Bag-In-Box Package Testing for Beverage Compatibility

Based on Proven Plastic Bottle & Closure Test Methods

Crochiere & Associates, LLC
Standard & Analytical Tests

• Sensory evaluation is subjective but it is the final word or approval.
• Analytical tests and specifications have to be calibrated to each beverage type.
• Reliable analytical tests speed development and reduce costs to all in the industry.
Sensory Testing

- Material effects
  - Migration and scalping
  - Contamination or “off” flavors
  - Missing flavors
  - Increase plastic surface area to accelerate test
  - Short term tests (weeks)

- Aging effects
  - Oxidation (reduction) of beverage
  - Color change
  - “Antioxidant” loss
  - Staling flavor
  - Use oxygen chamber to accelerate test
  - Long term test
  - Possible degradation of packaging
#### Package Compatibility Issues

<table>
<thead>
<tr>
<th>Added to Beverage</th>
<th>Removed from Beverage</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Ingress</strong></td>
<td><strong>Egress</strong></td>
</tr>
<tr>
<td>• Oxygen – Added during filling and ingress</td>
<td>• Carbonation</td>
</tr>
<tr>
<td>• Environmental contaminants – TCA</td>
<td>• Moisture – Weight loss</td>
</tr>
<tr>
<td>• Light – “Light Box Test”, %T</td>
<td></td>
</tr>
<tr>
<td>• Heat</td>
<td></td>
</tr>
<tr>
<td><strong>Migration</strong></td>
<td><strong>Scalping</strong></td>
</tr>
<tr>
<td>• Plastics additives</td>
<td>• Flavor &amp; odor components absorbed by packaging</td>
</tr>
<tr>
<td>• Scavengers and by-products</td>
<td></td>
</tr>
<tr>
<td>• Polymer degradation products</td>
<td></td>
</tr>
</tbody>
</table>
Oxygen Ingress Issues

• Oxygen ingress through all plastic components
• Barrier and scavenger performance is very dependant on temperature and absorption by the plastic of water, alcohol, flavors, etc.
• If the beverage reacts with oxygen, it is impossible to determine ingress with wine, beer or juice in package
• Total Package Oxygen measurements on packages filled with deoxygenated water or alcohol solution provides ingress rate
• Ingress rate with oxygen scavengers is not always zero
• It is possible to “use up” scavengers before filling or end of product shelf-life
Effect of Relative Humidity on O2 Permeability of the Polymer

Huige
Effect of Storage Temperature on O2 Permeability for various Polymers

![Graph showing the effect of storage temperature on O2 permeability for different polymers. The x-axis represents temperature in degrees Fahrenheit (40 to 100), and the y-axis represents O2 permeability in cc.mil/100 in2.day.atm. The graph includes lines for PET, EVAL E, EVAL F, and Nylon MXD6, each with a different slope and intercept.](image-url)
Example of the effects of temperature and outside RH on shelflife obtained by model

For 0.5L, 3-layer bottle with 9% EVAL-F, t=20 mil

(Over estimated by 40% due to liquid contents)
Example of Oxygen Ingress Test on Plastic Bottles

- Plot Total package oxygen (ppm) vs. Time (days)
- Typical shelf life for beer is when it accumulates 1 ppm of oxygen
- Estimated shelf life for some juices is when it accumulates 6-7 ppm of oxygen
- Intersection of bottle oxygen plot with shelf life limits determines the shelf life of that beverage in that bottle. (example, Bottle A – 50 days for beer and 150 days for juice)
Bag-In-Box Oxygen Ingress Test Example

- Small number of bags filled to provide an example for this presentation.
- Examples of EV-OH and MetPET 3 liter bags evaluated.
- The manufacture of the bags, grades and thicknesses of the films and the type of taps are not being disclosed.
- Any variation in the bag manufacturing or materials will change the results.
- Bags pre-flushed with nitrogen before filling.
- Most were vented through valve to minimize air.
Bag-In-Box Oxygen Ingress Test Analysis

- Studied headspace effect on initial oxygen
- Some bags exposed to 100 F for 7 days
- Studied change in headspace or bubble volume
- Studied oxygen ingress over time for both types of bags at both temperatures
- Compared dissolved oxygen to total package oxygen measurements
- Compared ingress rate to other wine packages
Initial Total package Oxygen as a Function of Headspace Volume - 3 liter bag

y = 0.0382x + 0.5477
Oxygen Ingress for 3 Liter Bags Only Measuring Dissolved Oxygen

y = 0.0633x + 0.6878
y = 0.0989x + 0.5791

Dissolved Oxygen (ppm)

Days

EV-OH 72 F
EV-OH 100 F for 7 days
MetPET 72 F
MetPET 100 F for 7 days
## Results of Wine Packaging Tests to Date

<table>
<thead>
<tr>
<th>Package or Closure Type</th>
<th>Barrier Material Type</th>
<th>Package Size</th>
<th>Average Oxygen Ingress ppm/day TPO</th>
<th>Average Oxygen Ingress ppm/year TPO</th>
<th>Average Years to 5 ppm TPO</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>BIB</strong></td>
<td></td>
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<tr>
<td>Bag-In-Box</td>
<td>EV-OH</td>
<td>3000</td>
<td>0.070</td>
<td>25.6</td>
<td>0.2</td>
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<td>Bag-In-Box</td>
<td>MetPET</td>
<td>3000</td>
<td>0.110</td>
<td>40.2</td>
<td>0.1</td>
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<td><strong>Corks</strong></td>
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<td>Natural cork</td>
<td>Super Select 49mm</td>
<td>750</td>
<td>0.022</td>
<td>8.0</td>
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<td>Technical cork</td>
<td>1+1</td>
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<td>0.010</td>
<td>3.7</td>
<td>1.4</td>
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<tr>
<td>Synthetic Cork A</td>
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<td>0.021</td>
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<td>Synthetic Cork B</td>
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<td>0.018</td>
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<td>0.8</td>
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<td>Synthetic Cork C</td>
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<tr>
<td>Synthetic Cork D</td>
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<td>0.020</td>
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<tr>
<td>Synthetic Cork E</td>
<td>750</td>
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<td>0.012</td>
<td>4.4</td>
<td>1.1</td>
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<td><strong>Aluminum</strong></td>
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<tr>
<td>Optimum Tin</td>
<td>750</td>
<td>0.000001</td>
<td>0.002</td>
<td>2500</td>
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<tr>
<td>Optimum Saranex</td>
<td>750</td>
<td>0.00002</td>
<td>0.006</td>
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<tr>
<td>Optimum Polyester</td>
<td>750</td>
<td>0.00411</td>
<td>1.5</td>
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<td>0.01644</td>
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<td>Twist-on Tin 5</td>
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<tr>
<td>Roll-on Tin 7</td>
<td>750</td>
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<td>Roll-on Tin 8</td>
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<td>Twist-on Saranex 1</td>
<td>750</td>
<td>0.00008</td>
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<td>Twist-on Saranex 3</td>
<td>750</td>
<td>0.00029</td>
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<td>Roll-on Saranex 5</td>
<td>750</td>
<td>0.00069</td>
<td>0.253</td>
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<td>Roll-on Saranex 7</td>
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<td>Twist-on Saranex 9</td>
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<td>0.00238</td>
<td>0.888</td>
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<td>Roll-on Saranex 11</td>
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<td>0.00486</td>
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<td>Roll-on Saranex 13</td>
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<td>0.00593</td>
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<td>Roll-on Saranex 15</td>
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<td>Roll-on Polyethylene</td>
<td>750</td>
<td>0.02980</td>
<td>10.9</td>
<td>0.5</td>
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</tr>
</tbody>
</table>
Flavor Scalping

• Polymers absorb flavors and odors from the beverage.
• Polymers absorb materials with similar polarity or solubility parameters
• Lightly flavored beverages are most vulnerable
• Studied by Remco W.G. van Willige, G.D. Sadler, Australian Wine Research Institute from the food and beverage science point of view
Factors Effecting Flavor Scalping

- Polymer type and crystallinity
  - LDPE > HDPE > PP > PET > PEN > Saran
- Chemical properties of flavor/odor compound
  - Non polar more likely to scalp
- Relative amounts of plastic and flavor (wt/wt) affects amount scalped
- Polymer surface area to flavor amount affects rate of scalping
- Amount of flavor in beverage vs. amount scalped vs. flavor threshold determines significance
Flavor Scalping Analysis

- Sensory analysis
- Analytical testing of beverage for specific chemical compounds
- Analytical testing of packaging material for specific chemical compounds
- Program underway with International Society of Beverage Technologists to develop standard test for evaluating packages
- This work will lead to a similar test for wines
## Package Scalping Comparison

<table>
<thead>
<tr>
<th></th>
<th>Glass Bottle &amp; Synthetic Cork</th>
<th>Glass Bottle &amp; Screw cap</th>
<th>BIB</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Wine volume</strong></td>
<td>0.75 liters</td>
<td>0.75 liters</td>
<td>3 liters</td>
</tr>
<tr>
<td><strong>Plastic area</strong></td>
<td>2.8 cm²</td>
<td>3.1 cm²</td>
<td>1550 cm²</td>
</tr>
<tr>
<td><strong>Surface area/volume</strong></td>
<td>3.7 cm²/liter</td>
<td>4.1 cm²/liter</td>
<td>517 cm²/liter</td>
</tr>
<tr>
<td><strong>Plastic material</strong></td>
<td>LDPE</td>
<td>Saranex</td>
<td>LDPE or PET</td>
</tr>
</tbody>
</table>
Environmental Contamination

• Chemical contamination from materials in atmosphere where the package is located
• Relative barrier performance determined by storing filled packages in a concentrated environment
• Analysis for chemical in beverage by appropriate method (GC, HPLC, etc)
TCA Barrier Properties of Crown Liners

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Additional Tests Open for Discussion
Oxygen Pick-up and Oxygen Control

• Oxygen Pick-up
  – The amount of oxygen added to the package and beverage from the filling process, the closing or sealing process and ingress through the package.

• Oxygen Control
  – The process of controlling the amount of oxygen added through the expected life of the package and product
Oxygen Pickup vs. Process in Wine During Filling and Shelf Life (750 ml)

- Tank
- Filler
- Filled bottle
- Headspace
- 1st year ingress
- 2nd year ingress

Oxygen Pickup (ppm)
- Minimum (ppm)
- Typical cork (ppm)
- Typical screwcap (ppm)
- Maximum (ppm)

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Oxygen Control Corrective Actions

- Inert gas in tank & filler bowl headspace
- Filter from bottom to top
- Minimize turbulent flow in process
- Control filling process, dip tubes, valves, rates
- Pre-evacuate bottles with inert gas
- Headspace flush with inert gas or liquid nitrogen
- Vacuum corking
- Closure and liner selection
- Proper procedures

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Accelerated Aging

Heat versus Oxygen Chamber
Accelerate Aging – Heat Exposure

• Must be used with caution
• Degrades beverage
• Degrades polymers
• Increases migration, scalping, ingress & egress
• No measurable way to predict acceleration
Accelerated Aging – Oxygen Chambers

- Accelerates oxygen ingress and aging without heat side effects on plastic or beverage
- 95-100% oxygen atmosphere give approximately 4.7 times acceleration
- Effective on beverage and scavenger shelf life
Oxygen Chamber Bottle Test at Room Temperature

Total Package Oxygen (ppm) vs. Days

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Moisture Loss Through Plastic Packaging

• Moisture evaporates through plastic bottle
• Changes fill level and concentrations in beverage
• Determined by measuring weight loss on non-carbonated product
• Affected by temperature
Migration Analysis

- Global migration weight analysis
- Specific chemical analysis (lubricant)
- Sensory analysis
- Affected/accelerated by heat
- Accelerated by increased exposure
Literature references
