Bag-In-Box Package Testing for Beverage Compatibility

Based on Proven Plastic Bottle & Closure Test Methods

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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

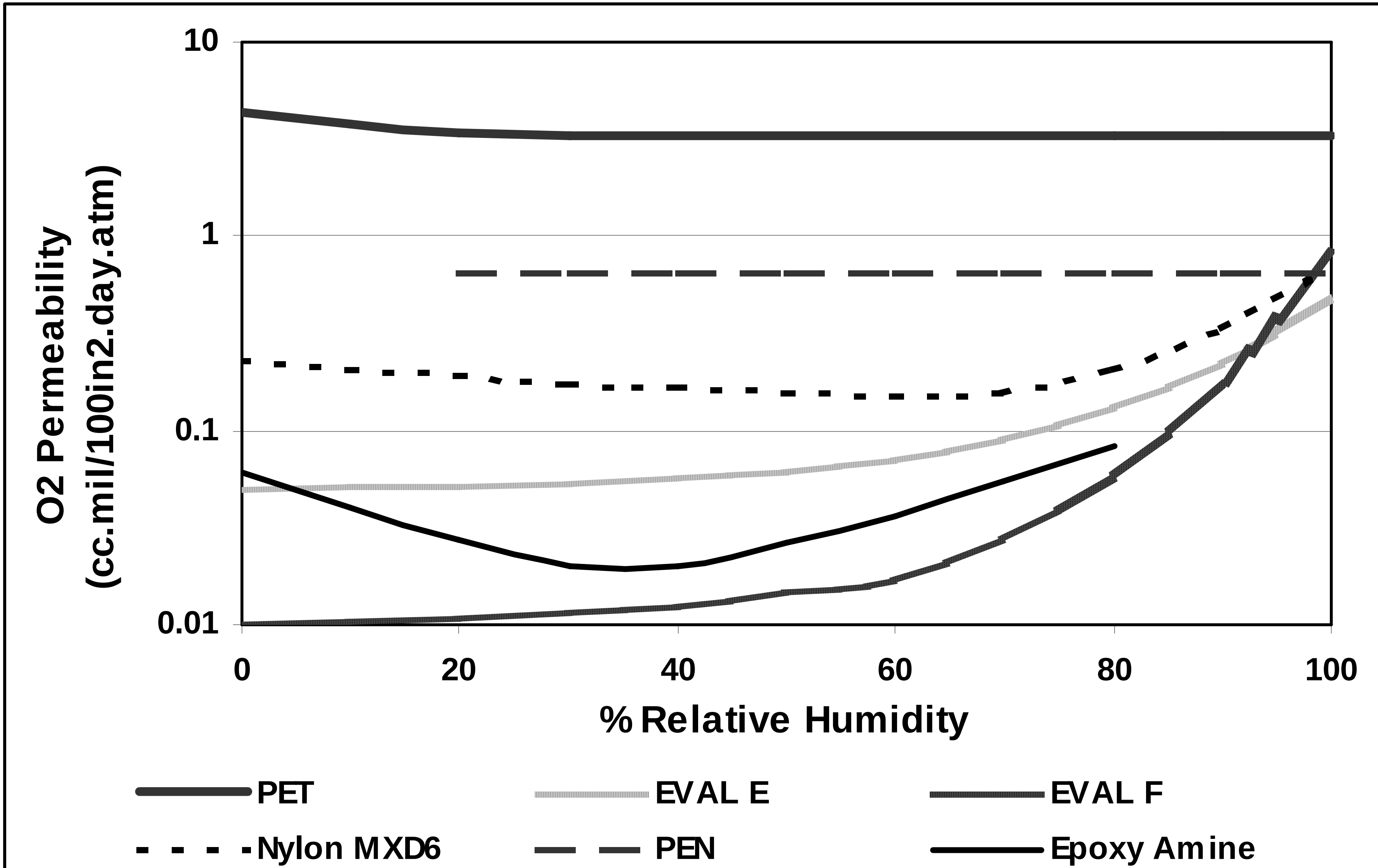
Added to Beverage	Removed from Beverage
Ingress •**Oxygen – Added during filling and ingress** •**Environmental contaminants – TCA** •Light – “Light Box Test”, %T •Heat	Egress •Carbonation •Moisture – Weight loss
Migration •Plastics additives •Scavengers and by-products •Polymer degradation products	Scalping •**Flavor & odor components absorbed by packaging**

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

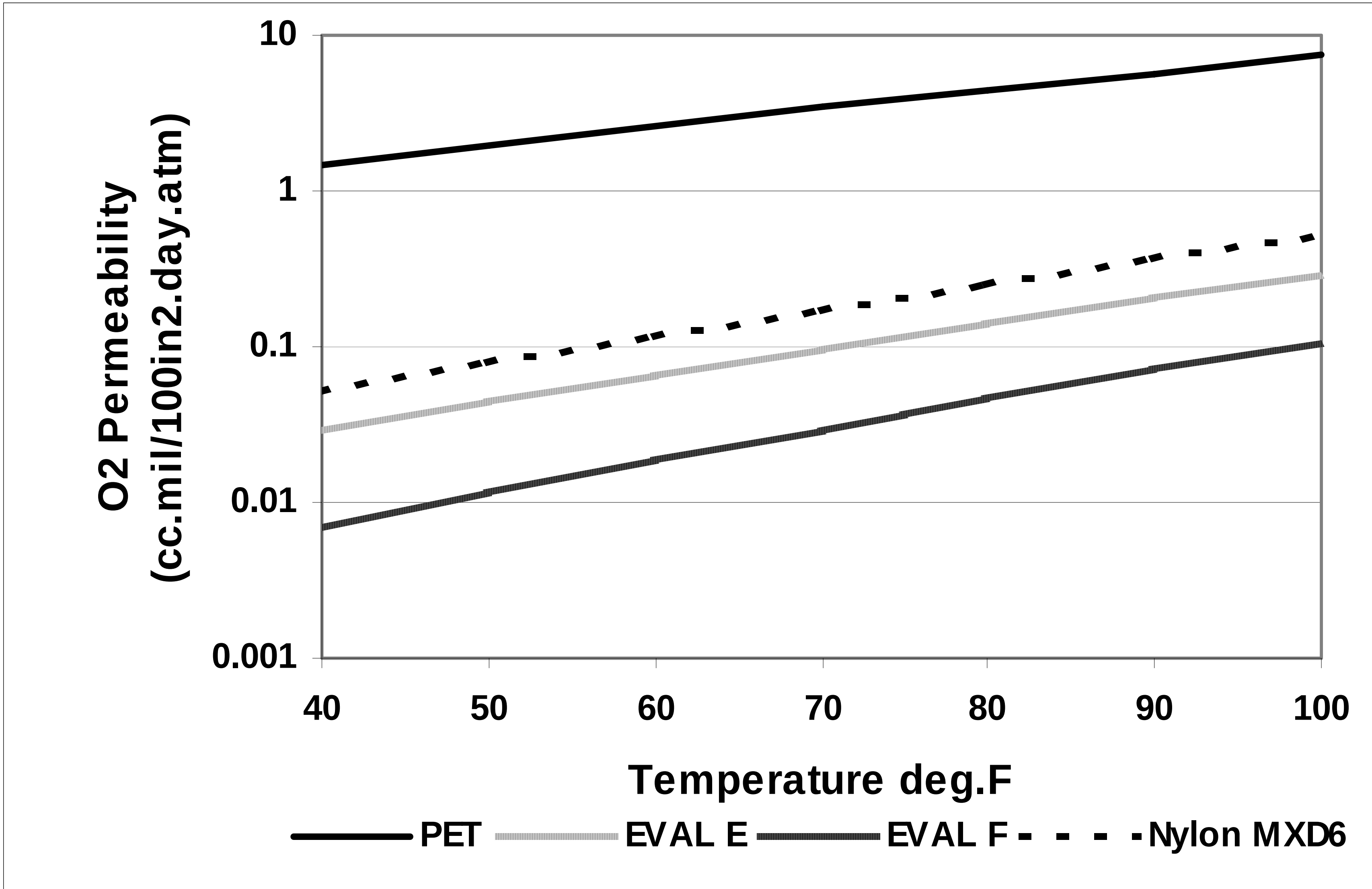
Effect of Relative Humidity on O2 Permeability of the Polymer

Huige



Effect of Storage Temperature on O2 Permeability for various Polymers

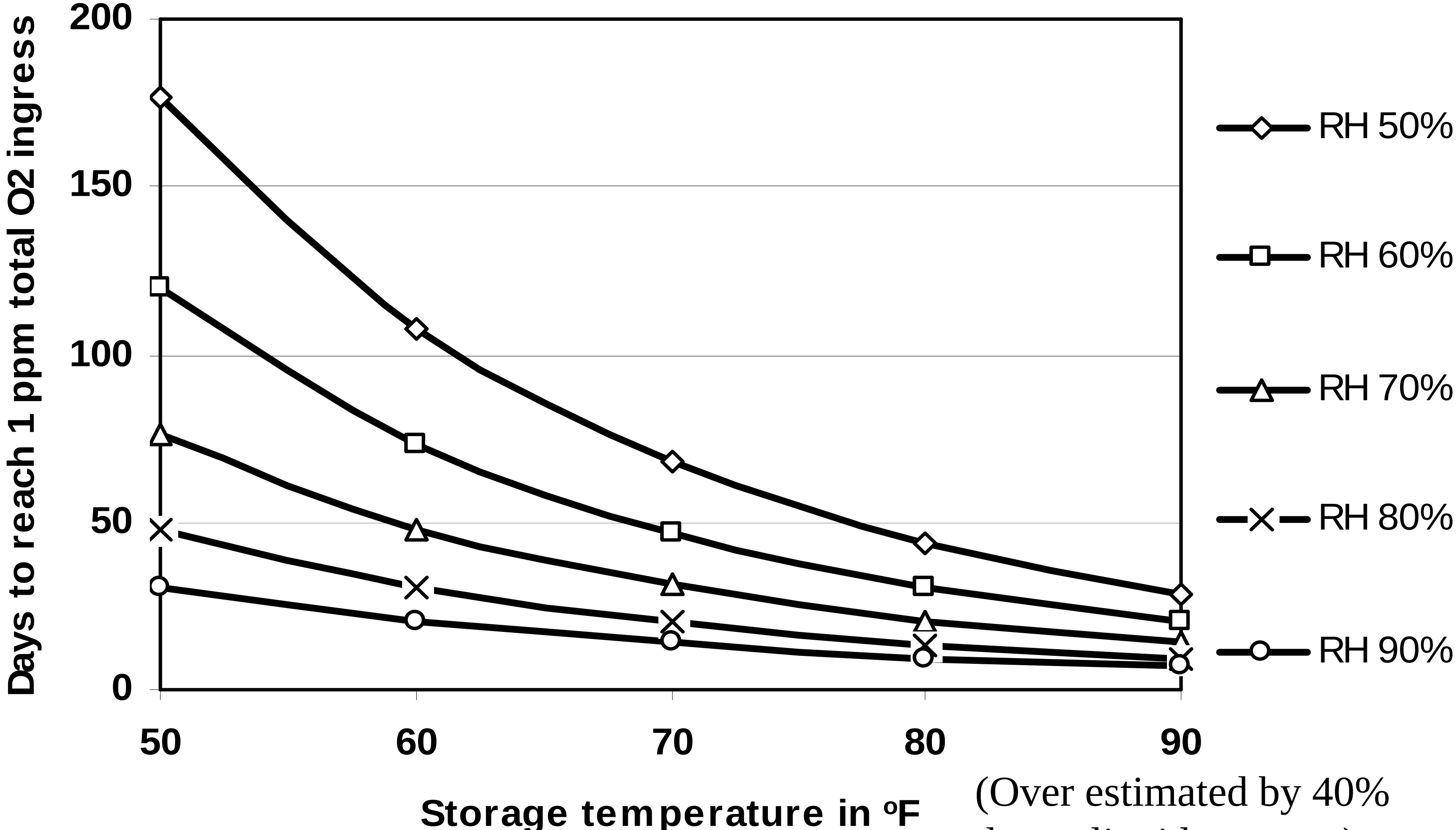
Huige



Example of the effects of temperature and outside RH on shelflife obtained by model

Huige

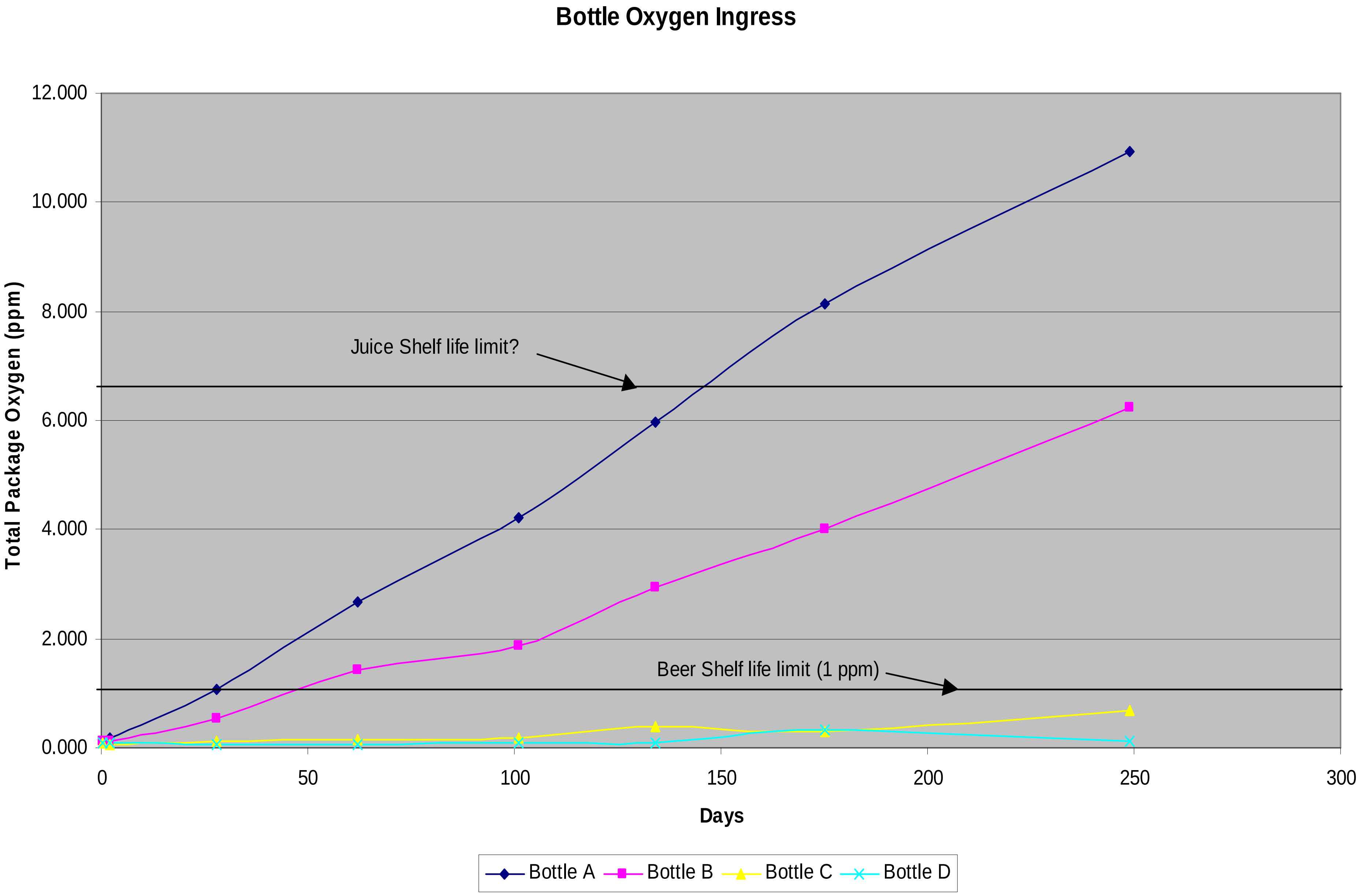
For 0.5L, 3-layer bottle with 9% EVAL-F, t=20 mil



(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)



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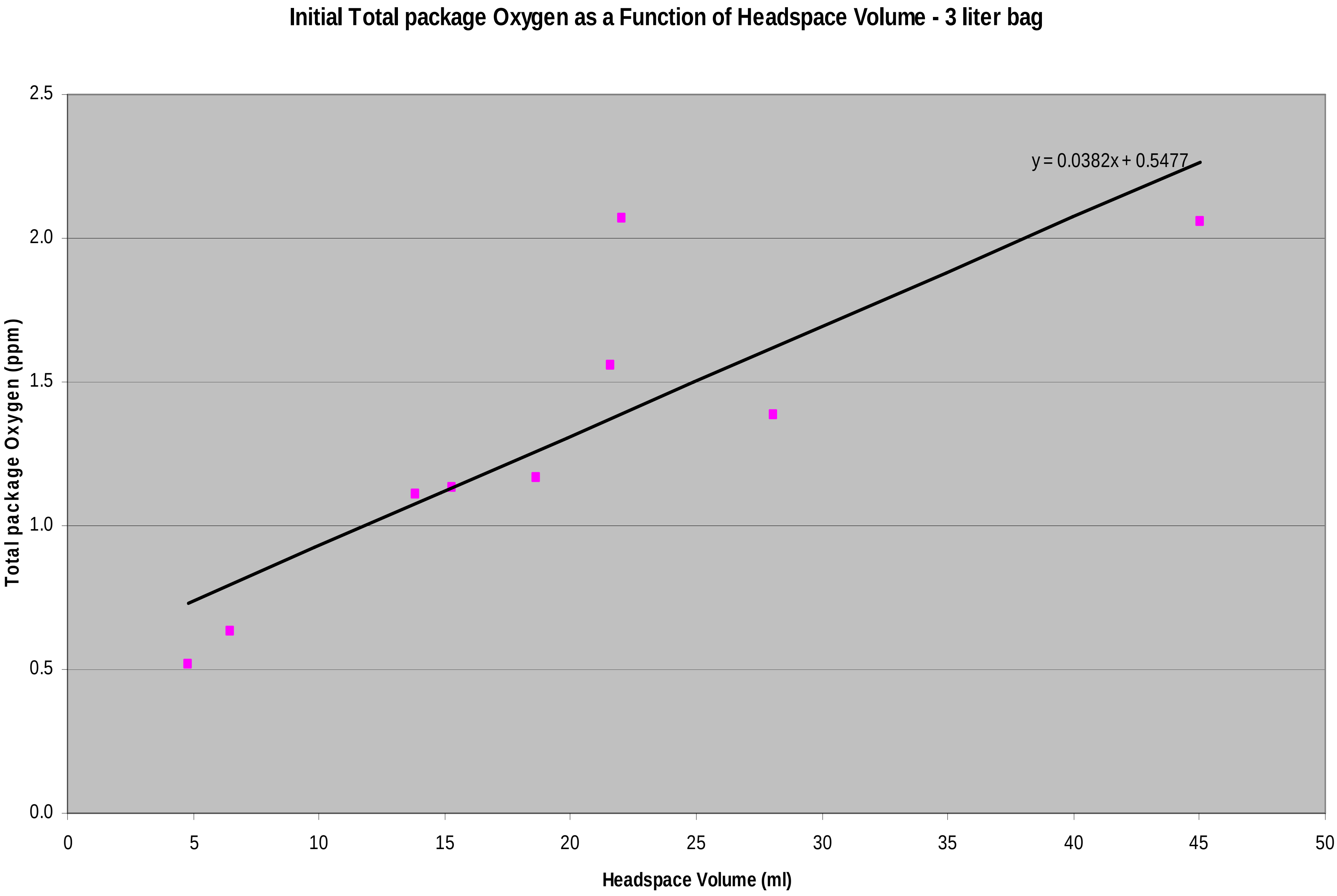
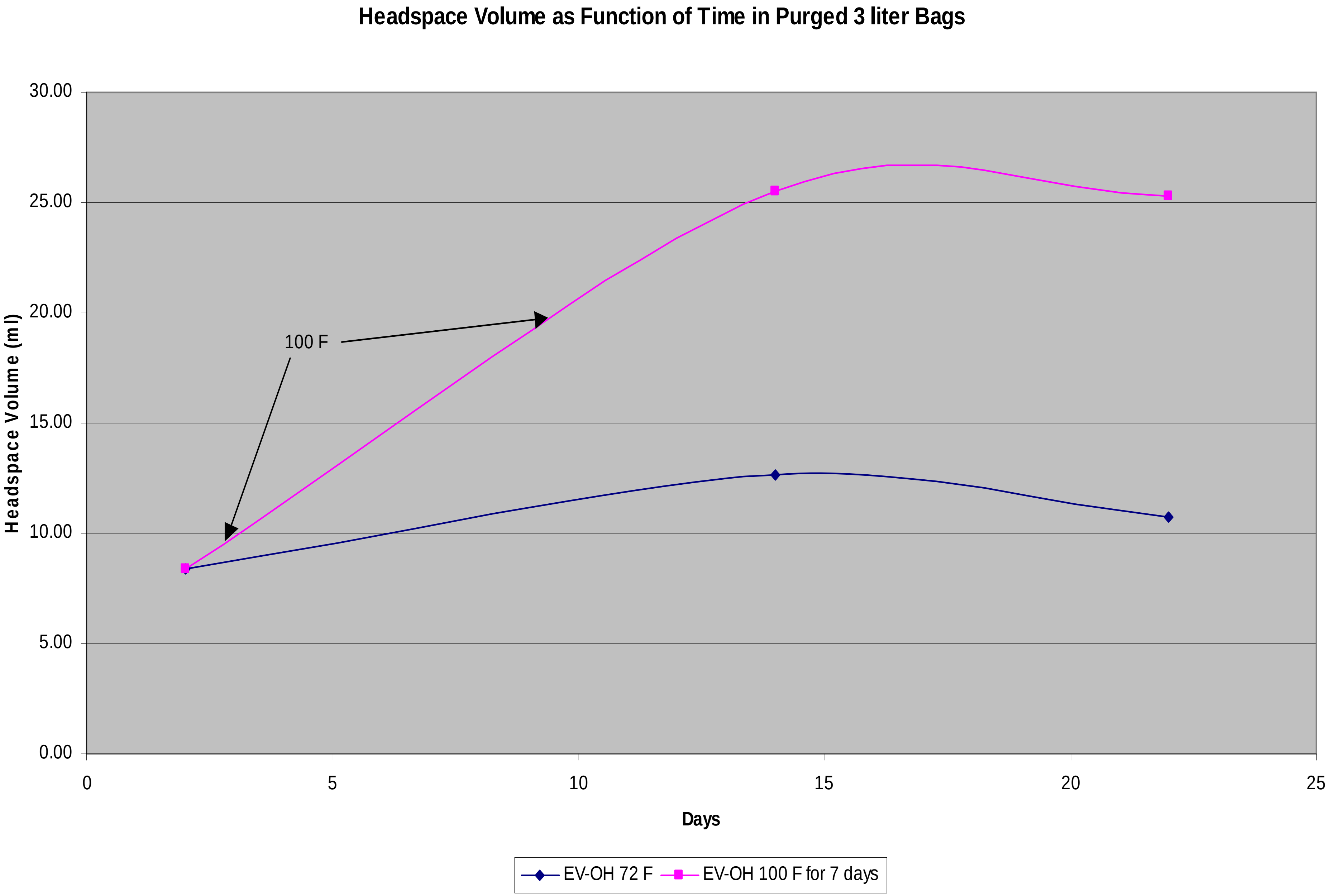
Bag-In-Box Oxygen Ingress Test Example

- 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
Total package Oxygen (ppm)
2.5
2.0
1.5
1.0
0.5
0.0
0
5
10
15
20
25
30
35
40
45
50
Headspace Volume (ml)



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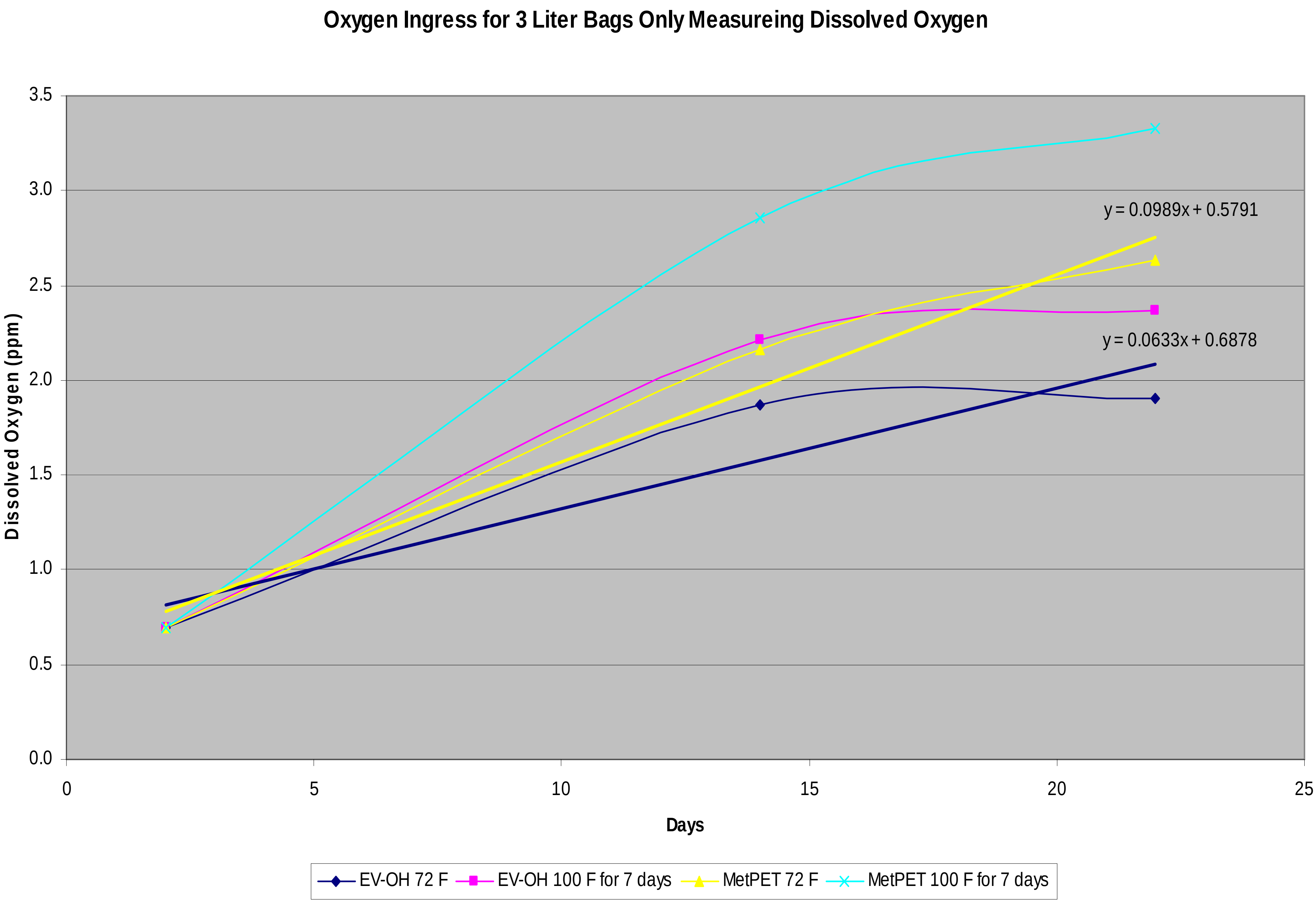

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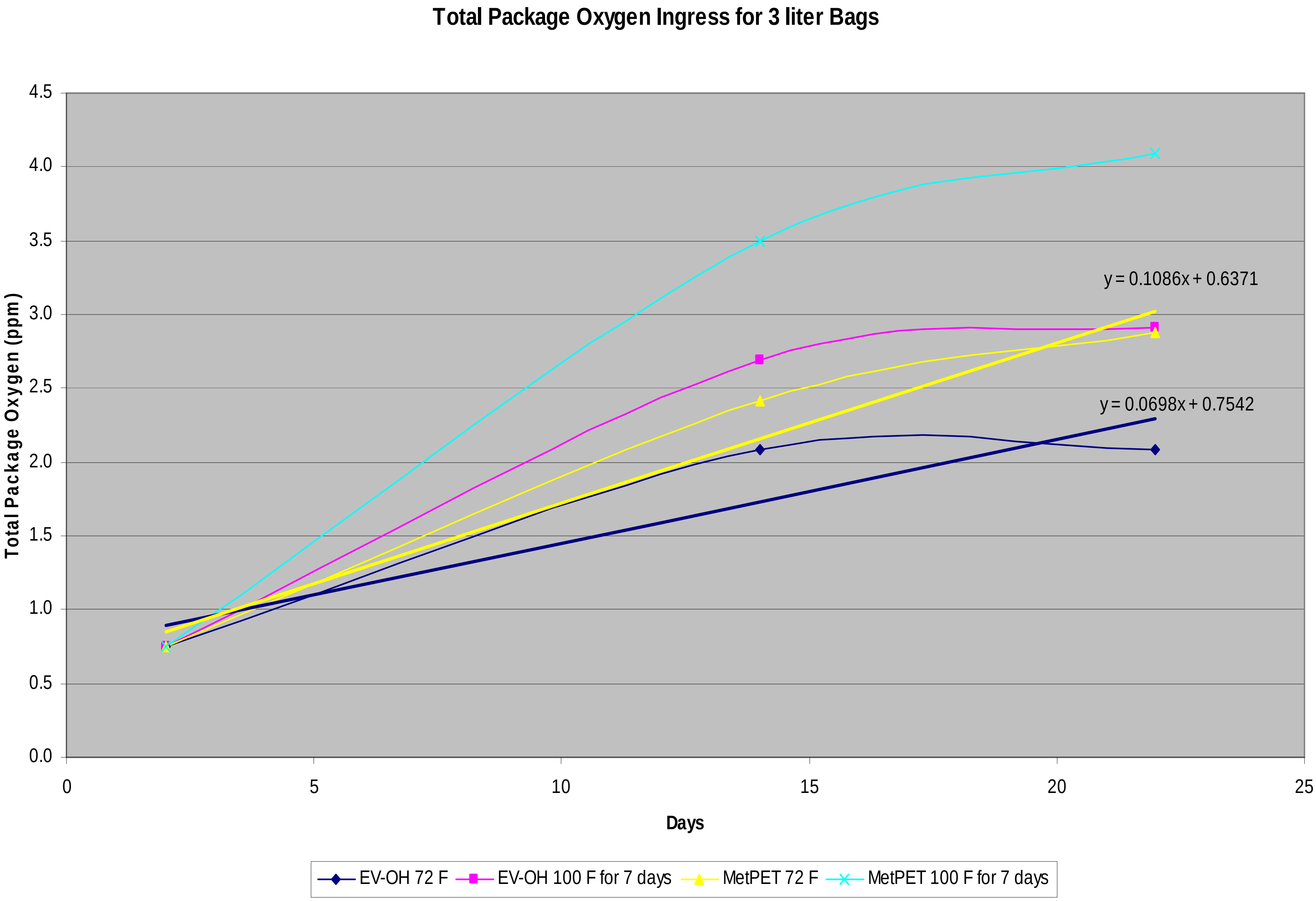

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Results of Wine Packaging Tests to Date

Package or Closure Type	Barrier Material Type	Package Size	Average Oxygen Ingress	Average Oxygen Ingress	Average Oxygen Ingress
		ml	ppm/day TPO	ppm/year TPO	years to 5 ppm TPO
BIB					
Bag-In-Box	EV-OH	3000	0.070	25.6	0.2
Bag-In-Box	MetPET	3000	0.110	40.2	0.1
Corks					
Natural cork	Super Select 49mm	750	0.022	8.0	0.6
Technical cork	1+1	750	0.010	3.7	1.4
Synthetic Cork	A	750	0.021	7.7	0.7
Synthetic Cork	B	750	0.018	6.6	0.8
Synthetic Cork	C	750	0.026	9.5	0.5
Synthetic Cork	D	750	0.020	7.3	0.7
Synthetic Cork	E	750	0.012	4.4	1.1
Aluminum					
Optimum	Tin	750	0.00001	0.002	2500
Optimum	Saranex	750	0.00002	0.006	833
Optimum	Polyester	750	0.00411	1.5	3.3
Optimum	Polyethylene	750	0.01644	6.0	0.8
Roll-on	Tin 1	750	0.00005	0.017	294
Roll-on	Tin 3	750	0.00007	0.025	199
Twist-on	Tin 5	750	0.00008	0.029	174
Roll-on	Tin 7	750	0.00013	0.046	109
Roll-on	Tin 8	750	0.00043	0.158	32
Twist-on	Saranex 1	750	0.00008	0.029	171
Twist-on	Saranex 3	750	0.00029	0.104	48
Roll-on	Saranex 5	750	0.00069	0.253	20
Roll-on	Saranex 7	750	0.00108	0.395	13
Twist-on	Saranex 9	750	0.00238	0.868	5.8
Roll-on	Saranex 11	750	0.00486	1.77	2.8
Roll-on	Saranex 13	750	0.00593	2.16	2.3
Roll-on	Saranex 15	750	0.01816	6.63	0.8
Roll-on	Polyester	750	0.01330	4.86	1.0
Roll-on	Polyethylene	750	0.02980	10.9	0.5

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

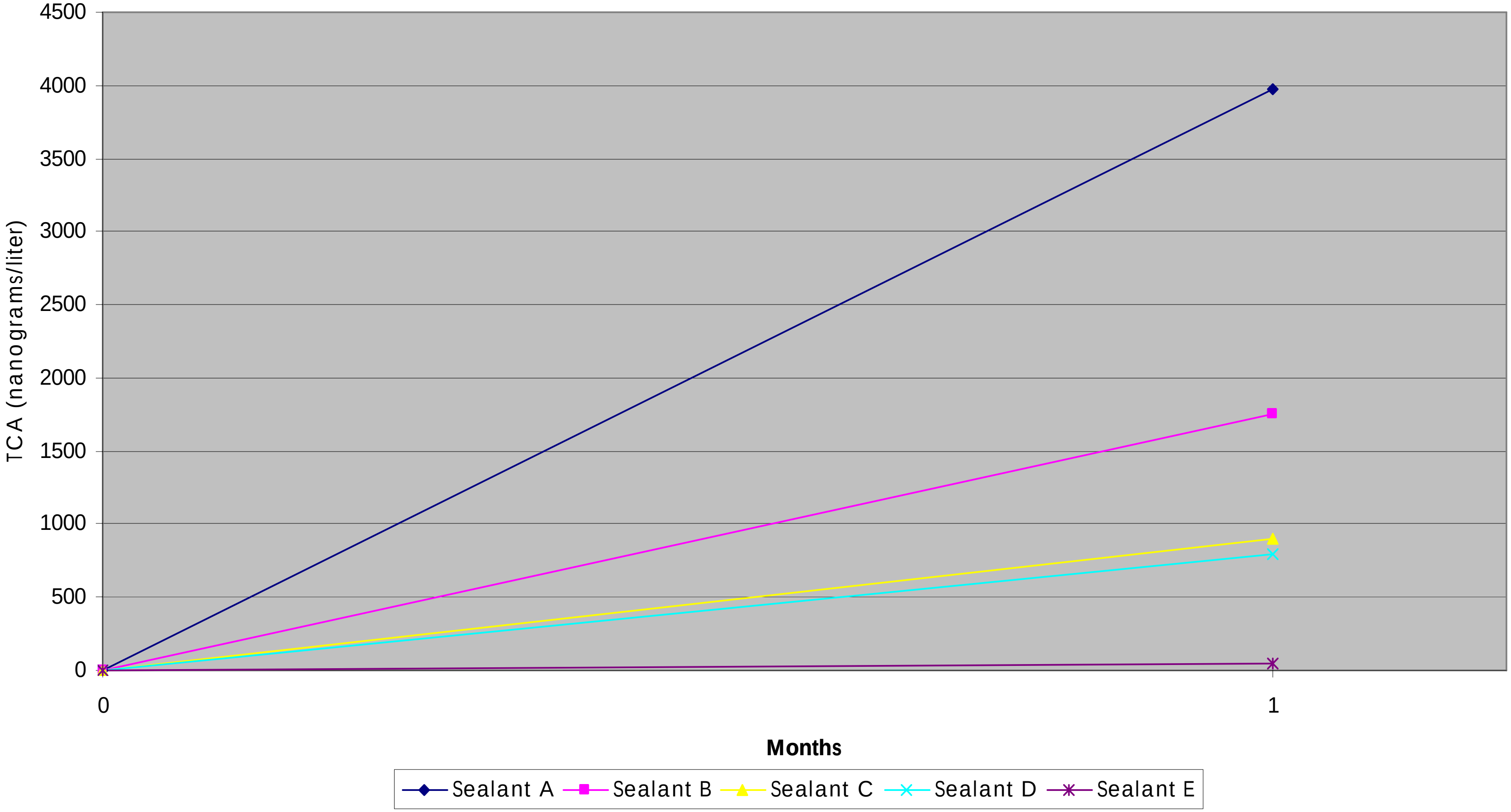
Package Scalping Comparison

	Glass Bottle & Synthetic Cork	Glass Bottle & Screw cap	BIB
Wine volume	0.75 liters	0.75 liters	3 liters
Plastic area	2.8 cm2	3.1 cm2	1550 cm2
Surface area/volume	3.7 cm2/liter	4.1 cm2/liter	517 cm2/liter
Plastic material	LDPE	Saranex	LDPE or PET

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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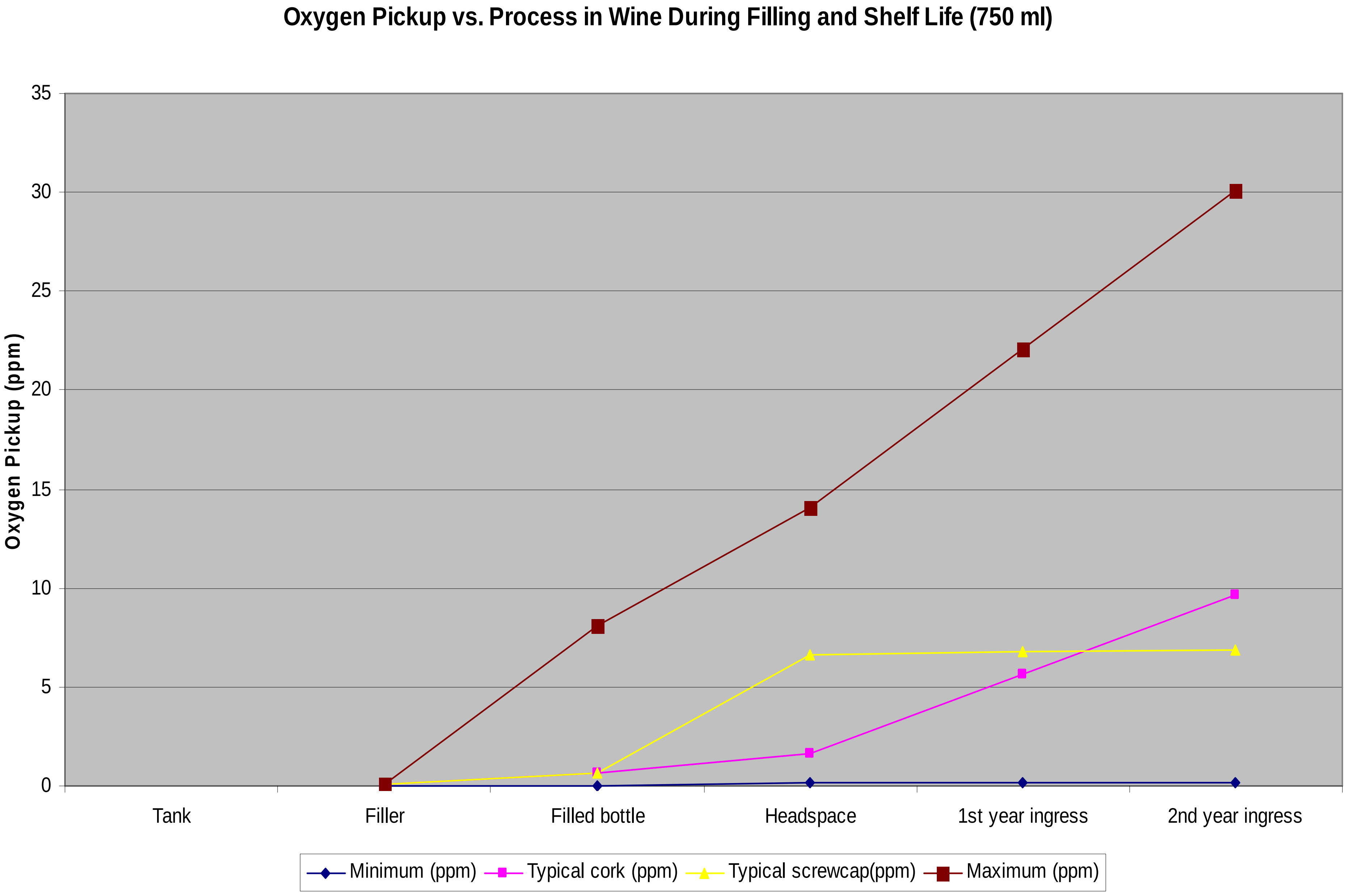
Additional Tests Open for Discussion

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



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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

Accelerated Aging

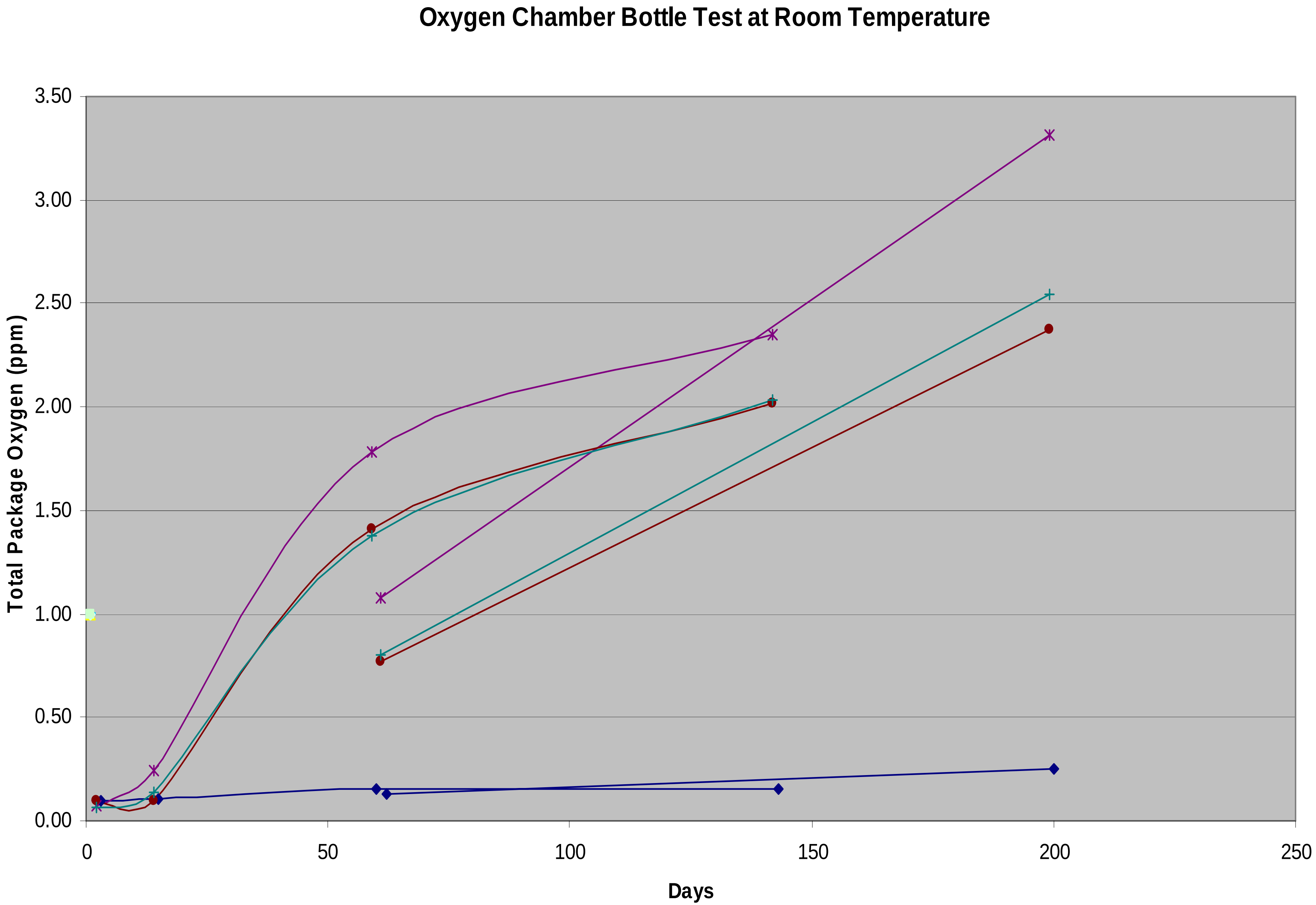
Heat versus Oxygen Chamber

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



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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

1. **Huige, N.J., MBAA TQ 2002, vol.39, no.4, pp218-230. Evaluating barrier enhancing and scavenger technologies for plastic beer bottles.**
2. **Remco W.G. van Willage, May 31, 2002, Effects of flavour absorption on foods and their packaging materials**