OXYGEN CLEANING

VERIFICATION METHODOLOGIES AND CHALLENGES

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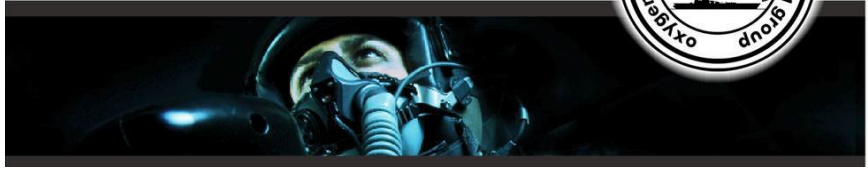
OSCG
Annual Meeting
Aug 20-22, 2024
Buffalo, NY

OSCG

oxygen standardization coordinating group



WHAT IS Precision Cleaning?

MEASUREMENT OF CONTAMINANTS

PARTICLES

- Small unit of solid material with definable shape or mass. A particle with a length-to-width ratio exceeding 10:1 is normally called a fiber (some specifications require fibers to have a minimum length of 100 µm).

NON-VOLATILE RESIDUE (NVR)

- Amorphous contaminants, typically oils & greases. Soluble material remaining after evaporation of a solvent, as mass per unit area. Hydrocarbon residues of greatest concern.

OTHER (end use dependent)

- Ionic contaminants, trace gasses, trace radioactivity, etc. Whatever will interfere with optimal performance & can be identified, cleaned, and measured.

OXYGEN CLEAN

SPECIAL CONCERNS

"Scrupulous cleaning is the most fundamental fire safety measure applied to oxygen-handling systems. One may not have to alter polymers or metals in some systems of low severity, but any system that is exposed to oxygen or oxygen enriched service will require scrupulous cleaning."

~ American Society for Testing and Materials (ASTM) G 93

"It is intended that oxygen equipment cleaning operations perform to a minimum level that will be suitable to preclude toxic and fire hazards for the application and conditions the equipment will be exposed to."

~ SAE ARP 1176A "Oxygen System and Component Cleaning" (SAE A-10 Aircraft Oxygen Equipment Committee)

REACTIVITY

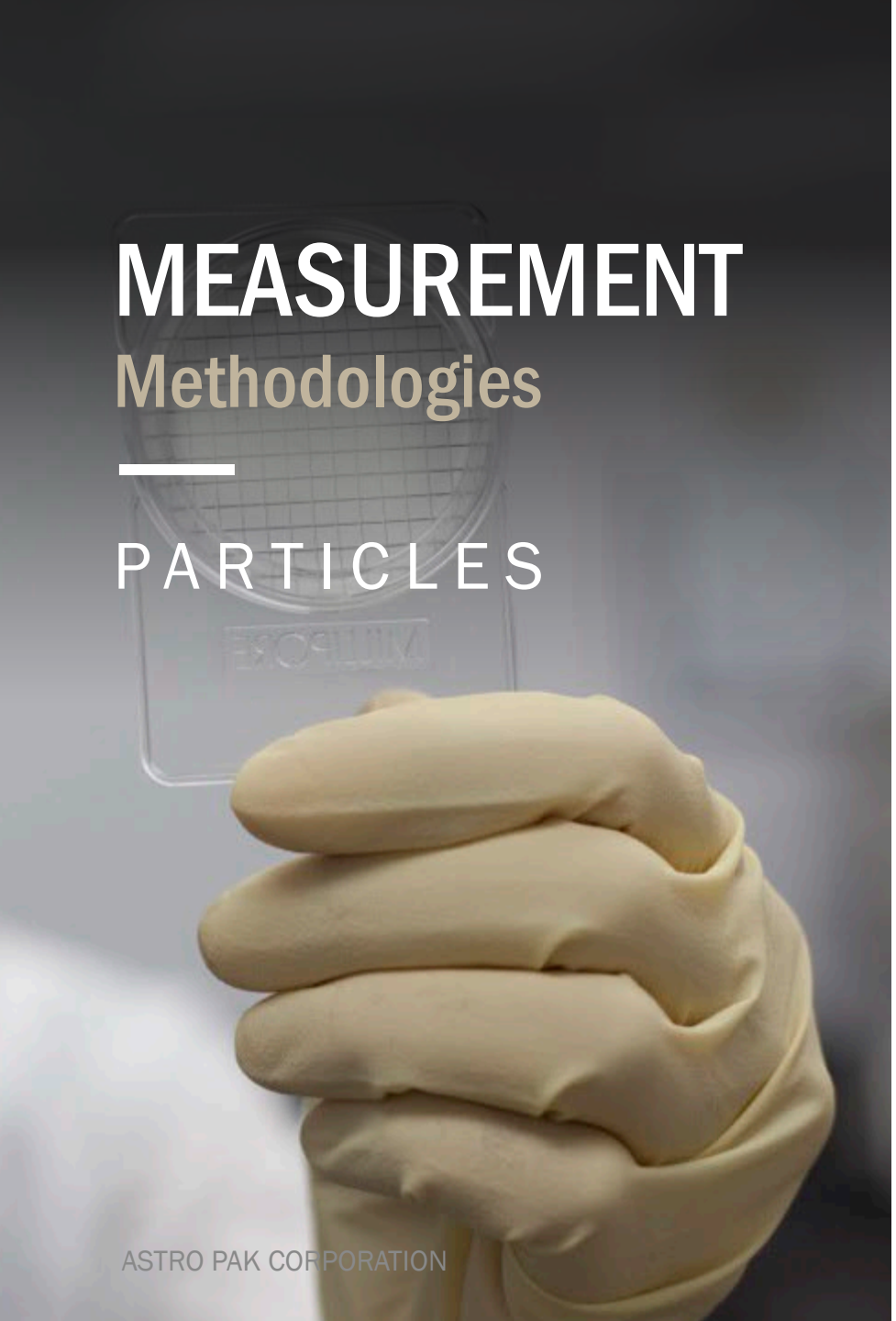
FIRE / EXPLOSION

NVR - GENERALLY HYDROCARBON

- Hydrocarbon-based oils & greases are flammable. Environments with increased O_2 concentration reduce temperatures necessary to cause ignition and increase the rate of combustion.

PARTICLE IMPACT IN GASEOUS OXYGEN

- Particles traveling in a rapidly-moving gas stream can impact tube or pipe wall or other in-line components with enough energy to cause friction heating that can ignite particle. Particle ignition can then ignite other metal in impact area.



MEASUREMENT
Methodologies

PARTICLES

PARTICLE COUNT (SIZE & NUMBER)

- Particles limited by size range & quantity per unit surface area.
- Measured post-cleaning & reported per size ranges defined in governing specification.
- SAE AS598, "Aerospace Microscopic Sizing and Counting of Particulate Contamination for Fluid Power Systems”

TOTAL FILTERABLE SOLIDS (Total Mass Of Solid Matter)

- Measured post-cleaning & reported as mass per unit surface area (mg per 0.1 m^2).

METALLIC vs NON-METALLIC

- Metallic particles often more strictly limited due to increased susceptibility to ignition.

MEASUREMENT METHODOLOGIES

NON-VOLATILE RESIDUE

- **GRAVIMETRIC NON-VOLATILE RESIDUE** - QUANTITATIVE
- **LONGWAVE ULTRAVIOLET LIGHT** *(wavelength 3200 to 3900 Angstrom units)* **INSPECTION** – SEMI-QUANTITATIVE (PASS/FAIL)
- **INFRARED ABSORPTION** – QUANTITATIVE, CONTAMINANT DEPENDENT
- **OPTICALLY STIMULATED ELECTRON EMISSION (OSEE), aka. PHOTO ELECTRON EMISSION (PEE)** – QUANTITATIVE but SURFACE DEPENDENT, SUBJECT TO OPERATOR ERROR

MEASUREMENT METHODOLOGIES

NON-VOLATILE RESIDUE

- **Fourier transform infrared (FTIR) spectroscopy with attenuated total reflection (ATR) accessory** – QUANTITATIVE but CONTAMINANT DEPENDENT

- **MESERAN** – radiocarbon 14 doped high vapor pressure evaporation rate measured via Geiger – Muller detector – QUANTITATIVE but CONTAMINANT & SURFACE DEPENDENT

- **NASA / JPL utilized FTIR/DRIFT to validate 1 nanogram per square centimeter for MARS 2020 sample tubes**, but this is experimental & has much uncertainty

MEASUREMENT METHODOLOGIES

SOLVENT EXTRACTION WITH GRAVIMETRIC NVR ANALYSIS

SOLVENT EXTRACTION for a **GRAVIMETRIC NVR** from the filtrate is still the most widely used quantitative verification of cleanliness for oxygen service because this methodology allows:

- Generation of quantitative results that are easily compared against design established requirements with little to no interpretation of data
- Accurate detection down to A/10 Level (0.1 micrograms per square centimeter)
- Apples to apples comparison of the level of cleanliness between differing substrates, components, and configurations
- Cost effective in terms of equipment, personnel, and time required to perform the analysis.

MEASUREMENT METHODOLOGIES

SOLVENT EXTRACTION WITH GRAVIMETRIC NVR ANALYSIS

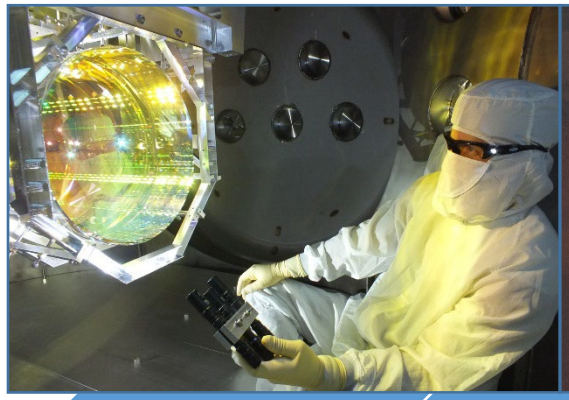
4 Factors Determine the Accuracy and Repeatability of Gravimetric NVR Analysis:

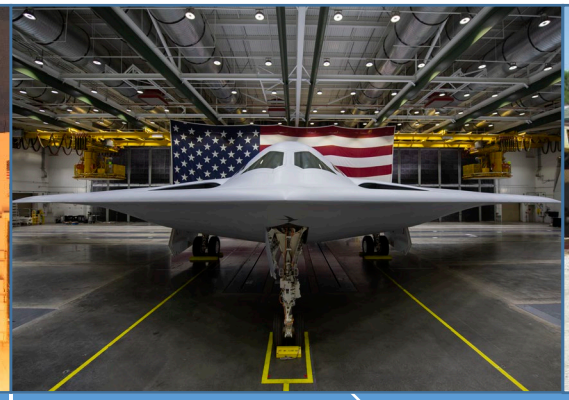
1. **Solvent Selection**
2. **Sample Collection Technique**
3. **Sample Filtration and Evaporation**
4. **Analysis of Results**

CHALLENGES
Facing the Industry

- VARIOUS STANDARDS
- SOLVENT SELECTION

VARIOUS STANDARDS






0.1mg/0.1m^2 | 0.5mg/0.1m^2 | 1.0mg/0.1m^2 | 3.0mg/ft^2 | 6.0mg/ft^2 | 47.5mg/ft^2

IEST-STD-CC1246E, Level A/20

5-75192-2

DPS 4.901

ASTM G93

STP0637

ARP1176

MIL-STD-1330

RA1110-025

BAC5402

CGA G-4.1

TDD 35D001F

KSC-C-123

AIPS-09-01-013

MSFC-SPEC-164

1P00071

ACS-PRS-2252

Various
Standards

Challenges

- Vastly different requirements exist between individual Prime and OEM manufacturers as well as different government agencies.
- Differing Materials Requirements
  - Cleaning solutions
  - Verification solvents
  - Packaging materials
- Differing Methodologies
  - Processing methods and parameters
  - Verification methods
- Increased costs and reduced commercial support available.

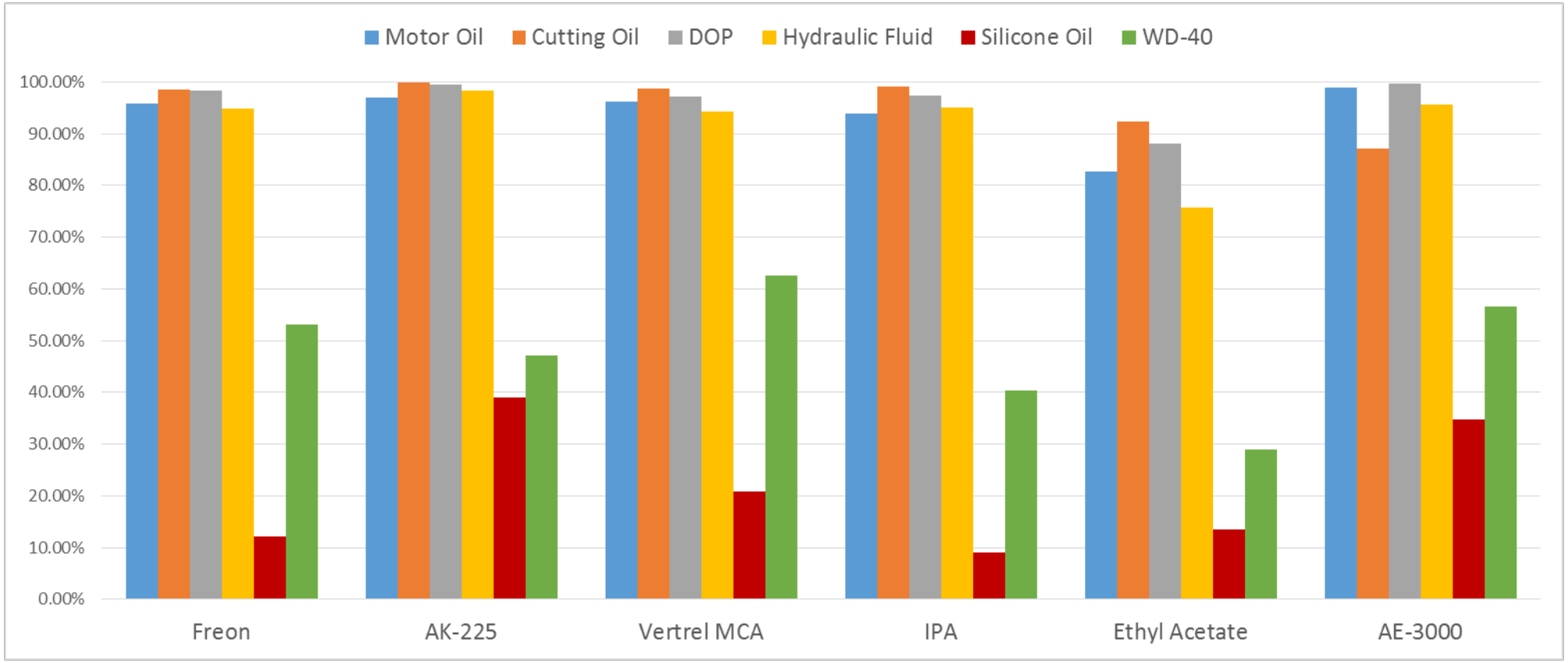
SOLVENT
Selection

CONCERNS

- MATERIAL COMPATIBILITY
- FLAMMABILITY
- TOXICITY
- ENVIRONMENTAL IMPACT
- COST
- EFFICACY

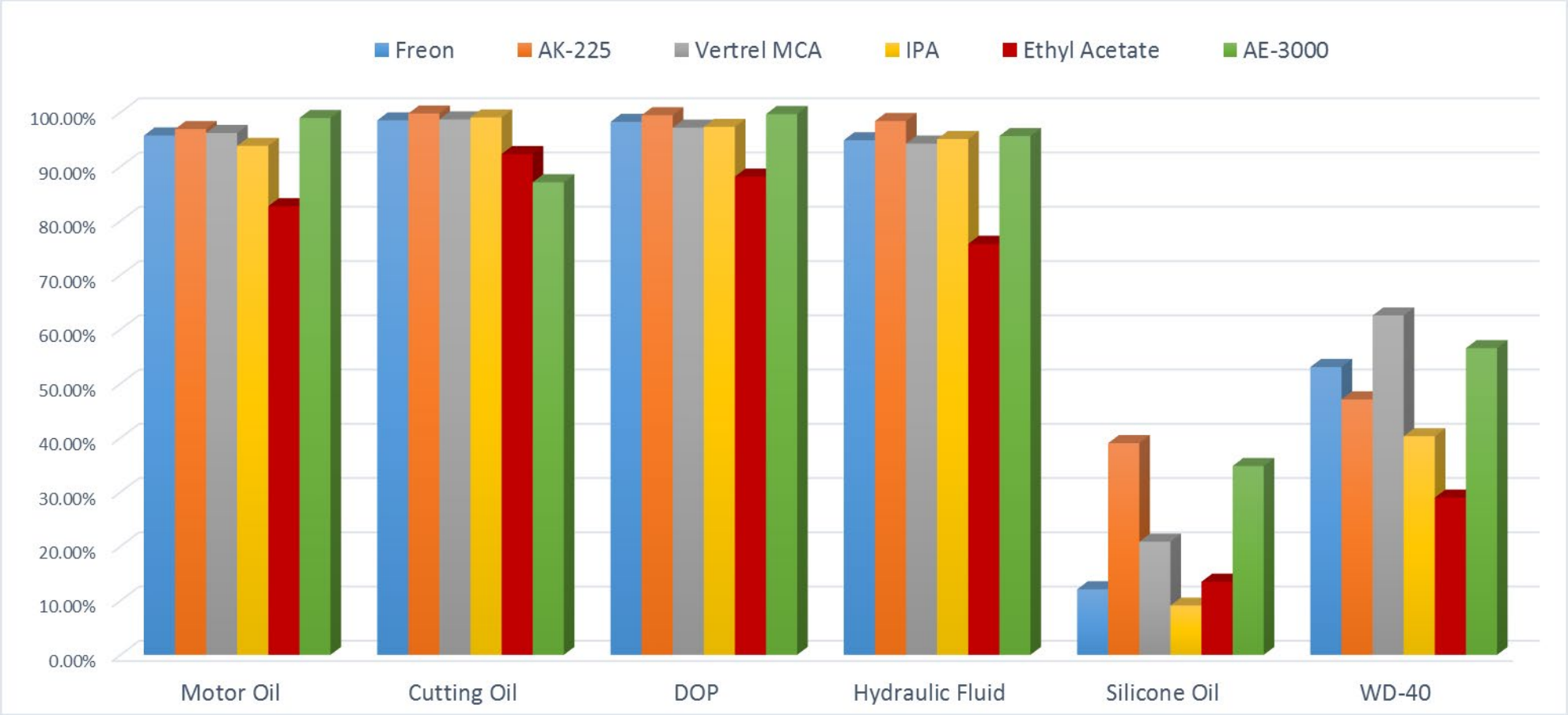
SOLVENT SELECTION

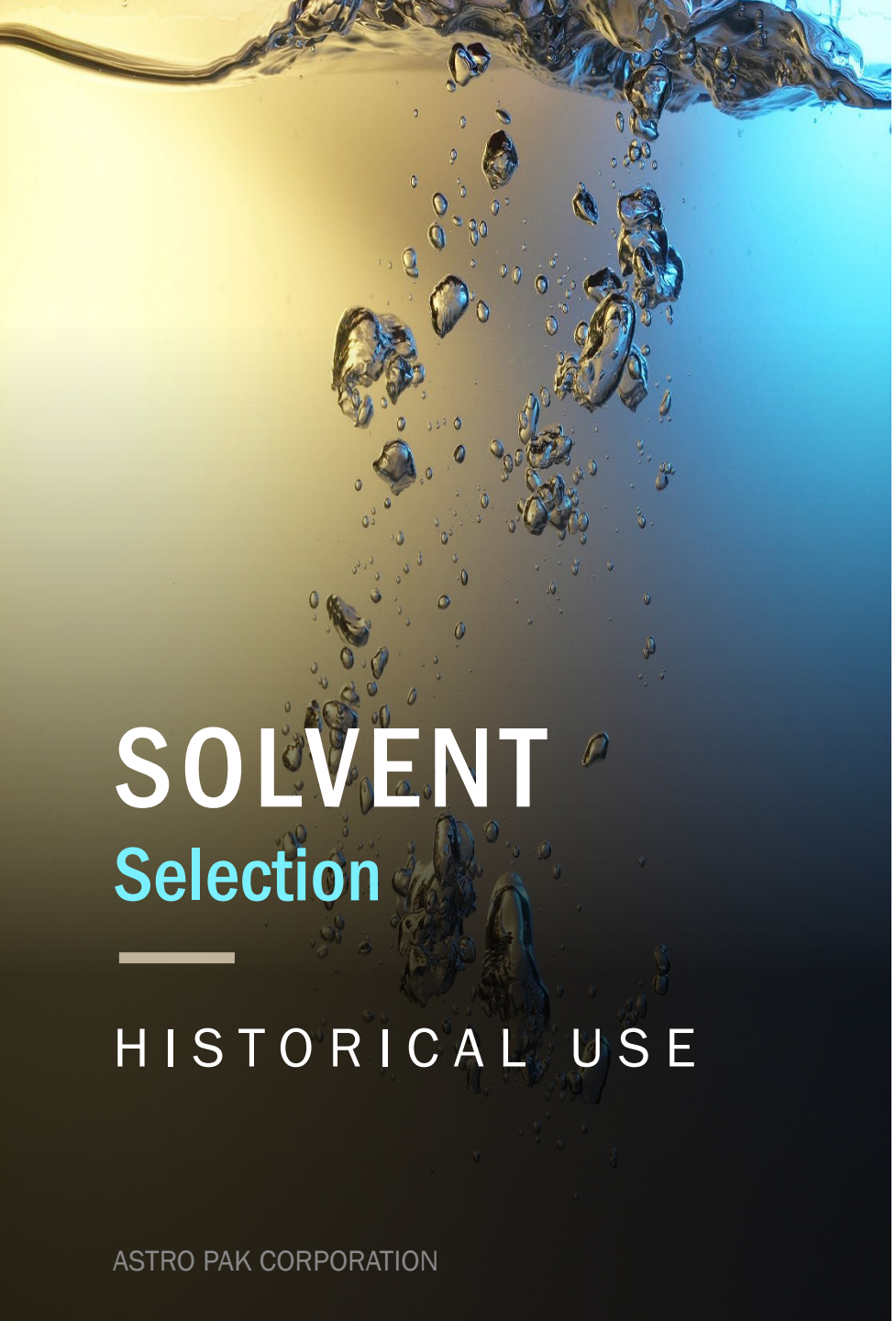
ABILITY TO EXTRACT CONTAMINANT



SOLVENT SELECTION

ABILITY TO EXTRACT CONTAMINANT





SOLVENT
Selection

HISTORICAL USE

- CFC-113 (Freon 113) was used for decades, nearly ideal NVR verification solvent, production banned due to environmental issues.
- AK-225 was a near-ideal replacement, but it had some toxicity & environmental issues, now production has also been banned
- HFE7100 being phased out by the end of 2025 due to manufacturer exiting PFAS manufacturing
- There is no universally agreed upon replacement! Different specifications call out a variety of solvents.

SOLVENT SELECTION

SOME CURRENTLY AVAILABLE SOLVENTS

HYDROCARBON

IPA

- Reasonably good efficacy, low cost
- Flammable
- Verify removal with photoionization detector (PID)

Ethyl Acetate

- Lower efficacy, moderate cost
- Flammable
- Verify removal with photoionization detector (PID)
- Noxious vapors, unpleasant odor

Cyclohexane

- Good efficacy for hydrocarbons, high cost
- Flammable
- Verify removal with photoionization detector (PID)

HALOGENATED

Vertrel MCA

- Good efficacy, very high cost
- Non-flammable*
- Some toxicity suspected

Solstice PF

- Good efficacy, very high cost
- Non-flammable
- Limited adoption
- A gas at room temperature

HFE7100 (not available after 2025)

- Lower efficacy for hydrocarbon, very high cost
- Non-flammable
- Low toxicity

*LOx impact test results borderline due to stabilizer in formula

CONCLUSIONS

- Verification of cleanliness using solvent extraction and gravimetric NVR analysis is likely to be the most used method for the foreseeable future.
- Solvent selection should be based on efficacy, material compatibility, environmental impact, toxicity, and cost.
- When the contaminant is known, test solvents & pick one with high efficacy. If contaminant(s) unknown, pick a solvent with a good spectrum of efficacy.
- Design considerations for systems and specifications must consider that there is currently no direct replacement for the old Freon, AK-225, or HFE7100 solvents.
- All performance parameters and cost considerations result in trade-offs.
- Coordinating to standardize cleaning methodologies and materials for Oxygen Service applications is critical to minimizing disruption from future solvent market changes.

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