Iowa Wine Lab Comparison Project

Summer 2022

Results compiled by the Midwest Grape and Wine Industry Institute

Summary

Participating Iowa wineries were provided bottles of the same commercially available wine with identifying information removed. The same wine was also sent to an external lab for analysis. Participants were given a list of chemical parameters to measure, but instructed to skip any that were not within their lab's capacity. Results were reported anonymously using Qualtrics.

Results were collected between June 22, 2022 and July 29, 2022.

Complete results for each parameter are included below, along with brief discussion for each.

Some results of note include:

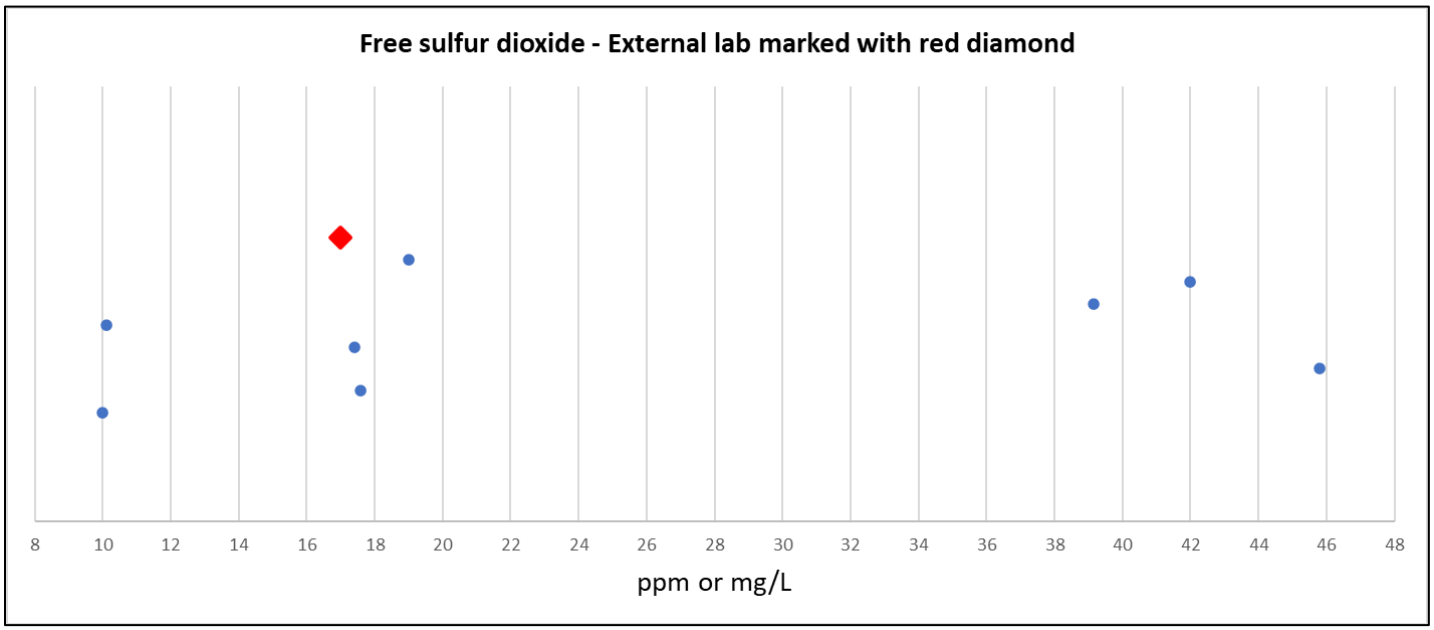
- All of the participating wineries reported results for free sulfur dioxide, pH, and titratable acidity.
- None of the participating wineries reported results for acetic acid / volatile acidity (VA).
- Alcohol results show good agreement among labs.
- pH results showed a surprisingly large discrepancy between labs.
- Free sulfur dioxide (FSO_2) results show a wide range of values, mostly due to two main categories of measurement methods.
- These results provide further context for the findings of the Survey of Iowa Wine Quality, reported in January 2021, which indicated substantial numbers of commercially available Iowa wines with low FSO_2 levels and sensory issues related to oxidation and VA.
- Extra attention is warranted for proper calibration, the choice of appropriate equipment for the application, and reporting with the correct units.

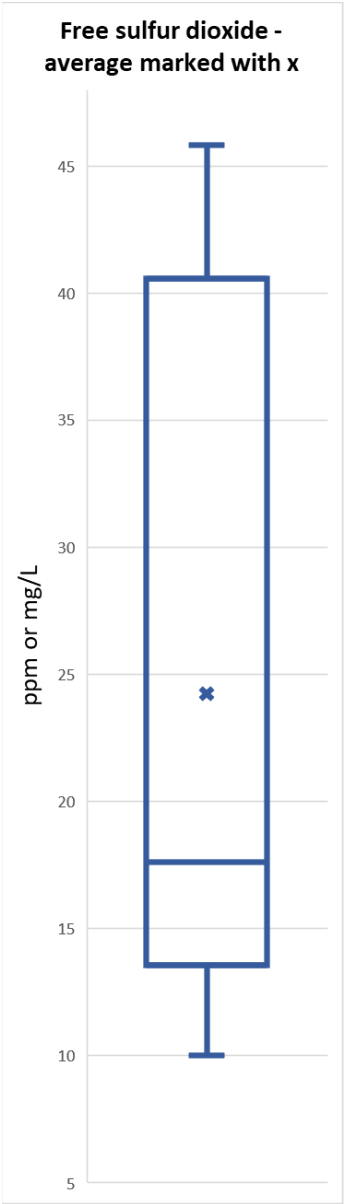
The MGWII is grateful for the participants who volunteered their time for this project.
We welcome any feedback to improve this program, or any of our other services and programs. If there is continued interest, a similar program may be offered again in the future and any suggestions for improvement would be appreciated.
MGWII staff are always available by phone or by emailing wine@iastate.edu with questions and concerns related to lab analysis or general winemaking.

If you did not participate in 2022 but would like to in the future, please let us know!

Looking forward, we plan to continue to focus on areas related to the fundamentals of wine quality. Based on these results, we will likely continue to emphasize the importance of accurately monitoring sulfur dioxide and VA in wines as two important wine quality parameters.

Free sulfur dioxide





Free sulfur dioxide		
Method	**Result**	**Unit**
Aeration/Oxidation (A/O)	10	ppm or mg/L
Aeration/Oxidation (A/O)	18	ppm or mg/L
Titrator	46	ppm or mg/L
Aeration/Oxidation (A/O)	17	ppm or mg/L
Aeration/Oxidation (A/O)	10	ppm or mg/L
Vinmetrica	39	ppm or mg/L
Vinmetrica Titration	42	ppm or mg/L
Aeration/Oxidation (A/O)	19	ppm or mg/L
External lab	17	ppm or mg/L
Average	24	
Standard deviation	14	

Free sulfur dioxide discussion

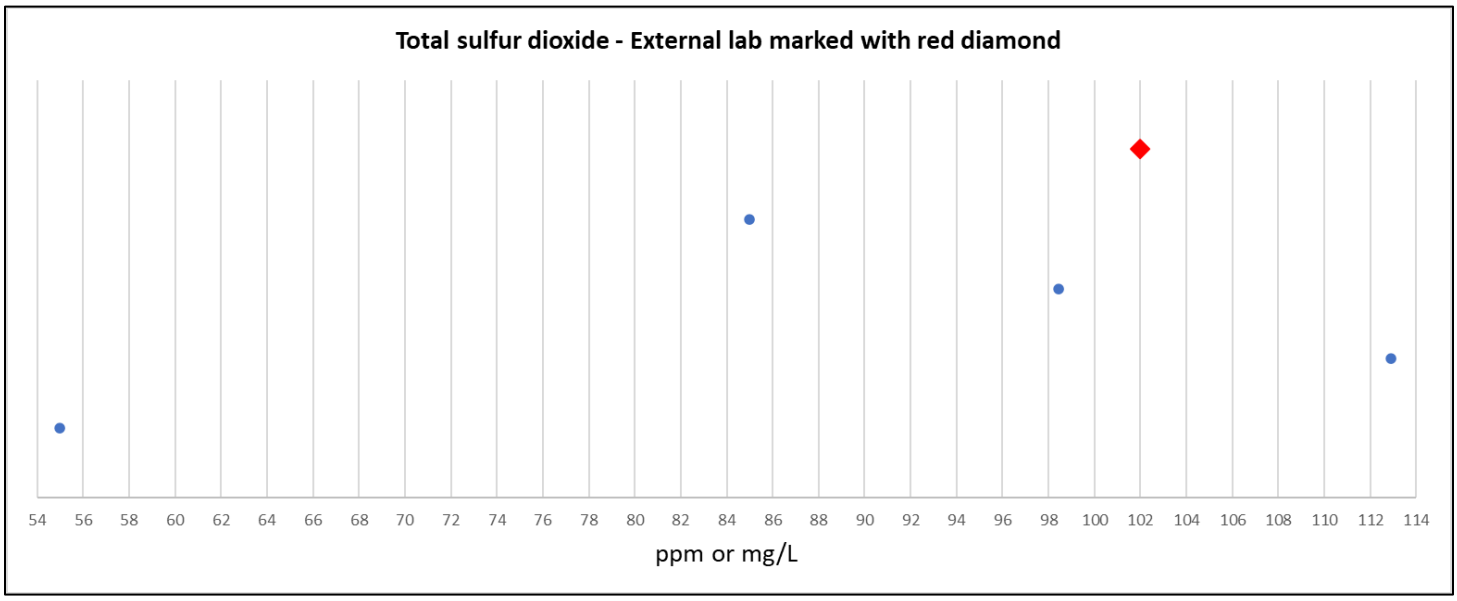
All participating wineries reported free sulfur dioxide.

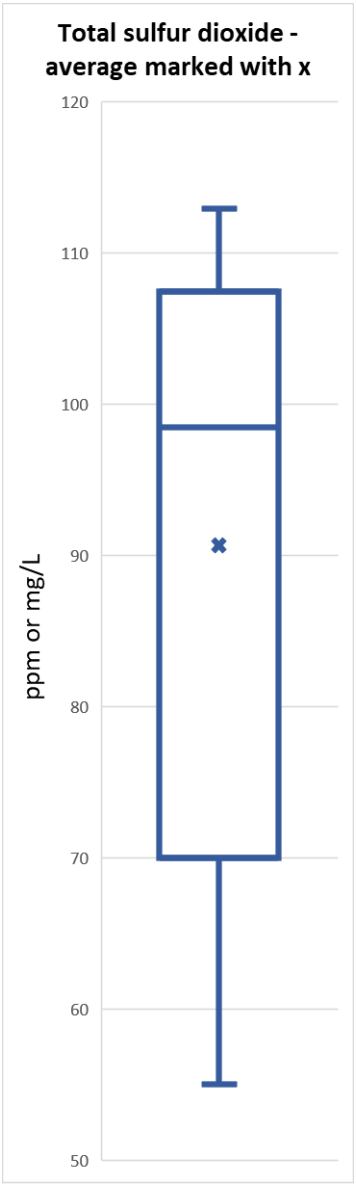
Labs used one of two basic categories of methods to measure free sulfur dioxide: aeration/oxidation (A/O) or a titration-based method. In the reported results, there is a clear separation between values obtained with one method vs. the other.

For comparison, the result reported by the external lab is grouped at the lower end of the range, along with the values reported by labs using A/O.

It is likely that labs using a titration-based method may overestimate the free sulfur dioxide level in their wines, which can have consequences such as higher risk of oxidation and microbial instability.

Total sulfur dioxide





Total sulfur dioxide		
Method	**Result**	**Unit**
Aeration/Oxidation (A/O)	55	ppm or mg/L
Titrator	113	ppm or mg/L
Vinmetrica	98	ppm or mg/L
Aeration/Oxidation (A/O)	85	ppm or mg/L
External lab	102	ppm or mg/L
Average	91	
Standard deviation	22	

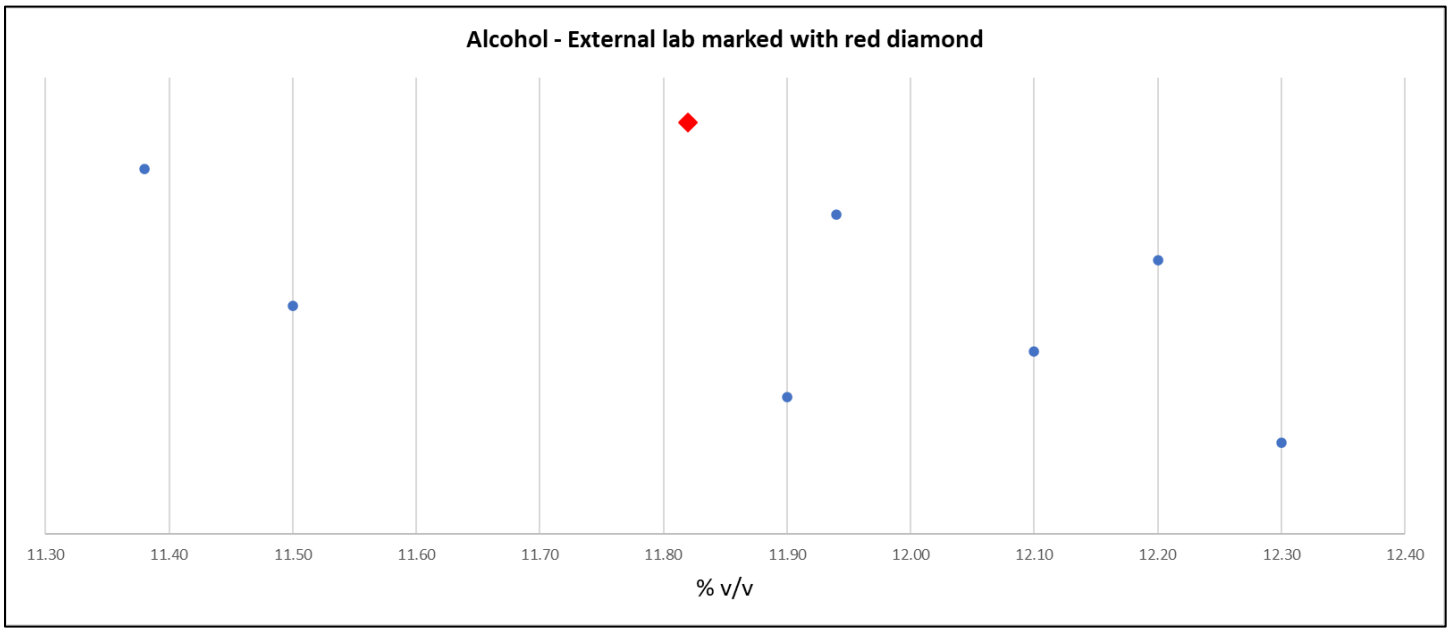
Total sulfur dioxide discussion

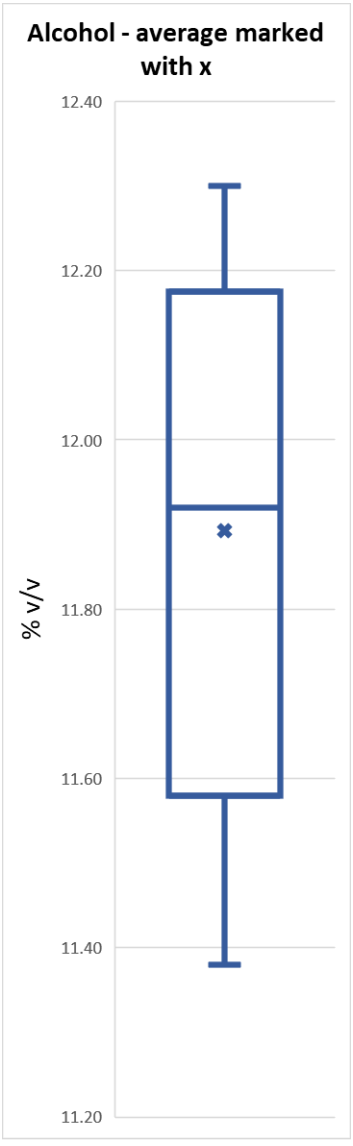
Fewer labs reported results for total sulfur dioxide than for free sulfur dioxide. Results ranged from 55 ppm to 113 ppm, with the result from the external lab (102 ppm) falling toward the higher end of that range.

Because total sulfur dioxide is less directly involved in protecting wine from oxidation and microbial spoilage, measuring it may be a lower priority for winery labs.

However, if winery labs underestimate the level of total sulfur dioxide in their wines, they may be at risk of exceeding the legal limit of 350 ppm set by the TTB (27 CFR § 4.22).

Alcohol / Ethanol





Alcohol / Ethanol		
Method	**Result**	**Unit**
Ebulliometer	12.30	% v/v
Ebulliometer	11.90	% v/v
Ebulliometer	12.10	% v/v
Ebulliometer	11.50	% v/v
Ebulliometer	12.20	% v/v
NIR	11.94	% v/v
HPLC	11.38	% v/v
External lab	11.82	% v/v
Average	11.89	
Standard deviation	0.32	

Alcohol / Ethanol discussion

The reported results for alcohol showed good agreement, with a total range of 11.38% to 12.30%, a difference of 0.92 percentage points.

The majority of wineries reported using ebulliometry to measure alcohol, and the results generated with that method do not show a clear separation from results obtained using other methods.

Acetic acid / Volatile acidity

None of the participating wineries reported results for acetic acid or volatile acidity (VA).

VA is a major quality parameter for wine, and the TTB has set legal limits in wine (1.4 g/L for red wine and 1.2 g/L for white wine).

VA is also a "red flag" compound for other quality issues in wine, including microbial spoilage and oxidation.

For more information, please see the Wine Faults Series: Volatile Acidity fact sheet at https://store.extension.iastate.edu/product/Wine-Faults-Series

VOLATILE ACIDITY

The Fault

Volatile acidity (VA) is defined as the total of steam distillable volatile acids in a wine, which is comprised almost exclusively of acetic acid (vinegar). Volatile acids contributing to a lesser extent are lactic, formic, butric, propionic, sorbic, carbonic, and sulfurous. Small amounts of acetic acid (0.2-0.4 grams per liter) are produced naturally through yeast metabolism during fermentation, and low levels can enhance a wine. An increased level of VA usually indicates spoilage in wine and is regarded as a fault at or near the sensory threshold of 0.7 grams per liter. The United States Alcohol and Tobacco Tax and Trade Bureau (TTB) sets the legal limits for VA at 1.2 grams per liter for white wines and 1.4 grams per liter for red wines. These limits determine the point that wine is considered spoiled to vinegar and not legal for sale.

Cause and Effect

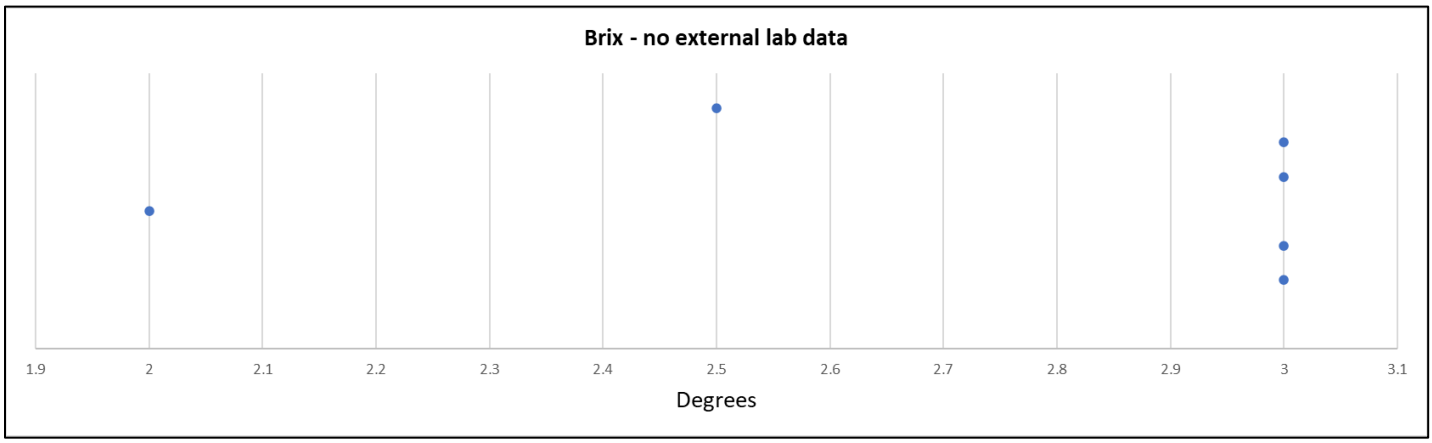
Damaged fruit coming in at harvest is the first instance where microbial spoilage and high VA can be an issue. In wines, acetic acid bacteria (AAB), lactic acid bacteria (LAB), and several species of non-saccharomyces yeast, *Hanseniaspora, Zygosaccharomyces,* and *Brettanomyces*, as well as the film-forming Pichia, all produce acetic acid. AAB are obligate anaerobes, meaning they require oxygen to grow. They convert glucose and ethanol to acetic acid and because they require oxygen to grow, the fault is often coupled with oxidation. LAB utilize glucose and pentoses to produce acetic acid and in some cases through metabolism of citric acid. Many of these yeast and AAB also produce another fault compound, ethyl acetate. When ethyl acetate is present, the VA is more noticeable. More information can be found on oxidation and ethy acetate in Iowa State University Extension and Outreach publications FS0040h – Oxidation, and FS0040c – Ethyl Acetate at store.extension.iastate.edu.

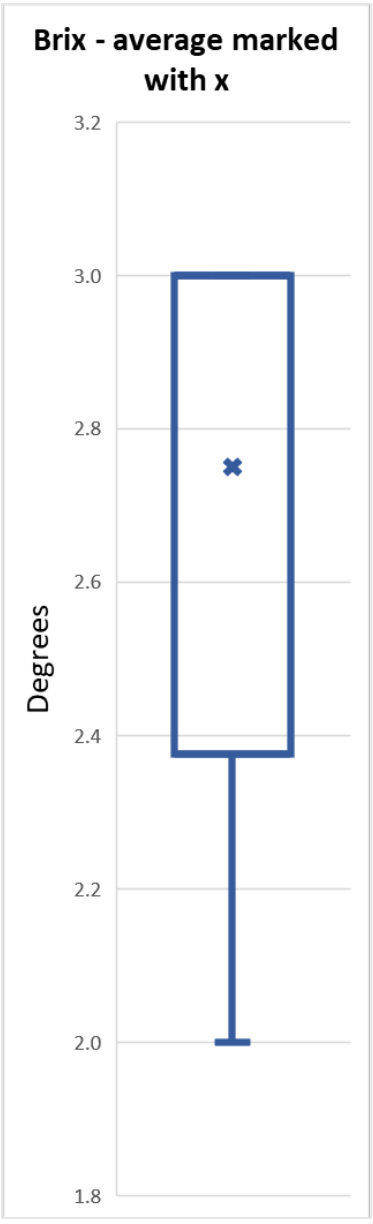
Prevention

Sound winemaking practices can go a long way in preventing high VA, including the proper use of sulfur dioxide (SO_2) and minimizing oxidation. Some of these practices include:

- Stringent cleaning and sanitation protocols for equipment and vessels.
- Use of healthy, clean fruit to reduce the number of microbes entering the winery.
- SO_2 added at crush (30-50 milligrams per liter for clean fruit) to reduce microbial populations.
- Keep tanks and vessels full, to minimize headspace.
- Vessel headspace regularly blanketed with gas (carbon dioxide or argon).
- Checking gauges, lids, and gaskets often on variable capacity tanks.
- Using SO_2 effectively to maintain 0.8 milligrams per liter molecular, based on wine pH.

Brix





Brix		
Method	**Result**	**Unit**
Hydrometer	3.0	Degrees Brix
Hydrometer	3.0	Degrees Brix
Hydrometer	2.0	Degrees Brix
Hand-held density meter (e.g. DMA)	3.0	Degrees Brix
Hydrometer	3.0	Degrees Brix
Hand-held density meter (e.g. DMA)	2.5	Degrees Brix
Average	2.8	
Standard deviation	0.4	

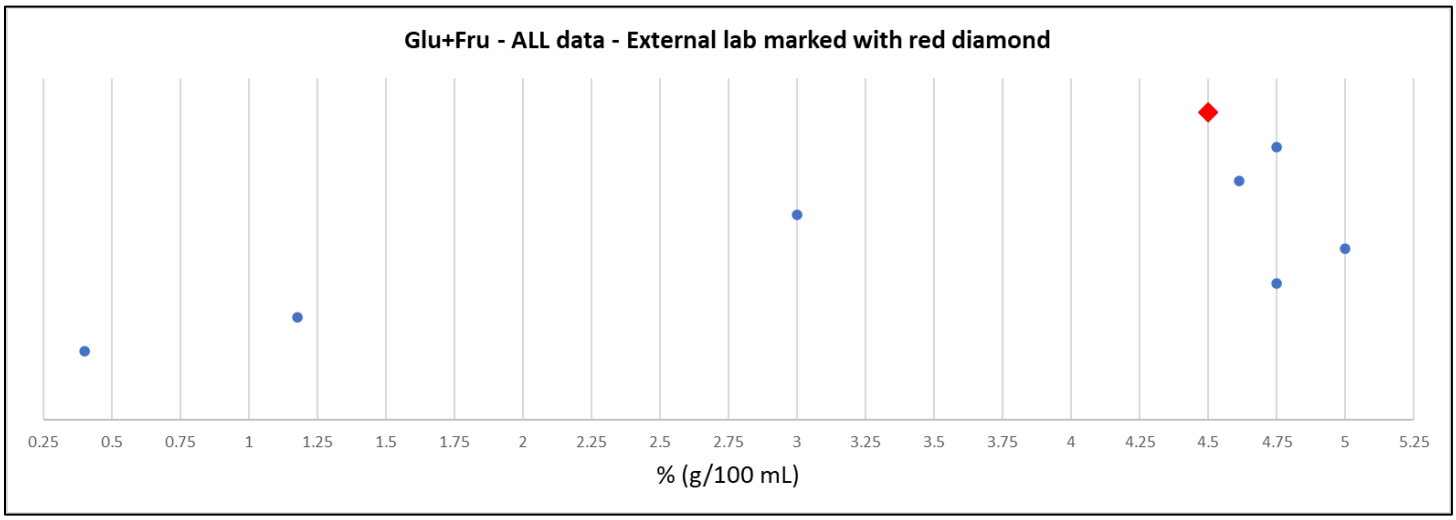
Brix discussion

Brix measurements ranged from 2 to 3 degrees brix.

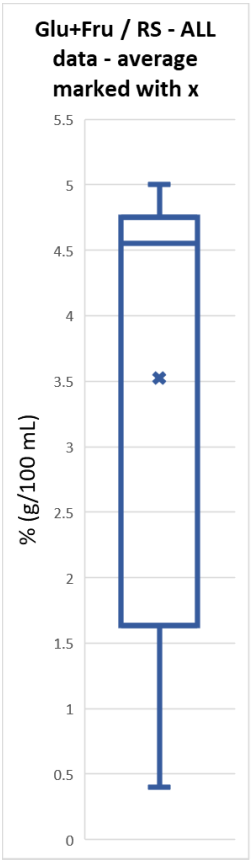
Brix is most useful for monitoring grape ripening and tracking the progress of fermentation, so exact values may not be crucial. However, accurate brix measurements are particularly useful for detecting dryness at the end of fermentation.

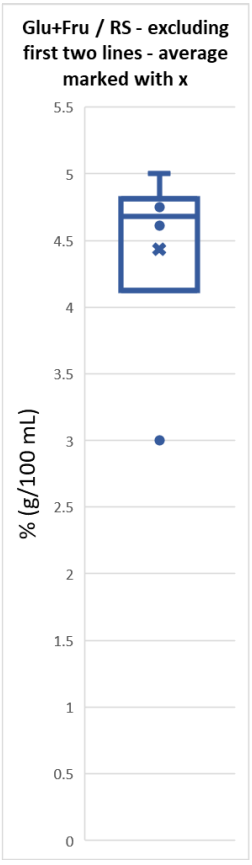
Not all wineries reported brix results, possibly due to volume limitations of the 750 mL wine bottle.

Glucose + Fructose / Residual sugar



Glu+Fru / RS				
Method	**Result**	**Unit**	**Notes**	
Clinitest (or similar)	0.4	% (g/100 mL)	Off by one order of magnitude; could be a reporting error using the wrong units	
Estimate Hydrometer + Refractometer	1.176	% (g/100 mL)	Reported in g/L, converted to %. Note that ethanol interferes with refractometer reading.	
Keto-Diastix + gut feeling	4.75	% (g/100 mL)		
Clinitest (or similar)	5	% (g/100 mL)		
Clinitest (or similar)	3	% (g/100 mL)		
Enzymatic	4.612	% (g/100 mL)		
HPLC	4.751	% (g/100 mL)		
External lab	4.5	% (g/100 mL)		
Average	3.52		Average excluding first two results	4.44
Standard deviation	1.81		Standard deviation excluding first two results	0.72





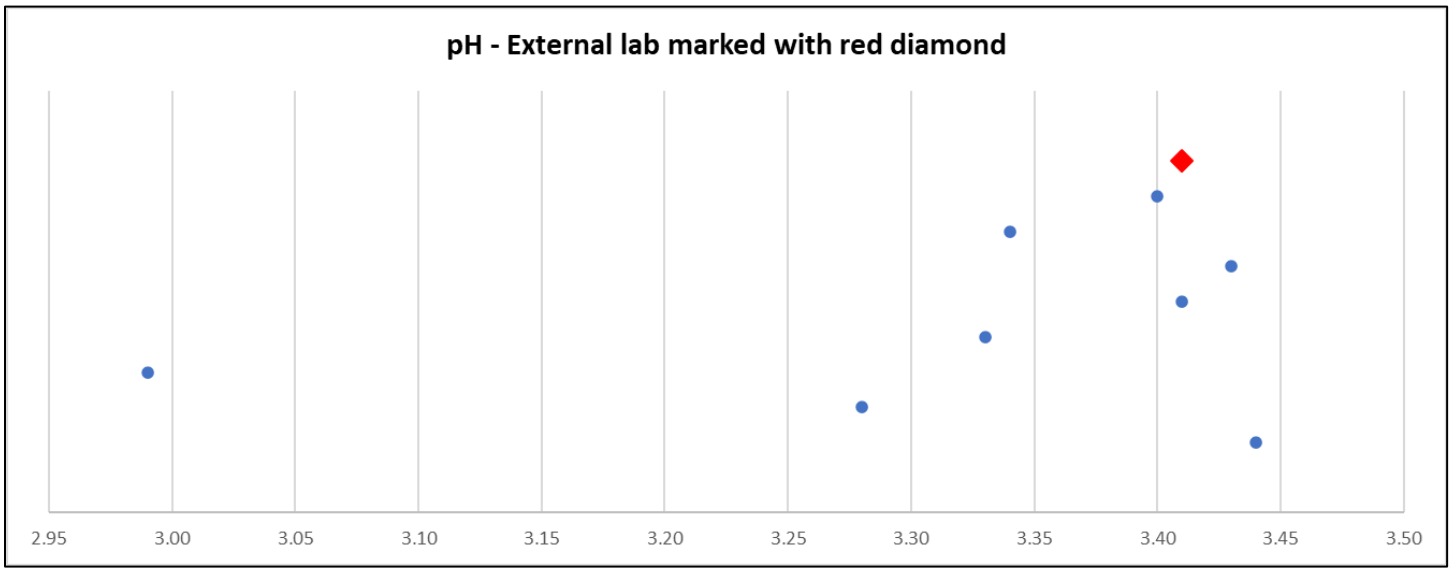
Glucose + Fructose / Residual sugar discussion

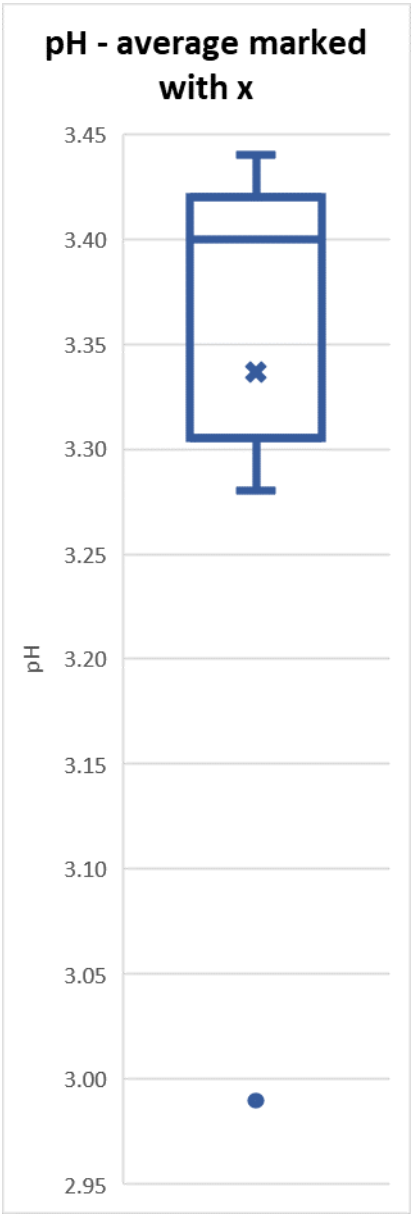
Note that the first two results appear to be outliers, for two different reasons. The first line, with a value of 0.4% residual sugar (RS), is likely a reporting or calculation error representing a shift by a factor of 10. The second line, with a value of 1.176% RS, was reported as 11.76 g/L and converted to % for comparison with the other values. The listed method includes refractometry, which cannot be used to measure sugar once ethanol is present in the sample due to the refractive index of ethanol.

Therefore, averages and box plots are presented both with and without the first two values.

Excluding the first two values, the RS values range from 3% to 5%, with an average of 4.44% showing good agreement with the external lab's reported value of 4.5%.

pH





pH	
Method	**Result**
pH probe and meter	3.44
pH probe and meter	3.28
pH probe and meter	2.99
pH probe and meter	3.33
pH probe and meter	3.41
pH probe and meter	3.43
pH probe and meter	3.34
pH probe and meter	3.40
External lab	3.41
Average	3.34
Standard deviation	0.14

pH discussion

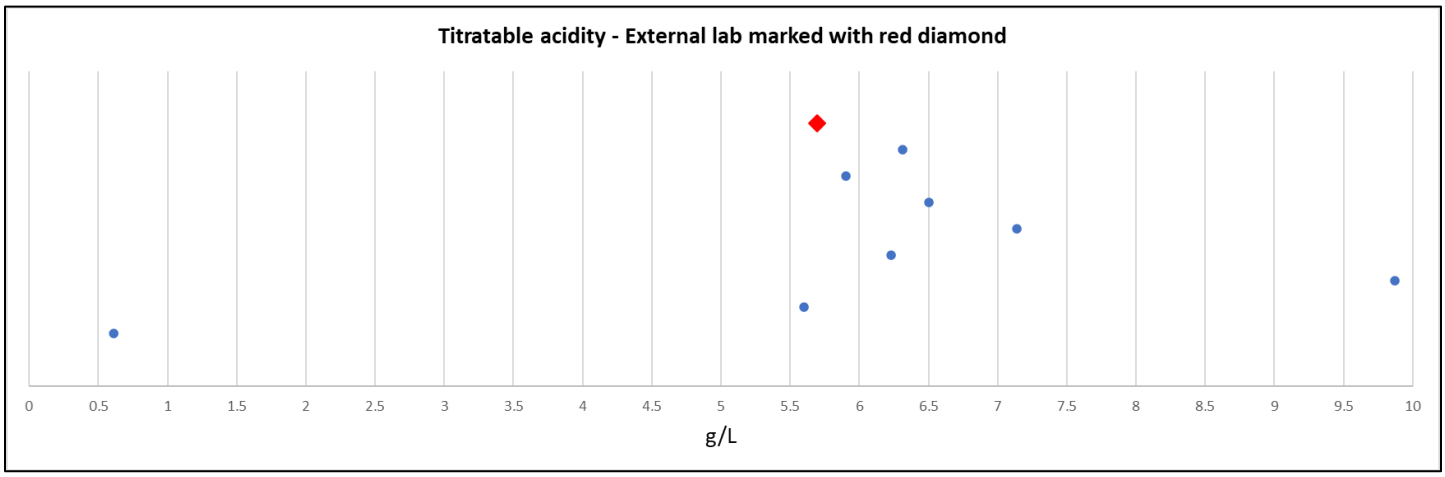
All participating wineries submitted results for pH.

The range of pH measurements was surprisingly wide, with the lowest reported as 2.99 and the highest reported as 3.44.

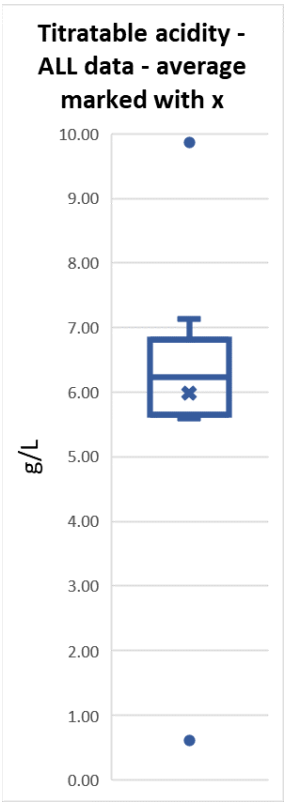
Accurate pH measurement has consequences for wine's microbial stability and the amount of free sulfur dioxide required to reach the protective level of 0.8 ppm molecular SO_2.

pH probes must be properly cleaned and stored, and probes and meters require calibration before each day of use.

Titratable acidity



Titratable acidity				
Method	**Result**	**Unit**	**Notes**	
Manual titration	0.61	g/L	Off by one order of magnitude; could be a reporting error using the wrong units	
Manual titration	5.60	g/L		
Autotitrator	9.87	g/L		
Manual titration	6.23	g/L		
Manual titration	7.14	g/L		
Manual titration	6.50	g/L		
Manual titration	5.90	g/L		
Autotitrator	6.31	g/L		
External lab	5.70	g/L		
Average	5.98		Average excluding first result	6.66
Standard deviation	2.40		Standard deviation excluding first result	1.39



Titratable acidity discussion

All participating wineries submitted results for titratable acidity (TA).

The value shown in the first line, 0.61 g/L, is an outlier that may be due to a reporting error by a factor of 10. Averages are calculated both with and without this value.

When the first value is excluded, reported values still range from 5.60 g/L to 9.87 g/L. When 9.87 g/L is also excluded as an outlier at the high end, the range becomes 5.60 g/L to 7.14 g/L, representing a difference of 1.54 g/L.

Malic acid and **Lactic acid**

Only one winery reported results for malic acid and lactic acid.
Using paper chromatography, they reported the presence of both malic and lactic acids.

The external lab also detected both acids, with a value of 1.17 g/L for L-malic acid and a value of 1.58 g/L L-lactic acid.

Sorbic acid
Dissolved carbon dioxide
Other (blank options)

No wineries reported results for these parameters.