Solstice PF Spray Cleaner (Aerosol) for Cleaning of LOX/GOX Components

RPT PMR at KSC February 14 – 15th, 2017

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Photo: NASA/SSC

SUMMARY

- **Acknowledgements**
- **Background**
- **Neat Solstice PF Spray NVR Testing**
- **Oxygen Reactivity Tests - WSTF**
  - Mechanical Impact Testing
  - Autogenous Ignition Temperature Testing
  - Heat of Combustion Testing
- **Laboratory Testing - SSC**
- **Nonvolatile Residue Removal Efficiency Test - MSFC**
- **On Site Vendor Demonstration**
  - **MSFC**
  - **SSC**
- **Conclusions**

Acknowledgements

- **Rocket Propulsion Test (RPT) Program for funding this test effort**
  - Roger Simpson (RPT Manager), Tim White (SSC-EA40), and a host of others
- **MSFC**
  - EM22 Contamination Team: Mark Mitchell (Project Lead), Kevin Edwards, Buford Moore, and Barry Morris
  - Budget Personnel: Nancy McNeill (ED11), Jim Smith (EM01), Tenina Bili (EM01), Trista Guthrie (EM01)
  - Test Area and Valve and Component Shop: Brannon Standridge (AS24), Nick Hensley (ET10), Tim Gautney (ET10), Bobby Hubbard (AS24), James Thomas (AS24), Judson Hudson (AS24), and a host of others
- **SSC**
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- **WSTF**
  - Susana Harper, Fred Juarez, John Bouvet, Steve Peralta, Christina Pina Arpin
- **Langley Research Center**
  - Steve Gentz - NESC-MSFC Chief Engineer
  - Mike Smiles - NESC-SSC Chief Engineer

BACKGROUND

- Statement of work entitled “FluoroSolv Testing” was funded by RPT in 2015
  - Tested several aerosol cans of neat FluoroSolv K1016A, which is 80% Honeywell Solstice PF (1233 ZDE) plus 20% Honeywell Propellant (1234 ZE), packaged by Miller Stephenson
  - Every can tested failed to meet the NASA non-volatile residue (NVR) requirement of <10ppm
- Discussed issue with Honeywell and Miller Stephenson
- Located other packagers that were marketing the 80/20 product (or willing to package for our testing)
  - Zip Chem Sure Prep 123 Aerosol
  - Microcare
  - KYZEN (Solstice PF pressurized with nitrogen)
- All aerosol cans failed to meet the NASA NVR requirement
- Project team decided to test the 80/20 aerosol product directly from Honeywell packaged in a cleaned 50lb cylinder, which met the tested NASA NVR requirements
  - Testing the aerosol product will provide all information necessary for use of the aerosol itself with our oxygen systems
  - If packagers provide a 80/20 product that NASA would like to use, NVR testing is all that is necessary to determine if it can be used with NASA systems
  - the FluoroSolv project name was really no longer applicable at this point since this was a particular packager of the product and it did not pass our NVR requirements

SOLVENT (NEAT) NVR REPORT - SSC

NVR reported by Honeywell for Each 50 lb. cylinder of Solstice PF Spray sent to White Sands Test Facility, Marshal Space Flight Center, and Stennis Space Center. NVR qualification conducted at MSFC & SSC.

Honeywell				Buffalo Research Laboratory			
Analyzed by:	Jason E. Lund				**Date Completed:**	5/12/16	
Customer:	Len Stachura				**Project Number:**	330303	
Material Tested:	50 lb. jugs of 1233zde (Pf-HP) + 1234ze for NASA						
Ticket Number:	27658				**Notebook / Page Number:**		42244 / 6

Total Residue (ppm by weight)

sample ID.	AR#	Gross	Tare	Sample	residue	ppm	+/-
42208-37-A	71322	299.16	0.00	299.16	0.0001	0.33	0.33
42208-37-B	71323	301.92	0.00	301.92	0.0005	1.66	0.33
42208-37-C	71324	313.21	0.00	313.21	0.0002	0.64	0.32

NOTE: 1. Total Residue determined using Method GP GEN-8C.

2. Precision determined using 0.0001 g. residue scale resolution devided by sample weight.

Stennis Space Center

Total Residue (mg / 200 ml)

Test Method	Test Run	NVR Results
Gravimetric	#1	0.5 mg / 200 ml
Gravimetric	#2	0.5 mg / 200 ml
Gravimetric	#3	0.5 mg / 200 ml
FTIR	(25 Runs co added)	< 0.5 mg / 200 ml

Honeywell Sample ID 42208-37-C
(Sample tested @ SSC)

Solvent Filter: Supplier –TEM, Part No. -TEM 911T
Filter Material - Two layers of Teflon PTFE Membrane in a 316SST Electro-polished Interior Housing. Effective Filter Area -19.75 cm2; > 99.9999% Efficient @ 0.003 micrometers

OXYGEN COMPATIBILITY TESTING

Oxygen Compatibility Tests Were Performed at WSTF June 6-12, 2016

- Retested Solstice PF (trans 1-Chloro, 3,3,3 trifluoroproplene)
- Analyze Solstice PF with Propellant (80% trans 1-Chloro, 3,3,3 trifluoroproplene & 20% 1,3,3,3 tetrafluoropropene)

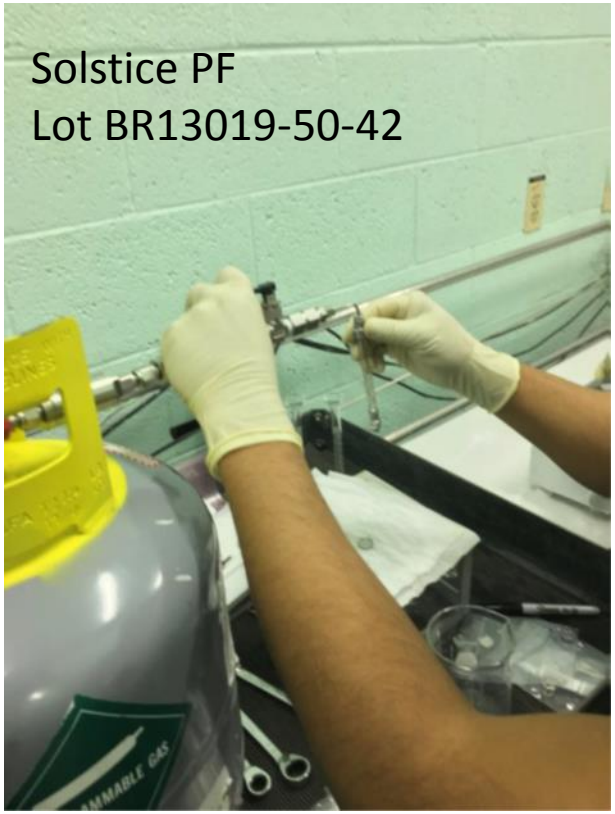
13A, LOX Impact Testing at Ambient Pressure Using Modified Test Protocols

- LOX Impact tests completed in June thru Aug of 2015 were repeated from the same cylinder to assess the repeatability of the modified 13A test protocol

ASTM G72/G72M-15, Standard Test Method for Autogenous Ignition Temperature (AIT) of Liquids and Solids in a High Pressure Oxygen-Enriched Environment. Note: Revised method incorporates increases in sample size for improved test resolution capability.

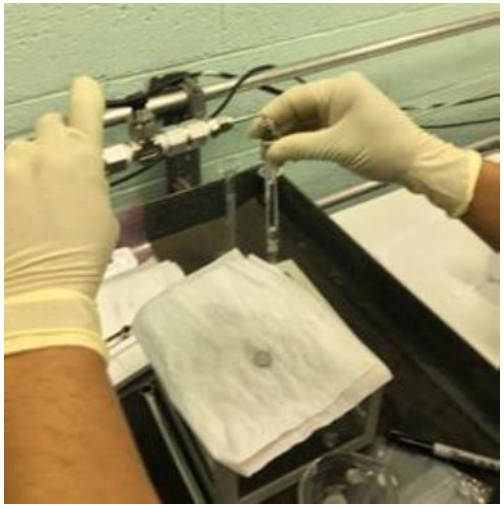
ASTM D240, Heat of Combustion (HOC) Testing

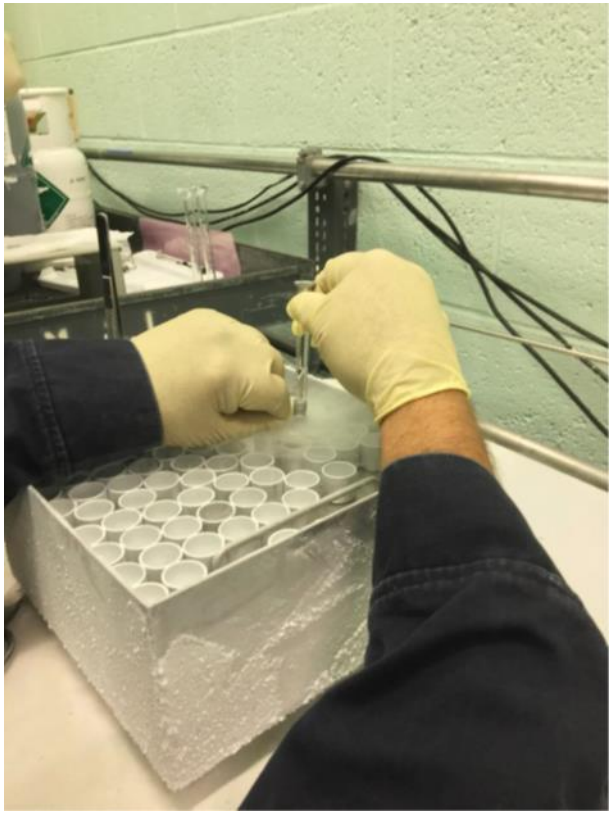
DISPENSING OF SOLSTICE PF & SPRAY PRIOR TO LOX SOAK



Filtered solvent is dispensed directly from the DOT container into a syringe equipped w/ a gas tight outlet valve via 0.003µ filter. (Filters changed out after each test)

Toggle valve provides precise flow control for delivering the solvent to the syringe. The syringe plunger is not used for filling the syringe and for dispensing the solvent.





0.4ml of solvent is measured into each aluminum cup. The cups are placed on a SST shelf that sits slightly above the LOX level.

LOX TEST SUMMARY

Standard Solstice PF and Spray tests for Heat of Combustion, Autogenous Ignition Temperature, and Mechanical Impact Testing were equivalent to the June 2015 results.

		Solstice (Propellant Version) Honeywell Lab ID 42208-37-B					Solstice PF Honeywell Lot BR 13019-50-42					Historical Data Solstice PF	
		Run 1	Run 2	Run 3	Mean	Stand. Dev.	Run 1	Run 2	Run 3	Mean	Stand. Dev.	Mean	Stand. Dev.
		(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)	(cal/g)
HOC		2250.3	2294.7	2346.3	2297.1	39.2	2311.4	2240.0	2304.3	2285.2	32.1	2447.7	22.4
		(˚F)	(˚F)	(˚F)	(˚F)	(˚F)	(˚F)	(˚F)	(˚F)	(˚F)	(˚F)	(˚F)	(˚F)
AIT (2ksi)	0.2 grams	TL	--	--	--	--	--	--	--	--	--	--	--
	0.5	TL	--	--	--	--	--	--	--	--	--	--	--
	0.7	402	--	--	--	--	--	--	--	--	--	--	--
	1	405	394	410	403	7	367	391	368	375	11	360	5
AIT (50 psia)	0.2	--	--	--	--	--	--	--	--	--	--	--	--
	0.5	--	--	--	--	--	--	--	--	--	--	--	--
	0.7	--	--	--	--	--	--	--	--	--	--	--	--
	1	456	472	492	473	15	463	497	461	474	17	464	16
13A Modified		0/60 (~45% RH/ 49-50 ⁰ F) [1]					0/60 (~45% RH / 49-50 ⁰ F) [1]					0/60 (10-15% RH) [1]	

[1]Test 13A (Modified) –Solvent was dispensed directly into the tall aluminum cups (no insert sample cups were used). A plummet catcher was used to eliminate rebound (uncontrolled) impacts.

NVR REMOVAL EFFICIENCY TEST

- Cleaning Effectiveness evaluated the efficiency of Solstice PF Spray to remove specific contaminants in ambient temperature. This method evaluated cleaning solvency without the use of heat or any additional mechanical action beyond the force of the aerosol spray. After spraying ~ 200 grams Solstice PF Spray, the sample was flushed with 200 ml AK-225G to liberate any remnant contaminants, thereby quantifying cleaning efficiency.
- Contaminant removal efficiency was compared to AK225G and previous results for standard Solstice PF.
- The following contaminants were tested:

1. Mineral Oil: RP-1 fuel, petroleum-based hydraulic & motor oil (CAS 8042-47-5)
2. Mobil DTE 25: Petroleum-based machine tool hydraulic fluid, ISO grade 46
3. Castrol Brayco Micronic® 882: Synthetic hydraulic fluid, MIL-PRF-83282
4. Di-2-Ethylhexyl Sebacate: Gauge calibration oil - Monoplex® DOS
5. Synthetic Sebum: Simulated sebaceous oils from skin
6. Fluorocarbon Grease: Krytox® 240AC
7. Fluorocarbon Grease: Christo-Lube
8. Big Red: Heavy paraffinic crane grease
9. WD-40: Penetrating oil (medium-heavy aliphatic hydrocarbons)

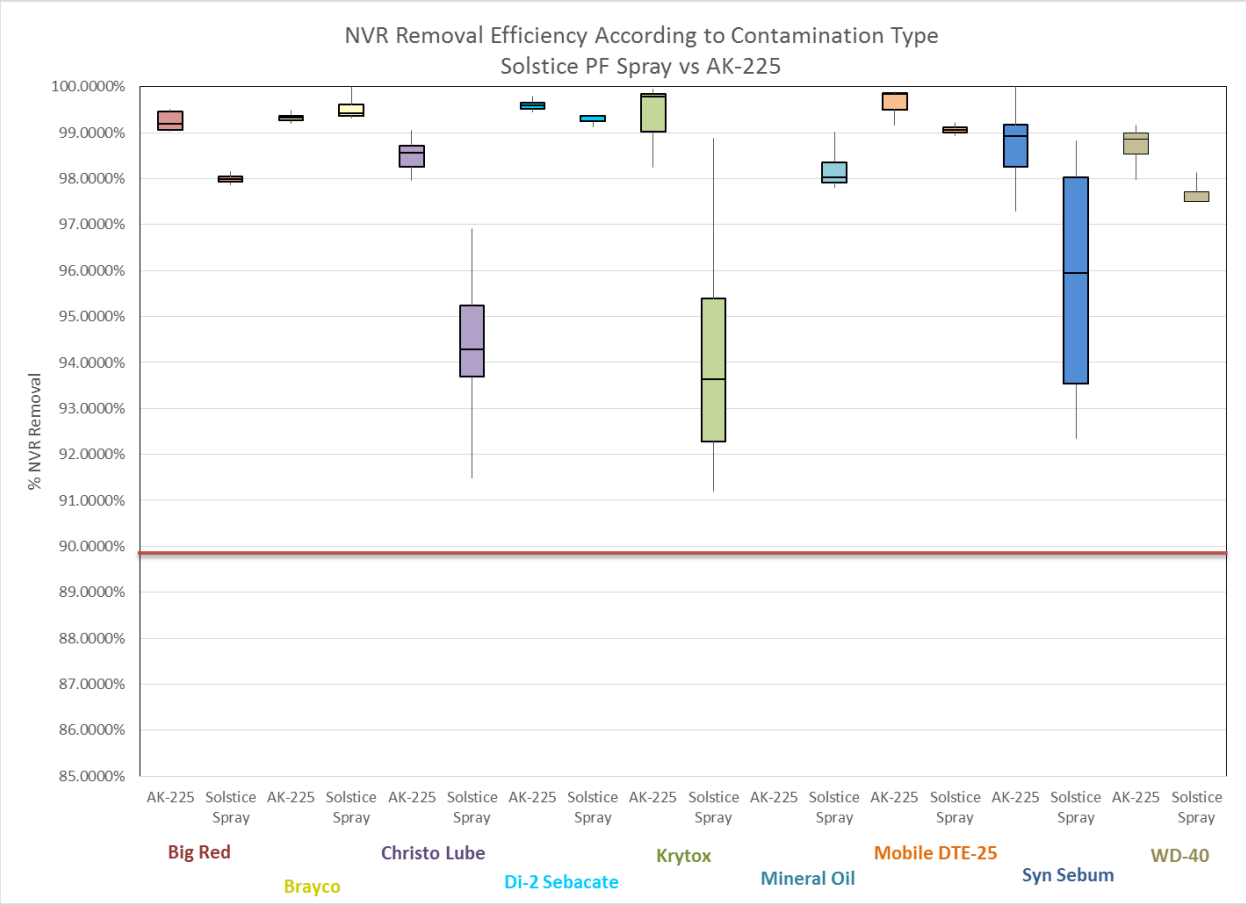
NVR REMOVAL TEST RESULTS

Solstice PF Spray and AK225 (used as the control) removed > 90% of NVR for the nine contaminant types tested.

Both solvents were statistically equivalent in removing Brayco Micronic 882, D-2 Sebacate, and Mobil DTE-25.

With a larger sample size, Big Red, Mineral Oil, & WD-40 would most likely be equivalent as well.

AK225 was more efficient than Solstice PF Spray when removing Christo Lube, Krytox, and Synthetic Sebum.

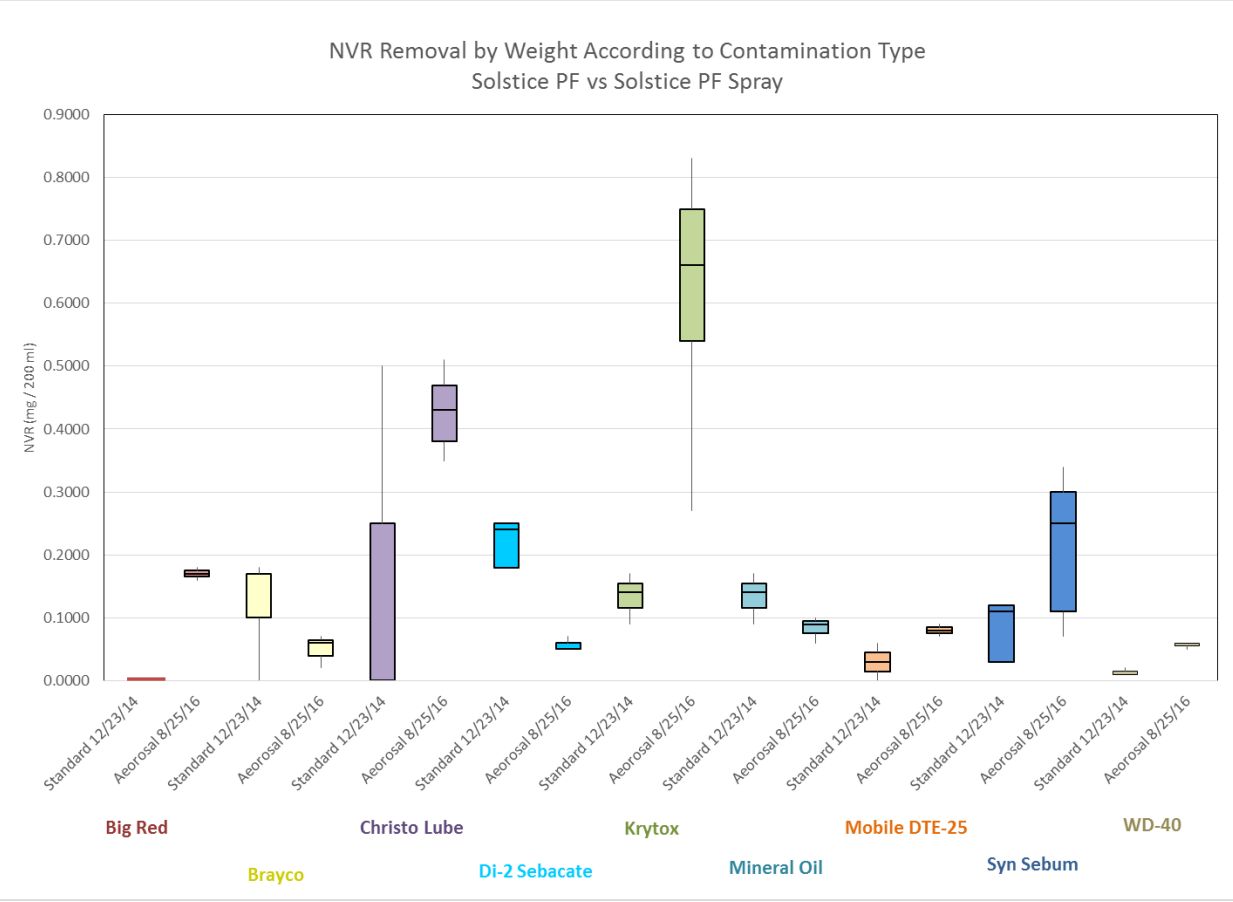


SOLSTICE PF & PF SPRAY VARIATION

Solstice PF Spray to Standard Solstice PF yielded similar results. Observed differences are most likely the result of low sample numbers and variations in actual contamination loading.

Note: The 1st Rinse, when using Standard Solstice PF, was captured and used to validate contaminant loading.

Solstice PF Spray, I.e. the 1st Rinse, was not collected on any of the tests as Splash back and evaporation of the solvent made this impossible.



SOLSTICE PF SPRAY FIELD TEST- MSFC

Solstice PF Spray was evaluated in the MSFC test area using raw LOX fittings that had not been cleaned. Typically, Sure Shot sprayers are used to dispense low pressure solvent before dismantling the part.

Observations:

- LOX fittings were heavily contaminated.
- Technicians prefer low pressure dispensing method over high pressure when breaking connections in the field.
- Solstice PF Spray removed Krytox grease from fittings to a visibly clean state.
- However, a final degreaser process would be preferred for heavily contaminated parts that are pre-cleaned.
- Pressure relief and flow control on the Solstice PF Spray delivery system would be advantageous.

LOX Fitting with Contamination

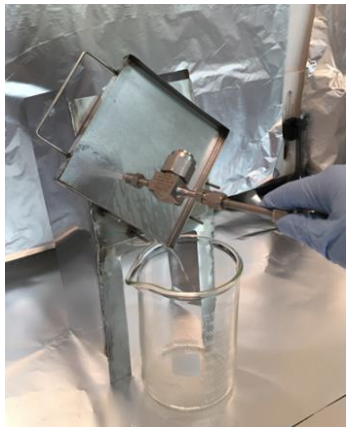
Sure Shot Can and LOX Fittings

SOLSTICE PF SPRAY FIELD TEST- MSFC

Following the MSFC Field Test, parts were taken back to the lab to evaluate heavy contaminant loading cleaning capability using the Solstice PF Spray System.

- Big Red was selected as a result of the anticipated challenges of removing a high density / viscosity contaminant.
- Solstice PF Spray clearly removed contaminant as observed in the color change in the captured effluent. (The initial effluent stream was orange from the tubing used on the hand wand. It was later determined that the solvent was leaching the tubing, which resulted in a tubing material compatibility test and change out.)
- Following cleaning, a contamination film still remained. This was easily removable using a swab and indicates that additional optimization / cleaning action may be required depending on the contamination type and level of contamination.

Sample Tray coated with Big Red



Spray activity using Solstice PF Spray

Contaminated Big Red / Solstice Spray effluent

Removal of Contamination Film following Solstice PF Spray and Swab

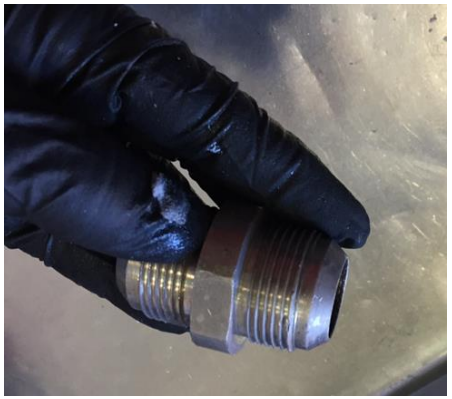
SOLSTICE PF SPRAY FIELD TEST - SSC

Solstice PF Spray was also evaluated at the SSC LOX Cleaning facility where the following observations were made:

- The LOX fittings tested were relatively clean. Solstice PF Spray easily removed Krytox that was on the threaded region of the fittings.
- Particles and oily residue were observed in the catch pan following cleaning.
- Solstice PF Spray delivery system final recommendations:

1) Pressure regulator / flow control to control spray.
2) Bleed off to drain solvent from line.
3) Change tubing material for compatibility. Utilize flared fittings, not metal ferrules, for any non-metallic tubing to prevent leaks. (Recommend SST Teflon lined hose.)
4) Safety on gun to prevent inadvertent spray action.

Solstice PF Spray Cleaning of LOX Fittings



LOX fitting post Solstice PF Spray Clean

Solstice PF Spray Delivery System Setup

SOLSTICE PF SPRAY FIELD TEST - SSC

Solstice PF Spray field cleaning was also conducted outside on a 10’ section of LOX piping where the ambient temperature exceeded 90° F. The pipe was 3” in diameter and was slanted during cleaning to aid in drainage.

Observations:

- The pipe cooled when the solvent first touched it. The solvent would vaporize until the temperature of the pipe was below the solvent’s boiling, at which point the solvent would remain in it’s liquid form.
- Metal particulate and residue were easily flushed from the pipe.

LOX Piping used in Solstice PF Spray Field Cleaning Test

Particulate and Oily Contamination removed from the LOX piping during the Solstice PF Spray Field Cleaning Test

SUMMARY / RECOMMENDATIONS

- Solstice PF Spray has been qualified as comparable to Standard Solstice PF and AK225G in the removal of typical aerospace contaminants.
- Utilize Solstice PF Spray for applications where preferable over use of Standard Solstice PF.
- MSFC-Spec-3709 is the material spec that will be used for purchase of Solstice PF. It is now approved and includes nitrogen as an approved pressurant and 1234 ZE as an approved propellant at 20% concentrations.
- The grade is specified based on the allowable level of nonvolatile residue (NVR) contamination in the solvent as follows:
  - Grade A – High Purity (HP), low NVR level for precision cleaning and NVR verification sampling.
  - Grade B – Standard Purity, commercial standard NVR level for general use. May be packaged and distributed by KYZEN under product name Metalnox® 6920.
- ASTM G04 committees are in the process of revising test methods for LOX impact & AIT testing for solvents based on this project's developmental work.
- Solstice PF implementation is pending. All implementation costs would require additional funding. This will be addressed in a supplemental presentation.

No claim is made here regarding the safety or efficacy of Honeywell Solstice™ PF Spray with materials or contaminants other than those tested.

No claim is made here regarding the suitability of Honeywell Solstice™ PF Spray for use with breathing oxygen systems.

Any Questions?

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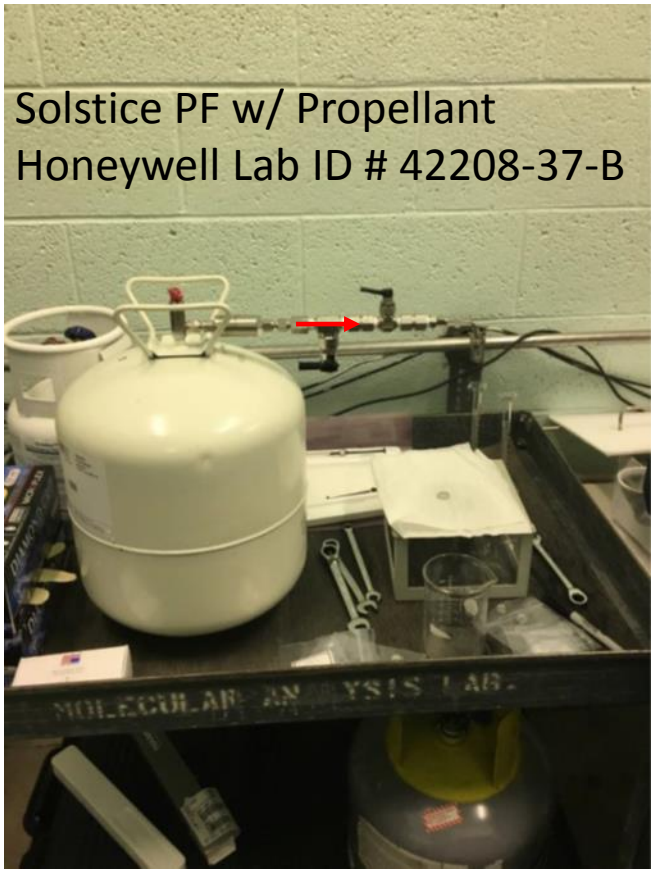
Back-Up Charts

SOLSTICE PF SPRAY DISPENSING

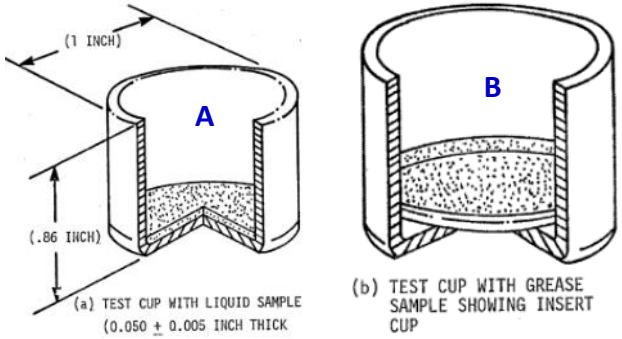


Solvent flows through a 0.003 micron filter as shown by the red arrow.

Filtered solvent is dispensed directly from the cylinder into a syringe equipped with a gas tight valve at the outlet.

LOX MODIFIED TEST PROTOCOL

LOX testing methodology was modified to eliminate variations that were observed in 2015 AK225 replacement study analysis.



Solvent samples were dispensed into the tall aluminum sample cup (**A**). The insert sample (grease) cups were not used for the modified tests (**B**).

- All impacts were near dead center to eliminate side wall impacts.
- Eliminating rebound impacts reduced the different point loadings.
- Excluding the insert sample cups eliminated any edge promoted reactions due to the plummet striking the raised edge (rim and rim wall) of the insert sample cup. Also, movement of the insert sample cup after placement in the impact tester would often result in a non-centric impact.
- Removing the insert sample cups also minimized the %RH effects (atmospheric moisture) during sample preparation. At high humidity, frost would accumulate on the rim of the insert cup and could quench reactions

IR DISCUSSION / RECOMMENDATION

- The IR solvent vapors were collected in a 10cm gas cell
- The gas cell available for this study has a Pyrex body and is designed to operate at 1 atmosphere. Since the propellant has a significantly higher vapor pressure and a much lower boiling point than Solstice PF, the propellant component escaped quickly from the gas cell. Consequently, the propellant spectral peak observed at 1094 can vary greatly in intensity as a function of time after collecting the solvent vapor.
  - If the spectrum is not acquired immediately after introducing the propellant vapor into the cell, the absorbance band at 1094 can disappear. To obtain reproducible spectra, the vapors would have to be captured in a HP gas cell to prevent the solvent vapors from escaping.
  - The best option is to obtain an IR spectrum of the solvent as a liquid in a HP short path length cell. The solvent would flow through the HP liquid cell from the solvent cylinder. After a liquid flow is established through the cell, the inlet and outlet valves on the cell would be closed. The solvent would be contained as a liquid while obtaining the IR spectrum. This option would provide the most reproducible results with greatly improved sensitivity. This cell would have to be purchased to provide the additional test data.

SEALED LIQUID CELL FOR IR ANALYSIS -SSC

- High Pressure Liquid Cell for the spectroscopic analysis of liquid samples under conditions of high pressure and from sub-ambient temperatures (-15°C) to 180°C would be ideal.
- The High Pressure Liquid Cell manufactured in EN58 stainless steel for chemical resistance and durability, with a choice of Zinc Selenide, Sapphire, and BaF2. These windows are permanently sealed in their window housing assemblies using perfluoroelastomer O-Rings. The cell path length is fixed at0.5, 1.0, 2.0, 5.0, or 10 mm as standard, and two 1/16” stainless steel flow tubes are brazed to the cell body to introduce the sample. Valves can be added to the flow tubes.
- The High Pressure Liquid Cell can be used for ambient temperature studies when mounted on the standard 3'' x 2'' back plate (provided as standard) inside an infrared spectrometer. For heated or sub-ambient applications, the cell can be used in conjunction with the Specac Variable Temperature Cell Holder (GS21525), Electrically Heated Jacket (GS20730), or Water Heating Jacket (GS20710).

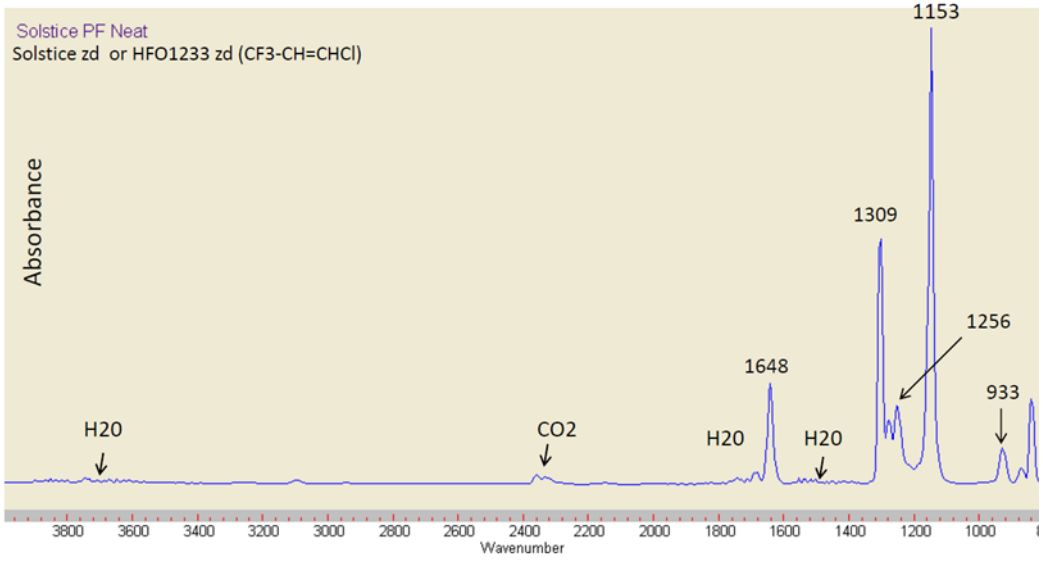
High Pressure Liquid Cell for Spectroscopic Analysis

FTIR VAPOR SPECTRUM

Solstice PF Neat

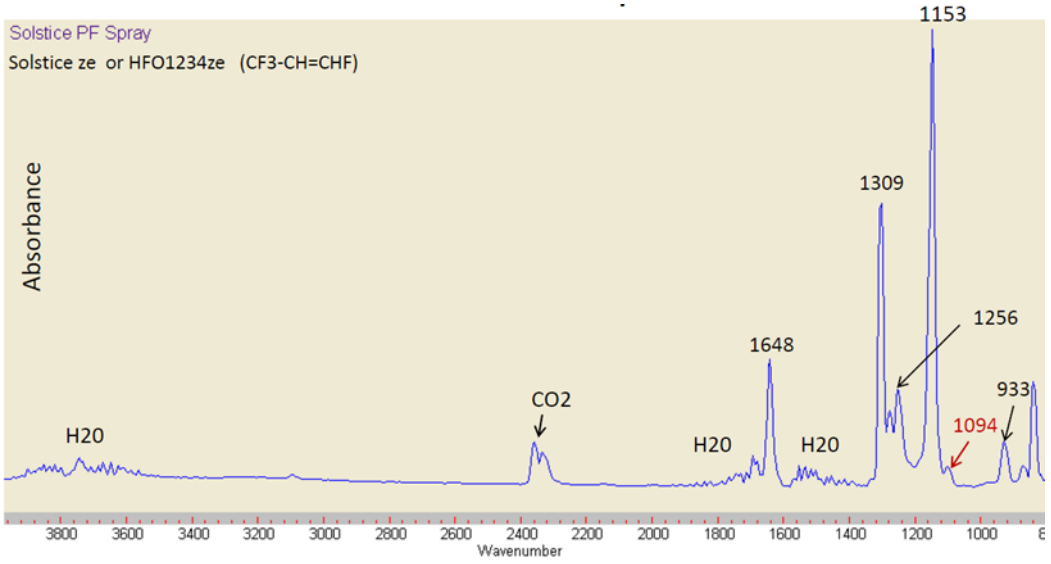
FTIR spectrum was obtained in a 10 cm gas cell equipped with BaF2 windows.

The CO2 doublet and the H2O fringe lines are spectral artifacts from the atmosphere.



Solstice PF Spray

The only difference in the spectra is a minor absorbance band at 1094, which is associated with the HFO 1234ze (propellant).



LOX IMPACT RESULTS @ 72 FT. LBS

- Test results of the Solstice PF Spray & Standard Solstice PF are equivalent to the current and historical tests.

Solvent / Lot ID #	Test Method	Test Facility	Reaction Proportion	% RH During Sample Preparation
	(Categorical Variable)	(Categorical)	(Frequency)	
Solstice PF Spray Test Run		*June 9, 2016*		
Solstice PF w/ 20% Propellent, 42208-37-B	D2512-82 (Revised)	WSTF	0 / 60	45-47%
Solstice PF, BR13019-50-42	D2512-82 (Revised)	WSTF	0 / 60	45-47%
Previous Tests		*June - August 2015*		
Solstice PF, BR13019-50-42	D2512-82 (Revised)	WSTF	0 / 60	10-15%
	D2512-82 (Revised)	MSFC	0 / 60	50-55%
	G86-98	WSTF	5 / 65	10-15%
	G86-98	MSFC	3 / 60	50-55%
	G86-98 Modified	WSTF	3 / 60	10-15%
	G86-98 Modified	MSFC	2 / 60	50-55%

- G86-98 used insert sample cups to sample liquids. The former method, D2512-82 did not, thus reducing any edge promoted reactions and minimizing the influence of relative humidity that can quench the reactivity.
- A plummet catcher was used with the revised procedure to eliminate rebound (uncontrolled) impacts.
- No historical test data is available for Solstice PF with the Propellant.

SOLSTICE PF TEST DATA SHEET

Test 13A (Modified) – Solvent was dispensed directly into the tall aluminum cups (no insert sample cups were used). Also, the plummet catcher was used to eliminate rebound (uncontrolled) impacts.

ASTM G86 TEST DATA SHEET - NASA-STD-6001.B TEST 13

WSTSF No. 16-46885 B
Material: 16 Solstice
Project TON 5506
Test Pressure: 12.4 psia
Test Temperature: ____ °F
Test Cell: 102

Test Sample Dimensions: Diameter: ____ in. Thickness: ____ in.
Weight: ____ g Other: ____
Test Media Constituents: 1,1,2 TCE
Phase: Liquid: ✓ Gas: ____
Prep RH: Start: 45 %
End: 45 %
Prep Temperature: 64 °F

Date	TC	Amb. Temp (°F)	Test RH (%)	Height (in.)	Energy (ft-lbf)	Impacts 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
6/9/2016	MJD SC	49	49 / 49	43.3	72.0	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O
						46.46	46.50	46.53	46.51	46.52	46.48	46.51	46.54	46.43	46.48	46.46	46.40	46.34	46.35	46.50	46.49	46.42	46.41	46.53	46.33
6/9/2016	MJD SC	49	49 / 47	43.3	72.0	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O
						46.46	46.35	46.48	46.[illegible]	46.60	46.40	46.54	46.43	46.43	46.41	46.49	46.45	46.54	46.41	46.38	46.53	46.45	46.30	46.55	46.49
6/9/16	MM CG	49	47 / 47	43.3	72.0	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O
						46.46	46.44	46.34	46.44	46.46	46.46	46.47	46.46	46.32	46.43	46.49	46.52	46.26	46.47	46.46	46.34	46.43	46.33	46.41	
			____ / ____	39.0	65.0																				
			____ / ____	36.0	60.0																				
			____ / ____	33.0	55.0																				
			____ / ____	30.0	50.0																				
			____ / ____	27.0	45.0																				
+=Reaction O=No reaction *=Blanks						1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

A - Audible Report
C - Sample Discolored/Charred
F - Flash
M - Melting of Specimen or hardware
O - Odor (note:odor alone is not considered evidence of reaction)
P - Press increase
S - Stains/scorch marks on hardware
T - Temp increase

SOLSTICE PF SPRAY TEST DATA SHEET

Test 13A (Modified) – Solvent was dispensed directly into the tall aluminum cups (no insert sample cups were used). Also, the plummet catcher was used to eliminate rebound (uncontrolled) impacts.

WSTSF No. 16-1[illegible] A
Material: Solstice PFOP
Project TON SSOL
Test Pressure: 7.4 psia
Test Temperature: ~83 °F Test Cell: IDS

Test Sample Dimensions: Diameter: ____ in. Thickness: ____ in.
Weight: ____ g Other: Solstice
Test Media Constituents: ~299 LCV
Phase: Liquid: 100% ✓ Gas: ____
Prep RH: Start: 45 % Prep Temperature: 54 °F
End: 45 %

Date	TC	Amb. Temp (°F)	Test RH (%)	Height (in.)	Energy (ft-lbf)	Impacts 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
6/04/16	SB mm	83	50	43.3	72.0	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O
			51			46.58	46.70	46.46	46.46	46.46	46.53	46.48	46.46	46.57	46.49	46.51	46.60	46.47	46.52	46.50	46.46	46.53	46.45	46.56	46.43
6/04/16	SB mm	49	52	43.3	72.0	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O
			52			46.58	46.58	46.45	46.42	46.40	46.48	46.40	46.44	46.55	46.59	46.46	46.59	46.37	46.41	46.58	46.56	46.54	46.59	46.56	46.43
6/09/16	LB mm	49	52	43.3	72.0	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O	O
			49			46.58	46.48	46.48	46.50	46.49	46.51	46.42	46.42	46.52	46.40	46.48	46.46	46.44	46.52	46.42	46.42	46.43	46.45	46.49	46.43
				39.0	65.0																				
				36.0	60.0																				
				33.0	55.0																				
				30.0	50.0																				
				27.0	45.0																				
+=Reaction O=No reaction '=Blanks						1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20

A - Audible Report
C - Sample Discolored/Charred
F - Flash
M - Melting of Specimen or hardware
O - Odor (note: odor alone is not considered evidence of reaction)
P - Press increase
S - Stains/scorch marks on hardware
T - Temp increase

PROPERTIES OF SOLSTICE PF & PF SPRAY

Chemical properties provided by Honeywell.

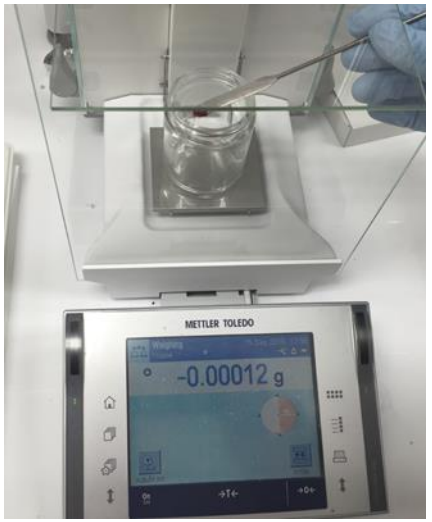
Property	Solstice PF
Chemical Name	trans-1-chloro-3,3,3-trifluoro-propene
Molecular Formula	$CF_3 - CH = CClH$
Molecular Weight	130
Boiling Point	66°F 19°C
Latent Heat of Vaporization at Boiling Point	83.4 BTU/lb 194 kJ/kg
Freezing Point	-161°F -107°C
Vapor Pressure at 68°F (20°C)	15.8 psia 109 kPa
Liquid Density at 68°F (20°C)	10.6 lb/gal 1.27 g/cc
Surface Tension at 68°F (20°C)	12.7 dyne/cm
Liquid Viscosity at 68°F (20°C)	0.503 cP
Solubility of Water in Solvent at 25°C	460 ppm
Solubility of Solvent in Water at 25°C	1900 ppm
KB Value	25

Property	Solstice Propellant	
Molecular Formula	$CHF=CHCF_3$	
Molecular Weight	114	
Boiling Point	-2.2°F	-19°C
Vapor Pressure at 70°F/21°C at 130°F/54°C	49 psig 147 psig	3.4 bars 10 bars
Liquid Density at 70°F/21°C	1.17 g/cc	
Vapor Flame Limits (Vol.% in Air) Measured at 70°F	None	
Solubility of Water in 1234ze at 68°F/20°C	225 ppm	
Solubility of 1234ze in Water at 68°F/20°C	373 ppm	
Dipole Moment (debye)	1.44	
Dielectric Strength (Vapor at 1 atm.)	11.7kV/0.1 inch	
Heat of Combustion	4385 BTU/lb.	10.2kJ/g
Heat of Vaporization @NBP	84 BTU/lb.	195kJ/kg

NVR SAMPLE METHODOLOGY

1) Stainless Steel Tray and Aluminum Pan Preparation (Prior to use)
   a) Clean sample trays and pans via 3 rinses of each of the following solvents
      - Acetone, Ethyl Alcohol, Hexane, AK225
   b) Weigh / record pans

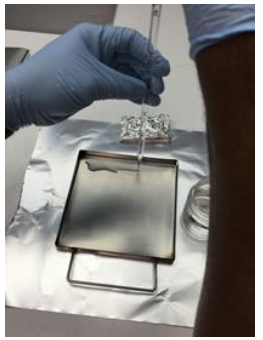
2) Sample Preparation
   a) Weigh 100 ml sample bottle to obtain the tare weight
   b) Add > 200 grams of contaminant to the bottle
      i. This is done while the bottle is being weighed
      ii. Solid grease contaminates tend to stick to the measuring tool, thus making exact measurements difficult
   c) Record contaminant solution details on the bottle
      i. Contaminant weight
      ii. Name & ID number
   d) Add 100 ml of AK225 to the sample.
   e) Vigorously shake the bottle until the contaminant is dissolved



Pipet Dispersal of Contaminant Solution

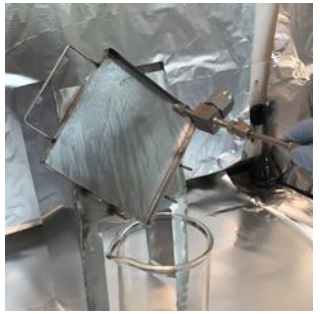
NVR SAMPLE METHODOLOGY

3) Contaminant Doping
   a) Place 5 ml of contaminant solution onto the tray via pipet
   b) Allow the contaminant to air dry onto the sample tray
   c) Place the sample tray in the oven dry for 2 hours @ 55°C
   d) Store the dried sample plate in a desiccator over night

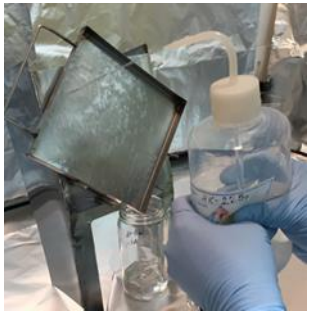


Pipet Dispersal of Contaminant Solution

4) Contaminated Tray Rinse
   a) Place contaminated sample tray on test stand
   b) 1st Wash / Rinse - 200 grams of Solstice PF Spray
      i. Wash / Rinse sample tray using a top / down, side to side motion
      ii. Insure that all surfaces are covered
      iii. Spray amount tracked via weight decrease of solstice container
      iv. Rinse is not captured as a result of splash back / evaporation loss
   c) 2nd Rinse – 200 ml of AK225 Control
      i. Rinse sample tray using the same top / down, side to side motion
      ii. Insure that all surfaces are covered
      iii. Capture the 1st 180 ml in a sample beaker
      iv. Capture the 2nd 20 ml in graduated cylinder w/ glass stopper (Used to flush sample pan)



Solstice PF Spray Wash / Rinse

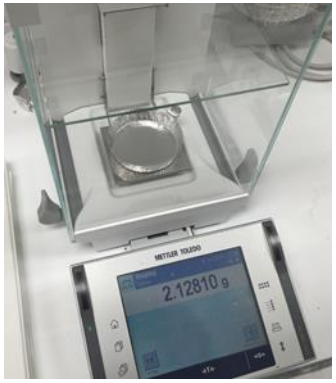


AK225 Rinse

NVR SAMPLE METHODOLOGY

5) NVR Weight Measurement
   a) Place opened 180 ml beaker sample under the flow hood
   b) Allow the sample solution to evaporate to 10 ml
   c) Transfer all 10ml of contaminated solvent into the Al pan.
   d) Rinse the empty 180 ml beaker using the graduated cylinder sample solution
      i. Rinse 4 times, using 5 ml of solution while rotating the beaker
      ii. Insure that all surfaces are rinsed
   e) Allow the sample solution to evaporate on the sample pan
   f) Place the sample pan in the oven for 1 hours @ 105°C to dry
   g) Store the dried sample plate in a desiccator for 30 minutes
   h) Weigh the Sample Pan + NVR and note in the log book

Sample Beaker Rinse using 20 ml Graduated Cylinder Sample



Sample Pan + NVR Weight Measurement

NVR REMOVAL TEST RESULTS

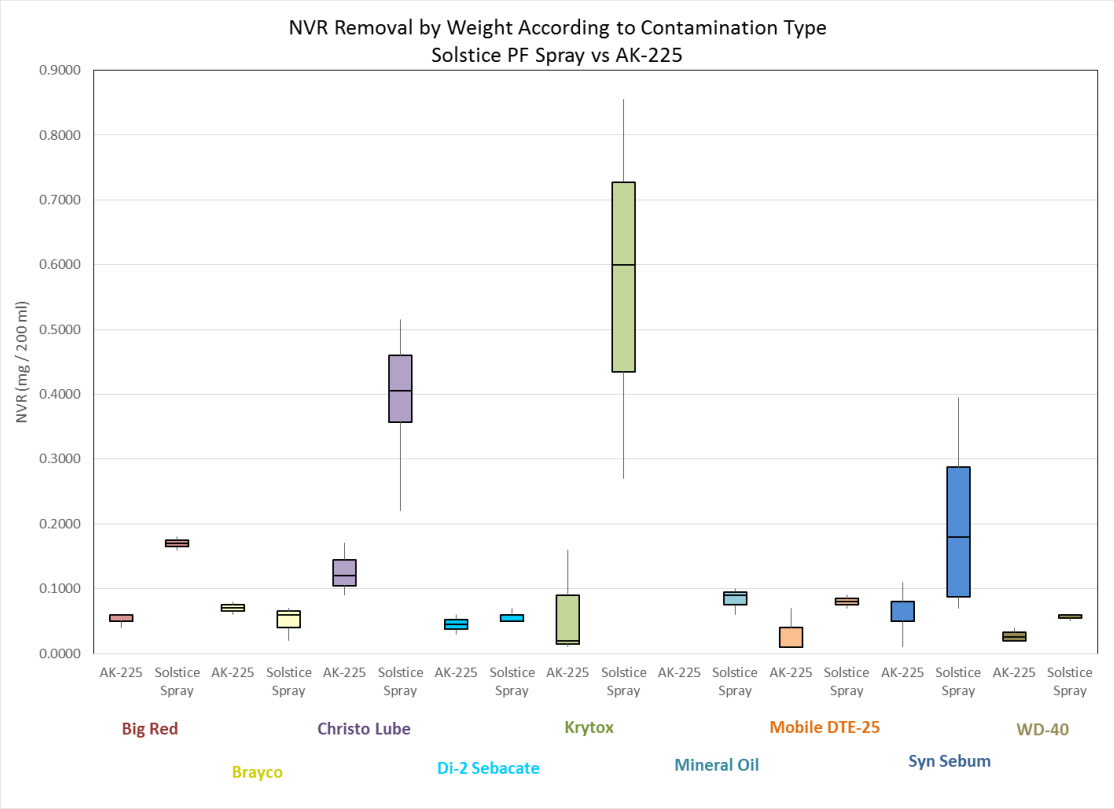
Analysis of the weight measurement method was evaluated to rule out tester / operator variability. 24 NVR pan samples @ T= 0 and T = 3 days were analyzed using Big Red, Mineral Oil, Synthetic Sebum, and WD-40 samples.

This was an additional pan only test to validate the NVR contamination deposition method.

The measurements were identical with 95% confidence. Thus, the measurement and storage of the sample pan is not a factor.

3 Day Weight Measurement Variation:

- Average: 0.00001
- STDEV: 0.00002
- Maximum: 0.00005

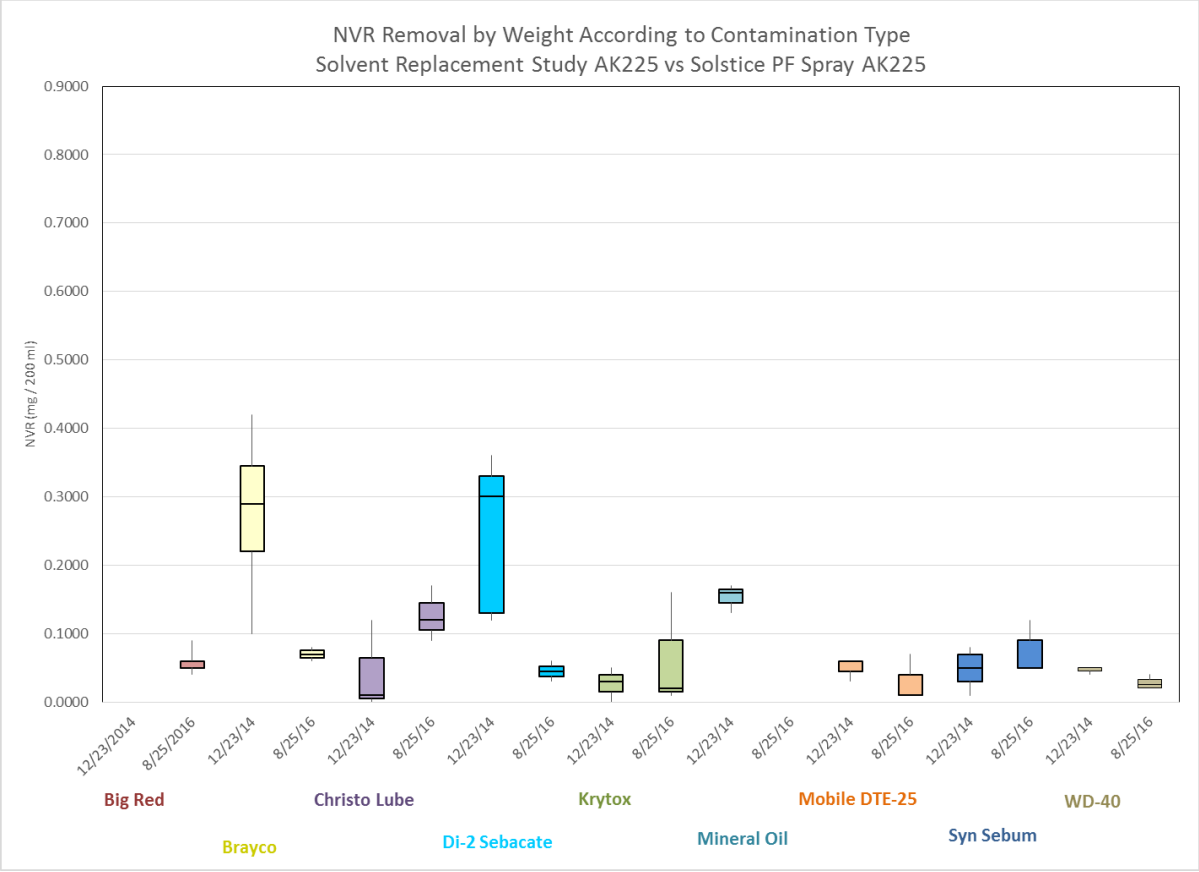


AK225 TEST VARIATION

AK225 data analysis was conducted to correlate the results from the 2015 solvent replacement study with the results from this study of the Solstice PF Spray.

Variability existed with the AK225 NVR removal, specifically with Big Red, Di-2 Sebacate, and WD-40 contaminants. This variability is the result of low sample sizes, subtle difference of application, and overall contamination loading.

(Note: Brayco and Mineral Oil sample quantities were low, so these values are not statistically relevant.)



NVR TEST CONTAMINANTS

1. Mineral Oil: RP-1 fuel, petroleum-based hydraulic & motor oil (CAS 8042-47-5)
2. Mobil DTE 25: Petroleum-based machine tool hydraulic fluid, ISO grade 46
3. Castrol Brayco Micronic® 882: Synthetic hydraulic fluid, MIL-PRF-83282
4. Di-2-Ethylhexyl Sebacate: Gauge calibration oil - Monoplex® DOS
5. Synthetic Sebum: Simulated sebaceous oils from skin
6. Fluorocarbon Grease: Krytox® 240AC
7. Fluorocarbon Grease: Christo-Lube
8. Big Red: Heavy paraffinic crane grease
9. WD-40: Penetrating oil (medium-heavy aliphatic hydrocarbons)