Laboratory Evaluation of Drop-in Solvent Alternatives to n-Propyl Bromide for Vapor Degreasing

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Acknowledgements

• This study was performed for the U.S. Army Research Laboratory
  — MIPR Number: MIPR2AO80BW013

• Alternative solvents for these tests were supplied by:
  — 3M
  — DuPont Fluoroproducts
  — AGC Chemicals Americas, Inc.
Ground rules for this study

- Test solvent effectiveness in the vapor phase only
  - Effectiveness using spray, immersion, ultrasound, etc. were not evaluated in this study

- Alternative solvent candidates must:
  - Have lower expected toxicity than nPB
  - Not be a Hazardous Air Pollutant (HAP)
  - Not be an Ozone Depleting Substance (ODS)
  - Have no flash point
  - Be compatible with existing vapor degreasers
Solvents Tested

- **Ensolv® n-Propyl Bromide** (baseline)
- Alternative solvents tested were all azeotropes or azeotrope-like blends of trans-1,2 dichloroethylene with other solvents.
  - tDCE is an effective solvent on greases and oils but is too flammable for use in vapor degreasers
  - Non-flammable solvents are blended with tDCE to suppress flammability while maintaining solvency
  - Blending may also lower VOC content, GWP and cost, and improve exposure limits.
Alternative Solvents Tested:

- Novec™ HFE 72DE (3M) 113°F
- Vertrel® SDG (DuPont) 109°F
- Azeotrope A1 R&D Solvent (DuPont)* 118°F
- AE3000ATE (Asahi Glass Co., Ltd)* 108°F
  (nPB 156°F)

*These solvents are not yet approved by the EPA for use in the United States. Samples were provided by the suppliers “for laboratory use only”.

Note: Perfluorobutyl Iodide was to be included in this study but a suitable sample was not available in the required time frame.
What is an Azeotrope?

- A mixture of two or more liquids at a ratio where, when boiled, the resulting vapor has the same composition as the liquid.
- This lends stability to maintain the properties of the blend over time, critical in vapor degreasing applications.
Materials Compatibility Tests

- Test coupons were immersed in boiling solvent for 30 minutes; observed and weighed before & after
- Materials Tested:
  - Aluminum 7075-T6
  - Magnesium AZ31B-H24
  - Steel Maraging C-250
- No degradation was observed with any of the solvents.
Cleaning Effectiveness Tests

- A standard contaminant was applied to aluminum 2219 coupons and baked for 2 hours at 130°F.
- All coupons were photographed and weighed:
  - Before contamination
  - After contamination and baking
  - After vapor degreasing for 30 minutes
- Photos were taken in bright white and long wave ultraviolet light
- Clean control coupons, degreased and not degreased, were included.
Standard Contaminant per ADS-61A-PRF*

Mixed, brushed on, and baked two hours at 130°F:

2 parts* MIL-PRF-83282
   Fire resistant, synthetic hydrocarbon base hydraulic fluid

1 part* MIL-PRF-81322
   General purpose aircraft grease

1 tenth* part Carbon Black

*by weight

*ADS-61-PRF Performance Specification, Cleaners, Aqueous and Solvent, For Army Aircraft
Contaminant applied to test coupons

Aluminum 2219 sheet – 2.5 in. x 6 in.
Cleaning Results – Set 1

Smooth coupon surface, contaminant removed same day as applied
(Typical visual appearance and average percent removal)

- Ensolv nPB: 98.2% removed
- Novec HFE 72DE: 97.3% removed
- Vertrel SDG: 99.4% removed
- Azeo A1: 99.2% removed
- AE3000ATE: 99.2% removed
Cleaning Results under UV – Set 1

Smooth coupon surface, contaminant removed same day as applied
(Typical appearance under UV and average percent removal)

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Percent Removed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ensolv nPB</td>
<td>98.2%</td>
</tr>
<tr>
<td>Novec HFE 72DE</td>
<td>97.3%</td>
</tr>
<tr>
<td>Vertrel SDG</td>
<td>99.4%</td>
</tr>
<tr>
<td>Azeo A1</td>
<td>99.2%</td>
</tr>
<tr>
<td>AE3000ATE</td>
<td>99.2%</td>
</tr>
</tbody>
</table>
Cleaning Results – Set 1

Three solvents show very similar results.
Cleaning Results – Set 2, aged contaminant

Smooth coupon surface, contaminant removed 7 days after application
(Typical visual appearance and average percent removal)

Ensolv nPB 96.2% removed
Novec HFE 72DE 94.8% removed
Vertrel SDG 99.1% removed
Azeo A1 97.5% removed
AE3000ATE 98.9% removed
Cleaning Results – Set 2, aged contaminant

Smooth coupon surface, contaminant removed 7 days after application
(Typical appearance under UV and average percent removal)
Cleaning Results – Set 2, aged contaminant

Cleaning Effectiveness Ranges and Averages Set 2

Cleaning efficiency

Solvent

- Ensolv nPB
- Novec HFE 72DE
- Vertra SDG
- Azeo A1
- AE3000 ATE
Cleaning Results – Set 3, rough surface

Grit blasted coupon surface, contaminant removed same day as applied
(Typical visual appearance and average percent removal)

Ensolv nPB 97.7% removed
Novec HFE 72DE 99.7% removed
Vertrel SDG 99.4% removed
Azeo A1 99.5% removed
AE3000ATE 98.5% removed
Cleaning Results – Set 3, rough surface

Grit blasted coupon surface, contaminant removed same day as applied
(Typical appearance under UV and average percent removal)

- Ensolv nPB 97.7% removed
- Novec HFE 72DE 99.7% removed
- Vertrel SDG 99.4% removed
- Azeo A1 99.5% removed
- AE3000ATE 98.5% removed
Cleaning Results – Set 3, rough surface

![Cleaning Effectiveness Ranges and Averages Set 3](chart.png)
Combined Cleaning Results

Cleaning Effectiveness Ranges and Averages - Combined

Cleaning efficiency

Solvent

Ensolv nPB (1)
Ensolv nPB (2)
Ensolv nPB (3)
Novec HFE 72DE (1)
Novec HFE 72DE (2)
Novec HFE 72DE (3)
Vertrel SDG (1)
Vertrel SDG (2)
Vertrel SDG (3)
Azeo A1 (1)
Azeo A1 (2)
Azeo A1 (3)
AE3000 ATE (1)
AE3000 ATE (2)
AE3000 ATE (3)
Cleaning effectiveness versus tDCE content

* tDCE% as shown in the Vendor Technical Data Sheet
** tDCE% as shown in the Material Safety Data Sheet
Results

- All solvents were compatible with metals tested
- All solvents cleaned in the range of or better than n-propyl bromide
  - Vertrel SDG cleaned the most consistently; AE3000ATE was very close.
  - All but Vertrel SDG showed reduced cleaning effectiveness on aged contamination
  - Cleaning effectiveness did NOT correlate with tDCE%
  - Cleaning effectiveness of any of these solvents may be adequate for the end use

- Results may vary with other materials, contaminants, and hardware configurations
Observations about the test method

- Both carbon black and ultraviolet light were useful visual indicators of contaminant residues.
- Despite the two-hour bake, contaminant aged just a few days was more difficult for some solvents to remove.
- Results varied between smooth and roughened test coupons.
- Contaminant aging had a more significant impact on cleaning effectiveness than surface roughening.
Conclusions

- Based on this limited laboratory study, solvent blends of trans-1,2 dichloroethylene with HFEs, HFCs, or PFCs appear to be viable alternatives to n-propyl bromide for vapor degreasing.
  - The lower boiling points of these blends may lead to greater solvent loss during use.
  - Additional factors must be considered when selecting a solvent substitute, including stability over time, VOC, GWP, toxicity, and business considerations.
Questions? www.nasa.gov