Method Validation for Selecting Baseline Solvent

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Outline

• Introduction & Background
• Objective
• Experimental Method
• Results & Discussion
• Conclusion
Introduction & Background
WSTF’s Experience

Replacing CFC-113 in the 90’s

- Collaboration
  - DOD
  - NASA
  - Contractors
  - Solvent Manufactures

- Solvents
  - CFC
  - HCFC
  - HFE
  - TCE

- Considerations for Solvents
  - Cleaning effectiveness
  - $O_2$ compatibility
  - Materials compatibility
  - Aerospace fluids compatibility
WSTF Cleaning Process

- **Pre-cleaning**
  - Ultra sonic
- **Visual inspection**
- **Final clean/ cleanliness verification**
  - DI water
  - HFE 7100
  - IPA
- **Process Validation**
  - AK 225G (baseline solvent)
Problem Statement

- WSTF Processes is dependent on AK-225G for process validation
- AK-225G limited availability in 2015
Objective

• To establish a standardized method for the evaluation of suitable replacement baseline solvent AK-225G.
Experimental Method
Standards for Testing Solvents


Attributes for a Baseline Solvent

ASTM Considerations

- Toxicity
- Carcinogenicity
- Recyclability
- Waste Disposal
- Ozone Depletion
- Inertness (Flammability and combustibility)
  - Oxygen compatibility
- Availability and technical support from supplier
- Corrosivity & material compatibility
- Cost effectiveness
- Compliance with local, state, and federal regulations
- Application and use of Solvent

Other Considerations

- HAP (Hazardous Air Pollutants)
- VOC (Volatile Organic Compounds)
- ACS reagent grade chemicals or higher
- Cleaning effectiveness
- Evaporation rate
So Many Choices!

**ASTM Test Method Options**

- **Contamination of Coupons**
  - Slurry
    - Contaminants: 1 vs. mixture
    - Concentration: 1 to 100 mg/mL
  - Application
    - 1 side vs. 2 sides
    - Pipette
    - Brush
    - Spray
    - Dip
  - Dry
    - Hang vs. laying flat

- **Cleaning of coupons**
  - Manufacture’s recommended use of solvent
  - Sonication
  - Elevated temperatures
Experiment Outline

Steps

1. Clean the coupons (standard)
2. Weigh the coupons (Tare weight)
3. Contaminate coupons
   a) Analyze slurry filter/NVR
   b) Oven dried
4. Weigh the coupon (Determine Contaminant)
5. Clean the coupon (test solvent)
   a) Analyze rinse (filter / NVR)
   b) Oven dried
6. Weigh the coupon (Residual contaminant)
7. Verify cleanliness of coupon (AK-225-G, Verification solvent)
   a) Analyze rinse (filter / NVR)
   b) Oven dried

Matrix

• Series 1
  – 1 Contamination Slurry for each solvent
  – 3 solvents tested
  – 7 coupons for each solvent

• Series 2
  – 1 contamination Slurry
  – 6 solvents
  – 1 coupon for each solvent
Contaminants

- Pump /hydrocarbon oil
- Hydraulic oil
- O₂ system lubricant
- Gage fluid
- Silicone grease
- Dye penetrates- particles
- Iron powder 60 Mesh- particles
Preparation of Contamination Slurry

- 5 slurry mixtures prepared
- 1 gram (+-.1g)
- 100 mL AK225-G
- Ultra Sonic 10 min
- Concentration 70 mg/mL
Contamination of Coupons

• 600 μL contamination Slurry
• Bake
  – 1 hr.
  – 45 ºC (+/- 5ºC)
  – Nitrogen purge
• Cool off 1 hr.
• Weighed
Cleaning & Verification

- Flush with 100 mL of solvent
  - NVR analysis
  - Particle count
- Cleaned with test solvent
- Verified clean with AK225G
Data Collection

- Counting Particles
- Weighing coupons
- Weighing NVR
- Weighing filters
Results & Discussion
Post Cleaned, Pre AK225G Validated Coupons

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>General</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S</td>
<td>Surface Area (cm²)</td>
<td>45</td>
</tr>
<tr>
<td>Ra</td>
<td>Surface Roughness (µm)</td>
<td>17</td>
</tr>
<tr>
<td>Spec. Bal</td>
<td>&gt;10g-205g (g)</td>
<td>0.002</td>
</tr>
<tr>
<td>Clean Bal</td>
<td>0 - 10 mg (+-g)</td>
<td>0.00005</td>
</tr>
<tr>
<td>Clean Bal</td>
<td>&gt; 10 mg - 1 g (+-g)</td>
<td>0.0001</td>
</tr>
</tbody>
</table>
Cleaned with AK-225G

Cleaned with CFC-113

Cleaned with D

Post Cleaned, Pre Verification Filter Papers
Cleaned with AK-225G

Post From AK-225G Verification Filter Papers

Cleaned with CFC-113

Cleaned with D
### Comparison of Contamination Analyzed on Coupons

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Solvents</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>*A (AK225G) (average)</td>
</tr>
<tr>
<td>MCA</td>
<td>Mass of Contaminant Applied (g)</td>
<td>0.0423</td>
</tr>
<tr>
<td>MCR</td>
<td>Mass of Contaminant Removed (g)</td>
<td>0.0410</td>
</tr>
<tr>
<td>CEF</td>
<td>Cleaning Effectiveness Factor (%)</td>
<td>97.11%</td>
</tr>
<tr>
<td>C</td>
<td>Amount of Contamination (mg/cm²)</td>
<td>0.9393</td>
</tr>
<tr>
<td>RC</td>
<td>Residual Contamination (mg/cm²)</td>
<td>0.0270</td>
</tr>
<tr>
<td>VNR</td>
<td>Verification Nonvolatile Residue Weight</td>
<td>0.0002</td>
</tr>
</tbody>
</table>

### Comparison of Removed Contamination Analyzed

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
<th>Solvents</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>*AK-225G (average)</td>
</tr>
<tr>
<td>SFW</td>
<td>Sample Filter contamination Weight (g)</td>
<td>0.0189</td>
</tr>
<tr>
<td>VFW</td>
<td>Verification Filter Contamination Weight (g)</td>
<td>0.0005</td>
</tr>
</tbody>
</table>

**Key:**
- **Baseline Solvents**
- * Series 1
- ** Series 2
Cleaning Efficiency of Solvents Continued

Discussion

- 3 potential baseline solvents identified
- Comparison to past test: “WSTF-IR-0134”
  - AK-225: 99% CEF
  - CFC-113: 97% CEF

Limited data on solvents, F,C,G,H,I
Conclusion
Conclusion

• The use of AK-225G in the validation step proves additional assurance that candidate solvents qualify as baseline solvents.

• Test results that provide percent cleaning efficiencies provide guidance into selecting baseline solvents.

• Future studies should consider adequate sample size to better define cleanliness efficiency.
Questions ?
Back up slides
NVR Measured in Solvent Blanks

- NVR criteria for solvents needs to be identified early on
- Evaporation rates role in analysis process
- Equipment compatibility with solvent
  - Beaker vs. pressure vessel

<table>
<thead>
<tr>
<th>Solvent:</th>
<th>Measured NVR (mg) in 100 ml</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-HFE- (average)</td>
<td>3.3</td>
<td>Sample from Pressure Vessel</td>
</tr>
<tr>
<td>C-HFE</td>
<td>0.3</td>
<td>Sample from new bottle</td>
</tr>
<tr>
<td>D-HFE</td>
<td>0.4</td>
<td></td>
</tr>
<tr>
<td>E-Solvent Blend</td>
<td>106700.3</td>
<td>Sample from Pressure Vessel</td>
</tr>
<tr>
<td>E-Solvent Blend</td>
<td>2.8</td>
<td>Sample from new mixture</td>
</tr>
<tr>
<td>F-HFE</td>
<td>0.1</td>
<td>Sample from bottle</td>
</tr>
<tr>
<td>G-HFE</td>
<td>0.2</td>
<td>Sample from bottle</td>
</tr>
<tr>
<td>H-HFE</td>
<td>0.1</td>
<td>Sample from bottle</td>
</tr>
<tr>
<td>I-HFE</td>
<td>0.3</td>
<td>Sample from bottle</td>
</tr>
<tr>
<td>J-Terpene</td>
<td>3.1</td>
<td>Sample from bottle.</td>
</tr>
<tr>
<td>K-HFE</td>
<td>0.1</td>
<td>Sample from bottle</td>
</tr>
</tbody>
</table>
Lessons Learned/Challenges

• Measure contaminants
• Identifying baseline, cleaning & verification solvent requirements.
• Testing location
  – Clean room
  – Vent hood
• Cross Contamination
• Experiment vs. Cleanroom practices.