OXYGEN CLEANING REQUIREMENTS AND METHODS, HOW THEY COMPARE AND HOW THEY ARE DIFFERENT







Why is oxygen cleaning needed?

 The primary requirement is the removal of hydrocarbon oils, greases and other combustible material, which could react violently, and with disastrous consequences, in the presence of oxygen.



 The secondary, but no less important, requirement is the removal of particles, which could, in a flowing stream of oxygen, impinge on a metallic surface, releasing sufficient energy to initiate a violent reaction.

Precision Cleaning Specifications

- CGA 4.1 (Compressed Gas Association)
- GAMPS 4121 (Gulfstream)
- KSC-C-123J (NASA Kennedy Space Center)
- MSFC-SPEC-164 (NASA Marshall Space Flight Center)
- RPTSTD-8070-0001 (NASA-Stennis)
- MAP-211025 (Orion Program)
- IEST-STD-CC1246 (Military Standard)
- ISO 14952 (International Standards Organization)
- 5-75192 (Lockheed Martin)
- STP 1012 (BOEING)

- STP 0637 (BOEING)
- SAE ARP1176 (Oxygen System and Component Cleaning and Packaging)
- RA1110-025 Aerojet Rocketdyne
- SPX-00006876 (SpaceX)
- ASTM G93 (International Standards Organization)
- 1P00071 (BOEING)
- MSFC-SPEC-164 NASA Marshall Space Flight Center
- P-103-56 Parker Hannifin
- GS-38 Praxair

Typical Oxygen Cleaning Process **Mechanical Cleaning**

Aqueous Cleaning

Water break free test

Passivation

Visual Inspection

Validation

Drying

Packaging/Protection

Aqueous Based Degreasing Solutions

Chemical	Liquid/Po wder	рН	Solution Strength	Тетр
Amway nonionic Legacy of Clean [®] L.O.C	Liquid	6.1 - 6.8	1/2 to 1.0 oz per gal	110-130 deg F
Turco 4215 NCLT	Powder	8.8	4 to 6 oz per gal	140-160 deg F
Micro90	Liquid	9.7	2.5 oz per gallon	145-165 deg F
Brulin 1990	Liquid	11 - 11.6	5-7% by volume	130-150 deg F
Brulin 815GD	Liquid	12	8%-10% by Volume	130-170 deg F
Navy Oxygen Cleaner (NOC)	Liquid	11-12	50% by volume	140-170 deg F
Trisodium Phosphate (TSP)	Powder	12-14	8.0 oz per gallon	110-130 deg F
BONDERITE C-AK 4181 L AERO	Liquid	>13	4 to 6oz per gal	120-160 deg F
Daraclean 282	Liquid	12.5	3 TO 25% by volume	80-180 deg F
Extreme SIMPLE GREEN Aircraft & Precision Cleaner	Liquid	10-11.5	8 to 10 oz per gallon	90-110 deg F
Blue Gold Industrial Cleaner	Liquid	11.1–11.9	2-5% by volume	104-140 deg F

Validation - Validation is performed to determine the fluid system wetted-surface cleanliness level

SAMPLE COLLECTION METHODS		
Flush Cleaning	Flow test fluid at a minimum average velocity of 1.25 mis (4 ft/s). Some specifications request higher flow rates	
Dip Sample	Small components, fittings, soft goods, etc., (items small enough to fit inside an 800-mL beaker) shall be individually dipped and agitated in 500 mL of test fluid.	
Impingement spray	Impinge all interior and exterior surfaces of items with validation solvent. Sampling distance between the spray gun and the hardware shall be 6 in ±3 in. The distance shall be visually estimated NOTE: Typically 50 ml is the minimum sample size necessary to be taken from each part but the quantity shall be sufficient to remove all contaminants and yield a valid sample of all surfaces of the part.	
Fill & Aggitate	Items such as bottles, vessels, pipes, etc., which are of sufficient size to hold the proper amount of approved test fluid and the test fluid can be "locked in", will be filled with solution and agitated to assure wetting of all significant surfaces and the solution drawn off for sample	

Cleanliness is verified (Validated) through measurement of:



Particulate contamination -

unintended presence of extraneous, mobile and undissolved particles



Nonvolatile Residue (NVR) - soluble or suspended material and insoluble particulate matter remaining after evaporation ASTM F312 – Standard Test Method for sizing and counting particles from Aerospace fluids on membrane filters -Absolute Scanning Method

Manual Count:

The absolute (100 percent) scanning method for sizing and counting particles shall be used for examination of sample filter discs. Systematically scan and count all particles on the entire effective filtering area of the sample filter disc, scanning each specified size range separately.



ASTM F312 – Standard Test Method for sizing and counting particles from Aerospace fluids on membrane filters -<u>Absolute Scanning Method</u>

Automatic Particle Count using Olympus (CIX 100)

- · Data is saved automatically
- Counts all particles in all specified ranges (NO TNTC)
- Compliant results are customized to the selected industry standards



Allowable Particulate Counts



IEST-STD-CC1246E Level 100 5 to 15 - 1519 15 to 25 - 186 25 to 50 - 67 50 to 100 - 9 >100 - 0



IEST-STD-CC1246E	INSTITUTE OF ENVIRONMENTAL SCIENCES AND TECHNOLOGY
ISO 14952	INTERNATIONAL STANDARD
KSC-C-123J	NASA, KENNEDY SPACE CENTER
RPTSTD-8070-0001, REV. 3	NASA, STENNIS
SAE ARP 1176, Rev. B	SAE AEROSPACE
MIL-STD-1330E	DEPARTMENT OF DEFENSE
CMSP-02292	BLUE ORIGIN
SPX-00021833	SPACEX
STP0637, Rev. C	BOEING

**The above specifications mirror or are very close to the same particle counts listed in IEST-CC-1246E, Level 100

Validation of Nonvolatile Residue (NVR) (Flash Evaporator)

ASTM F331 - Standard Test Method for Nonvolatile Residue of Solvent Extract from Aerospace Components (Using Flash Evaporator)





Infrared Spectrometric Transmission Method for Nonvolatile Residue Analysis Fourier Transform Infrared (FTIR)

This method involves evaporating an aliquot of solvent (Solstice, HFE 7100, etc.), or the entire sample volume and picking up the residue in tetrachloroethylene and analyzing the solution with an infrared spectrometer.



Technical Procedure (TP) 151

Infrared Spectrometric Transmission Method for Nonvolatile Residue Analysis

Fourier Transform Infrared (FTIR)



THIS DOCUMENT CONTAINS HAZARDOUS PROCEDURES.

Cattera

Quality Manager Approval

Engineering Manager Approva

Precision Fabricating & Cleaning Co., Inc. 3975 E. Railroad Ave. Cocoa, FL 32926 (321) 635-2000 www.precgroup.com



Notification of Proprietary Information or Business Confidential Information: This document includes information that will not be duplicated, dispersed, used, or disclosed, in whole or in part, other than the intended use of this procedure for our employees and direct customers. All information contained within this document is subject to this restriction.

Specifications that reference/allow FTIR for Nonvolatile Residue Analysis

KSC-C-123	NASA Kennedy Space Center, Surface Cleanliness of Ground Support Equipment Fluid Systems, Specification For
RPTSTD-8070-0001	NASA Stennis, Surface Cleanliness Standard of Fluid Systems for Rocket Engine Test Facilities of the Nasa Rocket Propulsion Test Program
IEST-STD-CC1246E	Product Cleanliness Levels - Applications, Requirements, and Determination
5-75192	United Launch Alliance, Cleanliness Criteria, Critical Components
MIL-STD-1330E	Department of Defense, Standard Practice for Precision Cleaning and Testing of Shipboard Oxygen, Helium, Helium-Oxygen, Nitrogen, And Hydrogen Systems
SN-C-0005	NASA Johnson Space Center, Contamination Control Requirements
GAMPS 4121	Gulfstream Aerospace Material Process Specification
ASTM G93	Standard Practice for Cleaning Methods and Cleanliness Levels for Material and Equipment Used in Oxygen-Enriched Environments
EIGA Doc 33	Cleaning of Equipment for Oxygen Service (EUROPEAN INDUSTRIAL GASES ASSOCIATION AISBL)
MIL-STD-1622B	Cleaning of Shipboard Compressed Air Systems
SPX-00008185-v2.0	SpaceX, Supplier Cleaning, Inspecting, and Packaging Requirements
CMSP-02292	Blue Origin, Cleanliness Verification and Packaging

COMPARISON OF ALLOWABLE NVR LEVELS PER SPECIFICATION

The most common allowable NVR level is 1mg/sqft

ASTM G93M-19	INTERNATIONAL STANDARD
CMSP-02292	BLUE ORIGIN
GAMPS 4121	GULFSTREAM
IEST-STD-CC1246E	INSTITUTE OF ENVIRONMENTAL SCIENCES AND TECHNOLOGY
ISO 14952	INTERNATIONAL STANDARD
KSC-C-123J	NASA, KENNEDY SPACE CENTER
RPTSTD-8070-0001, REV. 3	NASA, STENNIS
SPX-00021833	SPACEX

All specifications do not have the same allowable NVR level

MIL-STD-1359B	MILITARY STANDARD	3
SAE ARP 1176, REV. B	SAE AEROSPACE	3
MIL-STD-1330E	DEPARTMENT OF DEFENSE	5
MSFC-SPEC-164E	MARSHALL SPACE FLIGHT CENTER	5
CGA 4.1 (2018)	COMPRESSED GAS ASSOCIATION	20



This is an example of a **failed 1mg/sqft NVR**, typically, if you can visibly see residue, the allowable NVR level has been exceeded.

Oxygen Cleaning Validation Test Fluids

- Discontinued Validation Test Fluids:
 - CFC 113 Solvent of Choice in the 90's
 - AK 225 Discontinued in 2013
 - TRICHLOROETHANE banned from use in 1996
- Current selection of Oxygen Cleaning Validation Test Fluids
 - Ethyl Acetate
 - HFE 7100
 - Solstice
 - Perchloroethylene



Elhyl Acetale



SOLVENTS

- HFE 7100 Go to industry standard for oxygen cleaning. THE LAST SHIPMENT WILL BE DECEMBER 31, 2025, OR
 SOONER IF THE FACTORY RUNS OUT OF RAW MATERIALS. THE FACTORY WILL CANCEL ANY UNFULFILLED ORDERS
 IF THEY RUN OUT OF RAW MATERIALS BEFORE DECEMBER 31, 2025. CUSTOMERS ARE URGED TO TRANSITION TO AN
 ALTERNATIVE FLUID AS SOON AS POSSIBLE.
- SOLSTICE Acceptable as an Oxygen cleaning solvent when cleaning IAW MIL-STD-1330E, NASA Stennis, Gulfstream
- ETHYL ACETATE Must be followed by Oxygen compatible solvent flush, stringent solvent removal detection processes
- CYCLOHEXANE Highly flammable, <u>Must</u> be followed by Oxygen compatible solvent flush, stringent solvent removal detection processes
- ETHYL ALCOHOL Not used by PFC, not Oxygen compatible
- PERCHLOROETHYLENE Carcinogen and may cause neurological, kidney and hepatic disorders, Not used by PFC
- TRICHLOROTRIFLUOROETHANE (CFC 113) Solvent of Choice in the 90's, phased out
- AK 225 Discontinued in 2013
- TRICHLOROETHANE banned from use in 1996
- ISOPROPYL ALCOHOL (TT-I-735), not Oxygen compatible
- VERTREL MCA nonflammable, not used by PFC

Specification differences regarding allowable oxygen cleaning solvents

Wording from various specifications;

- 1. The test fluids shall not react with, combine with, etch, or otherwise cause immediate or latent degradation of the item being tested
- 2. The test fluids shall be compatible with the fluid used in the system or components being tested
- 3. Vertrel MCA does not meet the liquid oxygen compatibility requirements
- 4. Isopropyl alcohol and ethyl alcohol shall not be used as the test fluid for oxygen systems and hardware.
- 5. Alcohol solvents can be used to perform particulate analysis, but it shall not be used as a test fluid for NVR analysis.
- 6. HFE 7100 has low solubility and low solubility transport for keeping the NVR in suspension. HFE-7100 is a poor solvent for hydrocarbon oils and greases including silicone
- HFE-7100 may be used to perform NVR analysis if a correction sensitivity factor of 3.0 is applied to correct for the efficiency of HFE 7100 to remove non-volatile (hydrocarbon-based residue) from critical surfaces.
- 8. Items or systems with NVR requirements shall be flushed with HFE-7100, AK225, or Solstice PF or PF-HP
- 9. HFE-7100 is the final particulate cleanliness verification liquid for oxygen system hardware following acceptable NVR verifications.
- 10. Ethyl Acetate (when compatible with the base material) shall be the NVR verification solvent when sampling smooth wall detail items lacking liquid entrapment areas. NVR analysis shall be followed by a non-flammable oxygen compatible solvent as the final particulate cleanliness verification.

Solvents, continued

- Vertrel MCA contains HFC, which is on a global phase down schedule due to its GWP. HFC's will no longer be available after about 2030
- HFE-7100 3M exiting the market, Chinese-made "analogs" are available – all are PFAS per EPA definition.
 - US EPA is scrutinizing and may require reporting of Chinese HFEs (and other PFAS)
- Solstice the logical choice!

Solstice[®] **PF**

Nomenclature

- Trans-1-chloro-3,3,3-trifluoroprop-1-ene
- 1233zd(E)
- HFO: hydrofluoro-olefin

Performance

- Good solvency towards cutting oils, silicone oils
- Low surface tension, 12.7 dyne/cm
- Good stability, material compatibility, LOX compatible

Safety & Environmental

- Non-flammable by ASTM E-681
- Very low order of Toxicity: OEL 800 ppm (WEEL)
- Best overall combination of Environmental Properties
 - GWP = 1 (100yr ITH)
 - Very low MIR and POCP values / US EPA & South Coast AQMD VOC Exempt

Mobil 600W oil streaming off a metal part in Solstice PF

Solstice PF

Regulatory

- TSCA Inventory
- Added to the US EPA SNAP list as a Solvent
- US EPA VOC Exempt
- South Coast AQMD VOC Exempt
- EU REACH: Registered for volumes > 1,000 tons
- Registered in Canada, Japan, South Korea, China, Australia



PFAS is defined as including at least one of these three structures:

- R-(CF₂)-CF(R')R", where both the CF₂ and CF moieties are saturated carbons;
- $R-CF_2 OCF_2 R'$, where R and R' can either be F, O, or saturated carbons; and
- $CF_3 C(CF_3)R'R''$, where R' and R'' can either be F or saturated carbons.

Solstice PF – Physical Properties

Property	Solstice PF
Chemical Name	trans-1-chloro-3,3,3-trifluoropropene
Molecular Formula	$CF_3 - CH = CCIH$
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 gm/mL
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
KB Value	25

NASA MSFC-SPEC-3709 – Standard



MSFC TECHNICAL STANDARD EM22 Title: Solvent, Cleaning, Trans-1-Chloro-3,3,-Trifluoropropere, Solstice[®] PF Document No: MSFC-SPEC-3709 Baseline Effective Date: 10-JAN-2017 Page: 5 of 12

1.0 SCOPE

1.1 Scope

This specification establishes the requirements for Honeywell Solstice[®] Performance Fluid (PF) solvent, trans-1-chloro-3,3,3-trifluoropropene. This material is intended for use for cleaning and cleanliness verification for spaceflight hardware and related ground support equipment and test systems.

1.2 Classification

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 $^{\oplus}$ 6920.

2.0 APPLICABLE AND REFERENCE DOCUMENTS

2.1 Applicable Documents

The following documents of the revision listed (or latest revision if no revision is listed) form a part of this document to the extent specified herein.

- ASTM D2109 Standard Test Methods for Nonvolatile Matter in Halogenated Organic Solvents and Their Admixtures
- ASTM D2988 Standard Test Methods for Water-Soluble Halide Ion in Halogenated Organic Solvents and Their Admixtures
- ASTM D2989 Standard Test Method for Acidity-Alkalinity of Halogenated Organic Solvents and Their Admixtures
- ASTM D3401 Standard Test Method for Water in Halogenated Organic Solvents and Their Admixtures
- ASTM D6806 Standard Practice for Analysis of Halogenated Organic Solvents and Their Admixtures by Gas Chromatography
- ISO/IEC 17025:2005 General Requirements for the Competence of Testing and Calibration of Laboratories

CHECK THE MASTER LIST VERIFY THAT THIS IS THE CORRECT VERSION BEFORE USE

US Navy





DoD Cleaning Spec

MIL-STD-1330E(SH)

16 May 2022

DEPARTMENT OF DEFENSE STANDARD PRACTICE

- PRECISION CLEANING AND TESTING OF SHIPBOARD OXYGEN, HELIUM, HELIUM-OXYGEN, NITROGEN, AND HYDROGEN SYSTEMS
- Solstice Performance Fluid approved in this spec



Small Footprint Continuous Solvent Recycle System

- Used by PFC to recycle used Solstice to low NVR
- General process Flush solvent is collected in a suitable container
- When container is full, it is moved and attached to the Recycle system, cylinder of clean recycled solvent is put into service.
- Since solvent is continuously cleaned, contamination levels remain low.
- Cost savings = labor, virgin solvent, waste disposal

Example Schematic



Conclusions

- Oxygen system cleaning is imperative for safety of all involved
- Verification of cleanliness is equally important
- PFC and KYZEN have worked together to optimize the processes!





Questions and Contact Information

Beth Bivins - KYZEN *Global Key Account Manager – Solvents* Phone: (904) 676-5547 Email: <u>Beth Bivins@kyzen.com</u>

James Bowers - PFC Manager, Cleaning and Testing Operations Phone: (321) 394-2828 Email: james.bowers@precgroup.com

