

Wine Analysis for Small Scale Wineries

How to set up an effective lab on a small budget

by *Jean Jacobson* (*/search/?q=jean%20jacobson&sort=document-date*)

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There are no secret formulas, lists or guidelines to go by as to what wine analysis you should or could do according to the size of winery you have. The important matters to consider are the analytical results you need to make your wine, the volume of tests that will be performed, your ability and desire to take on the task of performing the analysis and, of course, the budget.

For hundreds of years the only instrument required to make good wine was a good palate. That still holds true today, but it is wise to remember that during those days wineries expected to have a certain amount of "ruined" wine every year. With the cost of farming and winemaking, those gallons of "ruined" wine can make an impact on a small winery. A good palate with the addition of wine analysis is an awesome duo. Wine analysis can detect potential problems or prevent problems from developing. In this day and age wine analysis is indispensable for the production of premium wines.

The more analytical data you can generate, the better picture you will have of the stage and status of your wine's life cycle. The "how and where" to obtain the all important data is the heart of this article. Small wineries (500 cases and under) with a hardy budget may opt to take samples to a professional laboratory on a regular basis, while those wineries on a tight budget will need to be more creative. Wineries approaching a 1,000-case production will be generating a significant volume of analysis and will benefit from as

much in-house analysis as possible, not only from an economic point but also to have prompt access to the resulting data.

Analysis performed at a professional lab is not inexpensive, but the results are as accurate (close to the "real" value) and precise (repeatable) as possible. This data you can hang your hat on. More creative methods can save you money, but, in turn, the accuracy is diminished and in many cases you will get a "ball-park" value to work with. Taking on performing the wine analysis yourself can save you money in the long run and give you accurate results if you put the time into learning the methods from top to bottom and are able to maintain the accuracy level.

The Right Analysis for the job

First things first: get out a pad and pencil and list the varieties you are producing. For each of those varieties, what method of winemaking will you use? Is it stainless steel Chardonnay with primary (alcohol) fermentation and no secondary (malic acid) fermentation; or will it be Cabernet Sauvignon going through primary and secondary fermentation with two to three years' barrel maturation? Will you have large lot sizes or a few barrels per lot?

For each of those steps, list the analysis you will need to make your winemaking decisions including pre-harvest, harvest, fermentation, post-fermentation, maturation, fining and bottle preparation. Figure in the number of lots and the frequency of testing to get the volume of tests that minimally will be performed. Cost each test based on tests performed in a professional laboratory. Estimate an affordable yearly budget for analysis. Now compare the two and determine where the two may or may not meet. The aim is to be able to obtain as much of the analytical data you can with the funds you have available. This is where you may need to be creative.

From my experience in "Guerilla Winemaking" (low to no analysis budget), in addition to tasting everyday, I would suggest the following very basic analysis:

- °Brix of berry field samples to determine sugar levels and harvesting
- °Brix of fermenting must to monitor the primary fermentation and estimate dryness
- L-malic Acid (LMA) levels (if conducting secondary fermentation) to monitor and estimate dryness
- Confirmation of dryness for primary and secondary fermentation via LMA levels and Residual Reducing Sugars (RRS) via enzymatic analysis
- Titratable Acidity (TA) and pH
- Alcohol level
- Sulfur Dioxide (SO₂) levels
- Volatile Acidity (VA)

Albeit minimal, the results can give you a sound basis to work with. Repeating analysis at certain stages is greatly beneficial to note trends that may indicate a potential problem.

Two Common Scenarios

Referencing the above list, let's drill down and look at the different methods, products and equipment available to obtain the data we need, their pros and cons, and costs. Assuming the best results will come from the professional laboratory with much more sophisticated instrumentation, I will indicate the method most appropriate for two user levels:

A = On-premises analysis for 1,000- case winery or less requiring additional laboratory skills resulting in an acceptable level of accuracy

B = Low budget wineries (500 cases or less) requiring limited laboratory skills resulting in more "ball park" or slightly better accuracy.

Wine Analysis for Small Scale Wineries

Analysis	Accuracy	Product	Measurement Type	Price	Notes	Additional Product Cost	Cost per Year
Monitoring Sugar Levels in Field	A & B	Hand-held Refractometer	°Brix	Less than \$50	Can read for both juice and second fermentation. Can only be used on non-fermenting juice.	\$0	\$0
Monitoring Primary Fermentation	A & B	Hydrometer	°Brix	Less than \$50	Requires more elimination of CO ₂ . Proper technique is imperative.	\$0 to \$200	\$0
Monitoring Secondary Fermentation	A	Accurate Quick Test Hydrometer	°Brix	Easy to use	Not as accurate as other methods.	\$0	\$0
Monitoring pH	A	Hand-held pH Refractometer	pH	Easy to use, digital read-out	Not as rugged instrument. A few models may be needed.	\$70	\$1.40
		Hand-held pH Refractometer	pH	Accuracy verified	Requires periodic calibration.	\$70	\$1.40
		Hand-held pH Refractometer	pH	Easy to use and accurate	Requires pH 7 and pH 4 buffer solutions for calibration.	\$100	\$0
Monitoring Specific Gravity	A	Hand-held Hydrometer	Specific Gravity	Accuracy to ± 0.001	Requires periodic maintenance. Can be used only for pH.	\$70	\$1.40
		Hand-held Hydrometer	Specific Gravity	Accuracy to ± 0.001	Requires good laboratory skills. Requires daily calibration. Requires periodic maintenance.	\$100	\$0.50
Monitoring of Sulfite Levels	A	Hand-held Hydrometer	Sulfite	Accuracy to ± 0.05	Requires good laboratory skills. Requires periodic maintenance.	\$100	\$0.50
		Hand-held Hydrometer	Sulfite	Accuracy to ± 0.05	Requires good laboratory skills. Requires periodic maintenance.	\$100	\$0.50
Monitoring of Alcohol Levels	A	Hand-held Hydrometer	Alcohol	Accuracy to ± 0.05	Requires good laboratory skills. Requires periodic maintenance.	\$100	\$0.50
		Hand-held Hydrometer	Alcohol	Accuracy to ± 0.05	Requires good laboratory skills. Requires periodic maintenance.	\$100	\$0.50
Monitoring of Acidity	A	Hand-held Hydrometer	Acidity	Accuracy to ± 0.05	Requires good laboratory skills. Requires periodic maintenance.	\$100	\$0.50
		Hand-held Hydrometer	Acidity	Accuracy to ± 0.05	Requires good laboratory skills. Requires periodic maintenance.	\$100	\$0.50

(/content/File/0709_WineAnalysis_Matrix.pdf) A few weeks before the anticipated harvest date, monitoring the sugar levels in field samples is fast and easy. Using a hand held refractometer can give you instant accurate results in °Brix for juice only. You simply put a drop of juice on the lens, close the cover, point it at a light source and read it. Okay, it is not quite that simple. You may need to calibrate the instrument before the season starts, make sure the lens is always clean and not scratched, and note the temperature of the juice which will directly affect the reading. Calibration usually takes only a few minutes. To be as accurate as possible you need to take the temperature of the juice, take your reading and then make adjustments to your °Brix, reading up or down accordingly.

The good news is you can buy a refractometer with automatic temperature compensation (ATC) built into the instrument. Refractometers are widely used throughout the industry and appropriate for both A and B users.

Pros: Most instruments have a rugged design for outdoor use providing years of service with good care. **Cons:** Must be kept clean and scratchfree and can be used only for non-fermenting juice. The prices range from around \$50 to over \$100.

Monitoring primary fermentation is a must (no pun intended). Having a controlled fermentation is paramount to developing the desired aroma and taste profiles you are looking for as well as preventing development of undesirable components. The traditional methods of monitoring fermentation are the soluble solids and density methods using a hydrometer gauged to °Brix, °Baumé or °Oechsle.

The United States uses mainly °Brix which is a soluble solids method and temperature dependent. Juice is put into a cylinder and shaken to release the CO₂; the hydrometer is slowly lowered into the cylinder and the reading taken. The temperature is also taken and adjustments made to the reading. There are no solutions or calibrations, and you can buy hydrometers with an integral thermometer.

I would suggest using two ranges of hydrometers: one from 0 to 35°Brix to measure the entire scale and a second from 0.5 to -0.5 for better accuracy at the critical lower scale. A °Brix reading in the negative range over three consecutive days is a good indication of dryness.

Hydrometers are widely used throughout the industry and appropriate for both A and B users. **Pros:** With proper care the hydrometers can be used year after year providing accurate results. **Cons:** Samples require elimination of CO₂ prior to testing, proper technique is imperative, and hydrometers are glass and will break requiring a back-up set especially during harvest. The cost ranges from \$20 to \$30 per hydrometer--but if you want a certified hydrometer with papers, it will cost you more.



Monitoring secondary fermentation is a bit more difficult. There are several choices for the B user to establish a baseline and monitor the progression of malic acid fermentation. The **Accuvin Quick Tests™**, **MegaQuant™** meter and the **Merck RQflex™** 10 meter should be considered.

The Accuvin Quick Tests test strips are a very economical way to monitor the fermentation trends at around \$3 a test.

The Accuvin Quick Tests test strips rely on color matching a test strip to an enclosed scale. This is an easy and simple method to use but does rely on timing and your ability to discern depths of color. The accuracy of the Accuvin Quick Test in the lower range of 30 mg/L is ± 10 mg/L (33 percent). **Pros:** Easy to use if you follow directions. **Cons:** Less than optimum results.



Next in line for our adventurous B users is the Merck RQflex 10 Reflectometer. Remission photometry is the method this meter centers on. Keeping it simple by utilizing a test strip; the RQflex 10 measures the light emitted from the reactive test strip and converts the intensity of the light into a concentration.

The meter's price is in the \$750 range plus the cost of test strips. Test strips are supplied in quantities of 50 and cost about \$2.20 each. Accuracy is approximately ± 10 percent

for the RQflex 10.

Pros: Fairly easy to use digital read-out; for an additional cost it can be interfaced to a PC, and the accuracy is not bad.

Cons: Sample preparation may require dilutions and/or de-colorization; it's not a rugged instrument; a bar code strip is used to prepare the meter for testing; and the meter may require periodic calibration--something to remember if the program is cleared somehow, and you need to run tests and can't find the strip or it is damaged.



The MegaQuant colorimeter and test kit is a step-up from the strips. The cost per test is approximately \$3.60 after an initial investment of \$700 or more for the meter. The MegaQuant method is based on the difference of light absorption due to colorimetric changes of a test sample.

Using the MegaQuant meter and test kit is more involved requiring several steps, possible dilutions, precise measurements and calculations. The test reactions take place in a small test tube which is inserted into the meter for the absorption readings. The accuracy of the MegaQuant system can be a difference of .01 to .02 absorbance units

according to the product information. Just for comparison, an enzymatic assay via spectrophotometer usually has an absorption difference of .003. The MegaQuant is appropriate for A and very motivated B users.

Pros: Reagents are in tablet form and are stable for two years, the meter is portable and runs on batteries, and results are fairly accurate. **Cons:** Requires good laboratory skills and additional laboratory equipment such as a micro-pipettor, volumetric flasks, etc.

The A users should consider performing enzymatic analysis utilizing a visible spectrophotometer, which sells for around \$2,000. Spectrophotometry is a very accurate and approved method for determination of LMA via light absorbance. This instrument provides the 340 nm spectral range needed for this test. Enzymatic kits are purchased containing enzymes and buffers that are required to isolate the analyte for analysis.

The cost per test is dependent on the number of tests run due to the utilization of the chemicals; the more tests performed the less waste, the lower the price. An estimate of cost per test would be \$2.25 for heavy users to \$5 or \$6 for infrequent users. Accuracy is very good with a difference of .003 absorbance units.

Pros: Provides accurate results with a lower cost per test for higher volume users. **Cons:** High level of laboratory skill is required to perform the assay; and precise measuring equipment is required such as a micro-pipettor, volumetric flasks, etc. (For more information on spectrophotometers, reference the July 2008 issue of *Wine Business Monthly* "Choosing the Best Spectrophotometer for Your Winery.")

Confirmation of dryness for both primary and secondary fermentation requires testing the RRS and LMA. Accurate measurements of RRS and LMA are critical to insure the wine has completed the fermentation level you want to achieve. B level users should take a sample to a professional laboratory for analysis. The A users can continue to utilize a

spectrophotometer and enzymatic analysis for both LMA and RRS.

TA and pH are essential analyses during the fermentation process, at the conclusion of fermentation and throughout maturation. The B level user can utilize the Accuvin Quick Tests™ or the RQflex 10 pH and TA test strips (see above for method description). Accuvin Quick Tests strips sell for about \$2.00 each, and the pH scale reads in 0.1 pH units. The strips for the RQflex 10 will cost \$1.40 to \$1.60 each.

Another option for the B user is the digital **Hanna pH/ATC Portable Meter** having a cost around \$100. **Pros:** Easy to use and calibrate, accurate to ± 0.15 pH units, waterproof and with proper care will provide years of service. **Cons:** Requires pH 4 and pH 7 buffer solutions to calibrate, requires periodic electrode replacement and can be used only for pH.

Accurate measurements of TA and pH should be obtained from a professional laboratory periodically when using the above methods.



To measure TA and pH for A users will require a simple bench top style pH meter similar to the **Orion 2-Star pH Meter**, a titration assembly set-up, glassware, a stirrer and stir bar, and chemicals. Total set-up would be around \$700.

Pros: Accurate results for both TA and pH; and with proper care this set-up can be used year after year providing accurate results. **Cons:** Requires a higher level of laboratory skills, requires daily calibration, hand calculations and periodic electrode replacement.

Determination of alcohol levels at mid-fermentation to calculate potential alcohol, cessation of fermentation, blending and for labeling are the critical points where very accurate alcohol measurements are needed. Measurement of alcohol during a stable

maturation process does not change all that much unless there is dilution. The RQflex 10 provides a \$2.60 test strip for alcohol estimations that would be beneficial for the B user. Critical measurements would need to be performed at a professional laboratory.



New on the market is **Hanna Instruments HI 83540 Alcohol Meter** with integral stirrer. The meter utilizes an alcohol probe to measure the alcohol level while stirring the sample. Results are digital, and the meter is supplied with reagents for 50 tests, syringes, beaker and stir bar for around \$775. The accuracy stated is within the **Alcohol Tobacco, Tax and Trade Bureau** (TTB) allowable limits of 1.5 percent for 14 percent and under and 1 percent for above 14 percent.

The A user and possibly B users can employ ebulliometry as a method to determine alcohol. This method has been used for many years and was approved by the TTB. Ebulliometry is based on the Churchward technique or the depression of the boiling point in a water and alcohol mixture. The higher the alcohol level the lower the boiling point. The Ebulliometer uses an alcohol burner to heat a metal chamber containing either the wine or the water sample and brought to a boil. Each boiling point temperature is read and compared; a scale is provided to interpret the differences in boiling points.

Ebulliometry is fairly accurate up to around 16 percent v/v alcohol with a \pm of 0.5 percent. An Ebulliometer will cost just under \$1,000.

Pros: Inexpensive method for accurate alcohol determination with proper instruction, fairly easy to use and with proper care will provide years of service. **Cons:** There are several areas that can pose problems resulting in inaccurate readings, the alcohol burner emits fumes and should be vented, and good laboratory practice and safety are important.

There are faster and more accurate methods available for alcohol determination, but the cost of the instrumentation jumps up drastically making it difficult to justify for smaller wineries at this time.

VA analysis on a regular basis is the best tool to alert the winemaker to potential spoilage/contamination problems. Currently the most accurate method of determining VA is using distillation via Cash still. A distillate is produced from heating a treated wine sample (that volatilizes the acid) then passing the steam through a cooling chamber. The distillate is then tested for the amount of acid the volatilization produced.

You will find the Cash still is widely used throughout the industry and appropriate for both A and B users. Using the Cash still is not difficult but does require good laboratory skills and instruction as there are problem areas that can affect your results. The cost of a Cash still set-up will be around \$800. You will also need chemicals and a titration set-up which will cost about

\$100.

Pros: Accurate results, inexpensive after initial investment and with proper care will provide years of service. **Cons:** Stills are made of glass and can be damaged, cracked or broken; and good laboratory practice and safety are important.

Acetic acid is the most volatile acid comprising VA but not the only acid. There is a close correlation between acetic acid results via the spectrophotometer enzymatic analysis and VA up to approximately 0.050 g/100ml (as acetic acid). Enzymatic acetic acid results can be used as a substitute for VA up to that point. Depending on the quantity of samples, the cost of enzymatic kits may prohibit using this method.

Protecting the wine from spoilage organisms during maturation is primarily accomplished by the addition of SO₂. You want to add just the right amount of SO₂ to produce enough free SO₂ (unbound to elements in the wine) to protect the wine without adding too much. To do this you need an accurate pH, the SO₂/pH scale and a good accurate source of SO₂.

SO₂ analysis is probably the most common analysis performed in a winery. B users will be happy to know that Accuvin Quick Tests has a test for free SO₂ as does the RQflex 10. The Accuvin Quick Test uses small vials rather than test strips for this test which costs \$2.73 per test and has a low range and high range color matching chart. Sulfurous acid (as free SO₂) is measured via test strip using the RQflex 10 for \$1.80 a test with a range of 1 to 50 parts per million (ppm). Both methods, as stated above, will give you estimated concentrations.

The Ripper method can give you more accurate results (± 10 percent) than the above but does require a higher level of laboratory skill. The Ripper method is based on oxidation-reduction reactions. A solution of hydrogen sulfate is added to the wine sample to reduce the oxidation of the polyphenols, a starch solution is added to the wine sample as an indicator; then iodine (oxidizing agent) is titrated into the sample oxidizing the free SO₂ creating iodide ions which will turn the starch blue. The amount of iodine used to barely turn the starch blue is used to calculate the free SO₂ present. The total SO₂ can be determined with the same method except just before you add the iodine you add a quantity of sodium hydroxide which releases the bound sulfites.

In addition to glassware you will need chemicals and precise measurement and titration equipment. Titration sets will cost about \$100.

Pros: Fast, inexpensive, fairly accurate and not too difficult. **Cons:** Must be able to reach the same end points consistently and requires a slightly higher level of laboratory skills.

Hanna Instruments has come out with the HI 84100 Mini Titrator with integral stirrer for the determination of free and total SO₂. The method used is based on the Ripper with a bit better accuracy of ± 5 percent. The titrator includes pre-measured reagent sets, beakers, stir bar and tubes for around \$575. This unit would be appropriate for both A and B users.

Pros: Pre-packaged solutions and reagents, digital read-out of results and better accuracy than manual Ripper method. **Cons:** Cost per test is higher.

The A user may want to determine free SO₂ via their spectrophotometer. This is a fast and fairly accurate method, but again, the quantity of analyses will determine the cost per test.

The most common method to determine free SO₂ is aeration and oxidation (A/O). This method has been approved by TTB for the measurement of free and total SO₂. There are set-ups available on the market to perform the A/O (see vendor information), or you can build your own. Basically you add a small amount of phosphoric acid to a wine sample which helps release the free SO₂. The sample is kept in an ice bath to reduce evaporation and dissociation of bound SO₂. A continuous air flow over the sample carries the volatile free SO₂ with it through a volume of hydrogen peroxide where the SO₂ is oxidized into sulfuric acid. The sample is titrated with sodium hydroxide back to the hydrogen peroxide pH. Color indicators are used to mark the point. The A/O set-up, along with a titration set-up, chemicals and vacuum, can vary in pricing, but I would count on a few hundred dollars.

Pros: Accurate results, inexpensive after initial cost of set-up and with care the set-up can be used year after year. **Cons:** Glassware breaks, it must be able to reach the same end points consistently, requires a slightly higher level of laboratory skills and hand calculations.

Conclusion

For the B users there are methods that can be utilized to help defer laboratory costs. By following the trends of the results from these less expensive methods and taking their wine samples to a professional laboratory at critical points they can keep a handle on the condition of their wine. As money becomes available, they can invest in equipment and instrumentation for

more accurate methods and set up their own laboratory. The A users can take from both sides, utilizing the inexpensive methods in some areas while working towards equipping their laboratories with more advanced instrumentation where they really need accuracy and sample throughput.

Getting the proper instruction and learning how to perform the analysis is paramount in obtaining the best results even from the simplest test. Quality control and checking out your methods from time to time with a professional laboratory will give you confidence in your results allowing you to make the winemaking decisions that will produce the best wine possible. **wbm**

Michael Ramsey, teaching laboratory manager for the Department of Viticulture and Enology, **UC Davis**, has more than 20 years' experience with alcoholic beverages. Ramsey conducts laboratory-based classes geared to the home and smaller scale wineries as well as classes geared for students with more laboratory experience.

"Students range from home winemakers to winery owners," Ramsey said. "There are some students who want to learn to do their own analysis to save money, but the majority of students are interested in the methods and learning the techniques."

Even the simple tests such as using a chemistry strip to measure pH and other methods that can be performed outside a professional laboratory will have step-by-step instructions to follow to obtain the best results from that method. Ramsey and I agree that doing your own analysis without following or obtaining proper instruction is a recipe for disaster.

In my book, *Introduction to Wine Laboratory Practices and Procedures*, I have a section with step-by-step procedures to follow for most of the common wet chemistry methods along with troubleshooting. The UC Davis classes Ramsey teaches are one-day events and are held on the weekends. Contact UC Davis at www.extension.ucdavis.edu/wine (<http://www.extension.ucdavis.edu/wine>) for more information. **wbm**

Resources

Accuvin Quick Tests www.accuvin.com (<http://www.accuvin.com>) 707-255-2029

Adams & Chittenden Scientific Glass--Cash Still www.adamschittenden.com (<http://www.adamschittenden.com>) 510-843-5277

Hanna Instruments HI 83540 www.hannainst.com (<http://www.hannainst.com>) 800-426-6287

Megazyme MegaQuant www.vinotecnapa.com (<http://www.vinotecnapa.com>) 707-953-7072

Merck RQflex 10 www.merck.com (<http://www.merck.com>) 908-423-1000

ThermoScientific, Orion 2-Star pH Meter www.thermo.com (<http://www.thermo.com>)

by Jean Jacobson Jean L. Jacobson is a writer and author of the book, *Introduction to Wine Laboratory Practices and Procedures*, published by Springer Publishing, NY, NY. Jacobson was laboratory director for Kendall-Jackson Wine Estates for many years and is currently working in winemaking.

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