Wine testing and analysis J. Richard Sportsman, Ph.D. Vinmetrica



Today's talk

- Vinmetrica and me
- Wine pH and TA
- Controlling SO₂
- New tools for better guidance



Vinmetrica

- Started 2010
 - Preceded by 16 years of amateur winemaking
 - Need for better SO₂ analysis
- Developed the SC-100 system for SO₂
- Wine lab tools that are
 - Easy
 - Accurate
 - Affordable





Vinmetrica Today

- Over 6000 analyzers in use worldwide
- Instrument kits for SO₂, pH & TA, Malic acid
- Test systems for RS, YAN, DO & %ABV
- Lab Services
 - Wine chemistry
 - Microbiology





Rich Sportsman

- Ph.D. analytical chemistry U Arizona 1982
- Bay area biotech 1992 2008
- Amateur winemaker since 1993
- Started Vinmetrica 2010
 - Commercialize my sulfite toy
- Started Little Oaks Winery 2012
 Hobby out of control





Measuring pH

- <u>Don't</u> stir or swirl solution continuously
 - Mix briefly and then let the electrode sit still
 - Former recommended stirring not needed with new electrodes
- Calibrate system daily
 - Set with 4.01 (or 3.00) and 7.00 reference solutions
 - Best to have everything at same temperature
 - Tip: check with Cream of Tartar
 - AKA potassium hydrogen tartrate or bitartrate
 - Fresh saturated solution ($\frac{1}{4}$ tsp/20 mL H₂O) gives pH 3.55 at 25°C
- Take pH reading.
 - Don't add water!



Measuring TA

- Take measured sample (5 mL)
 - remove CO₂ by shaking or vacuum, if needed
 - Musts: homogenize first?
 - reds probably yes
 - whites probably no
 - Add water as needed
- Titrate with 0.10-0.20 M NaOH to pH 8.2 (7.0?)
- Reported as g/L or g/100 mL (%) tartaric acid

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Measuring Sulfite (SO₂)

- Critical sulfite levels in winemaking
 - Before primary
 - After primary before MLF inoculation
 - Monthly during barrel aging
 - At bottling
- Methods
 - Ripper titration
 - Aeration Oxidation (Monier-Williams)

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Measuring Sulfite (SO₂)

"Ripper titration": reaction of SO₂ and iodine

 $SO_2 + I_2 + H_2O \rightarrow 2I^- + SO_3 + 2H^+$

Endpoint iodine detected by starch (blue color) or electrode (Hanna, Vinmetrica).

Aeration Oxidation (AO)

 $SO_2(g) + H_2O_2 \rightarrow H_2SO_4$

Air bubbled through acidified wine sweeps SO₂ into peroxide. Resulting sulfuric acid is titrated at the end with NaOH.

• What is the most reliable method?

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Comparison of SO₂ methods

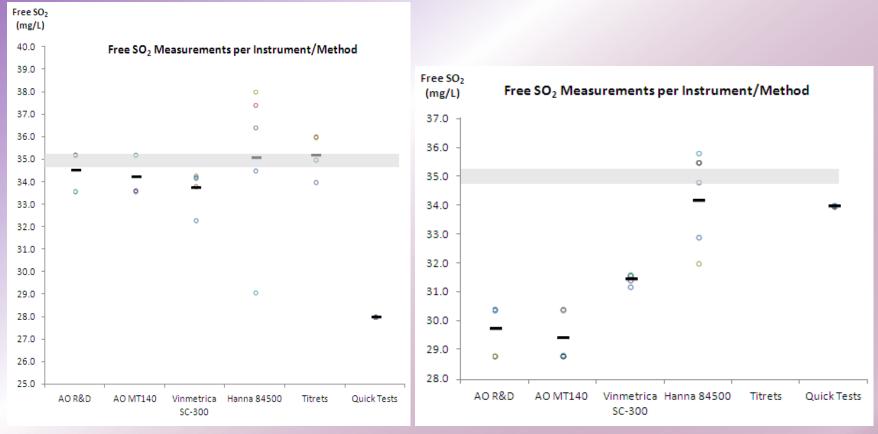
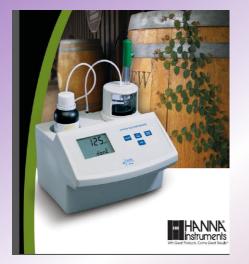


Figure 1. Free SO₂ on 35 mg/L

Figure 3. Free SO₂ on 35 mg/L with grape tannins, 2 g/L



Price/performance for SO₂



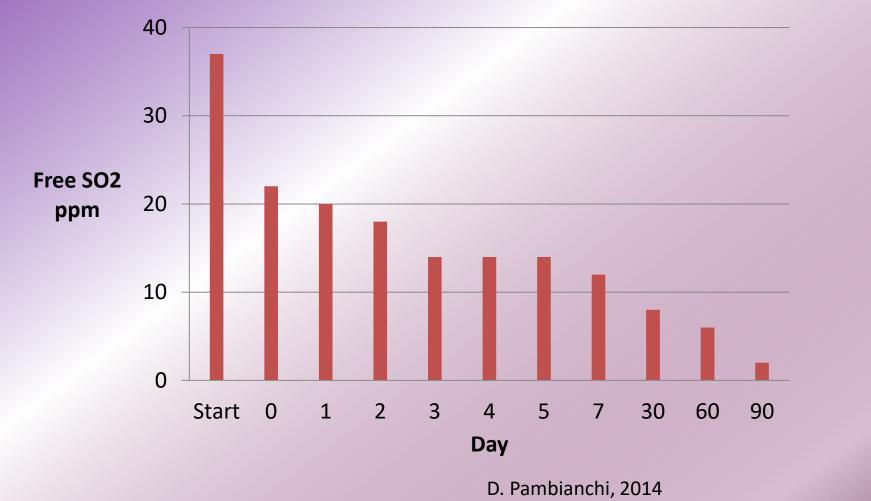
Hanna autotitrator \$900 2 min Ripper titration/ORP Aeration Oxidation Setup \$200 20 min Aeration- H_2O_2 oxidation/ NaOH titration



Vinmetrica SC-300 (+ pH & TA) \$485 2 min Ripper titration/Ampero



Free SO2 levels drop!



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"A Review of Sulfite Management Protocols Based on SO2 Levels and Type of Wine" <u>https://morewinemaking.com/web_files/morebeer.com/files/SO2</u> <u>Management Protocol.pdf</u>

Adjusting SO₂

- Potassium metabisulfite 10% solution
- SO₂ gas or liquid
- Target levels depend on pH and desired "molecular SO₂" target (0.5 to 0.8 ppm)
 – 20-40 ppm free SO₂ typical
- Use sulfite calculators (e.g.WMM's or FermCalc)
- Freshly-added SO₂ should be re-checked

 Loss due to binding or dO₂ over a few hours

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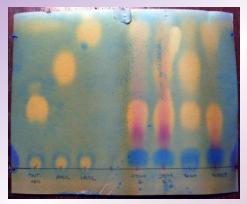
New Tools

- Malic acid testing for MLF completion
- dO₂ electrode
- ABV analysis
- Sentia[™] Analyzer

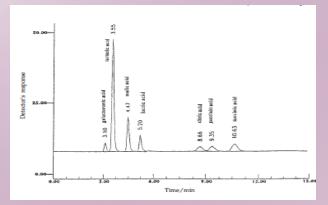


Malic methods

- Need to know when MLF is done for timely SO₂ adjustment
 Measure malic acid levels.
- •Want < 0.1 g/L







Paper chromatography 24 hours – qualitative Inexpensive, simple Detects 0.2 g/L

Enzymatic - photometry 1 hour - quantitative Expensive, technical Detects 0.01 g/L

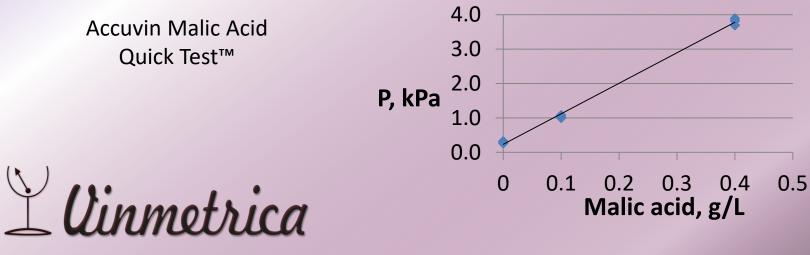
HPLC <1 hour - quantitative very expensive & technical Detects 0.01 g/L



Malic Methods







Dissolved Oxygen System

- Managing O₂ levels in must and wine is critical.
- dO₂ electrode: dip and read
- Target < 2 mg DO/L @ bottling.

Test	mV	"pH"
0% DO	+8.0	6.67
100% DO	+302	1.48
Wine	+50	5.92
sample		

DO, % saturation = 100 * (50-8.0)/(302-8.0) = 14%Or = 100 * (5.92-6.67)/(1.48-6.67) = 14%.

Saturating oxygen at RT and pressure is ~8.5 mg/L => 8.5 mg/L x 14% = 1.2 mg/L





Alcohol by Volume

- Current accurate methods are expensive:
 - FT-IR based systems
 - Gas chromatography
 - Ebulliometry
 - Distillation followed by titration or other
- Inaccurate methods
 - Difference in RI or SG
 - Bioassay

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ABV assay

- Ethanol in the sample volatilizes into the reaction chamber
- Diffuses into and reacts with the Oxidant underneath.

- Non volatile components not detected
- Accurate, simple titration for wine, beer, spirits
- 4-24 hour incubation





The ABV Reaction

ABV: Quick titration, and done

 $Cr_2O_7^{2-} + 14H^+ + 6I^- \rightarrow 2Cr^{3+} + 3I_2 + 7H_2O$

Convert leftover dichromate to iodine (Developer)

 $2 S_2 O_3^{2-} + I_2 \rightarrow S_4 O_6^{2-} + 2I^{-}$

Titrate the iodine with thiosulfate (ABV Titrant)

- Accurate to within 0.3% ABV
 for wines and beers; 0.5% in spirits
- Cost per test ~ \$3.00





SENTIA[™] Wine Analyzer

- \rightarrow Free SO₂, Malic Acid, RS results in less than a minute
- \rightarrow Handheld size, 1 drop sample
- \rightarrow Upload results and update system via <u>Wi-Fi</u>
- \rightarrow Touchscreen display easy and intuitive
- \rightarrow Over 100 tests on a single charge
- → Reproducible results, comparable to other methods
- \rightarrow No calibration needed
- \rightarrow Only \$3.50 per test





Sentia Free SO₂ compared with Vinmetrica

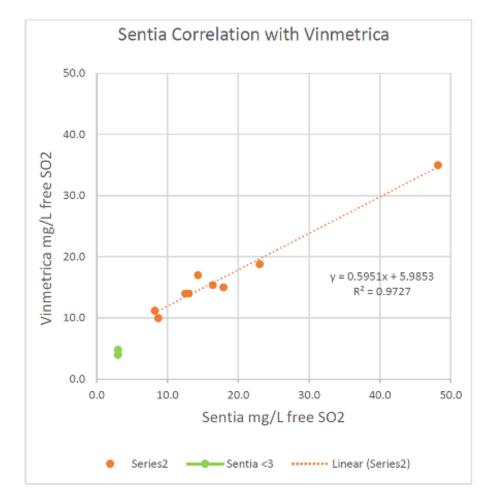
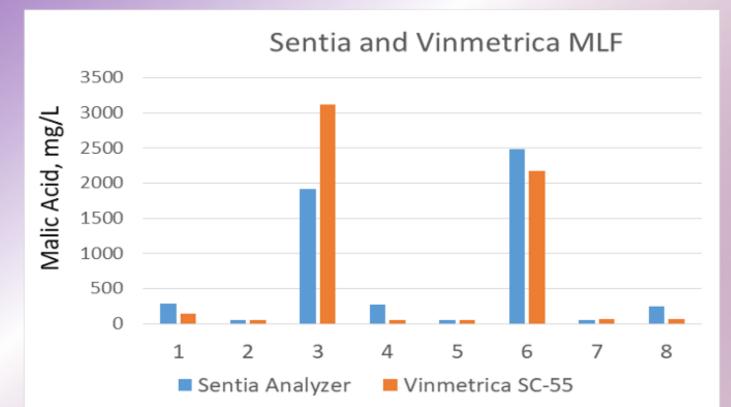


Figure 1. Correlation of Sentia and Vinmetrica results on the wines listed in Table 1.

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Sentia Correlation with Vinmetrica: Malic Acid



Eight wine samples (2 white, 6 red) were analyzed on the <u>Sentia</u> Analyzer and on the Vinmetrica SC-55. Samples 2, 5, and 7 were just at, or below, minimum detectable concentration (50 mg/L) by both methods.

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Summary

- Vinmetrica: Fast, inexpensive and accurate tests
- -Service and Support
- -Wine analysis services also
- Control sulfite and acidity with quick feedback
- Sentia analyzer for SO₂, MLF, RS
- -Rapid, simple, test-in-place
- TA, VA in development

