

**Alternative
Green Solvents
Project**

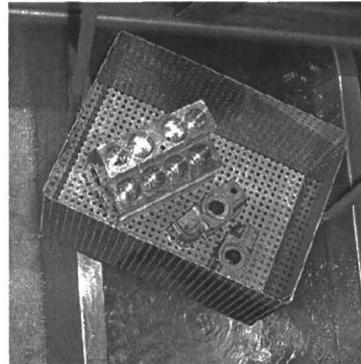
Phillip Maloney

Background

- Necessary for safe and proper functioning of equipment
- Mainly halogenated solvents
 - Carbon tetrachloride
 - Trichloroethylene (TCE)
 - CFC-113
- No longer used due to regulatory/safety concerns

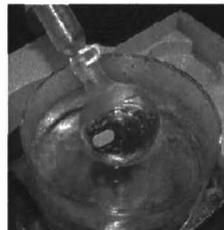
Precision Cleaning at KSC

- Small % of total parts
- Used for liquid oxygen (LOX) systems
- Dual solvent process
 - Vertrel MCA (decafluoropentane (DFP) and *trans*-dichloroethylene)
 - HFE-7100
- DFP has long term environmental concerns



Specifications and Analysis

- Highest level at KSC – 25A
- Verified by particle counting and NVR analysis



| Particulate Matter Contamination Levels | | | NVR Contamination Levels | | Visible Contamination Levels | |
|-----------------------------------------|---------------------------------------|----------------------------------------------------|--------------------------|--------------------------------------|------------------------------|-------------------------------------------------------------|
| Level | Particle Size Range (µm (micrometer)) | Maximum Number of Particles per 0.1 m ² | Level | Maximum NVR (mg/0.1 m ²) | Level | Definition |
| 25 | <5 | Unlimited* | A | 1.0 | GC | Freedom from manufacturing residue, dirt, oil, grease, etc. |
| | 5 to 15 | 19 | | | | |
| | >15 to 25 | 4 | | | | |
| | >25 | 0 | | | | |

Project Goals

- Identify potential replacements
 - 22 wet chemical processes
 - 3 alternative processes
- Develop test procedures
 - Contamination and cleaning
 - Analysis
- Use results to recommend alternative processes

Candidate Process Criteria

- Good solvency
- Low toxicity
- LOX compatible
- Environmentally friendly
- Low surface tension
- High vapor pressure
- Inexpensive



Processes Evaluated

| Pure Solvents | Proprietary Solvent Formulations | Alternative Technologies |
|-----------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------|
| Acetone, ethanol, cyclohexane, ethyl acetate, isopropyl alcohol, NMP, limonene, ethyl lactate, 1-bromopropane, methyl myristate, tert-butyl acetate | Entron Aero, Citricolv, Inland Isoprep, Inland AV-OP-125, Inland Breakthrough, Inland Skysol, Steposol SB-W, Steposol SB-D, Steposol SC, Steposol M-8-10, Vertec Bio | Atmospheric plasma glow discharge (AGPD), supercritical carbon dioxide (SCCO ₂), carbon dioxide snow |

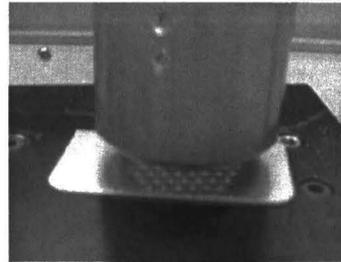
Candidate Solvents

- Easiest to integrate with existing process
- Avoided highly halogenated solvents
- Augmented with sonication, directed pressure, etc.
- May be recycled to reduce waste stream



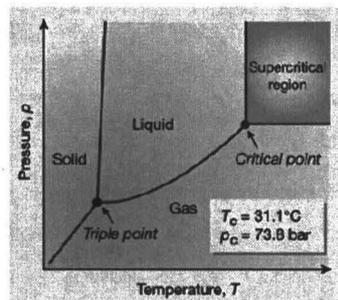
Plasma Cleaning

- Plasma is an ionized gas (TVs, lightning, etc.)
- Created by applying electrical field to a gas
- Clean via sputtering and/or chemical reaction
- Important parameters: gas type, exposure time, energy



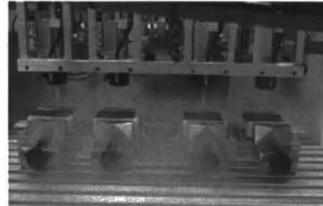
Supercritical CO₂

- Has properties of both liquid and gas
- Exists at temps >31.1°C and pressures >1072 psi
- Dissolves nonpolar molecules and hydrocarbons well
- Cleaning can be enhanced by the use of cosolvents



CO₂ Snow

- Cleans via thermo mechanical shock or dissolution
- CO₂ density determines type of cleaning



- Technique is similar to aqueous pressure washing
- Adjustable parameters: nozzle design, velocity, additional propellants

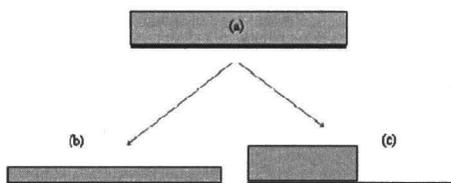
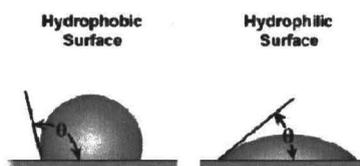
Solvent Testing Method

- 2" x 2" Al coupons
- Rough and smooth textures
- Sprayed with 5 contaminants
- Rinsed with 15mL of solvent and dried for one hour or...
- Cleaned by alternative process



Analytical Methods

- Problems w/ NVR
- Gravimetric
- XPS
- Contact angle



Top 5 Solvent Results

| Solvent | 83282 | 5606 | Diocetyl Sebacate | Krytox | Braycote |
|-------------------|-------|-------|-------------------|--------|----------|
| Vertrel | 102.7 | 83.3 | 98.5 | 98.6 | 89.0 |
| 1-bromo propane | 98.4 | 117.1 | 101.0 | 54.9 | 20.0 |
| T-butyl acetate | 99.1 | 102.5 | 99.3 | 54.1 | 29.2 |
| Ethyl acetate | 93.2 | 77.8 | 95.5 | 75.6 | 33.6 |
| Isopropyl alcohol | 100.4 | 79.0 | 98.8 | 66.6 | 15.6 |

Values listed are in % cleaning efficiency determined by gravimetric analysis

Contact Angle Measurements for Alternative Technologies

| Technology | 83282 | Krytox | Braycote |
|----------------------|--------|--------|----------|
| Vertrel MCA | 88.7° | 88.5° | 96.5° |
| Plasma | 44.4° | 59.6° | 79.3° |
| SCCO ₂ | <10.0° | 109.9° | 10.0° |
| CO ₂ Snow | 91.5° | 105.7° | 108.8° |

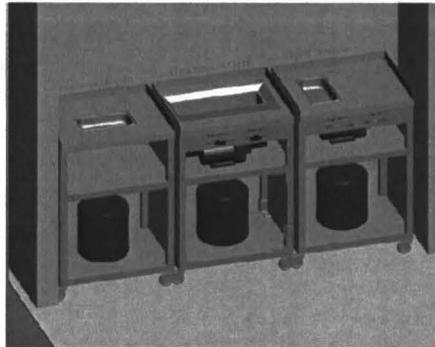
Values listed are in degrees as determined by contact angle measurements

Conclusions

- No alternative matched Vertrel in this study
- No clear second place solvent
- Hydrocarbons – easy; Fluorinated greases – difficult
- Fluorinated component may be needed in replacement solvent
- Process may need to make up for shortcoming of the solvent
- Plasma and SCCO₂ warrant further testing

Continuing Efforts

- Test blends with fluorinated component .
- Further testing of plasma and SCCO₂
- Clean complex hardware



Continuing Efforts

- Test compatibility with soft goods
- Round robin testing with partnering facilities
- Scale up of technologies
- Assess benefit to other government agencies and private partners