Bottling Shock
A review of wine and equipment preparation techniques

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1. Wine Stability Treatments and Pre-Bottling Analyses
2. Bottling Line Sanitization
3. Bottle/Cork/Label QC and Bottle Storage
4. Bottling Line Demo & Discussion
Issues to be discussed:

- Cold/heat stability
- Re-fermentation rules
- Pre-bottling analyses
- Oxygen pick-up @ filtration and bottling
- Re-contamination points
- Chlorine vs. hot water
- Cork sampling/sterility
- Bottling temperature and headspace
- Bottle/case position
- Storage temperatures: cellar vs. TR
Bottling Shock

- Wine Stability Treatments and Pre-Bottling Analyses
- Bottling Line Sanitization
- Bottle/Cork/Label QC and Bottle Storage
- Bottling Line Demo & Discussion
Heat Stabilization vs. Grape Proteins!

Fining with Bentonite

The grape berry contains a large variety of nitrogen compounds mainly amino acids, peptides (short amino acid chains) and proteins (long amino acid chains). They serve various biological functions within the grape such as enzymes, cell wall components, etc. The nitrogen content of grapes varies greatly by variety, rootstock, vintage, climate, pruning and crop levels, fertilization practices, etc.

Amino acids are soluble and can be used by wine yeasts to grow and ferment the grape’s sugars into alcohol. Peptides and proteins cannot be metabolized as easily available nitrogen and their solubility decreases with the wine’s alcohol content. This may lead to precipitation of aggregated proteins in the form of a mobile amorphous haze. This effect is accelerated or triggered by exposure to elevated temperatures, e.g., when a customer buys a bottle of wine in the tasting room and forgets it in the trunk of his or her car over the weekend.

Protein hazes are purely aesthetic, visual problems in wine as they cannot be tasted. However, while it is a natural effect, most consumers prefer a wine free from unappetizing looking protein instabilities.

The Winemaker’s Options Prevent Protein Instabilities

Bentonite clay in different forms, (e.g., powder on sodium and calcium bentonite) can reversibly adsorb various sizes of proteins and has been the protein floculant of choice. It takes about six times the quantity of clay to take out the relevant amounts of protein. Protein content of wine ranges from around 10 to 300 mg/L, bentonite additions from 60 to 1,000 mg/L.

- Heat exposure such as a high temperature - short time (HTST) treatment can denature proteins in unfermented must and limit the need for additional fining of the wine. An HTST treatment, similar to a milk pasteurization at 80°C (176°F) for 5 seconds, does generally not affect the quality of the fermented wine. Such a treatment is also advisable for juices from grapes with heavy Botrytis.
Potassium Bitartrate Stability

Temperature (°F)

Alcohol (%)

g/L Bitartrate

Temperature (°F)
### Potassium Salt Additions

<table>
<thead>
<tr>
<th>Salt</th>
<th>K⁺</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bitartrate</td>
<td>41</td>
</tr>
<tr>
<td>Bicarbonate</td>
<td>257</td>
</tr>
<tr>
<td>Sorbate</td>
<td>70</td>
</tr>
<tr>
<td>Metabisulfite</td>
<td>30</td>
</tr>
</tbody>
</table>

**mg/L aka “ppm” aka “parts”**

=> Cold stability compromised!

=> For bottled-fermented *sparkling wine dosage* use SO₂ *gas* dissolved in water, not potassium metabisulfite.
Cold Stabilization

Wine Cold Stability Issues

"Cold stabilizing" means to add a wine of unstable potassium bitartrate ("cream of tartar"), the naturally occurring salt of the grape's tartaric acid, the major contributor to a wine's perceived tartness. Since potassium bitartrate is more soluble in water than in alcohol, the amount soluble in wine is smaller than the one in grape juice. However, due to the presence of colloidal materials in wine, such as macromolecules, pectins, and other polysaccharides, the unstable tartrate may not precipitate unless the wine is chilled or aged significantly in tank or barrel which might not be appropriate for a particular wine style.

In rigorous conditions, the deposit of the hom- and tetrahydrate potassium bitartrate as visible crystals on the bottom of the bottle or wine glass may cause the consumer to mistake these "wine diamonds" for glass splinters. While only an aesthetic issue that requires wine consumer education more than winemaker intervention, it has raised concerns about potential frivolous lawsuits. Thus, the winemaker stabilizes the wine to simulate what would happen if a wine consumer is pouring a bottle of (white) wine into a very cold refrigerator to chill it down to, for example, 32°F.

Since the solubility of potassium bitartrate is also much reduced at lower temperatures, the winemaker usually chooses to "cold" stabilize a wine at a temperature just above the freezing point of the individual wine. This is a giant waste of energy and a sad example of unnecessary over-processing of a natural product. The wine's freezing point is related most importantly to its alcohol content. The approximate freezing point of wine at 10% ethanol by volume is 25°F; at 12% it is 23°F, and 21°F at 14%. This means that the winemaker has a maximum stabilization window of 6 to 11°F which translates into a solubility difference for potassium bitartrate of between 200 and 300 mg/L, equal to 41 to 62 mg/L of potassium.

Commercial Winemaking Series

Coming soon @ www.indianaquality.org
Re-fermentation Rules

Re-Fermentation Prevention

How much residual sugar will cause visible yeast growth or carbonation in the bottle?

Residual Sugar
The recognition threshold for sweetness from residual sugar (R.S.) in a dry wine of average acidity is about 5 g/l (0.5%), while concentrations of 1 to 4 g/l can smooth a wine without making it taste off-dry. In very acidic wine styles, these numbers are significantly higher and a method traditional sparkling wine may taste flat even at 15 g/l R.S.

Microbial Stability
However, sensory perception of dryness is quite different from microbial stability of a particular wine, and cannot be used as a gauge for re-fermentation potential. After fermentation, a wine is commercially considered dry when its combined Saccharomyces fermentable sugars (glucose and fructose) are below 1 g/l (0.1%) and the wine laboratory tests for R.S. by measuring all reducing sugars, including pentoses, then 2 g/l (0.2%) is commonly used.

Those numbers practically assure that the wine is safe from noticeable re-fermentation, i.e., cell growth and carbonation by Saccharomyces yeast strains. Nonetheless, any R.S., even below 2 g/l, can still serve as a substrate for our wine microbes such as Brettanomyces yeast or lactic acid bacteria.

Gas Production
Glucose and fructose are converted into roughly half ethanol and half CO₂, i.e., 1,000 mg/l residual fructose can produce almost 500 mg/l CO₂ gas. As mentioned above, the solubility of CO₂ in wine is very high compared to other gases such as nitrogen or oxygen. A carbonation that is strong enough to push corks would occur beyond the saturation concentration of about 1,600 mg/l at room temperature (20°C) and strongly influenced by bottle headspace volume and closure type. A perceptible spritz may be felt at 500 mg/l, CO₂ which would require 1.5 g/l, re-fermentable fructose or glucose.

Haze Formation
Saccharomyces may grow if the recommended doses of sorbate and SO₂ are not met, or the sterile filtration prior to bottling was compromised (see bubble test). It has been reported that even 100 mg/l residual pentoses can lead to a visible Brettanomyces yeast haze if proper SO₂ management and filter integrity tests are.

Commercial Winemaking Series
Coming soon @ www.indianaquality.org
Re-fermentation Rules

Yeast

Residual Sugar
• Recognition threshold for sweetness is about 5 g/L (0.5%)
• 1 to 4 g/L can smoothen a wine without making it taste off-dry.

Microbial Stability
• Sensory perception of dryness is different from microbial stability
• Wine is dry when its combined fermentable sugars are below 1 g/L (0.1%).
• Considering all reducing sugars, incl. pentoses, 2 g/L (0.2%) is dry.
• Even below 2 g/L, R.S. can serve as a substrate for our spoilage microbes.

Gas Production
• 1,000 mg/L R.S. can produce almost 500 mg/L CO₂ gas.
• To push corks about 1,400 mg/L at room/bottling temperature are required.
• Strongly influenced by headspace volume and closure type.
• A perceivable spritz may be tasted at 800 mg/L CO₂ (from 1.6 g/L R.S.).

Haze Formation
• S.c. may grow if the recommended doses of sorbate and SO₂ are not met.
• Or if sterile filtration prior to bottling was compromised (use bubble test!).
• Even 100 mg/L residual pentoses can lead to a visible Brett haze.
• Visible haze due to S.c. must be expected above 1,000 mg/L R.S.
Re-fermentation Rules

**Bacteria**

**Malic Acid**
- Grapes at harvest contain between 0.6 and 6 g/L of malic acid
- Malolactic bacteria turn malic acid into lactic acid and carbonic acid (CO$_2$)
- Tartaric acid cannot be metabolized by wine bacteria.
- Absence of a malic acid spot on a paper chromatogram indicates less than 30 mg/L.
- A barely visible spot about 200 mg/L.
- In wine which completed MLF, residual malic acid is less than 300 mg/L (0.3 g/L).

**Gas Production**
- Malic acid is converted into two thirds lactic acid and one third CO$_2$
- 300 mg/L residual acid could produce 100 mg/L gas.
- A perceivable spritz may be tasted at 800 mg/L CO$_2$ (from 2.4 g/L malic).

**Haze Formation**
- Visible haze in a white wine due to the growth of Oe. oeni above 300 mg/L.
Sorbic Acid

**Basics**

- Yeast growth inhibitor: 200 mg/L
- Legal limit: 300 mg/L
- Sensory threshold: 135 mg/L
- Some yeasts are resistant!
- **NO** effect against bacteria
- Added as *potassium* salt (*Sorbate*)

=> Watch cold stability!
I mixed up potassium sorbate and citric acid together to make a sorbate addition and an acid adjustment prior to bottling.

An amorphous white precipitate has formed and is floating on top of the mixture.

What is it and what should I do about it?
Geranium Off-Odor

Cause

- Sorbic acid + Malolactic bacteria

Prevention

- Avoid sorbate as preservative
- Use sorbate only with proper SO₂
- Add no earlier than day before bottling
- Always bubble test/sterile filter
- NO removal option from wine
Oxygen Pick-up @ Bottling

Just vacuum at filler + Headspace sparging + Vacuum at corker + Bottle sparging w/N₂
Oxygen and $\text{SO}_2$ at Bottling
Oxygen Basics

**Air-O$_2$ solubility (68 °F):** 8 mg/L

**Air-O$_2$ uptake at bottling:** 0.17 - 8 mg/L

**Uptake via topping (per year):** 20 mg/L

**Uptake per racking:** 20 mg/L

**Uptake via cork (per year):** 0.1 mg/L

**Young red wine:**

**Total O$_2$ uptake capacity:** 4,000+ mg/L

**Optimum O$_2$ uptake:** 60 - 130 mg/L
Wine Oxygen Pickup at Bottling

<table>
<thead>
<tr>
<th>[mg O₂/L]</th>
<th>Added at the filler</th>
<th>Added via headspace</th>
<th>Added Total O₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gravity flow filler only</td>
<td></td>
<td></td>
<td>8.00</td>
</tr>
<tr>
<td></td>
<td>6.60</td>
<td>1.40</td>
<td>8.00</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Removed at the filler</th>
<th>Removed from headspace</th>
<th>Removed Total O₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Vacuum pulled at filler</td>
<td></td>
<td>6.00</td>
</tr>
<tr>
<td>2. Bottle sparged with N₂</td>
<td>0.51</td>
<td>0.02</td>
</tr>
<tr>
<td>3. Vacuum pulled at corker</td>
<td>-</td>
<td>0.15</td>
</tr>
<tr>
<td>4. Headspace sparged with N₂</td>
<td>-</td>
<td>1.15</td>
</tr>
</tbody>
</table>

#1- #4 implemented 6.51 1.32 7.83 = 98%
SO₂ Loss at Bottling

**O₂** uptake at bottling: 0.17 - 8 mg/L

\[ 8 \text{ mg O}_2 + 16 \text{ mg SO}_2 \Rightarrow \text{Sulfate} \]

= Free SO₂ Loss

+ additional losses during filtration etc

\[ \Rightarrow \text{Add 10 - 25 mg/L EXTRA SO}_2 \text{ before bottling} \]
Free $\text{SO}_2$ required vs. pH

- $\text{SO}_2$ molecular
- pH range: 2.9 to 4.1
- $\text{SO}_2$ concentration (mg/L) range: 0 to 150
pH and Required SO$_2$

Free SO$_2$ = 0.85 • (1 + 10$^{\text{pH} - 1.83}$) required

Or see charts @ indianaquality.org

+ Additional losses during filtration/bottling

⇒ Add 10–25 mg/L EXTRA SO$_2$ pre-bottling
Bottling Temperature

At room temperature ($\approx 68^\circ F$)!

Why?

- Lower oxygen solubility
- Better filtrability
- Less thermal expansion
- Less bottle headspace pressure
• Wine Stability Treatments and Pre-Bottling Analyses
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Bottling Shock

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Pad Rinsing/ Sanitation

w/ out counter pressure  with counter pressure
## Pore Seize vs. Flow Rate

### Seitz Filter Sheet Grade vs. Nominal Micron Retention Rating

<table>
<thead>
<tr>
<th>Seitz Filter Sheet Grade</th>
<th>Nominal Micron Retention Rating</th>
<th>Suggested Uses for Wine Filtration</th>
</tr>
</thead>
<tbody>
<tr>
<td>K900 or K1500</td>
<td>9-10</td>
<td>Rough-polishing filtration for retention of yeast and larger micron-sized particles</td>
</tr>
<tr>
<td>K800</td>
<td>7-8</td>
<td>Rough-polishing filtration for use as above.</td>
</tr>
<tr>
<td>K700</td>
<td>5-7</td>
<td>Polishing filtration for use as above.</td>
</tr>
<tr>
<td>K300</td>
<td>3-4</td>
<td>Polishing - clear wine filtration.</td>
</tr>
<tr>
<td>K250</td>
<td>2-3</td>
<td>Polishing clarity filtration.</td>
</tr>
<tr>
<td>K200</td>
<td>2.0</td>
<td>Higher polishing clarity filtration of clear wines</td>
</tr>
<tr>
<td>K150</td>
<td>1.5</td>
<td>Higher polishing clarity filtration of clear wines</td>
</tr>
<tr>
<td>K100</td>
<td>1.0</td>
<td>Higher polishing clarity filtration of clear wines</td>
</tr>
<tr>
<td>KS80</td>
<td>0.8</td>
<td>Beginning of grades for ‘sterile’ filtration. This grade for yeast-sterile filtration.</td>
</tr>
<tr>
<td>KS50</td>
<td>0.5</td>
<td>Grade for yeast sterile bottling filtration. Pre-membrane (0.65μ) filtration.</td>
</tr>
<tr>
<td>EK</td>
<td>0.45</td>
<td>A slightly tighter grade for ‘sterile’ bottling filtration. Pre-membrane (0.45-0.65μ) filtration.</td>
</tr>
<tr>
<td>EK1</td>
<td>0.35</td>
<td>A tighter grade for ‘sterile’ bottling filtrations. Pre-membrane (0.45-0.65μ) for difficult-to-filter wines.</td>
</tr>
<tr>
<td>EKS</td>
<td>0.25</td>
<td>Tightest grade for ‘sterile’ bottling filtrations. Pre-membrane (0.45-0.65μ) for difficult-to-filter wines.</td>
</tr>
</tbody>
</table>

### Permeability [L m⁻² min⁻¹]

- EKS: 29
- EK1: 41
- EK: 68
- KS50: 93
- KS80: 113
- K100: 146
- K150: 185
- K200: 213
- K250: 785
- K300: 925
- K700: 1275
- K800: 1700

The graph illustrates the permeability of different Seitz Filter Sheet Grades against flow rate.
Membrane Filtration

- Mount
- Integrity Test → Bubble Point
- Sterilize
- Bottle
- Re-Test

- Flood membrane with water
- Pressurize to 80% with $N_2$
- Increase pressure slowly
- Record when bubbling starts
- Compare to specifications

Results:

- Membrane integrity
- Correct mounting
- Correct filter size
- NOT tested: downstream sterility
Downstream Sterility?
Sources of Re-Contamination

- Hoses, Lines, Gauges
- Valves
- Gaskets
- Inert Gas
- Vacuum Line
- Corks
- Filtered Air
- Hopper
- Vacuum Line
- Corker Jaws
- Rinse Water
- Filler Bowl
- Bottles
- Conveyors
- Condensate
- Filler Heads

Millipore Corp. in PW&V 1989
Bottling Line Sanitation

Water Issues

• No chlorine residues in city water allowed

• Soften/iron- remove well water

• Sterile filter rinsing water

• Cell kill at 180°F
  = 10x more effective than at 170°F

• Ozonated water cannot reach dead spaces as steam can
Bottling Line Sanitation
Chlorine

Commercial Winemaking Production Series
Chlorine Use in the Winery
Why not to use any chlorinated products anywhere in the winery

By Christian Battle
Coatings Professor for the Indiana Wine Grape Council
Department of Food Science
Purdue University
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Hypochlorite
Cleaning products that contain hypochlorite (NaOCl) should not be used anywhere near the winery, especially the production and hospitality areas, specifically the tasting room.

Formation of 2,4,6-trichloroanisole
Presence of chloroform is one of the two major contributions to the production of 2,4,6-trichloroanisole (TCA), the compound that causes a moldy, musty cork taint. TCA’s sensory threshold is one of the lowest in nature at around 1 to 5 n-hexanoic per liter. The second requirement for TCA formation is the presence of molds. They are commonly found in wine cellars and barrels due to frequent mixing of tanks and floors and the desirably high relative humidity (80 percent or more) in barrel rooms, which minimizes evaporative losses of wine. Chlorinated and mold-methylated phenolics from materials such as wood or cork bark are known as chloroanisoles, and their equally potent brominated analogues are bromoanisoles.

Airborne TCA
Dirty floor drains in particular can become a potential source for TCA formation in the winery as they combine chlorine residue from wines with the rich microbial activity needed for its formation.

If TCA is subsequently present in the cellar air, it can be introduced into the wine when barrels or tanks are emptied and refilled. The tiny amount of TCA that it takes to spoil a wine lot corresponds to equally small residues of chlorine from sanitization operations. TCA is also easily absorbed by cork shavings in the bottling line hopper and by open bags of potassium or filter aids, so proper and segregated storage of all processing aids is crucial.

Chlorinated cleaning products
Unfortunately, it is not always easy to immediately recognize that a product contains hypochlorite. Look closely at the ingredient list on dishwashing detergents (for testing glasses), kitchen and bathroom cleaners, disinfecting wipes, and anti-allergen and sanitizing sprays. You should also watch out for fabrics and textiles that were treated with proprietary coating techniques that bind hypochlorite and prolong the presence of chlorine bleach. Because it is easily activated on contact with organic matter, chlorine often bleach the dirt without removing it, while leaving a “sweat” (only by associative) smell behind.

Water quality
In addition to eliminating hypochlorite-based cleaning products, winemakers
Filter Fouling

“undesirable accumulation of materials on the surface of the filter”

• Yeast/Bacteria Cells (alive or dead)
• Biofilm Formation (Polysaccharides)
• Colloidal Materials (Pectine, Protein etc.)
• Fining Agent Residue (Bentonite etc.)
Filter Fouling
During Bottling Run

- Don’t by-pass the sterile filtration unit
- Make sure you have an extra cartridge
- Make sure you re-sanitize the unit
- Don’t back-wash filters
- Save money for a cross-flow unit
Membrane Storage
Filterability Testing

- How many pads/cartridges do we need?
- Will it go through the membrane?
- How much wine can we filter?
- Can we keep up with the bottling line?
- When can we go home?

Suggested reading: www.winerysolutions.com/filter.html
• Wine Stability Treatments and Pre-Bottling Analyses
• Bottling Line Sanitization
• Bottle/Cork/Label QC and Bottle Storage
• Bottling Line Demo & Discussion
Bottle Glass QC
By all means, get a pressure sensitive labeler!
Cork QC
Conclusion: TCA is not related to visual quality. Please note, however, that oxygen permeability is in a large part related to visual quality.
Cork Taint

T'ain't the bottle

T'ain't the wine
Cork QC

In short:

For each 5,000-cork bale and when using the simplest fixed-sample-size plan applicable to wine closures (ANSI/ASQC Z 1.4-1993 sampling plan for normal inspection, general inspection level I; supplier with good track record), the winemaker has to sample, soak and sniff 80 corks.

If 4 or more of those 80 corks were tainted, the lot should be rejected and send back to the supplier.
Proper (TCA-free) Storage

Filter Pads/DE

Bentonite
Bottle Storage

SIDeways

![Diagram of a wine bottle lying on its side with a red cross over it, indicating it is not the correct way to store a bottle.](image)
Flow Through Porous Media

Important Factors:

- *Gas permeability* of the cork
- *Layers* of alternative media
- *Variability in porosity* \(^*\) from cork to cork
  (*fraction of volume which is occupied by gas*)
- *Distribution* of pore sizes
- *Gas viscosity* is 1/100\(^{th}\) that of water
Giving Headspace

• $\text{SO}_2$ Permeation = $f(\text{Headspace})$!

• Headspace $\uparrow$ exponential with Temperature
Effect of Headspace Volume on Bottle Pressure
(and Permeation)

Wine Temperature (°C)

Bottle Pressure (Atm)

Time of Day (Hours)

Effect of Headspace Volume on Bottle Pressure
5 C Rise and Fall, 750 mL

Wine Temperature

3 mLs
4 mLs
5 mLs
6 mLs
Bottle Storage Temperatures

Heat Exposure

Storage Time

<table>
<thead>
<tr>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Never</td>
</tr>
<tr>
<td>Spikes of 30 min or less</td>
</tr>
<tr>
<td>1-4 weeks</td>
</tr>
<tr>
<td>Long-term storage</td>
</tr>
</tbody>
</table>

Cold Exposure

Storage Time

<table>
<thead>
<tr>
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Bottling Line Demo & Discussion

- Dave Schrodt - Brown County Winery
- Jason Heiligenberg - Huber's OWV
- Dennis Dunham - Oliver Winery
- John Doty - French Lick Winery
- Mark Easley - Easley Winery
The Wine Grape Action Team is a cooperation between Purdue University’s Department of Food Science and the Indiana Wine Grape Council to serve the State’s vintners and growers and help propel the Indiana wine/grape industry into world-class competitiveness. The 4-member team is available at any time to troubleshoot emerging issues in your vineyard and winery:

- Bruce Bordelon
- Christian Butzke
- Jill Blume
- Jeanette Merritt

Wine Grape Action Team Extension Publications:

- Wine Aroma Descriptor Manual (Christian Butzke)
- Commodity Storage Manual: Wines (Christian Butzke)
- Midwest Grape Production Guide (Bruce Bordelon)
- Growing Grapes in Indiana (Bruce Bordelon)
- Grape Varieties for Indiana (Bruce Bordelon)
- Starting a Commercial Winegrape Vineyard (Bruce Bordelon)
- Ethyl Carbamate Preventative Action Manual (Christian Butzke)
- NOPA Method for Yeast Assimilable Nitrogen (Christian Butzke)
- Cork Sensory Quality Control Manual (Christian Butzke)

Events organized or contributed to by members of the Wine Grape Action Team:

- Wine Grape Action Team & Indiana Wine Grape Council launch Indiana’s first signature wine: TRAMINETTE
  
  Try On Traminette campaign

Press coverage:

- Wineries promote Traminette as signature Indiana wine
- Indiana wine industry pops the cork on new signature wine
- State selects signature wine
- Traminette selected as state’s signature wine
- Indiana gets its own wine
- State will market signature wine
- Indiana wine industry selects turkey friendly Gewürztraminer hybrid as state’s 1st signature wine