IMPORTANCE OF O$_2$ AND SO$_2$ MANAGEMENT IN WINE PRODUCTION: PRELIMINARY BOTTLING LINE AUDIT RESULTS

T.E. STEINER
DEPT. of Horticulture and Crop Sciences
The Ohio State University/OARDC
Wooster, Ohio 44691
THANK YOU!

- 2014 Michigan Annual Wine Conference
- Conference Organizing Committee
- Nancy Oxley
  - Initial Contact
- Linda Jones
- Barb Mutch
GO BUCKEYES!
OUTLINE

• Overview of oxygen benefits and problems

Oxygen Management Status: Overview of written Ohio commercial winery survey results

• Developed action plan

• Preliminary bottling line audit results

• Sulfur dioxide use and oxygen interaction

• Recommended actions
OXYGEN UPTAKE
BENEFITS OF OXYGEN

- Hyper-oxidation
  - Oxygen exposure to must/juice exercised in prevention of further post fermentation browning reactions

- Alcoholic Fermentation
  - Oxygen is essential during the initial stages of alcoholic fermentation for successful yeast propagation and fermentation.
    - Residual oxygen is completely removed by CO$_2$ during fermentation

- Micro-oxygenation
PROBLEMS OF OXYGEN PICKUP

- Generally, oxygen is detrimental for wine quality from the end of fermentation, cellaring, bottling and aging of wine
- Enzymatic and chemical oxidation
- Microbial issues
  - AAB, film yeast, “Brett”
PROBLEMS OF OXYGEN PICKUP

• Loss of varietal character in aroma and flavor over time (especially aromatic whites)

• Aging potential and wine stability in the bottle
2012 OHIO COMMERCIAL OXYGEN MANAGEMENT & STATUS SURVEY

- Consisted of 3 short surveys sent in July and August of 2012 covering critical cellar practices concerning excess oxygen absorption into wine
- Sent to all Ohio commercial wineries
- Data comprised from a less than satisfactory response of 34 wineries (21%) of Ohio wineries
  - Therefore may not represent a true representation of active cellar practices throughout the industry
    - However, trends look to be true for the industry
2012 OHIO COMMERCIAL OXYGEN MANAGEMENT & STATUS SURVEY: RESULTS

- Wineries measuring dissolved oxygen currently who participated in the survey
  - Yes – 08 percent
  - No – 92 percent
Survey results indicate several key areas of concern for excess oxygen pickup into wine:

- Wine movement: racking and pumping
  - Transfer lines and receiving tanks
- Headspace in tank and barrel
- Filtration
- Cold Stabilization
LIMITING OXYGEN EXPOSURE

- Oxygen can range in wine from 6 to 9 mg/L
- Oxygen will dissolve in wine more rapidly at lower temperatures however, oxidation occurs more rapidly at warmer temperatures
  - Cold stabilization becomes a concern
WINE TRANSFER AND O₂ PICKUP AT DIFFERENT TEMPERATURES

<table>
<thead>
<tr>
<th>Operation: Wine Transfer</th>
<th>Temperature</th>
<th>Average Mg/L O₂ Pickup</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bottom Tank Pumping</td>
<td>70° F</td>
<td>0.5</td>
</tr>
<tr>
<td>Bottom Tank Pumping</td>
<td>50° F</td>
<td>1.3</td>
</tr>
<tr>
<td>Splashing and Racking</td>
<td>Not Indicated</td>
<td>7.0</td>
</tr>
</tbody>
</table>

Observe a 3-fold pickup in oxygen due to racking at lower temperatures as viewed above

Source: J. Gallander, Oxygen Uptake at Several Winemaking Steps – A Review, OGEN Newsletter
• SO$_2$ use and monitoring
  • Critical from post-primary fermentation through bottling to be at .8 ppm molecular (whites) or .6 ppm (reds) based on pH

• Bottling:
  • Wine transfer, filtration, filling, headspace levels of the bottling tank, filler bowl and bottle
  • Temperature of wine at bottling
Closure

- Two major factors affecting oxygen pickup in wine relating to specific closures is 1) closure recovery time from compression and 2) the rate of oxygen permeation

- Level for oxygen permeation is lowest for screwcaps and “technical” corks, intermediate for natural cork stoppers, and highest for synthetic closures

- Furthermore, oxygen pickup varies between grades of each closure (Lopes et al., 2007)
*Percent Closures Used by Reporting Wineries

- Natural: 16
- Technical: 36
- Synthetic/Plastic: 20
- Screwcap: 16

*More than one closure being reported by a given winery in exceeding 100 percent
OSU/OARDC OXYGEN MANAGEMENT ACTION PLAN: FOUR FOCUS AREAS

• 1) Initial survey determining oxygen levels in white, red and rosé wines received shortly after bottling

• 2) Determine the effect of bottling operations on oxygen absorption of table wines – Particularly:
  • Holding tank, pump outlet, filter outlet, DO, HS and TPO

• 3) Evaluate oxygen absorption in wine involved with key cellar/vinification operations
OSU/OARDC OXYGEN MANAGEMENT
ACTION PLAN: FOURTH AREA OF INTEREST

• 4) Type of Closure
  • Aging potential – more longer term study
THE OSU/OARDC ENOLOGY OXYGEN MANAGEMENT ACTION PLAN GOALS

• Identify how important oxygen management in addition to proper sulfur dioxide concentrations are to the Ohio commercial wine industry

• Ultimately help increase wine quality and aging potential through these research and extension projects
THE OSU/OARDC ENOLOGY OXYGEN MANAGEMENT ACTION PLAN

- Recently purchased a “Nomasense Trace Unit” to quantitatively measure dissolved oxygen at all stages of the winemaking process.
- Works on Oxo-Luminescence Nomasense™ technology which specially enables analysis of oxygen in bottled wine nondestructively.
NOMASENSE LUMINESCENCE TECHNOLOGY
NOMASENSE LUMINESCENCE TECHNOLOGY
DISSOLVED O$_2$ STATUS OF BOTTLED OHIO COMMERCIAL WINES – PROTOCOL

- Purpose: To determine the oxygen status of bottled commercial wines in Ohio
- Asked to provide 3 types (white, red, rosé) as close to bottling as possible
DISSOLVED $O_2$ STATUS OF BOTTLED OHIO COMMERCIAL WINES – PROTOCOL

- Developed a procedure to evaluate DO of wine sent to us at OARDC after opening the bottle
  - Does not take into account initial DO, HS, TPO and initial $SO_2$
- 36 wines which were analyzed in replicate of the three wine types
  - Limited participation for this initial study
Most wines were found to be below 1 ppm with many in the ppb range.

- These wines were anywhere from 1 – 6 months in bottle.

Two wines were found to have higher DO values of 1.5 – 2.0 that were both bottled 1 week prior to analysis.
DISSOLVED O₂ STATUS OF BOTTLED OHIO COMMERCIAL WINES - RESULTS

• Oxygen and SO₂ interaction takes place quickly
• At least one wine type from each winery had lower than desired (below 10 ppm) SO₂ representing a concern for chemical oxidation, microbial instability and shortened aging potential
• This initial study/survey expresses the need to further examine the bottling line process of Ohio commercial wineries
BOTTLING LINE AUDITS

• Develop a protocol to evaluate oxygen pickup at the bottling line in Ohio commercial wineries

• Training occurred during August of 2013 with Wes Ward of Nomacorc/NomaSense™ utilizing the NomaSense trace unit

• Identify all key areas of the bottling line and determine the amount of oxygen absorption occurring at these points
THE BOTTLING LINE AUDIT PROCESS: PRE-PREPARATION

- Worked with 4 wineries (small, medium & large)
  - Small < 5,000 - Medium ~ 30,000 - Large ~ 120,000 gallons
- The bottling line audit involves pre-preparation
  - Obtain exact bottles used for bottling run
  - Determine correct headspace levels and internal bottle diameter
  - Based on previous winery runs
THE BOTTLING LINE AUDIT PROCESS: PRE-PREPARATION

- Place sensor dots in middle of bottle and headspace area in neck of bottle
- Place sensor dot in sight glass (2)
  - Used in line after pump and pad filter (If applicable) prior to bottling line
THE BOTTLING LINE AUDIT PROCESS: AT THE WINERY

- Determine temperature of wine at bottling tank and initial DO reading
- Once bottling line starts take reading at site glass after pump and filter
- The size of the bottling line determines amount of bottles to be analyzed
  - One bottle recommended for each fill spout/head
- Run reps through beginning, middle and end of bottling run
THE BOTTLING LINE AUDIT PROCESS: AT THE WINERY

- Pull bottles off line after corking to determine:
  - DO and HS
- Place data in spreadsheet to come up with TPO
• Holding Tank
  • Contained mostly lower levels (below 1.05 ppm)
  • Also an indicator of cellar O$_2$ levels
    • High levels demand additional cellar evaluation
  • One winery contained levels approximately 4.9 ppm
    • Due to temperature which presents a real concern
Vine Temperature:

Wine temperature is an essential factor at the time of bottling.

- The correct wine temperature (68-70°F) at bottling helps provide less dissolved oxygen, thermal expansion, closure headspace pressure and appropriate fill level’s.
• **Wine Temperature:**

  • Two of the four wineries were bottling at much lower than desired temperatures ranging from 42 to 46 °F.
    • These lower temperatures can result in a 3 fold increase in O\textsubscript{2} absorption into the wine prior to bottling
    • Represented the two wineries with the highest DO for the bottling run at 1.05 and 4.90 ppm
BOTTLING LINE AUDIT: RESULTS

• Filtration:
  • Initial testing indicates this may be a problem
  • One winery increased DO from 1.0 – 1.6 (60%)

• Filler Heads:
  • Oxygen measurements indicate this as a major source of DO variability
  • Small gravity fill operation observed approximately 3.0 ppm variability between filler heads
  • One large winery fairly consistent with lower DO range of 1.1 to 1.8 ppm
• Headspace Oxygen (HS):
  • Headspace represents the air space between the wine and closure
  • Oxygen measurements showed a lot of variability for all wines
    • Winery avg. 0.70, 1.4, 2.1 and 2.8 ppm
    • Takes 4, 6, 8 and 11 ppm free SO$_2$ for this alone
BOTTLING LINE AUDIT: RESULTS

• Headspace Oxygen (HS):
  • Measurements showed an extreme variation between all wines with a mean range for all analysis of 0.3 and 4.9 ppm
  • HS $O_2$ levels dependent on:
    • wine temperature, fill volume, inert gas sparging and providing a vacuum at corking
• Bottling Run DO: (Recommended below .5ppm)
  • DO tends to be high at the beginning ⅓ and last ⅓ while being lowest in during the middle ⅓ of the run
    • One winery - (4.6 beginning, 3.0 mid & 3.6 end)
  • DO for the 4 wineries were: 1.5, 2.8, 3.5 and 5.7 ppm
    • All higher than desired causing good potential that undesirable sensory attributes can form unless $\text{SO}_2$ is at appropriate levels
BOTTLING LINE AUDIT: RESULTS

- Total Package Oxygen (TPO):
  - Represents the sum of DO and HS oxygen
  - Some literature generously states best below 3.0 ppm TPO
  - More important than DO (tells the whole story)
  - This preliminary study indicates that 3 out of 4 wineries had higher than desired levels
BOTTLING LINE AUDIT: RESULTS

• Total Package Oxygen (TPO):
  • Mean values of the 4 wineries varied between 2.2, 4.2, 6.4 and 7.8 ppm.
  • Three wineries were above the recommended level.
  • The 3 wineries above the recommended TPO are in jeopardy of binding all free sulfur dioxide leaving none for protection from chemical oxidation, microbial growth and aging.
BOTTLING LINE AUDIT: RESULTS

- Total Package Oxygen (TPO):
  - It takes 31 ppm SO$_2$ to react with 7.8 ppm of O$_2$
  - Need at least 10 ppm free (minimum) SO$_2$ left in bottle to protect against chemical oxidation
  - Therefore the winery should have a minimum of 31 ppm plus enough free SO$_2$ based on wine pH at .8ppm molecular level
### Chemical Data - DO and S0₂

#### DO And Free S0₂ Levels At Various Stages Of Storage

<table>
<thead>
<tr>
<th>Chemical Data</th>
<th>Reps</th>
<th>Holding Tank</th>
<th>Bottling</th>
<th>Storage Days</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>ppm</td>
<td>ppm</td>
<td>3</td>
</tr>
<tr>
<td>Free S0₂</td>
<td>1</td>
<td>40</td>
<td>37 ppm</td>
<td>11 ppm</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>38</td>
<td>6 ppm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>35</td>
<td>8 ppm</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td>36 ppm</td>
<td></td>
<td>7 ppm</td>
</tr>
<tr>
<td>DO</td>
<td>1</td>
<td>6.0</td>
<td>2.62 ppm</td>
<td>76 ppb</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>5.9</td>
<td>2.60 ppm</td>
<td>60 ppb</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>5.6</td>
<td>2.56 ppm</td>
<td>51 ppb</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>5.7</td>
<td>2.63 ppm</td>
<td>35 ppb</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td>5.8</td>
<td>2.60 ppm</td>
<td>56 ppb</td>
</tr>
<tr>
<td>HS</td>
<td>1</td>
<td>2.07</td>
<td>1.99 ppm</td>
<td>1.48 ppm</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>2.06</td>
<td>1.98 ppm</td>
<td>1.50 ppm</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>2.03</td>
<td>1.94 ppm</td>
<td>1.46 ppm</td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>1.98</td>
<td>1.88 ppm</td>
<td>1.50 ppm</td>
</tr>
<tr>
<td>Average</td>
<td></td>
<td>2.035</td>
<td>1.95 ppm</td>
<td>149 ppm</td>
</tr>
</tbody>
</table>
IMPORTANCE OF SULFUR DIOXIDE
IMPORTANCE OF SULFUR DIOXIDE

• As an antioxidant
  • Protects musts and wines from browning
  • Binding of oxygen and acetaldehyde

• Antiseptic activity
  • Prevents microbiological spoilage in wines from microorganisms such as acetic acid bacteria, lactic acid bacteria, molds, and wild yeast
FREE SO$_2$ BINDING SITES

- Oxygen
- Acetaldehyde
- Pyruvate
- 2-Ketoglutarate
- Malvadin-3-glucoside
- Glucose
- Other wine constituents

Source: www.foodsci.purdue.edu/research/labs/enology
ESSENTIAL TIMES FOR SO₂ ADDITION

- Crushed grapes or must
  - Amount based on condition of grapes, temperature and pH
- Immediately after alcoholic fermentation
  - Amount based upon wine style and variety
- Wine storage
  - Treat wines at regular intervals with additional amounts to prevent oxidation and spoilage
- Pre-bottling
  - Adjust to .8 ppm molecular based on wine pH
ADDITON OF SO$_2$ PRIOR TO BOTTLING TO OBTAIN .8 PPM (MOLECULAR)

<table>
<thead>
<tr>
<th>pH</th>
<th>Free SO$_2$</th>
<th>pH</th>
<th>Free SO$_2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.9</td>
<td>11</td>
<td>3.5</td>
<td>40</td>
</tr>
<tr>
<td>3.0</td>
<td>13</td>
<td>3.6</td>
<td>50</td>
</tr>
<tr>
<td>3.1</td>
<td>16</td>
<td>3.7</td>
<td>63</td>
</tr>
<tr>
<td>3.2</td>
<td>21</td>
<td>3.8</td>
<td>79</td>
</tr>
<tr>
<td>3.3</td>
<td>26</td>
<td>3.9</td>
<td>99</td>
</tr>
<tr>
<td>3.4</td>
<td>32</td>
<td>4.0</td>
<td>125</td>
</tr>
</tbody>
</table>

Source: C. Smith, Enology Briefs, Feb/March, 1982, Univ. of Calif., Davis
Other role of SO$_2$: Molecular SO$_2$ as anti-microbial

Molecular SO$_2$ can be calculated from pH and free SO$_2$

0.6-0.8 ppm (mg/L) molecular SO$_2$ typical rec for dry wines

2 ppm is approximate sensory threshold of molecular SO$_2$

Source: Dr. Gavin Sacks, 2014 Ohio Grape and Wine Conference
ADJUSTING SO$_2$ AT BOTTLING

• Adjust SO$_2$ to .8 mg/L (whites) and .6 mg/L (reds) based on wine pH prior to bottling

• Also adjust SO$_2$ levels to account for oxygen pickup during bottling
  • 4 mg/L of SO$_2$ will react with 1 mg/L of oxygen
  • If we assume 3 mg/L oxygen pickup at bottling we would need an additional 12 mg/L SO$_2$ prior to bottling in addition to amount to equal correct molecular concentration
ADJUSTING SO₂ AT BOTTLING

• Length of bottle aging, variety and vintage
  • White wines meant for consumption at 6 months may not need additional SO₂ then described above
  • However, wines meant for longer aging periods may require an additional 10 to 15 mg/L SO₂ added

<table>
<thead>
<tr>
<th>Wineries and Wines</th>
<th>Ohio Wine Competition Data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of Wineries</td>
<td>27</td>
</tr>
<tr>
<td>Number of Wines</td>
<td>215</td>
</tr>
<tr>
<td>Number of White Wines</td>
<td>129</td>
</tr>
<tr>
<td>Number of Red Wines</td>
<td>86</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Medal Winners</th>
<th>Ohio Wine Competition Data</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of Medals for White Wines</td>
<td>70</td>
</tr>
<tr>
<td>Percent of Medals for White Wines</td>
<td>54%</td>
</tr>
<tr>
<td>Number of Medals for Red Wines</td>
<td>48</td>
</tr>
<tr>
<td>Percent of Medals for Red Wines</td>
<td>56%</td>
</tr>
</tbody>
</table>

Source: J. Gallander, T. Steiner, and P. Pierquet, derived from the (2010-2012) Ohio Wine Competition results and analysis

<table>
<thead>
<tr>
<th></th>
<th>Free SO₂ Concentration (ppm.)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>White Wines – Free SO₂</strong></td>
<td></td>
</tr>
<tr>
<td>Avg. Free SO₂</td>
<td>20</td>
</tr>
<tr>
<td>Avg. Free SO₂ at 0.8ppm molecular</td>
<td>25</td>
</tr>
<tr>
<td>Avg. Free SO₂ Medal Winners</td>
<td>25</td>
</tr>
<tr>
<td>Avg. Free SO₂ Non-Medal Winners</td>
<td>15</td>
</tr>
<tr>
<td><strong>Red Wines – Free SO₂</strong></td>
<td></td>
</tr>
<tr>
<td>Avg. Free SO₂</td>
<td>21</td>
</tr>
<tr>
<td>Avg. Free SO₂ at 0.6ppm molecular</td>
<td>28</td>
</tr>
<tr>
<td>Avg. Free SO₂ Medal Winners</td>
<td>25</td>
</tr>
<tr>
<td>Avg. Free SO₂ Non-Medal Winners</td>
<td>16</td>
</tr>
</tbody>
</table>

Source: J. Gallander, T. Steiner, and P. Pierquet, derived from the (2010-2012) Ohio Wine Competition results and analysis

• **Take Home Points:**

• Appears to be a correlation between sensory quality and medal winners relating to free sulfur dioxide concentration
  - 0.8 ppm molecular for white wines
  - 0.6 ppm and above for red wines

• A fact not listed in the information above indicates that over 50% of wineries had difficulty in achieving the correct SO$_2$ concentration

• **Results:**

• These results would also have a direct correlation with the amount of dissolved oxygen content which was not examined for this case study

• Sulfur dioxide and oxygen interaction could effect chemical oxidation, microbial issues, varietal aroma and flavors in addition to shelf life potential as described previously in this presentation
BOTTLING LINE AUDIT RESULTS: TAKE HOME POINTS

• Limit major sources of oxygen pickup at bottling
• If oxygen content is too high prior to bottling, sparging wine with an inert gas like nitrogen or carbon dioxide is possible
  • Nitrogen/Argon is preferred just prior to bottling
BOTTLING LINE AUDIT RESULTS: TAKE HOME POINTS

• Wine transfer: pumping & filtration
  • Use appropriate inert gas in purging transfer lines and bottling tank prior to filling
  • Fill from bottom of tank to bottom of receiving vessel or holding tank
  • Top any headspace (not recommended) with inert gas in holding tank
BOTTLING LINE AUDIT RESULTS: TAKE HOME POINTS

- Wine transfer: pumping & filtration
  - Operate filtration equipment according to manufacturers directions
  - Make sure all connections and pads are tight
  - Purge air from filter pads and transfer lines

- Filler:
  - Filler bowl should also be blanketed with an inert gas in reducing oxygen pickup
BOTTLING LINE AUDIT RESULTS: TAKE HOME POINTS

• Filler:
  - DO Varies on length of fill spout, type and force of jet
  - Therefore, it is advisable to have fill spouts as long as possible depending on the bottle
  - Providing a vacuum to the bottle and flushing with 2 to 3 volumes of nitrogen has been shown to lower oxygen absorption (Boulton, et al., 1999)
BOTTLING LINE AUDIT RESULTS:
TAKE HOME POINTS

• Bottle Headspace
  • Oxygen dissolving into wine is due to variation in bottle headspace related to wine temperature, solubility of gases, bottle size and shape
  • Injection of nitrogen or CO$_2$ can effectively reduce oxygen in headspace
  • A bottling line supplied with a vacuum filler is also effective in reducing the amount of oxygen in the headspace
BOTTLING LINE AUDIT RESULTS:
TAKE HOME POINTS

• Similarly, a controlled dose of liquid nitrogen into the wine is another good option of driving oxygen out of the bottle headspace for screw cap closures (Crochiere, 2007)

• Corking
  • If set up properly, setting up a vacuum at corking can also reduce the absorption of oxygen into the wine

• Closure
  • Choose correct closure depending on winery needs and available scientific data based on $O_2$ ingress
BOTTLING LINE AUDIT RESULTS:
TAKE HOME POINTS

• Be cognizant of wine temperature while transferring wine and bottling
• Proper use of sulfur dioxide is critical
ACKNOWLEDGEMENTS

The OSU/OARDC Enology Program:
Todd Steiner, Enology Outreach Specialist and Program Manager
Dr. James Gallander, OSU/OARDC Professor Emeritus
Patrick Pierquet, Enology Cellar and Laboratory Manager
Lisa Robbins, OSU/OARDC Enology Graduate Student

Specifically:
Dr. James Gallander: help with extension articles and co-coordinating the oxygen management project
Patrick Pierquet, assistant coordinator of Ohio Wine Competition, laboratory analysis, data preparation and assistance with oxygen management project
Lisa Robbins, assistance with oxygen management project
THANK YOU!

Todd Steiner
Enology Program Manager and Outreach Specialist
OARDC
Dept. Of Horticulture & Crop Science
Phone: (330) 263-3881
E-mail: steiner.4@osu.edu