NASA Rocket Propulsion Test Replacement Effort for Oxygen System Cleaner – Hydrochlorofluorocarbon (HCFC) 225

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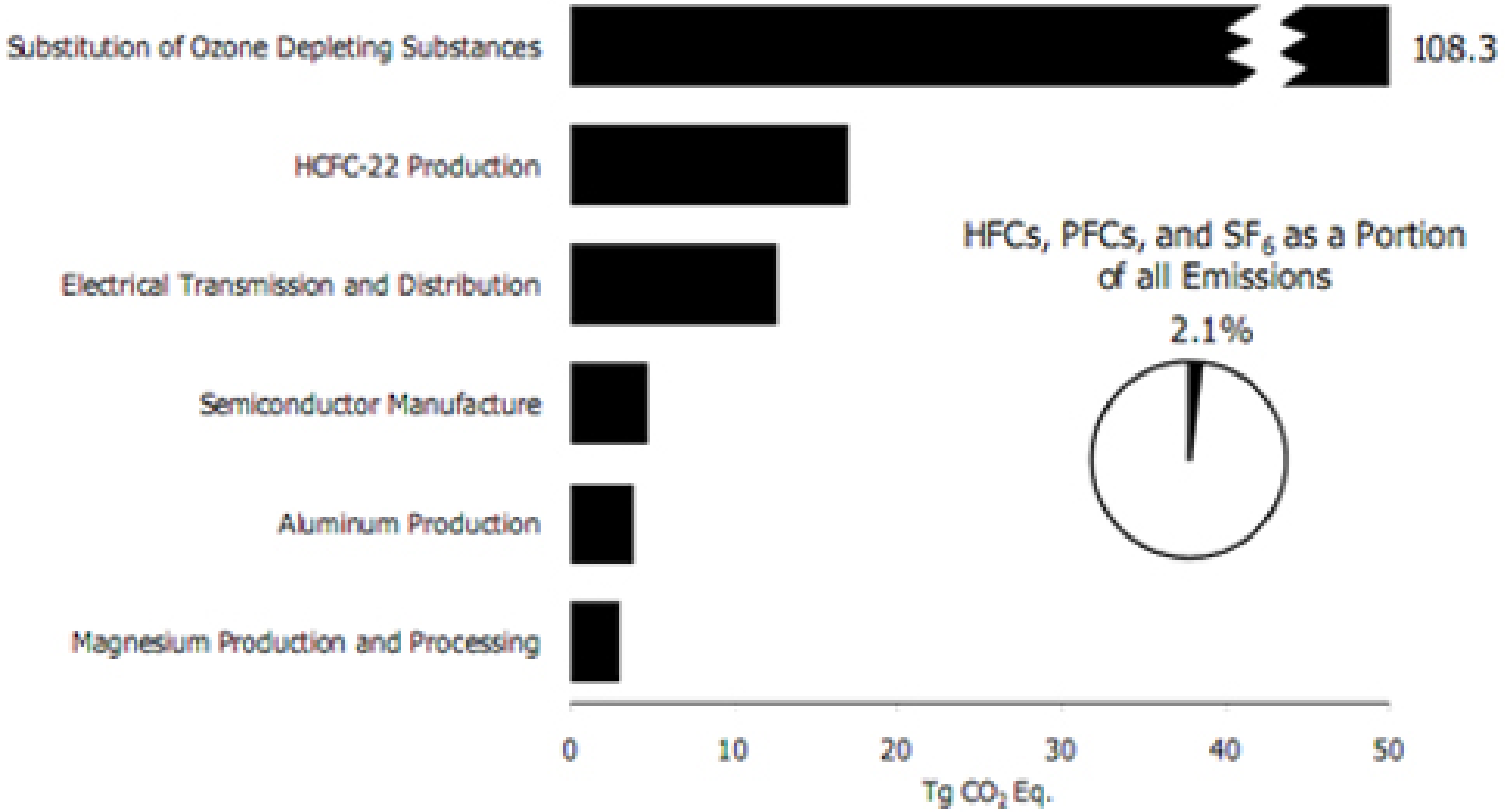
- **Background**
- **Problem Scope**
- **Current Applications**
- **Replacement Search and Test Strategy**
- **Implementation**
- **Look Ahead**

Background – Ozone Depleting Substances (ODS)

- Freon - CFC-113 historically used in oxygen system applications
- International and domestic environmental legislation were drivers for 1996 production phase-out
  - Montreal Protocol of 1987
  - Clean Air Act Amendments of 1990
- HCFC-225 implemented as an *interim* replacement for CFC-113 in LOX/GOX cleaning / verification applications.
  - Production phase out Jan 1, 2015 - no longer available for procurement after 2014 – may be reclaimed/recycled
  - Mission critical requirements for most HCFCs after 2014 would have to be negotiated with the Environmental Protection Agency (EPA) if material is available.

Relative US Contribution of High Global Warming Potential (GWP) Gases

- ODS Substitutes, while a small fraction of overall greenhouse gases (GHG), are potent greenhouse sources.



Source: 2009 U.S. Greenhouse Gas Inventory Report, EPA. 2009

Effect of ODS Reduction on GWP Sources

- As use of ozone depleting CFCs has gone down, use of long-lived Hydrofluorocarbons (HFCs) has gone up.
- Many HFCs are potent greenhouse gases.

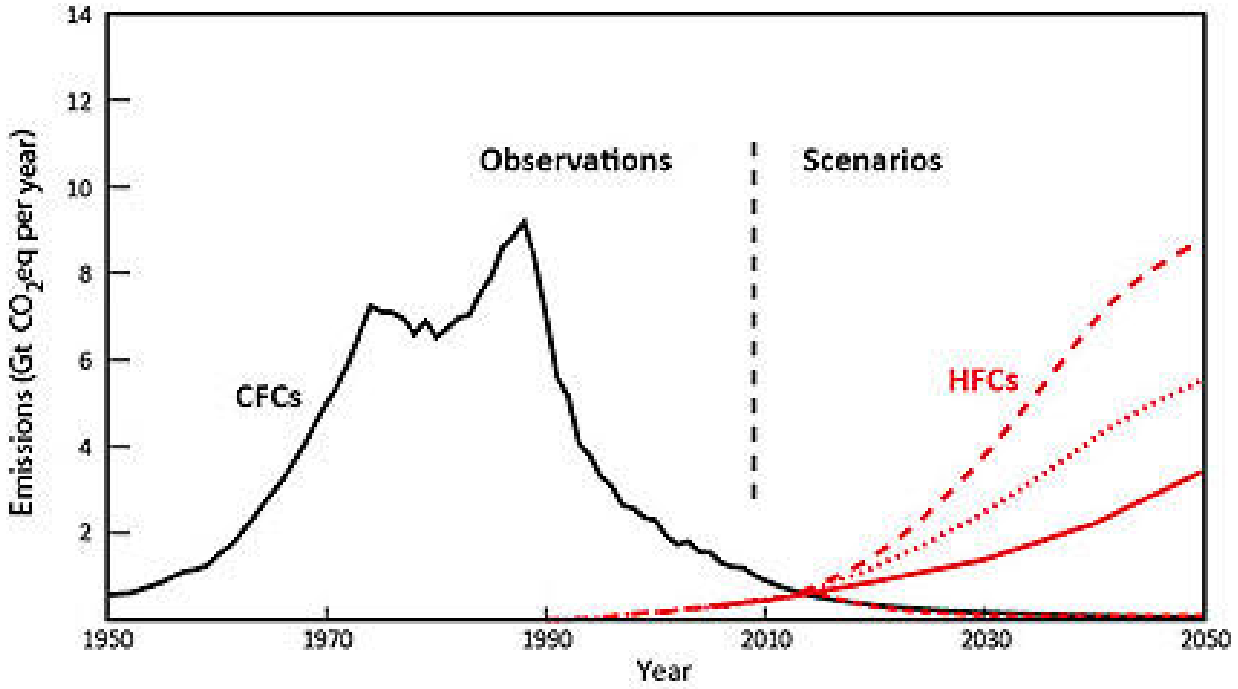


Image Source: Science Daily Feb. 24, 2012
http://www.sciencedaily.com/releases/2012/02/120224110737.htm

Problem Scope

- HCFC-225 has been replaced in most small component NASA applications but is still the primary material used at MSFC and SSC to clean propulsion systems and their test stands.
- At present, no alternative has been identified meeting all criteria:
  - Non-flammable with LOX / GOX
  - Compatible with materials used in LH2/LOX propulsion systems and test stands
  - An effective cleaner on the soils that must be removed
  - Usable on large scale hardware without very high capital equipment costs
- Testing is required to identify and qualify a replacement before stockpiles are depleted at MSFC and SSC.

Applications - MSFC and SSC

- **MSFC:** Cleaning/Verification of propulsion systems, complex geometries and large components. Baseline solvent for comparative testing.
- **SSC:** Cleaning /Verification of valves , large components, propulsion systems and test stands. Baseline solvent for comparative testing.
- **WSTF and others:** Baseline solvent for comparative testing.

Space Launch System (SLS) Program Agreement

- **Proposed approach to address HCFC 225 availability**
  - Identify candidate cleaners
  - Conduct test program to select HCFC-225cb (Asahiklin AK-225G) replacement for cleaning and verification of propulsion oxygen systems.
  - Down-selected cleaner(s) implementation plan
    - Develop material specifications
    - Identify facility modification associated with new materials
    - Procure support hardware, facility modifications, filter/distillation systems, etc
    - Update paperwork – procedures, specifications
- **Risk Mitigation – stockpile to allow implementation time**
  - MSFC maintaining HCFC 225 ~ 10 year stockpile - estimate considering current usage rate
  - SSC does not maintain a stockpile therefore SLS funded 5 year stockpile (now on hand)

Replacement Search and Test Strategy

Scoping the Replacement Program

- **Extensive literature search**
  - Previous testing references - program plans and data
  - Projected EPA restrictions
  - Replacement solvent developer data
  - Environmental Office – HCFC 225 users and quantities
  - ASTM Test Methods
- **Personal contacts and surveys**
  - Agency working groups
  - Local Environmental Office & Population-Environment Research Network (PERN)
  - Agency and commercial aerospace oxygen system operators/users
  - Solvent developers/venders
  - Propulsion hardware developers, cleaning contractors, etc
  - MSFC, SSC, and Michoud Assembly Facility (MAF) users

Scoping the Replacement Program

- **Identify solvent applications**
  - Review current applications and acceptance criteria
  - Identify any unique concerns / limitations
  - Review existing oxygen compatibility data
  - Review similar user application solutions
  - Solvent vender experiences
- **Test program definition**
  - Identify common and most critical materials
  - Identify contaminants that must be removed
  - Solvent candidates identified
  - Baseline characterization of solvent candidates
  - Pre-screen candidates for contaminant solvency
  - Material compatibility testing
  - Oxygen compatibility testing
  - Cleaning Effectiveness
  - MSFC and SSC operator's component level applications testing

Literature Search

•**SMC-TR-28 "*Nonvolatile Residue Solvent Replacement, March 1, 1995, Arnold, G,S. and Uht,J.C."***
•**Joint Test Report J-99-CL-015-R "*Validation of Alternatives to Ozone Depleting Chemicals Used in Oxygen Line Cleaning", Sept. 4, 2002, Engineering & Technical Services for Joint Group on Pollution Prevention.***
•**NAVSEA Project Number 72530/ *"Evaluation of Solvent Alternatives for Cleaning of Oxygen Systems, 1999." (Beeson, H:Biesinger, P. Delgado, R. Antin)***
•***"Elimination of Ozone Depleting Chemicals-Cleanliness Verification Alternatives", August 6-8, 1997, Aerospace Environmental Technology Conference; Douglas, V.***
•***ASTM G 127-95 (2008) "Standard Guide for the Selection of Cleaning Agent of Oxygen Systems".***

Solvent Replacement Project Experience

- Space Shuttle Program
  - Solid Rocket Motor Program - Ozone Depleting Chemical Effort
  - Shuttle Environmental Assurance efforts
- U.S. Army Research Laboratory, “Laboratory Evaluation of Alternatives to n-Propyl Bromide for Vapor Degreasing”
  - Four HFC/HFE azeotropes with tDCE were tested vs. nPB
  - Vertrel SDG, AE3000ATE, and Vertrel Sion (Azeo-A1) performed well.
  - ARL still seeking vapor degreasing solvents that are VOC exempt and low GWP.
- Defense Logistics Agency (DLA) - Aviation Hazardous Minimization and Green Products Branch, “Solvent Replacement for HCFC-225 for Cleaning Oxygen System Components”
  - DoD user customers include USAF, NAVSEA, and NAVAIR.
  - Propulsion system and breathing oxygen system cleaning
  - 3M L14780 (HFE7000/tDCE) and Honeywell Solstice PF (1233zd(E)) being tested versus AK-225cb and Capstone 4-I

Surveys - Replacement Community

Multiple NASA organizations are engaged in working groups/communities comprised of stakeholders in industrial cleaning and aerospace precision cleaning:

- MSFC, SSC, WSTF - Department of Defense Joint Service Solvent Substitution (JS3) Working Group (Army, Navy, Marines, EPA, NASA)
- MSFC, SSC, WSTF - NASA Precision Cleaning & Contamination Control (PC3) Working Group
- KSC, MSFC, SSC, WSTF - NASA Technology Evaluation for Environmental Risk Mitigation (TEERM) Principal Center
- MSFC/Jacobs - ASTM Committees:
  - **G4** on Compatibility and Sensitivity of materials in Oxygen Enriched Atmospheres
    - Task groups on cleaning oxygen systems
  - **E21** on Space Simulation and Applications of Space Technology
    - Subcommittee **E21.05** on Contamination
- MSFC - Established technical communications with key solvent suppliers at DuPont, 3M, Asahi Glass Co. (AGC Chemicals), Microcare, Petroferm

Considerations - Solvent Balancing Act

Safety, Health, and Environmental Requirements

Environmental

- ODP - ozone depleting potential
- VOC - volatile organic compound
- HAP – hazardous air pollutant
- GWP – global warming potential

Safety and Health

- Human Toxicity
- Flammability (human safety)

International Influence – Registration, Evaluation, Authorisation, and Restriction of Chemicals (REACH)

Performance Requirements and Cost Considerations

Materials Compatibility

- Nonmetals – swelling, cracking
- Metals – corrosion, Stress Corrosion Cracking (SCC)

Cleaning effectiveness

- Common contaminants- Greases, oils
- Effective cleaner (cold, flush, etc.)
- How is the solvent dried/removed?

Oxygen compatibility

Cost Considerations

- Cycle time (manufacturing schedule impact)
- Capital equipment
- Energy usage
- Solvent stability/recyclability/disposal

Restrictions are expected to increase with time

Applications - What Are Other User Solutions?

User	Hardware cleaned	Design solution	Limitations for MSFC/SSC
WSTF	Test equipment, life support systems	Aqueous ultrasound immersion	Ultrasound does not scale up for large items, cost, corrosion
KSC	Ground Support Equipment	Two step co-solvent process of HFE71DE followed by HFE7100 / Aqueous ultrasound	Cost and cycle time, not fully LOX compatible
PWR	Rocket engines	Cyclohexane/ Dry / Lock-up and Sniff Test	Highly flammable, safety precautions are costly
ULA	Rocket LOX tanks and components	Trichloroethylene (TCE) vapor degreasing – sealed system	Carcinogen, VOC, HAP, high capital equipment cost
MAF	External Tank LOX tank	Aqueous spray (tanks) w/TCE flush verification	Very high capital cost, TCE hazards
VACCO	LOX valves	Aqueous with AK225 or HFE7100 verification	Scale up/ corrosion, HFE7100 not very effective cleaner
Navy	Breathing oxygen	Navy Oxygen Cleaner (NOC) - aqueous	Scale up/ corrosion
Hamilton Sunstrand	Orion Environmental Control and Life Support System (ECLSS)	Vertrel MCA & HFE7100 co-solvent process	Cost and cycle time, scale up
USAF	Breathing oxygen systems	Perfluorbutyl iodide (PFBI)	Corrosive to aluminum

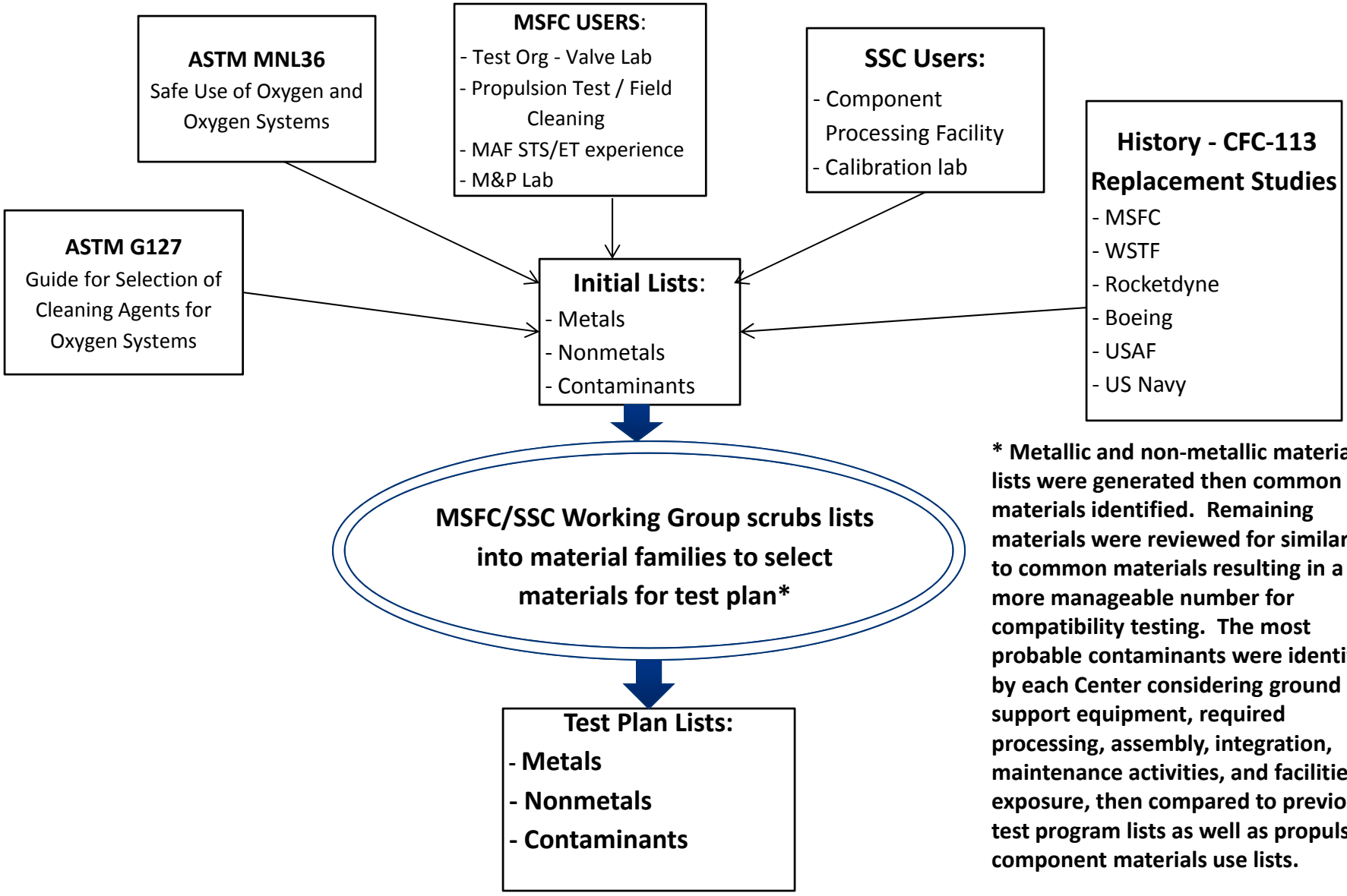
Drivers for Solvent Replacement Efforts

- Several issues are driving the need for cleaning solvent replacements at NASA and DoD:
  - HCFC-225 is a Class 2 ODS
    - No procurement or new use after 12/31/2014
    - Used mainly for cleaning of oxygen systems at MSFC & SSC
  - N-Propyl Bromide revised toxicity ratings
    - OSHA/NIOSH Hazard Alert issued 7/2013
    - Used mainly for vapor degreasing
    - Used by the MSFC ES43 Electronic Parts Fabrication Lab
  - Vertrel MCA groundwater contamination at KSC
    - KSC is seeking non-halogenated alternatives
  - Several currently used solvents are potent GHGs.
  - Many Centers are still using stockpiled CFC-113
    - Expensive to purchase and stock will eventually be depleted.

What Are Our Critical Selection Criteria?

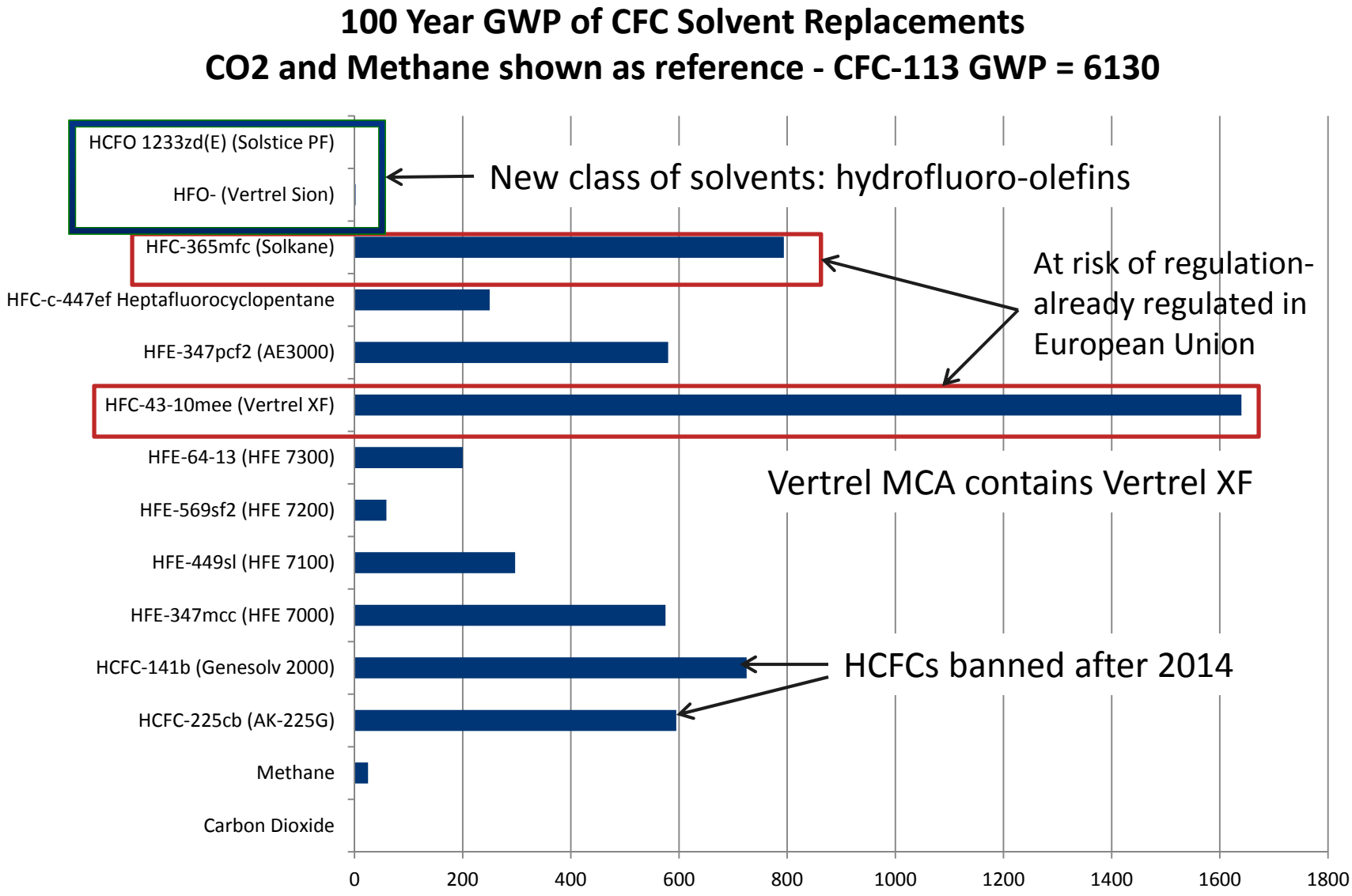
Must address:
1. Oxygen Compatibility / Flammability: Test of neat solvent and residue to ***NASA-STD-6001 13A / ASTM D2512.*** Auto-ignition temperature test per **ASTM G72** for GOX application.
2. Materials Compatibility: Non-metals *:* Embrittlement, Leaching, Crazing, Reversion , hydrolosis. ***Metals:*** Corrosion, Stress Corrosion Cracking, Embrittlement, Masking of Crack-like Indications. Characterize changes after immersion in candidate solvents at the use temperature.
3. Cleaning Effectiveness: Demonstrate effective removal of key contaminants to below 1 mg/ft^2 requirement (***MSFC-SPEC-164***) per **ASTM G 121- *Standard Test Method for Evaluating the Effectiveness of Cleaning Agents (modified)*** and end item configurations.
4. Processing Evaluation: Utilize candidate cleaners in component and field cleaning applications at the use site. Obtain support personnel opinions as to performance.

Materials and Contaminants Selection



*** Metallic and non-metallic material lists were generated then common materials identified. Remaining materials were reviewed for similarity to common materials resulting in a more manageable number for compatibility testing. The most probable contaminants were identified by each Center considering ground support equipment, required processing, assembly, integration, maintenance activities, and facilities exposure, then compared to previous test program lists as well as propulsion component materials use lists.**

High GWP Solvents are at risk of regulation



Most Probable Replacement Solvents

Single Component	KB*	AEL-8hr (ppm)	Caveats
AGC Chemical AE3000 (new) HFE-347pc-f2	13	50	Low KB may not clean well, toxicity
Honeywell Solstice PF (new) HFO-1233zd (E)	25	300	Boiling point of 66°F – requires pressurized containment, higher vapor losses expected
Dupont Capstone 4-I Perfluorobutyl iodide	No Data	375	Not compatible with Aluminum, expensive, short supply
Solvay Solkane 365mfc	14	1000	Unusual flammability characteristics, low KB, may not clean well
Azeotrope			
AGC Chemical AE3000AT (new) 45% tDCE / 55% AE3000	32	200/50	Expected to clean well, may not pass LOX test, toxicity
3M L-14780 (re-eval) 22% tDCE / 78% HFE-347mcc3 (3M HFE-7000)	Similar to MCA	200/250	Boiling point 82°F – higher vapor losses expected, toxicity, Performed well in past tests
Dupont Vertrel MCA (re-eval with new stabilizer) 38% tDCE / 62% HFC-43-10mee	20	200	Cleans well but borderline LOX compatible on past tests. Low AIT at high GOX pressure.
Solvay Solvokane (new) 30% tDCE / balance HFC-365 mfc	25	200/1000	Boiling point 97°F, individual components are flammable

**KB=kauri-butanol value per ASTM D1138, a measure of hydrocarbon solvent power*
Highlighted solvents leveraged from Defense Logistics Agency-Aviation test program

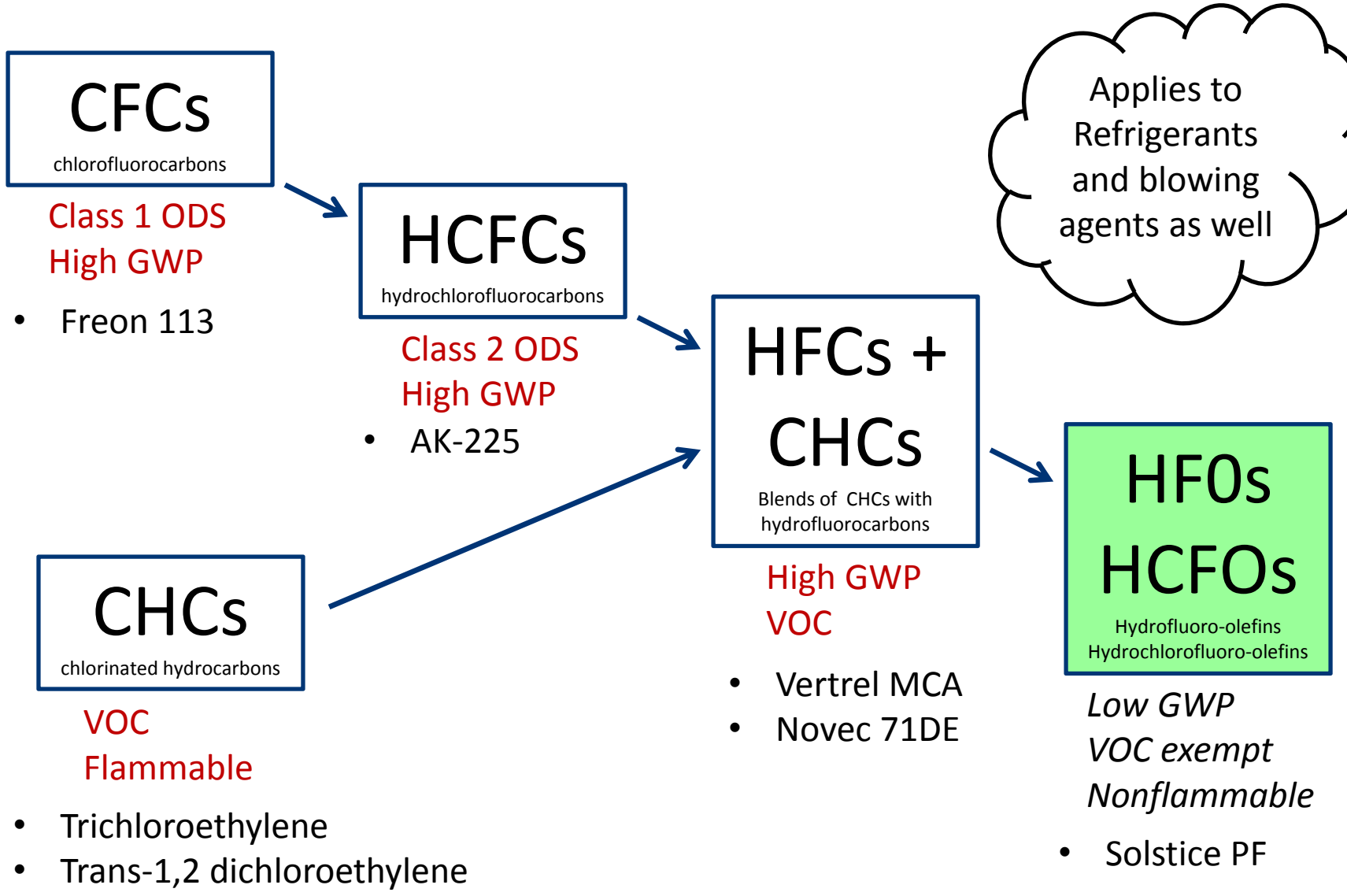
Solvent Candidates Regulatory Status

Solvent	ODS SNAP Approved	VOC Exempt	100 Yr GWP	Comments
AK-225G (HCFC-225cb)	No - Class II ODS	Yes	595*	NA after 2014
Honeywell Solstice PF (1233zd(E)) Trans-1-chloro-3,3,3,-trifluoroprop-1-ene	Yes	Yes	4.7 to 7	GWP per EPA site. Vendor claims < 1
3M L-14780 22% tDCE /78% HFE-347mcc3 (3M HFE-7000)	Yes	No**	575*	HFE7000 is VOC exempt
DuPont Vertrel MCA 38% tDCE/ 62% HFC-43-10mee 1,1,1,2,2,3,4,5,5,5-Decafluoropentane (Vertrel XF)	Yes	No**	1640*	Vertrel XF is VOC exempt
Solvay Solvokane 30% tDCE/70% HFC-365mfc 1,1,1,3,3 Pentafluorobutane (Solkane)	Yes	No**	794*	Solkane is VOC exempt

*GWP from the Fourth Assessment Report of the Intergovernmental Panel on Climate Change, 2007. GWP is for the non-tDCE portion of the solvent. Reference 100 year GWP for CO2 = 1

**These solvents are azeotropes with trans-1,2 dichloroethylene. tDCE has negligible GWP, but is NOT eligible for VOC exemption.

Evolution of Cleaning Solvents



Recent Solvent Development – HFOs & HCFOs

- The fluorocarbon industry has recently focused development on hydrofluoro-olefins for low GWP
- HFOs contain a double bond that breaks down in the atmosphere in a matter of days rather than years, yielding low greenhouse potential. HCFOs also contain 1 or more chlorine atoms
- DuPont and Honeywell have new HFO / HCFO refrigerants, blowing agents, and solvents.
- Solvents:
  - Honeywell Solstice PF (HCFO 1233zd(E))
  - DuPont Sion (HFO w/ ≈ 80% trans-1,2 dichloroethylene)

Testing Results Summary

What we've learned:

- The only "non-flammable", non-aqueous cleaners are halogenated.
- Bio-based solvents are flammable VOCs.
- Most new fluorinated solvents are poor cleaners.
  - DuPont Vertrel XF and HFX, 3M Novec HFEs, Asahi AE3000, Solvay Solkane.
  - These are blended with trans-1,2 dichloroethylene (tDCE) for cleaning power. tDCE is a VOC.
  - Blending suppresses the flammability of tDCE.
  - Also blended with alcohols for flux removal.
  - Only azeotropic blends are stable in composition over time.
  - Blends high in tDCE% are not oxygen compatible.
- Many fluorinated solvents have very high GWP.

Sustainability

- Efforts to qualify replacement solvents support the **NASA Strategic Sustainability Performance Plan:**
  - **Goal 1: Greenhouse Gas Reduction** by seeking solvent alternatives that will reduce Scope 1 Greenhouse Gas (GHG) emissions.
  - **Goal 8: Agency Innovation and Government-Wide Support** by collaborating with multiple NASA Centers and DoD agencies to test and qualify safer and greener cleaning solvent alternatives for numerous federal applications.

Look Ahead

- Bigger Picture / Materials Obsolescence in the future
- Currently no Agency wide materials obsolescence working group
- The SLS Program has a new effort with emphasis on materials obsolescence and environmental regulations.
  - Team is smaller than SEA.
  - Less opportunity for inter-element collaboration on testing of replacement materials.

BACKUP

SSC HCFC 225 Stockpile/Requirements

- SSC does not maintain a stockpile of HCFC 225
  - 7 drums are maintained in the warehouse for use (current verified supply)
  - Replacement drums are purchased to replenish supply as stock is used
- Annual SSC HCFC 225 usage ~ 12-14 drums
- SSC is requesting a five year usage stockpile ~70 drums, should cover needs through 2019
- Required Funding: $1M*
  - Includes costs for 70 drums, long term storage barrels (one time purchase), material sampling, and distillation system maintenance/ops
- Five year stockpile allows time for evaluation and implementation of replacement alternative(s)
  - Update procedures/processes
  - Procure capital equipment
  - Train personnel

* Costs based on present day HCFC 225 market price, may increase as phase out approaches

References

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2. Allen, Gale J., and Fishell, Kenneth A., *Aqueous Cleaning and Verification Processes for Precision Cleaning of Small Parts*, Proceedings of the Aerospace Environmental Technology Conference, Huntsville, AL, March, 1996, NASA Conference Publication 3298.
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4. MIL-DTL-24800, *Cleaning Compound – Aqueous – Oxygen System Components*, Naval Sea Systems Command, Department of the Navy, September 30, 1996.

   Note: This compound is commonly referred to as Navy Oxygen Cleaner (NOC).
5. MIL-STD-1330D(SH) w/Change 1, *Precision Cleaning and testing of Shipboard Oxygen, Helium, Helium-Oxygen, Nitrogen, and Hydrogen Systems*, Naval Sea Systems Command, Department of the Navy, June 28, 2007.
6. 3M™ Novec™ 7100 Engineered Fluid, methoxy-nonafluorobutane, proprietary solvent, 3M Electronics Markets Materials Division, 3m Center, St. Paul, MN, www.3M.com.
7. KSC-C-123J, *Surface Cleanliness of Ground Support Equipment Fluid Systems, Specification for*, NASA John F. Kennedy Space Center, July 17, 2009.
8. Hesselroth, David A., HFEs in the Aerospace Industry: A Logical Alternative to CFCs and other Halogenated Cleaning Solvents, Proceedings of the 4th Conference on Aerospace Materials, Processes, and Environmental Technology, Huntsville, AL, September 2000, NASA/CP-2001-210417.
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10. Beeson, Harold, Beisinger, P, Delgado, R., and Antin, N., *Evaluation of Solvent Alternatives for Cleaning of Oxygen Systems*, Huntsville, AL, Proceedings of the Third Aerospace Environmental Technology Conference, Huntsville, AL, June 1998, NASA/CP-1999-209258.

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12. M&P-3-1667, Eichinger, Eric, *Evaluation of Solvent Cleaners for Oxygen Systems*, Boeing-Huntington Beach Materials & Processes Laboratory Report, 13 September 2005.
13. Campbell, Mike*, Implementation of Precision Verification Solvents on the External Tank*, Proceedings of the Third Aerospace Environmental Technology Conference, Huntsville, AL, June 1998, NASA/CP-1999-209258.

    Note: At the time of this publication, M. Campbell planned to replace TCE with HCFC-225 for verification of the LOX Tank. This was not done because it was found that HCFC-225 was too volatile for capture of an adequate NVR sample in the LOX Tank. Reference: Personal communication with Mike Campbell, March 3, 2008.
14. Environmental Protection Agency (EPA) Integrated Risk Information System (IRIS), Substance Code: 0199, Trichloroethylene; CASRN 79-01-6; 09/28/2011.
15. National Emission Standards for Hazardous Air Pollutants (NESHAP): Halogenated Solvent Cleaning, 59 FR 61801.
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Alternatives

Alternative Approach	Limitations
Aqueous ultrasonic agitation with verification of NVR by analysis of Total Organic Content. Reference ASTM G 144 [1] - This test method requires full component immersion and ultrasonic agitiation in a tank of deionized water. - Used by NASA-WSTF, NASA-KSC, and Pratt & Whitney Rocketdyne (PWR) for cleaning and verification of small components.	• Ultrasound does not scale up for large components. This test method is limited to components that are both smaller than one square foot (0.09 m^2) of surface area and weigh less than 3.3 lbs (1.5 Kg). [2][3] • Not feasible for components installed on test stands and for long tubes and feedlines that will not fit into an immersion tank. • Water is corrosive to components where complete drying cannot be assured
Aqueous cleaning by immersion, ultrasonic, spray or flush with an alkaline cleaning solution (Navy Oxygen Cleaner, MIL-DTL-24800 [4]) Reference MIL-STD-1330 [5] - Used by US Naval Sea Systems Command for cleaning of approved shipboard oxygen systems and shore base facilities.	• Not suitable for final cleaning of instruments and gauges such as flowmeters and dead-end pressure switches and transducers, liquid oxygen generating plants and plant piping; not suitable for field wiping (reference MIL-STD-1330 [5] sections 4.4.1.1, 5.4, 5.8.2, and Table E-I)

Alternatives

Alternative Approach	Limitations
Two step process: Clean with a non-oxygen – compatible solvent or aqueous agent, rinse and verify with nonflammable solvent (3M Novec 7100 [6]) - Used by NAVSEA (MIL-STD-1330 [4]), NASA-KSC (KSC-C-123 [7]), Royal Australian Air Force [8]	• Costly, requires additional equipment and doubles required solvent stocks • Less reliable verification, higher risk. Novec 7100 is a poor solvent for hydrocarbon oils and greases including silicone and must be used with mechanical agitation or pressure, may not detect all specifies of contaminants (references [5] Table E-I note 5, [8], [9], [10])
Use flammable solvents such as cyclohexane, ethyl acetate, or isopropyl alcohol, for cleaning or for verification after cleaning. - Used by PWR [3] [11]	• High risk where complete drying to remove solvent cannot be assured or where processes such as vacuum oven drying are not feasible [3] [11]
Clean and/or verify with perfluorbutyl iodide (PFBI) - Qualified by USAF for depot cleaning of aviator's breathing oxygen systems. (There is no aluminum in these high pressure systems.)	• PFBI is corrosive to aluminum, an important light weight metal in for launch vehicle LOX tanks and housings. [12] Corrosion testing is now in progress at MSFC to determine the extent of this risk.

Alternatives

Alternative Approach	Limitations
Use trichloroethylene (TCE) for cleaning or for verification after cleaning. • Was used for the Space Shuttle External Tank LOX tank cleanliness verification at the NASA Michoud Assembly Facility until program cancellation. [13] • Reported to be used for LOX tank vapor degreasing at United Launch Alliance.	• Carcinogen [14], Hazardous Air Pollutant [15]. Unacceptable worker exposure expected and unacceptable release during field cleaning operations. • Will self-ignite in 2000 psi pressurized oxygen when temperature reaches 77^{o}C (170.6^{o}F). [10]. Not safe for pressurized systems.
Clean and/or verify with stockpiled CFC-113, a Class I ODS.	• No current stockpile at MSFC or SSC. • Available stockpiles may not be sufficient, losses will occur and deplete stock. • Not a long term solution.
Clean with an alternative non-ODS solvent that meets all performance criteria for oxygen compatibility, cleaning efficiency, and materials compatibility. - No Users.	• No suitable alternative has been identified to date for MSFC/SSC propulsion test oxygen system applications. [12][16][17]

National Aeronautics and Space Administration

EM50 Solvent Replacement Efforts

MSFC Green Team - *January 22, 2014*

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