MICROWAVE ASSISTED SOLVENT EXTRACTION OF POLYVINYL CHLORIDE (PVC) FOR ANALYSIS OF PLASTICIZERS BY GAS CHROMATOGRAPHY
Objective

To demonstrate the application of Microwave Assisted Solvent Extraction (MASE™) for the analysis of Plasticizers in Poly Vinyl Chloride (PVC) wire wrap by GC/FID
Equipment Used

• MARS-X™ Microwave Sample Preparation System, equipped with fiberoptic temperature control, and safety features for use of organic solvents
• GreenChem™ Extraction Vessels
• VAF-100™ Vacuum Filtration Apparatus
• N-EVAP™ Nitrogen Evaporation Device
• Varian STAR 3400 Cx GC/FID
MARS-X Safety Features

- Solvent detector which monitors and reacts to solvent vapor levels in the microwave cavity
- Exhaust fan which exchanges air through the microwave cavity
- Air flow switch which assures constant air exchange
- Vessel design incorporating over pressurization venting mechanism
- Reinforced microwave door
MARS-X Cavity, Vessels and Exhaust System

- Microwave Cavity
- Extraction Vessel
- Exhaust Fan
- Solvent Detector
- Air Flow Switch
GreenChem Extraction Vessels

- Innovative Frame Design
- Glass or Teflon™ inner liner with reinforced sleeve
- Fourteen position turntable for high sample throughput
- Autovent style sealing mechanism
- 200 ºC operating temperature
- 200 psi operating pressure
Sample Matrix

Wire Wrap cut into small pieces

Next Slide
Sample Matrix

- PVC wire wrap received as a three part completed wire
- Outer wrap was separated from internal wires
- Outer wrap was cut into small pieces
Analytes

Diisodecyl Phthalate (DIDP)
Ditridecyl Phthalate (DTDP)
Triisononyl Trimellitate (TINTM)
Traditional Soxhlet Procedure

- Weigh 0.5 grams of PVC into a Soxhlet thimble
- Add 150 mL solvent* and assemble Soxhlet apparatus
- Run for 16 hours
- Allow to cool
- Take extract to dryness
- Weigh Dry Residue
- Re-dissolve residue in THF
- Analyze by GC/FID

*solvent = di-Ethyl Ether
Microwave Extraction Method Development

% Extractables (Gravimetric)

- MASE 95 °C
- MASE 115 °C
- MASE 135 °C
- Soxhlet
Parameters for Microwave Extraction of Poly Vinyl Chloride Wire Wrap

- Sample Size: 0.5g
- Solvent: di-Ethyl Ether
- Extraction Temp.: 135 °C
- Extraction Time: 30 min.
Microwave Extraction Procedure

Weigh 0.5 grams of PVC into a GreenChem glass liner

Add 40 mL solvent* and assemble the vessel

Extract in the MARS-X for 30 min. @ 135 °C

Allow to cool and filter

Take extract to dryness

Weigh dry residue

Re-dissolve residue in THF

Analyze by GC/FID

*solvent = di-Ethyl Ether
## PVC Wire Wrap Results
### Gravimetric Analysis

<table>
<thead>
<tr>
<th>Technique</th>
<th>Average % Extractables*</th>
<th>% RSD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soxhlet</td>
<td>26.14</td>
<td>4.64</td>
</tr>
<tr>
<td>Microwave</td>
<td>25.23</td>
<td>2.87</td>
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</tbody>
</table>

* N=6
GC Operating Conditions

- 1/8” Stainless Steel Packed Column 3% SP-2100 on 100/120 Supelcoport™
- Flame Ionization Detector
- Injector Temp 330 ºC
- Detector Temp 335 ºC

Heating Profile

Initial: 70 ºC Hold 2 min.
Ramp: 30 ºC/min 300 ºC
Ramp: 5 ºC/min 320 ºC
Hold: 20 min.
## PVC Wire Wrap Results

**GC/FID Analysis**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Soxhlet Avg. (%)</th>
<th>Soxhlet (% RSD)</th>
<th>Microwave Avg. (%)</th>
<th>Microwave (% RSD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DIDP</td>
<td>18.68</td>
<td>0.0117</td>
<td>18.90</td>
<td>0.0167</td>
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<td>DTDP</td>
<td>2.08</td>
<td>0.0299</td>
<td>2.21</td>
<td>0.0214</td>
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<tr>
<td>TINTM</td>
<td>2.52</td>
<td>0.0349</td>
<td>2.82</td>
<td>0.0205</td>
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</tbody>
</table>

* N=6
Conclusions

• MASE is a fast, safe, economical technique to prepare PVC samples for plasticizer analysis by gravimetric and GC/FID methods.

• MASE yields acceptable results when compared to the standard Soxhlet method.

• MASE provides extraction with 1/3 the amount of solvent required for the traditional method.

• MASE requires 1/10 the time of the Soxhlet method.