Best Practices for Wine Analysis
CCC February 16, 2017

University of Wisconsin
Associate Outreach Specialist
Nick Smith
nsmith35@wisc.edu
Winery Analysis: Today’s Topics

- Advantages of an in-house laboratory
- Laboratory needs assessment
- Techniques and recommendations for optimal results
- Laboratory decisions for the commercial winery

http://www.chaikenvineyards.com
Session Expectations

- What are your expectations?
- Any specific questions?
- Where should we focus?
Value of the Laboratory

- No substitute for laboratory results and information
- Agricultural products vary greatly from season to season
- Wineries producing sub-par products often lack analytical capacity and capabilities
  - Commercial wine production is not large scale home winemaking
Advantages of a Laboratory

- Timely results
  - Improves efficiency of winery operations
- Objective insight into wine quality
- Improved quality
  - Quality control
- More information
  - Provides historical context to assist with future decisions
  - Greater understanding of why something works or doesn’t
- Problem solving
Winery Analysis: Today’s Topics

- Advantages of an in-house laboratory
- Laboratory needs assessment
- Techniques and recommendations for optimal results
- Laboratory decisions for the commercial winery

http://www.chaikenvineyards.com
Laboratory Needs Assessment

- When designing a laboratory, what do you need to consider:
  - Budget
  - Time
  - Expertise and skill
  - Size of production and number of wines
Budget

Fast

Cheap

Quality
Winemaker Checklist

- **Budget**
  - How much do you plan to invest in laboratory equipment?
  - Wide assortment of equipment available
    - Which piece of equipment is right for you?
      - Budget
      - Time
      - Expertise
      - Volume of analysis
Budget

- The lower your budget:
  - The more time and effort you will need to dedicate to analysis
    - Manual vs. automated
  - Increased use of outside services
  - Less information to work with
    - Riskier winemaking
    - Lower quality
Time

- An undervalued resource
- How much do you have available and are willing to commit to laboratory work?
- When time is tight and you have to make a decision on what gets done, how will you prioritize lab analysis?
- What areas of wine production do you tend to focus on?
Time

- When time is limited:
  - Consider more automated solutions
  - Hire someone to do the analysis
  - Schedule time to perform analysis
  - Use an outside laboratory
Expertise

- What is your background in science and chemistry?
- What is your interest level in learning about techniques and analytical test?
- How enjoyable do you find analytical analysis?
- Are you intimidated by the analysis?
Size of Production

- How large is your operation, and how many wines do you plan to produce?
  - Understand your analytical demands
    - Volume and quality
- Large volumes
  - Expensive if things go wrong
- Large product line
  - The more products, the more analysis
Size of Production

- The large your production, or the greater the number of products, the greater the need for high quality and easy to produce analysis
Winery Analysis: Today’s Topics

- Advantages of an in-house laboratory
- Laboratory needs assessment
- Techniques and recommendations for optimal results
- Laboratory decisions for the commercial winery

http://www.chaikenvineyards.com
Techniques for Quality Results

- Accuracy and precision
- Volumetric analysis/methods
- Reagent standardization
- QC methods for analysis
Accuracy and Precision

- Low accuracy, Low precision
- Low accuracy, High precision
- High accuracy, Low precision
- High accuracy, High precision
Volumetric Techniques

- Use volumetric glassware
  - Often calibrated to a specific volume
    - Pipets
    - Graduated cylinders
    - Flasks
    - Burets
- Available in different classes
  - Class A vs Class B
- A small error in measurement can magnify to large error at the commercial scale
- Balance (0.1 to 0.01 g)
Volumetric Pipettes

- Required for accurate dilutions and measurements
  - Stock chemicals
Volumetric Pipette

- TC vs TD

Class A pipette is accurate to within +/- 0.020 mL.

TD stands for “To Deliver.” That means by letting the liquid simply drain out, it will deliver 10mL in 25 seconds.

20°C (68°F) is the temperature where most accurate.
Volumetric Flasks

Setting the meniscus to the volume mark. Side view.
W/W vs W/V vs V/V

- **W/W**
  - Weight per Weight
  - Mass fraction
  - Brix

- **W/V**
  - Mass per unit volume
  - Titratable acidity (g/L)
    - Stock solutions
    - 5%: 5 grams of product, brought up to 100 mL of total solution
      - Not 5 grams plus 100 mL of solution

- **V/V**
  - ABV
Burettes

- High visible gradation lines
- Class A and class B
- Different sizes
  - 25 ml and a 10 ml
Standardization

- Process of measuring/validating the concentration of a stock solution
  - Stock solutions may not be stable over time
- Counter titrate with acid/base
  - HCl
- Test with a sample with a known concentration (calibrator/standard)
- Run calibrator/standard at start and end of an analytical run
- Troubleshooting to find errors/problems
Records

- Record and track analysis
- Record and track calibration information
  - Calibration values
  - Frequency
- Detailed notes
Winery Analysis: Today’s Topics

- Advantages of an in-house laboratory
- Laboratory needs assessment
- Techniques and recommendations for optimal results
- Laboratory decisions for the commercial winery

http://www.chaikenvineyards.com
Entry Level Analysis

- The basic tests
- What they measure
- What the numbers mean and how to use them
- How often to perform analysis
Tests

- Basic analysis and procedures for all wineries:
  - Soluble solids (Brix)
  - pH
  - Titratable acidity
  - Free SO₂
  - Estimate residual sugar
  - Estimate malic acid
  - General heat and cold stability
  - Bench trials
Tests

- Additional tests for intermediate/advanced laboratory:
  - EtOH
  - Malic acid
  - Residual/reducing sugar
  - Volatile Acidity
  - Advanced heat and cold stability
  - Microbial
Soluble Solids

- **Hydrometers**
  - Multiple scale ranges (+5/-5)
  - Useful for tracking fermentations

- **Refractometer**
  - Digital
  - Less useful for tracking fermentation

- **EtOH prediction**

- **Grape sampling/ripening**
Residual Sugar

- Residual vs. Reducing
- Need to understand how much sugar remains in wine
  - Bottle stability
  - Desired sweetness
- Stuck fermentations
- Can not rely on a hydrometer
- Estimate using Clinitest
- Send out for final value
  - Or measure using enzymatic testing
pH

- Purchase a quality bench top meter
  - Fisher/Orion, Mettler Toledo
- Ideally 3 point calibration, 0.01 pH units
- Reading will pause when stable
- Reports slope values after calibration
pH

- Important for microbial stability
  - SO2
- Color, particularly rosé wines (SO2)
- Berry samples – monitor ripening
- Must – start pH, make adjustments
- Fermentation – watch for pH spikes/changes
- Post fermentation – at least once a month
  - FSO2 additions, changes in pH could indicated spoilage
- Cold stability
  - 3.65 break point
- MLF
Titratable Acidity

- Important for measuring sourness
- Expressed TAE
- When to measure:
  - During ripening: Harvest Parameter
  - After crush/press
    - Adjust if necessary or interested
  - After fermentation
  - After cold stabilization
  - During aging
  - Prior to bottling
Titratable Acidity

- TA and pH may be related, but one cannot tell you what the other is
- Manual vs automated methods
  - Time, expertise, budget, precision
Titratable Acidity

- To de-gas or not to de-gas?
  - CO2 acts as an acid when dissolved in solution
  - High CO2 wines (during or just finished fermentations) should be degassed for best results
  - Heating up on hot plate
  - Microwave
  - Filter with a syringe filter
SO$_2$

- Free and total SO$_2$
- Important for:
  - Microbial stability
  - Oxidation
- Measure and adjust after fermentation
- Measure and adjust regularly (1x+ per month)
SO₂

- Multiple methods and equipment available
- Aeration / Oxidation
SO₂

- Other methods/equipment:
  - Iodine
  - Ripper
  - Automated/probe
SO2 and Oxidation

- Some oxidation prevention properties
  - Binding to precursors and oxidation products
  - Little to no scavenging of oxygen
- Ethanol in the presence of O2 can become acetaldehyde
- O2 can lead to aerobic bacteria growth (*Acetobacter*)
  - Acetic acid and ethanol leads to ethyl acetate
- SO2 is not an effective strategy against high oxygen pressure environments
- Sound cellar practices and equipment is best
Malic Acid

- Determining when MLF has completed
  - Avoid spoilage and other faults
- Perform regularly during MLF
- Paper Chromatography
  - Indicates if MLF has or is near finished
  - Sensitive to about 100 ppm
  - Hard to measure MLF progress
  - Takes approximately 24 hours
  - Less than desirable odor
- Send out for analysis
- Presence or absence of CO2 does not correlate to MLF activity or malate levels
Malic Acid

- Enzymatic testing with spectrophotometer
  - May need to decolorize sample with PVPP
  - Provides actual malic acid content
  - Can allow for tracking of MLF
  - Ensure proper residual malate levels are achieved.

http://schaechter.asmblog.org
Stability Testing

- Hot and cold stability
  - Cold Stability
  - Refrigerator/Cold storage test
    - Quick result
    - Filter while chilled
  - Place sample in refrigerator, observe over a week
  - Place sample at -4C, observe after 2-3 days
    - Use a small freezer with a temp controller
- Conductivity testing
  - Chill sample, add KHT, measure difference in conductivity
Stability Testing

- Heat stability
  - Usually conducted with a bentonite fining trial
  - Visual or turbidity assessment

- Open to debate:
  - Usefulness
  - Testing parameters
  - Interpretation

- Water bath
- Bentotest
- TCA
Bench Trials

- A bunch of same size containers
- Measure accurately
- Create a stock solution
  - Scale accurate to 0.1 to 0.01 g
- Use a micropipettor
- Attempt to duplicate winery conditions
  - Mimic filtration
- Purchase a new model
  - Avoid Ebay
- Start with a 100uL to 1000 uL
- (1 to 5 mL nice for juice TA)
- Eppendorf
- Rainin
Advanced Testing

- Ethanol
- Enzymatic analysis
- Color and phenolics
- Volatile acidity
- YAN
Ethanol

- Small winery:
  - Distillation vs. ebulliometer
- Cost and time
  - In house vs. outside laboratory
- Sweet wines and high alcohol wines
- Labeling requirement
- Troubleshooting
- Advanced:
  - NIR (Anton Paar – Alcolyzer)
  - GC
  - WineScan
  - Enzymatic?
Volatile Acidity

- Primarily acetic acid
- Cash still or enzymatic
- TTB limit
- A wine fault, often before TTB limit
- Can be measured prior to sensory detection
- QA/QC
- Measure after AF, throughout MLF, and regularly during storage
YAN

- Yeast Assimilable Nitrogen
- Two tests
  - Ammonia
  - Free Amino Nitrogen
- Ammonia
  - Enzymatic test
  - Formal Titration
  - ISE Electrode
- Free Amino Nitrogen
  - OPA
  - Spectrophotometer
Spectrophotometer

- Enzymatic assays available to measure a wide array of food and beverage components
  - Malate, RS, VA, YAN
  - Color and phenolics
- UV vs Vis
- Multiple micropipettes
Spectrophotometer

- Dealing with high color samples:
  - High color increases start Abs
    - Dilution may not be effective
  - Polyphenols may interfere with analysis
  - 0.1 g PVPP in 10 mL of sample
  - Malate
  - May be useful to consider for other spec analysis
Spectrophotometer

- Multiple options exist
- MegaQuant from Megazyme
- Unitech Scientific
Microbial

- Microscope
  - Measure yeast counts and viability
  - General identification of microbes
  - Assessment of sediment
- Camera
- Plating
  - Dedicated/controlled environment
- Illustrated Guide to Microbes and Sediments in Wine, Beer
- Microbiological Analysis of Grapes and Wine: Techniques
Books

- UC David Bookstore (Enology and Viticulture)
- Chemical Analysis of Grapes and Wine: Techniques and Concepts
- Wine Analysis and Production
Review

- Importance of laboratory analysis
- Analytical needs and capabilities for your winery
- Proper technique
- Appropriate analysis
Questions?

- Nick Smith
- NSmith35@wisc.edu