### Critical Issues in Wine Stabilization, Bottling, and Aging to Promote Shelf-Life Stability

#### T.E. STEINER

DEPT. of Horticulture and Crop Sciences The Ohio State University/OARDC Wooster, Ohio 44691





THE OHIO STATE UNIVERSITY

COLLEGE OF FOOD, AGRICULTURAL AND ENVIRONMENTAL SCIENCES

# Appreciation

The 2025 FWGGA Conference

The FWGGA Bord for the invitation to present

- Sue Elliot for her conference organizational efforts
- The FWGGA conference organizing committee for their effort in conference development and organization

#### **Conference Attendees**

• Taking valuable time out of your busy schedules

### Outline

Critical issues to promote wine stabilization & shelf life potential

• Grape condition, Winery hygiene, Temperature control

#### Wine Stabilization – Definition and Sources

- Wine Fining briefly mention
- Blending
- Oxygen uptake & elimination
- Bottling line sterilization
- Sterile bottling
- Chemical preservatives
- Bottle storage conditions

### **Fruit Quality and Wine Stability**

Process clean/sound fruit!!!

- Whether purchased or grown
- "You can't make a silk purse out of a sow's ear!"

Of special concern to grape and wine quality are:

- Oxidative yeast Candida, Hanseniaspora, Pichia
  - production of ethyl acetate and ethanol
- Acetic acid bacteria Gluconobacter, Acetobacter
  - production of gluconic acid and acetic acid

Produces enzymes called tyrosinase and laccase which causes browning and oxidation

Increases levels of glucan which effect filterability

Damage fruit leads to secondary spoilage by microorganisms through subsequent pass-through processing operations

An increase in rot elevates must pH (> 3.5)

- Greater chance for both chemical and microbial instabilities
- Also, tartrate, protein, color and SO2 effectiveness

Clean fruit has relatively low yeast and bacteria numbers

Leads to a better-quality wine with increased shelf-life stability





### Winery Hygiene

• Good winery hygiene practices involve planning, identifying critical control points, cleaning, sanitizing, sterilization and proper monitoring. Goal of keeping the winemaking facility clean and free of non-desirable microbes that cause concern for further "pass-through" winery operations.

• Therefore, proactive control in minimizing the risk of spoilage organisms is critical in promoting wine quality and shelf-life stability.



### Effect of Grape Cluster Rot on Wine Quality

% Rot	pН	% TA	Sensory Score
0	3.74	.56	13.9
5	3.79	.49	11.7
10	3.79	.53	11.0
20	3.72	.56	8.2
40	3.74	.61	6.1

Loinger (1977)

#### Temperature Control

One of the most important issues in the production of consistent quality/sound wines from harvest to bottling and storage



# Temperature Control Important For the Following Processing Practices:

Harvested grapes: 38 - 40°F

Must Processing: White skin contact: 33 – 38 °F, Red cold soak: <50°F

Primary Fermentations: White: 55 – 63°F, Red: 70 - 95°F

• Varietal dependent

Malolactic Fermentation: Near 70°F for optimum conditions

Cold Stabilization: 28°F for a period of 3-4 weeks ideal

• Time and temperature dependent variable upon procedure

Cellar aging: Effective range from 50 - 63°F

Bottling - Bottle near 68°F ideal

• Prevent undesirable internal pressure changes and increased oxygen absorption

Bottle storage: 55 - 68°F constantly all year round

• Pending wine type and style (Whites' cooler while reds can take a little warmer)





#### Chemical

- Cold Stabilization
- Heat Stability
- Heavy Metals

#### Colloidal

- Pectin
- Glucan
- Phenolic
- Other

### Microbial

- Yeast
  - Fermentative
  - Film
  - Brettanomyces
- Acetic Acid Bacteria
- Lactic Acid Bacteria

#### Chemical

- Cold Stabilization
- Heat Stability
- Heavy Metals

### Colloidal

- Pectin
- Glucan
- Phenolic
- Other

One of the most common problems I have encountered during my career in dealing with improperly stabilized wines resulting in lower quality wine

### Microbial

- Yeast
  - Fermentative
  - Film
  - Brettanomyces
- Acetic Acid Bacteria
- Lactic Acid Bacteria

# **Chemical Instability**

**Cold Stabilization** 

- Unstable shows presence of tartrates in bottle
- Perform cold stabilization via
  - Cold Temperature– Jacketed tanks or Mother Nature
  - Contact seeding involving chilling
  - Electrodialysis increased water use and expense
  - Additives such as CMC, Mannoproteins, Gum Arabic
    - Wine chemistry must be correct for these potential procedures
- Perform lab analysis test for cold stabilization efficiency

**Protein Stability** 

- Unstable shows protein haze or flocculation in wine
- Stabilize with Bentonite at recommended dosage rates
  - Based on lab trials (Bentotest) to determine optimum levels
  - Larger additions best done to must/juice with fine tuning to wine if needed
- Perform heat stability trials to check efficiency

# **Microbial Stability**

Two ways to achieving biological Stability:

- Physical (Filtration)
- Chemical or Fining
  - Sulfur dioxide
  - Lysozyme
    - Enzyme derived from egg whites that can be used in organic winemaking
    - Used against gram + bacteria including Lactic acid bacteria (no effect on yeast)
  - Chitosan
    - Non-allergenic and non animal based antimicrobial agent to control Brettanomyces spp., lactic acid bacteria and acetic acid bacteria pending formulation
    - Good in high pH wine environment
  - Sorbate
  - DMDC (Velcorin)

# **Post Fermentation Microbiology Concerns**

Oxidative yeast species:

- Brettanomyces, Zygosacccharomyces and various strains of Saccharomyces
  - Capable of rapid growth under proper conditions
  - (Pomace, spilled grape juice or wine etc.)
- Film Yeast: Hansenula, Candida and Pichia

Can produce off by-products in aroma and taste (Band-Aid®, barnyard, VA, acetaldehyde etc.)

# **Post Fermentation Microbiology Concerns**

Aerobic bacteria

- Acetic acid bacteria
  - Produce both vinegar and ethyl acetate aromas

Fermentative species

Lactic acid bacteria such as Lactobacillus kunkeii, Oenococcus and Pediococcus

Capable of producing off by-products in both aroma and taste (VA, sauerkraut, geranium etc.)

# **Colloidal Stability**

Generally, colloidal instability in wine refers to flocculation of:

 Pectin's/polysaccharides, glucan, tannins, phenolic complexes, iron and copper

Cold and heat stability can help reduce colloidal instability

Utilize enzymes with pectinase, cellulase, hemi-cellulase, protease, beta-glycosidase activity

• Glucans and are important colloids as they can affect filterability

Gum Arabic can also help reduce color loss in reds while helping to reduce astringency and improve mouthfeel

Wine stabilization must occur prior to bottling

Wine stabilization must occur prior to bottling.

After bottling, wine stabilization can occur only with a great deal of effort and cost

Wine stabilization must occur prior to bottling.

After bottling, wine stabilization can occur only with a great deal of effort and cost.

Unstable bottled wine can create an image of poor quality on the winery in the marketplace and the overall Florida commercial wine industry



#### Can help aid in wine stabilization

# Fining: Definition & Goals

Broad definition:

• To become pure or clean

In wine:

- To add an absorptive or reactive substance to reduce or remove the concentration of one or more undesirable products
- To aid in producing a product that is near perfect in terms of taste, color, bouquet and clarity
- The fining method should not take away from these attributes (taste, color, bouquet and clarity) but bring them together in expressing varietal character as much as possible
- Can be as simple as letting nature and time take its course or actively adding and assortment of fining agents at our exposure
  - This depends entirely on the individual must/wine and its components in addition to suspected time of bottling in meeting consumer demand

# **Reasons for Fining**

Aroma:

• H2S, oxidation, off varietal aroma, slightly flawed

Color:

• Intensity, browning

Flavor:

• Bitterness, astringency, off-balance

Palate:

Harsh (round and soften, improve phenolic profile)

Haze:

• Protein, heavy metals

Cold stabilize

# **Factors Improving Fining Action**

Low carbon dioxide (CO<sub>2</sub> impedes fining and settling)

Warm temperatures

• Simply warming a wine up in some cases can help settle out and clarify a wine (except protein instability)

Lower pH wines require less clarification time

- A low metal content (high can affect fining efficiency)
- Young wines are more forgiving in protein fining
- Dry wines
- **Clarified wines**

# **Essential Steps in Fining Wine**

Sensory evaluation

**Chemical Analysis** 

• pH, TA, VA, SO<sub>2</sub>, alcohol, heat stability, cold stability, % R.S. etc....

Laboratory trials

Cellar application

# **Cellar Applications**

Commercial companies such as Scott Laboratories have nice laboratory size fining trial packets available with good directions for cellar applications

Refer to these companies for a list of fining agents and their optimum treatment use

Perform laboratory trials and cellar applications the same: Fining agent, preparation methods, temperature, mixing and timing are all critical

• Procedural differences may result in over or under fining

# **Cellar Applications**

Effectiveness of fining can be reduced by 50% due to improper preparation methods

Entire volume of wine must come into contact with fining agent

- Therefore, efficient mixing is critical to the process
- May mix on daily basis pending fining agent process

Limit contact time to minimum amount required to perform purpose efficiently

Proteinaceous fining agents work better at colder temperatures (except bentonite)





Source: KJ.com

# Blending

Blending can be a powerful tool to pull together strengths and weaknesses from each variety, vintage or lot to increasing wine quality of the final blend

- Make good wines great
  - Enhance wines not complete in aroma, color, body mouthfeel, taste & finish
- Correct for excess negative attributes in wine aroma or taste
  - Herbaceous characters, diacetyl, harsh tannin and bitter phenols
- Adjust pH, acid/sugar ratio more stable
- Improve slightly flawed wines to consumer acceptable
- To create a wine which will fit your market, image or define your style

# **Fining and Blending Reminder**

Blending recommended earlier in cellar aging

Heat and cold stabilization trials should be performed after fining or blending is accomplished

Adjust sulfur dioxide levels based on wine pH

Limit headspace in tank or barrel to prevent unwanted oxygen from dissolving into wine after fining or blend is complete

Continue sensory evaluation of fined or blended wine in tank or barrel throughout aging until bottling

# Oxygen Management

One of the most important aspects to practice in the winery to produce quality wines free of faults









### **Oxygen Management – Extremely Important**

Generally, oxygen is detrimental for wine quality from the end of fermentation, cellaring, bottling and aging of wine

Enzymatic and chemical oxidation

- Enzymatic: Polyphenoloxidase (PPO) and Laccase (Botrytis fruit)
- Chemical: Reaction of wine components (polyphenols) with oxygen forming browning of color and acetaldehyde (Nutty, sherry-like aroma's)

Microbial issues

- AAB, film yeast, "Brett"
- Can be problematic throughout fermentation and especially during wine aging pending microbial issue'(s) at hand

Avoid potential sources for oxygen pickup cont.

- Cellar: Racking, excess headspace, pumping filtration and bottling
  - Use inert gasses to purge headspace, transfer hoses, receiving vessels etc.

# Limiting Oxygen Exposure

If oxygen content is too high prior to bottling, sparging wine with an inert gas like nitrogen or carbon dioxide is possible

- DO at Bottling/holding tank should be below 0.5 ppm
- Nitrogen/Argon is preferred just prior to bottling

Recommended levels of dissolved oxygen in wine after bottling

- Red wine  $\leq 1.25$  mg/L
- White, Blush & Rose wine  $\leq 0.60$

Monitor and Maintain sulfur dioxide base on wine pH to at least .8 ppm free  $SO_2$ 

• 4 ppm SO<sub>2</sub> needed to react with one ppm O<sub>2</sub>

### Importance of Sulfur Dioxide

• The proper use of sulfur dioxide from harvest to bottling is still one of the most important and critical aspects in producing quality wines free of faults for all commercial wineries regardless of size or experience



### **Importance of Sulfur Dioxide**

#### As an antioxidant

- Protects musts and wines from browning
- Binding of acetaldehyde
- Free SO<sub>2</sub> levels must not fall below 10 ppm

#### Antiseptic activity

- Prevents microbiological spoilage in wines from microorganisms such as acetic acid bacteria, lactic acid bacteria, molds, and wild yeast
- Usually, molecular levels of 0.6 to 0.8 ppm sufficient

At certain levels SO<sub>2</sub> may promote a rapid and complete clarification of must and wine





# **Essential times for SO<sub>2</sub> 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 (white's) .6 (red's) based on wine pH
  - Account for oxygen pickup at bottling in addition to bottle aging desired
  - 1 mg/L oxygen reacts with 4 mg/L SO<sub>2</sub>
  - If we assume 3 mg/L oxygen pickup at bottling, we will need an additional 12 mg/L SO<sub>2</sub> prior to bottling
  - Additional SO<sub>2</sub> may be needed if bottle aging is desired pending variety

# **Sulfur Dioxide and Wine Faults**

Much of the time our SO<sub>2</sub> levels are too low being directly responsible for many of our faults

• Also correlates considerably to increased amounts of oxygen

In many cases rectifying a wine involves the addition of sulfur dioxide before or after a reactive measure pending the actual fault

SO<sub>2</sub> levels can also be too high relative to wine pH which is also considered a fault

- Will overwhelm varietal character/expression with a pungent aroma and envelope the palate masking the delicate fruit attributes
- Therefore, lab analysis and proper application is critical
# Addition of SO<sub>2</sub> prior to bottling to obtain 0.8 ppm (molecular)

pН	Free SO <sub>2</sub>	pН	Free SO <sub>2</sub>
2.9	11	3.5	40
3.0	13	3.6	50
3.1	16	3.7	63
3.2	21	3.8	79
3.3	26	3.9	99
3.4	32	4.0	125

#### Other role of $SO_2$ : Molecular $SO_2$ as anti-microbial



Source: Dr. Gavin Sacks, 2014 Ohio Grape and Wine Conference





Since bottling is the last winemaking process, it is essential that all bottling operations including filtration be properly sanitized

The most efficient sterilization process is the use of steam or hot water at  $\geq$  180° F.

Although effective, chemical sterilization from chlorine-based compounds, iodophor's, sulfur dioxide/citric acid and ozone have limitations with Clean in place (CIP) operations

• Due to the inability of water-based compounds penetrating the submicron cracks of the bottling line

Once the temperature reaches 180 °F at the distal point (i.e.., the filler spouts) sterilization should occur for 20 minutes

• The flow can be reduced in saving energy cost

Important areas of microbiological growth and concern in the bottling room are the filler (48%), corker (28%), bottle sterilizer (10%), bottle mouth (8%) and sterilizing filter (6%) (Neradt, 1982)

Other sources of infection: filter drip trays, wine spills, and floor drains

All these areas should be focused on cleaning with the appropriate compounds and flushed with copious amounts of water

Special attention should be given to the filler spouts, bell rubbers and rubber spacers of the filling machine

 These areas can be hand sprayed with solutions of 25% iodophor, 90% isopropanol or 70% ethanol (preferred)

These solutions (70% ETOH) can be used to spray the corker jaws

It is a good idea to spray filler spouts and corker jaws every hour and after line stoppages

Mechanical maintenance of the bottling line including removing and greasing / cleaning all parts should take place the day before bottling It is good to develop a quality control plan at the bottling line for your sterilization procedure

- Take swab and air samples of the bottling line and room
- ATP Luminometer excellent tool for monitoring sterilization/sanitation process at bottling line
- Collect wine samples during bottling at regular intervals (i.e., beginning, middle and end) for microbial analysis and plating

Sterilization of the bottling line should take place both before and after each bottling

Best to use soft water during cleaning and sterilization to prevent scale buildup forming collection sites for organic debris and microbes

Hard water (scale) can also plug the filters and shorten the life and efficiency of the pre and final filter

## **Sterile Bottling**

Purpose is to remove all wine tolerant microbes that they do not present a post bottling threat or concern to the finished product Proper cleaning and sterilization of the bottling line and filter unit are critical for sterile bottling to occur

Involves proper sterile filtration set-up and practices

- 0.45-micron membrane filtration for white and Rosé
- 0.60-micron membrane filtration for red wine (dry)
- Must be able to perform integrity testing or bubble point

Most common troubleshooting issue stems from lack of efficient sterile bottling practices causing precipitates in the bottle and shortened shelf life potential. Also includes re-fermentation in bottled wine

## **Sterile Bottling: Filtration**

Depth filters

- Given a nominal rating in microns (µm)
- Not a true micron rating related to the size of microbe retained but representing the tightness of the filter pad
- Can also be expressed through rough, polish and sterile pad terminology
- Best used as a pre-filter in lengthening the life of the sterile filter



## **Sterile Bottling: Filtration**

Membrane Filter

- Considered best for sterile filtration
- Commonly supplied as cartridges but can also be used in the form of stacked disc filters
- Since they lack depth, they can plug up easily
- Rated on their membrane pour size





## **Sterile Bottling: Filtration**

Perform a fine pad or cross-flow filtration day before bottling (< 24hrs)

- beyond this time allows for colloids to redevelop and cause filtration fouling issues
- Same day of bottling could result in blow by or pad failure
  - Problematic for pre and sterile filtration plugging at bottling
- Filtration range from 0.65 0.45 pad or cross-flow
- All other additions i.e. SO<sub>2</sub>, sorbate and sugar should be done prior to this filtration up to several days before bottling
  - SO<sub>2</sub> should be stable several weeks prior to bottling

## **Sterile Bottling Filtration**

Filtration is critical to successful sterile bottling

The use of 0.45 to 0.60-micron membrane filter for both whites (0.45) and reds (0.60) critical for this process

- 0.45 Yeast and bacteria retention
- 0.60 Yeast not a guarantee for daughter cells
- Accomplished through membrane filter (absolute)
- Must be able to perform filter integrity checks
  - Bubble point test most common
    - Follow manufacturers directions

Locate final filter immediately after the pre-filter (cartridges)

Locate check valves for strategic microbiology sampling

- Sampling valves can be located between the pre and final filter in addition to immediately downstream of the final filter
- Place pressure gages both before and after the pre and final filter

### **Chemical Preservatives**

Sulfur dioxide (Previously mentioned) Sorbic acid, Potassium sorbate aka, (sorbate) Dimethyldicarbonate (DMDC)

The use of chemical preservatives and sterilant's should not be used as a substitute for sterile bottling but may be used in addition to sterile bottling if unsure about sterile bottling efficiency and procedures





## **Sorbic Acid / Sorbate**

Important as a chemical preservative

- sorbic acid is used to protect against yeast and mold spoilage generally in wines that contain some residual sugar
  - Higher sugar levels require additional

Sorbic acid behaves like sulfur dioxide in that the undissociated molecule is the most effective form in preventing yeast growth.

Therefore, as pH decreases, the greater the percent of undissociated acid and is more effective

Increased turbidity and or microbial load present, the more sorbic acid is required to be effective

## **Sorbic Acid / Sorbate**

Added in the form of a salt (potassium sorbate) of sorbic acid

In general, sorbic acid is added prior to bottling to prevent fermentations from occurring in the bottle

A general range of 100 - 200 ppm of sorbic acid is usually used at time of bottling.

Disadvantage:

- Certain people can detect sorbic acid levels normally found in wines
- Malolactic bacteria may convert sorbic acid to an undesirable odor geranium taint
- Not much activity against Zygosaccharomyces, Brettanomyces/Dekkera

## **Dimethyldicarbonate (DMDC)**

DMDC is a very effective yeast sterilant and has replaced sorbic acid in many larger wineries

Sold under the trade name Velcorin<sup>®</sup>

Lethal towards yeast and most lactic acid bacteria at maximum level of 200 mg/L (Fugelsang, 1997)

Larger wineries atomize it into the bottling line using a proportional pump

Smaller sized wineries will dissolve the appropriate amount of DMDC to be used in a small portion of absolute ethanol then add it directly back into the wine to be bottled

## **DMDC** Limitations

Although effective against most spoilage yeast, its killing properties is less effective on wine bacteria (Boulton et al., 1999)

- As a result, Important to monitor and maintain SO<sub>2</sub> levels appropriately
- DMDC is hydrolyzed rapidly with no residual activity
  - Secondary contamination can be a problem making sterilization procedures of the bottling line critical in keeping microbial activity down in bottled wine

DMDC needs to be handled properly since it can burn the skin and pose a health threat if ingested directly

Advisable to check out the cost efficiency in using DMDC from your winery perspective

#### **Bottle storage / aging conditions**



## **Bottle Storage Conditions**

Bottle storage

- Once bottled, easy for winemaker to go on to other cellar activities and forget about the importance of temperature during bottled case storage
  - storage during winter and summer months causes a serious concern with temperature extremes and fluctuations seriously affecting wine quality for storage areas not well insulated and lacking temperature control abilities
- High temperature concerns:
  - Loss of flavor and aroma compounds, loss of SO<sub>2</sub>, oxidation, protein haze, increased microbial activity, concern for increased levels of ethyl carbamate
- Low temperature concerns:
  - Tartrate precipitation, partial freezing of wine
- Temperature fluctuation concerns:
  - Heavy metals, protein, tartrates, oxygen ingress

## **Bottle Storage Conditions**

Bottle storage cont.

- Optimum storage for bottled white wines ranges from 55 - 63°F
- Optimum storage for bottled white wines ranges from 63 - 68°F – possibly higher for accelerated aging
  - Whites' cooler while reds can take a little warmer due to their phenolics/tannins
  - Helps extend shelf life and limits instability issues
- If two separate storage facilities are not feasible, one well insulated and temperature control facility at 63°F should suffice for both red and white wines



## **Bottle Storage Conditions**

Humidity

- Since it is customary to keep corks in an environment of 50 70% humidity, it is recommended to keep bottle storage / aging conditions the same
  - Although, corked wine is in an environment of 100% humidity
- After a period of 48 hours after bottling wines can be laid down horizontal if desired or upside-down during case storage
  - Does not matter for screw cap closures

Light

 Avoid direct sunlight which can cause precipitation issues with protein, copper and other heavy metals under reductive conditions

### **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



