSC CPF
9/19/2013

SSC Gas & Materials Science

Post Solstice PF Flush (Rough) Cleaning of an A/N Fitting that was grossly coated with Christo lube MCG111 grease (similar to Krytox 240 AC)

Passed Cleanliness Level “VC” .
The vapor pressure developed in the head space of the Solstice PF cylinder produced a solvent flow rate that resulted in a moderate impingement on the surface of the fitting.

Performed at the SSC CPF on 9/19/2013

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)* 01–04–2015

2. REPORT TYPE Technical Publication

3. DATES COVERED *(From - To)*

4. TITLE AND SUBTITLE
Replacement of Hydrochlorofluorocarbon-225 Solvent for Cleaning and Verification Sampling of NASA Propulsion Oxygen Systems Hardware, Ground Support Equipment, and Associated Test Systems

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
H.D. Burns, M.A. Mitchell, J.H. McMillian, B.R. Farner,* S.A. Harper,** S.F. Peralta,** N.M. Lowrey,*** H R. Ross,† and A. Juarez‡

5d. PROJECT NUMBER

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
George C. Marshall Space Flight Center
Huntsville, AL 35812

8. PERFORMING ORGANIZATION REPORT NUMBER
M–1396

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
National Aeronautics and Space Administration
Washington, DC 20546–0001

10. SPONSORING/MONITOR'S ACRONYM(S)
NASA

11. SPONSORING/MONITORING REPORT NUMBER
NASA/TP—2015–218207

12. DISTRIBUTION/AVAILABILITY STATEMENT
Unclassified-Unlimited
Subject Category 23
Availability: NASA STI Information Desk (757–864–9658)

13. SUPPLEMENTARY NOTES
Prepared by the Materials & Processes Laboratory, Engineering Directorate
*Stennis Space Center, **JSC-WSTF, ***Jacobs Technology, Inc./Jacobs ESSSA Group, Huntsville, AL, †A2 Research, ‡Jacobs Technology, Inc./MEI Technologies, JSC-WSTF

14. ABSTRACT

Since the 1990's, NASA's rocket propulsion test facilities at Marshall Space Flight Center (MSFC) and Stennis Space Center (SSC) have used hydrochlorofluorocarbon-225 (HCFC-225), a Class II ozone-depleting substance, to safety clean and verify the cleanliness of large scale propulsion oxygen systems and associated test facilities. In 2012 through 2014, test laboratories at MSFC, SSC, and Johnson Space Center-White Sands Test Facility collaborated to seek out, test, and qualify an environmentally preferred replacement for HCFC-225. Candidate solvents were selected, a test plan was developed, and the products were tested for materials compatibility, oxygen compatibility, cleaning effectiveness, and suitability for use in cleanliness verification and field cleaning operations. Honewell Soltice™ Performance Fluid (trans-1-chloro-3,3, 3-trifluoropropene) was selected to replace HCFC-225 at NASA's MSFC and SSC rocket propulsion test facilities.

15. SUBJECT TERMS
solvent, oxygen cleaning, launch vehicle, ozone depletion, HCFC-225, cleanliness verification, nonvolatile residue

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			STI Help Desk at email: help@sti.nasa.gov
U	U	U	UU	521	19b. TELEPHONE NUMBER *(Include area code)* STI Help Desk at: 757–864–9658

National Aeronautics and
Space Administration
IS20
George C. Marshall Space Flight Center
Huntsville, Alabama 35812