Practical Juice and Wine Analysis

The 5 (or 6) Most Important Tests to
Run In Your Own Lab (and why it matters)

Overview

- About Myself – I am not from Napa
- Setting Up a Vineyard & Winery Lab (for not much $)
  - hydrometers – pH meter – pipettes – burettes – paper chromotography – aeration/oxidation apparatus distillation apparatus – glassware – scale
- The 5 (or 7) Most Important Lab Tests for Wine Making
  - Density – pH – Titratable Acidity – Free SO2 ML Chromotography – Alcohol by Volume – Copper Sulfate
  - WHAT – HOW – WHY
  - Practical Demonstrations
- Thoughts About Cold Climate Winemaking
- Questions and Answers

About Myself

- Benjamin J Banks
  - 2006 - Degree in Graphic Design & Photography in Pittsburgh
  - 2007 - Family started a vineyard in Minnesota and I came to design labels and do website
  - 2008 – Made first batches of wine using commercial kits, no wine knowledge at all prior to this.
  - 2010 – First batches of wine at Sovereign Estate, apprentice with our MAST student from France
  - 2011 – Took a few VESTA classes & Clark Smith's Fundamentals
  - 2012 – Took over winemaking at Sovereign Estate
  - 2014 & 2019 – Estate La Crescent wins Minnesota's Governor's Cup

About Myself – Sovereign Estate

About Myself – Where I Work

About Myself – The Wine Lab

About Myself – The Wine Lab

Setting Up a Wine Lab

- Hydrometers – Measuring Brix, Fermentation, & Alcohol
  - Hydrometer Tube - $5
  - Triple Scale Hydrometer - $6
  - Narrow Range Hydrometer - $29 (optional)
  - Alcohol Hydrometer - $26

- pH Meter – Measure pH for Ripeness, Yeast Selection, Malolactic Suitability, Cold Stabilization, SO2 effectiveness
  - Handheld - $85
  - Bench - $360 (optional)
  - pH 4 & pH 7 Buffers for Calibration - $16
  - Cleaning & Storage Solution - $16

Setting Up a Wine Lab (cont.)

- Pipettes - for Titratable Acidity, Free SO2, Copper Sulfate
  - Volumetric – 1ml, 15ml & 20ml - $25
  - Serological Pipettes – 10ml - $18
  - Pipette Pumps – 10 ml, 10 ml, 25 ml

- Burettes – for Titratable Acidity, Free SO2
  - 25ml buret - $30
  - 25ml buret - $30 (optional)
  - Labware Stand - $17
  - Labware Clamp - $16

Setting Up a Wine Lab (cont.)

- Paper Chromotography – for measuring malolactic ferm.
  - Paper, Column, Capillary Tubes, Solvent, Acid Standards - $60

- Free SO2 – for measuring unbound sulfites in wine
  - Economy aeration/oxidation kit - $100 (deluxe $370)
    - Air Pump, Tubing, Flasks, Indicator Solution
  - Phosphoric Acid 25% - $11
  - Hydrogen Peroxide - $2
  - Sodium Hydroxide 0.01N - $10

  - ALTERNATIVE – Vinmetrica SO2 Analyzer - $300 (optional)

Setting up a Wine Lab (cont.)

- Alcohol by Volume
  - Distillation Apparatus - $140
    - 500ml Boiling Flask, Condensation Coil
  - Stand - $15
  - Clamp - $16
  - Propane Burner - $38

  - ALTERNATIVE – Ebulliometer - $900 - $2,600

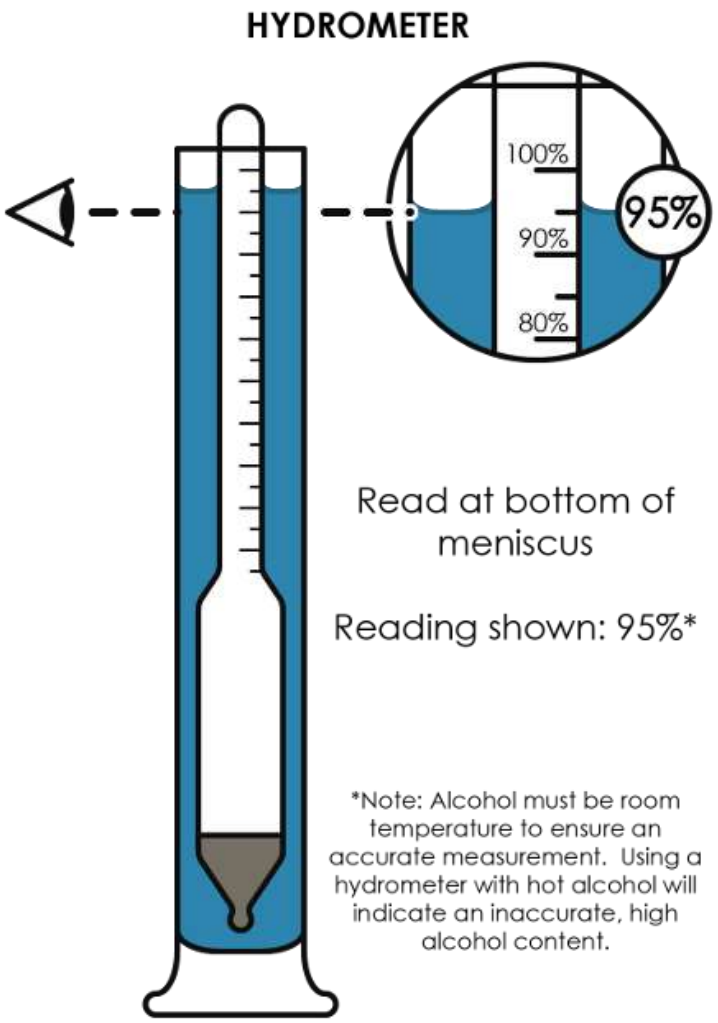
- Copper Sulfate Trial – to remove sulfurous odors
  - 250ml Volumetric Flask - $10
  - 100ml Volumetric Flask - $10
  - Copper Sulfate - $10

Setting up a Wine Lab (cont.)

- Other Glassware
  - At least 2 - 100ml graduated cylinders - $20
  - At least 5 – 150 ml glass beakers - $4 each - $20
  - 500 ml Volumetric Flask - $10
- Scale
  - 2kg x 0.1g - $30
- Total - $800 for essentials that will last for as long as they are taken care of

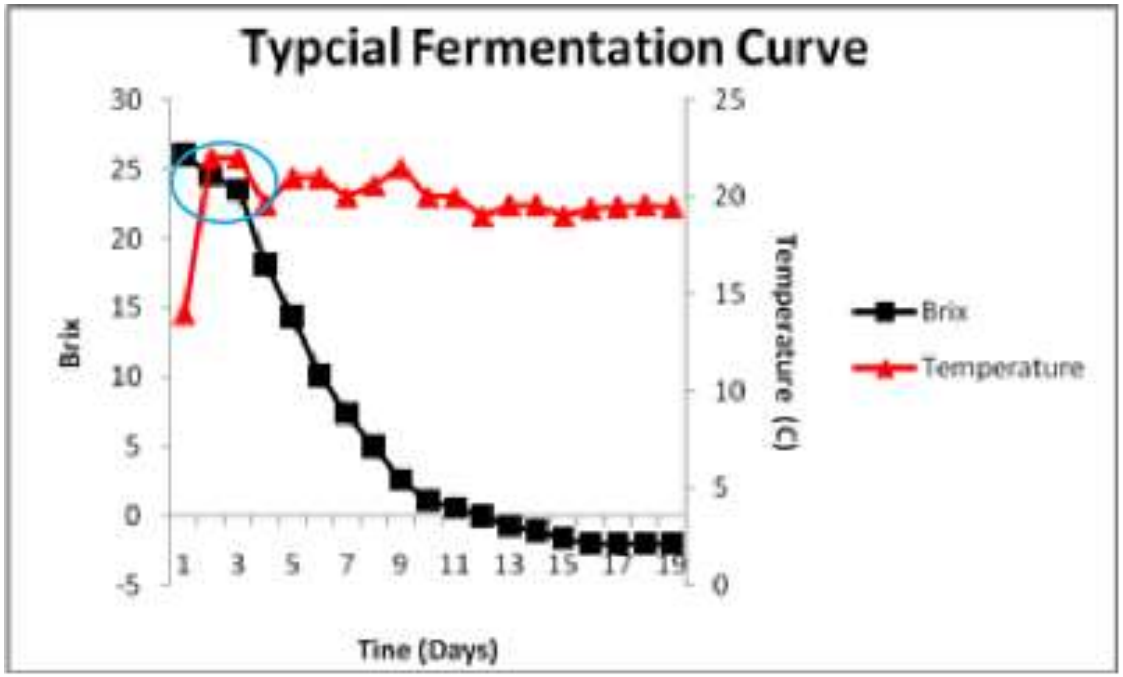
TEST #1 - Measuring Brix

- Hydrometer vs. Refractometer
  - Hydrometer requires a larger sample and is usually more indicative of actual juice, and more juice is needed to test pH and TA as well
  - Hydrometer is cheaper
- My preferred method
  - Collect whole clusters, about 3-5
  - Crush entire cluster and strain
  - Measure strained juice with hydrometer, and keep juice for testing pH and Titratable Acidity



TEST #1 – WHY measure Brix?

- WHY it is important
  - Easiest measure of grape ripeness (but not only!)
  - Knowing starting sugar lets you know potential alcohol
  - Measure the progress of sugar fermentation



TEST #2 - pH

- Electrode must be soaked 30 minutes before beginning
  - Benchtop meter has the advantage of always being immersed
- Calibrate with pH 7 buffer, rinse, then pH 4 buffer, rinse
- Immerse electrode in juice/wine and give it a minute to stabilize. Adjust for temperature if needed (20C – 68F)

TEST #2 – WHY measure pH?

- WHY it is important – probably most important!
  - Recommended Harvest Value between 2.9 to 3.3
  - It is possible to have a low sugar, high pH grape, such as during a very wet harvest. In this situation it would be better to pick the grapes using pH as the main harvest parameter
  - It's the easiest way to measure TA by titration, no need to watch for a color change
  - It affects yeast and malolactic bacteria choices
    - Many Malolactic Bacteria strains won't work at low pH

TEST #2 – WHY measure pH?

- Effectiveness of SO2 as an antimicrobial is determined by pH
  - When dissolved in water, SO2 exists in three forms
    - 2H & SO3 (bisulfite)
    - H & HSO3 (sulfite)
    - H2O & SO2 (molecular) ← Only the molecular form is antimicrobial
  - At wine pH, very little molecular SO2 is present, but its effect increases exponentially as pH gets lower
  - If pH is too low, the wine stings of sulfur
  - If pH is too high, the wine can spoil easily

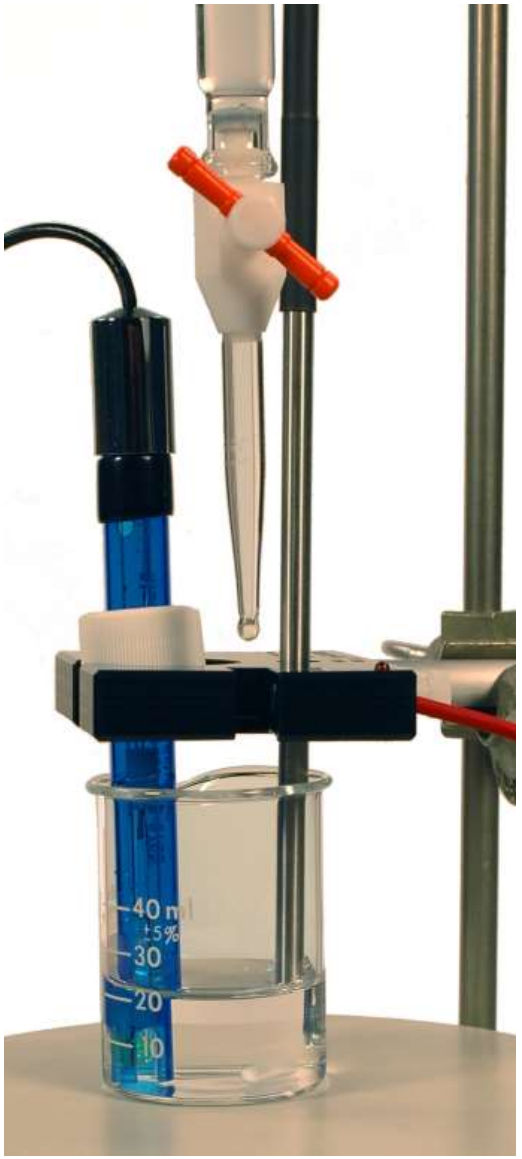
TEST #2 – WHY measure pH?

- Cold Stabilization balances on pH
  - Cold Stabilization of juice/wine below pH of 3.65 will see its pH get even lower
  - Cold Stabilization of juice/wine above pH of 3.65 will see its pH get even higher

- pH can determine your choices for deacidification
  - A high TA and low pH wine can use potassium bicarbonate, as it will raise pH
  - A high TA and high pH wine might require calcium carbonate and a double salt method so the pH doesn't get too high

TEST #2.5 Titratable Acidity

- 15 mL of wine in a small beaker
- Measure pH. With probe in the beaker, titrate with 0.2N Sodium Hydroxide (NaOH) until pH reaches 8.2 pH
- Being logarithmic, will start slowly and get faster as you approach 8.2
- mL of NaOH needed is equal to 1/10 % Titratable Acidity
  - 16.5 mL needed would equal 1.65%, for example
- Benefit of using pH instead of phenolthalein is that many hybrid juices are too pigmented to see a color change



WHY to test Titratable Acidity

- Acidity is the major hurdle in cold climate winemaking
- Measuring acidity during growing is a great indication of ripening
- With pH, gives you the information you need for deacidification methods you can use
  - Potassium Bicarbonate if pH is low, up to 0.3%, as long as pH doesn't get above pH 3.65
- Also informs on winemaking style
  - I tend to embrace acidity and use it
  - If acidity is below 1%, I will make it dry
  - If acidity is between 1% and 1.5%, I will make it semi-sweet
  - If acidity is above 1.5%, I will deacidify or use it for sparkling wine

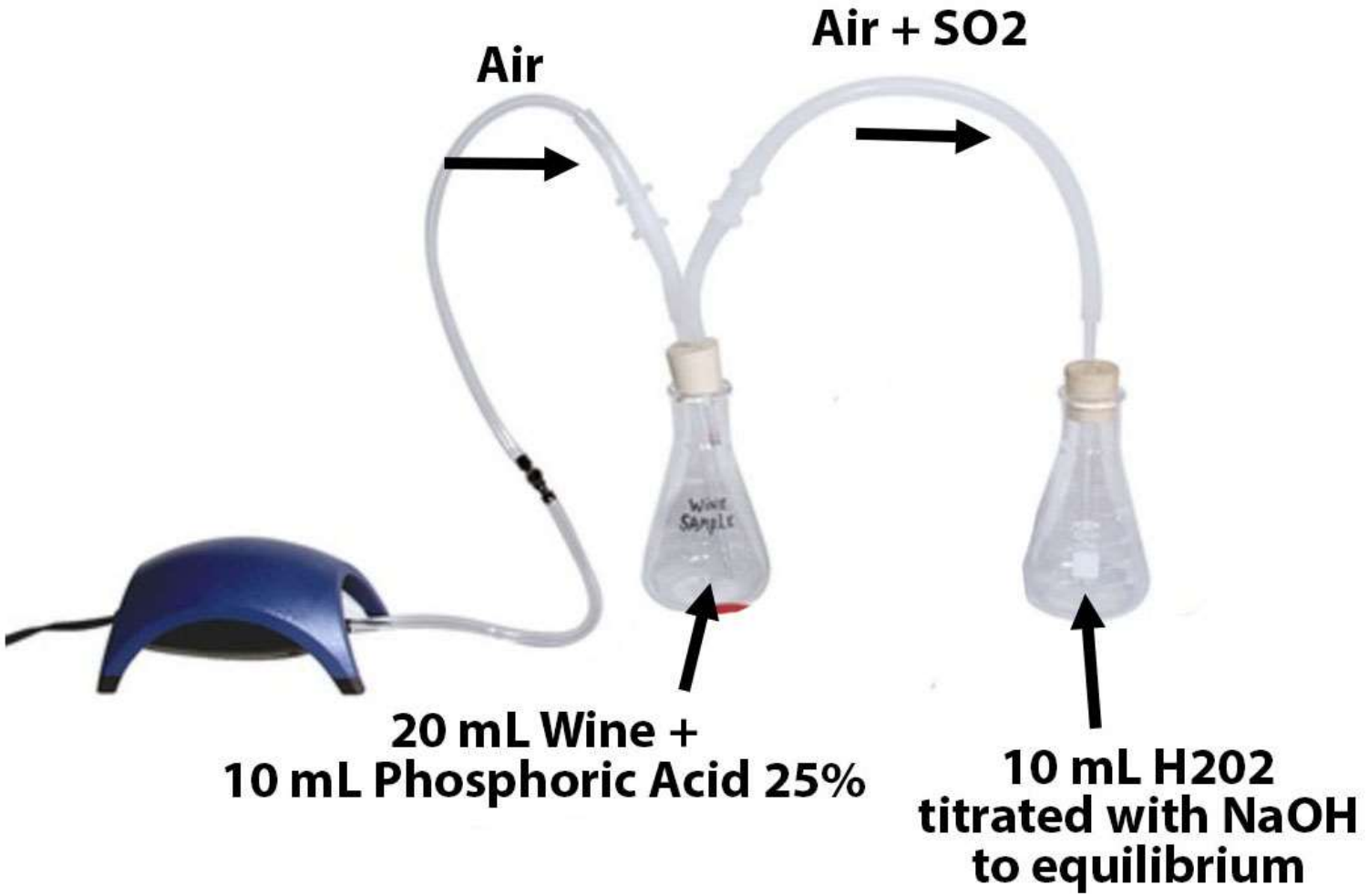
TEST #3 – Free Sulfur Dioxide

- My Favorite Test! Getting this test solid made the biggest increase in the quality of my wines.
- **I use the aeration/oxidation method.**
- The SO2 added to wine will bind with aldehydes. Once bound, they are no longer affective at preventing new oxidation or microbial suppresion
  - Aldehydes are formed EVEN IF the wine is kept completely away from oxygen. Polymerization of phenols can produce hydrogen peroxide which will oxidize the wine unless SO2 binds it first
- Very important to check SO2 of wines kept in bulk every few weeks or so, as it naturally will decrease

TEST #3 – Free Sulfur Dioxide

- The test works by dropping the pH of a wine sample so low that all the unbound SO2 will exist in its molecular form, which is volatile. It can then be moved with an air current to a separate vial of H2O2, which will immediately react with the SO2 to form sulfuric acid. The sulfuric acid can then be titrated with NaOH to an equilibrium point, which can then let you know how much SO2 was present in the wine

TEST #3 – Free Sulfur Dioxide

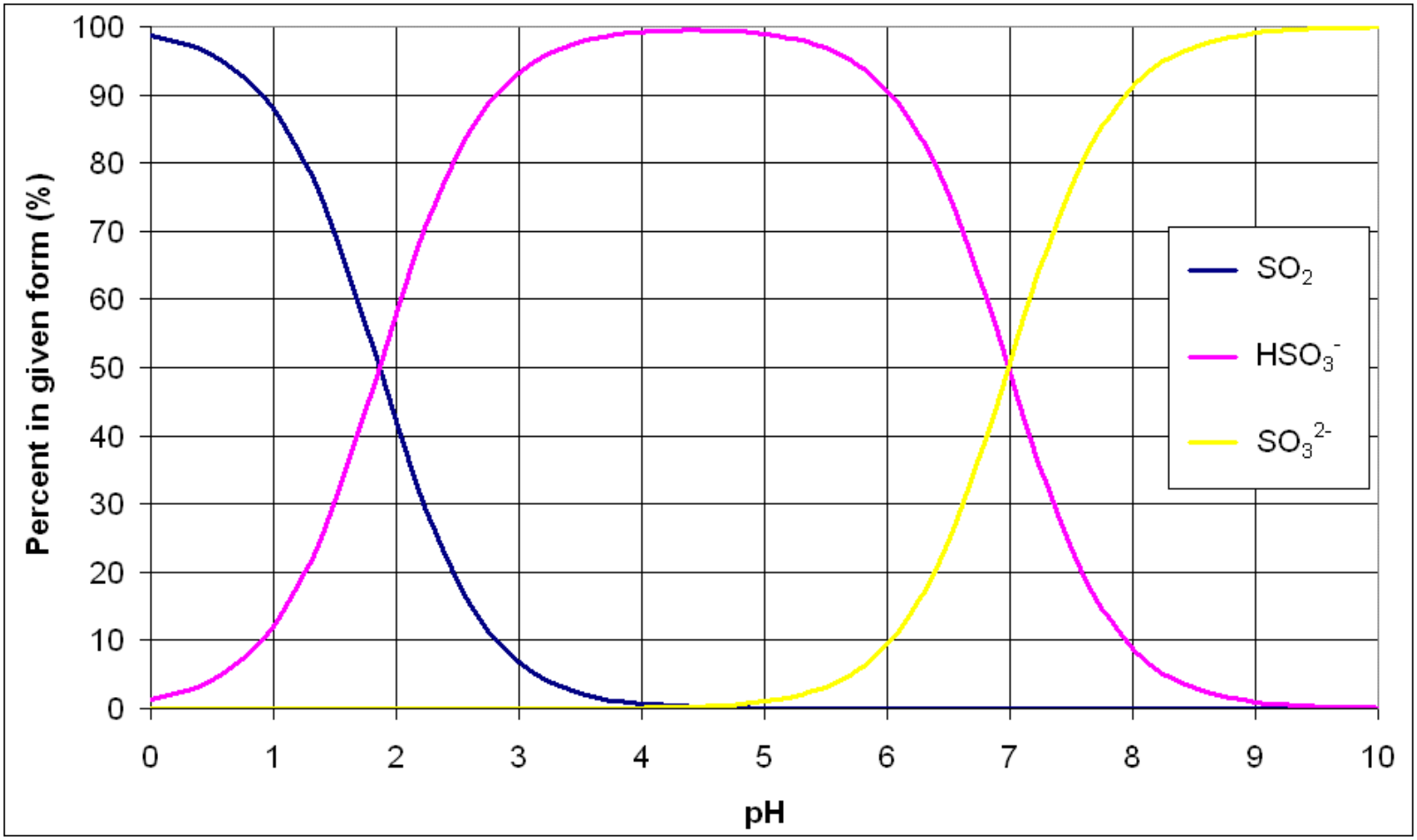


TEST #3 – Free Sulfur Dioxide

- Into the reaction vial
  - Add 10 mL of Hydrogen Peroxide (H2O2) 3%
  - Add 3 drops of indicator solution. The vial should be pinkish
  - Titrate the vial with 0.01N Sodium Hydroxide (NaOH) to equilibrium, which is when the vial turns bright green
- Into the test vial
  - Add 20 mL of the wine to be tested
  - Add 10 mL of Phosphoric Acid (H3PO4) 25%
- Attach the aeration so that a sealed stream of air bubbles through the test solution and flows into the reaction vial
  - Allow the air to flow for 10 minutes
- Titrate the reaction vial back to green using NaOH
  - mL of NaOH used x 16 = ppm free SO2 in sampled wine

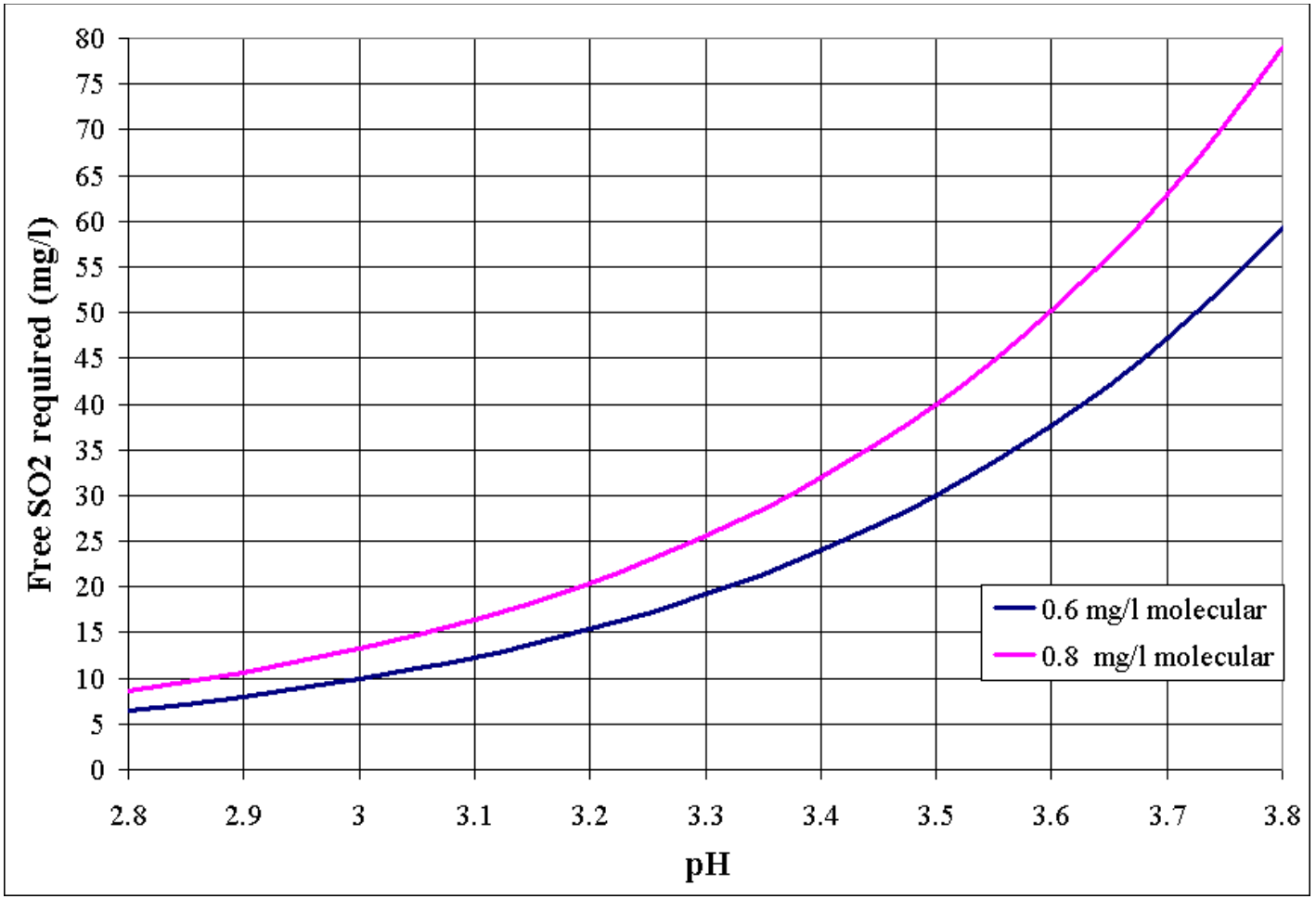
TEST #3 – Free Sulfur Dioxide

- Effectiveness of free SO2 is determined by pH!



Test #3 – Free Sulfur Dioxide

- 0.8ppm Molecular SO2 is needed for microbial inhibition



WHY test Free Sulfur Dioxide

- In my experience, the two biggest hazards to small wineries are oxidation and spoilage
- Knowing your Free SO2 and your pH lets you control the majority of oxidation and spoilage
- It allows you to have the proper dosage of SO2 at bottling so that the wine open up after uncorking

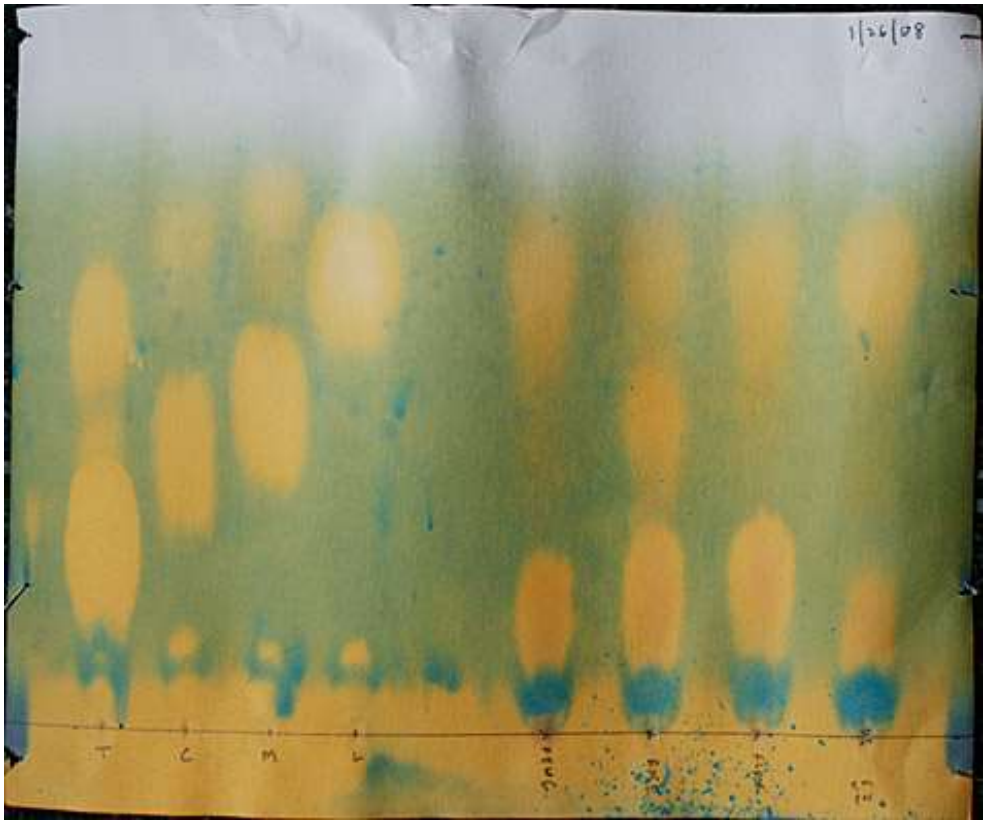
- MY APPROACH – 25g/ton SO2 at crush. Hit wine with very high SO2 as soon as fermentation is done, monitor its decrease after racking, top up as needed, bottle at 0.8ppm molecular for whites & rosés and 50ppm total free for reds.

TEST #4 – Paper Chromotography

- On a sheet of test paper, make a mark for each acid standard (tartaric, malic, lactic) and each wine being tested one inch from the bottom
- Using a separate capillary tube for each wine or standard, drop 7-10 drops on each mark, allowing to dry each time
- Put a half inch of solvent in the test chamber, and then put the test paper in such a way that it stands up in the chamber with the samples at the bottom
- Allow the paper to sit in the solvent for 8 hours, then allow to dry

TEST #4 – Paper Chromotography

- The solvent will pull the standards and the wine toward the top. By comparing the wine samples to the standards, you will be able to see if any malic acid is left.



WHY to do Paper Chromotography

- If a wine is exposed to malolactic bacteria at any point, such as a barrel or dirty hose, it will likely undergo a spontaneous fermentation unless it is sterile filtered
  - This is very bad if it is already bottled
  - It is doubly bad if you use Potassium Sorbate, it is the cause of geranium taint
- Knowing when MLF is done lets you get SO2 in the wine quicker for less likelihood of oxidation

TEST #5 – Alcohol by Volume

- Distillation Method – Cheap but Slow
  - Also the method used by the TTB
  - Works with sweet wine
  - Take a quantity of wine (250 mL) plus a small amount of water (100 mL) and distill it and collect 250 mL of distillate. Alcohol boils at a lower temperature than water, so all of the alcohol will be distilled and condensed.
  - This distillate can then be measured with a hydrometer to determine alcohol by volume

- Ebulliometer – Fast but Expensive
  - Works by measuring the difference in boiling temperature between pure water and wine

WHY measure Alcohol by Volume

- TTB Tax Classes
- Used to make decisions about chapitalization and consistency of wines
  - Different (and even the same) yeasts can have different conversion factors.

Test #6 – Copper Sulfate Trials

- Stressed Fermentations can produce a lot of hydrogen sulfide (H2S) which smells like bad eggs
- Reduced wines can produce sulfur mercaptans which is the smell used to detect natural gas leaks
- Oxidized mercaptans can produce disulfides which smell like petrol, garlic, or canned asperagus
- Copper Sulfate can be a useful tool to remove these odors if used correctly
  - It is poisonous in large doses, so it is critical to use the very smallest amount needed, and then filter out the precipitate

Test #6 – Copper Sulfate Trials

- First do a penny test
  - Get three beakers and two pre-1982 pennies (made of copper)
    - One beaker is just wine, one is wine plus the penny, one is the wine plus the penny plus 80ppm ascorbic acid
    - Copper will react with H2S and mercaptans, but not disulfides
    - Ascorbic Acid with break apart disulfides so that copper can react with them
- After determining weather ascorbic acid is needed or not, I follow the procedure outlined at https://www.extension.iastate.edu/wine/copper-sulfate-trial

Test #6 – Copper Sulfate Trial

- Make a 100 mL solution of 1% copper sulfate
- Take 1 ml of that solution, and add water to make 250 ml of 0.004% copper sulfate solution
- Fill five beakers with 100 mL of wine, marked 1-5
- Beaker 1 is a control. Beaker 2 get 0.5 mL of the 0.004% solution of copper sulfate, Beaker 3 gets 1.0 mL, and so on
- Cover the beakers and observe the next day
- Determine which concentration of solution was needed to eliminate the odor
- Dose the wine with that amount of copper sulfate, and then fine with bentonite/PVPP
- Filter the wine 3-5 days after dosing

WHY do Copper Sulfate Trials

- Sulfurous odors have a low sensory threshold
- Best to take care of as soon as noticed. Sometimes sulfurous odors can disappear without treatment only to reappear after bottling, making the wine harder to sell.
- Trials allow the minimum amount of copper to be used, which will precipitate and get filtered out leaving little to no residual

Cold Climate Winemaking Thoughts

- Very Different Grapes require very different approaches
- I noticed at the tasting last night that most of the wines were very good and also how much the different climate and soil affected the grapes from here versus Minnesota
- Figure out what the grape wants to be rather than push it toward something it doesn't

Frontenac

- Good Characteristics – deep color, intense fruit, strong grower
- Challenging Characterisics - high acid, no tannin, bitter green if overextracted
- To me these characteristics do not fit well with traditional red wine techniques
- In my opinion, they are pretty ideal for rose and port style wines

Frontenac Gris

- Good Characteristics – Versitility, intense and distinct fruitiness, strong grower
- Challenging Characteristics – Inconsistent color, high acid
- I have had Frontenac Gris as great white wines and roses. I haven't had it the same way twice, though, at least from my growers. Because of that unpredictability, it's hard to make into a flagship wine, in my opinion
- It is great for making fanciful wines, especially dry and sweet roses, as crowd-pleasers

Frontenac Blanc

- Good Characteristics – Very tropical aromas, easy to grow
- Challenging Characteristics – Phenolic can be a touch bitter, Last grape to ripen (at least for us), high acids, low pH
- I'm still trying to figure out what to do with this grape. I'm stuck between an oaked malolactic version and a traditional sparking method. Both have been good. It's also been very good as a very reductive dry white, like a New York Reisling

La Crescent

- Good Characteristics – Unmistakeable aroma, generally good harvest numbers (at least in Minnesota), unique
- Challenging Characteristics – Very hard to grow
- The aroma of La Crescent is so special I wouldn't do any technique that would hinder it, such as malolactic or oaking.
- It tends to do well with a very unstressed fermentation. I will often stop it before total dryness so that the yeast don't produce off aromas trying to ferment the last percent of sugar

Marquette

- Good Characteristics – hearty grower, rich color and aroma, touch of tannins
- Challenging Characteristics – can be hard to ripen, bitter if over extracted, doesn't play nice with oak flavors
- Despite being bred from Pinot Noir it does not like to be treated like Pinot Noir
- Enjoys lots of oxygen uptake during fermentation and aging but not the flavor of new oak – does well in older barrels

Thank You

I will be around to answer questions or show you how to use the lab equipment if you are new to it.