

Proficiency testing for wine analysis — so you can be sure...

BY Sue Caloghiris

articipation in a proficiency testing program can be a very powerful quality tool in any well-run laboratory. Below we will discuss a program that will provide both the winemaker and the technician with peace of mind.

What is proficiency testing?

Proficiency testing is the analysis of samples in conjunction with other laboratories testing the same or similar sample types. The process usually involves analysis of the sample for particular compounds, followed by collation and statistical analysis of the results. Proficiency testing allows you to compare your laboratory's analytical results with those of other laboratories who have analysed the same samples at the same time.

What is available to wine industry laboratories?

There are many programs available to the wine industry. Most offer analysis in a range of beverages that include wine and usually offer a limited list of compounds for testing. Others target things like agrochemical residues and are only valuable to those laboratories that have the facilities to test such things. There is one program, however, that is winespecific and is run by wine industry people for the wine industry.



Interwinery Analysis Group

The Interwinery Group is an Australian group that commenced

formally in 1983 after almost 20 years of informal operations. The group is run by a committee of laboratory staff from wineries in many wine regions of Australia and has almost 250 members, including several from wine regions in the U.S., France, South Africa, and New Zealand. This program offers an opportunity to perform 17 analyses. These are:

- Free SO₂
- Total SO₂
- pH
- Titratable acidity
- Volatile acidity
- Malic acid
- Alcoholic strength
- Residual sugar
- Specific gravity
- Citric acid
- Potassium
- Copper
- Iron
- Sodium
- Calcium
- Carbon dioxide
- Turbidity

Note that the ability to analyse all these parameters is **not** required — in fact some members submit results for only two or three analytes.

How does this program work?

Each participating laboratory receives one case of samples per year. The wine samples are labelled in pairs marked YY-01-06 A and B, where YY is the year, 01 is the round number and A and B are the sample pair (i.e. the samples for this month's round would be labelled 13-01 A and 13-01 B).

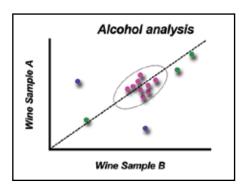
Analysis is performed by all participating laboratories during the same 7-day period and the results are entered via a portal on the Group's website. The results are then collated

and run through a powerful statistical program, which allows complex statistical processes to be interpreted in very simple graphic analysis. This gives the participating laboratories clear information about the quality of their results.

Each analysis is graphed in a similar fashion to the one shown below. The A and B results for each sample are plotted one against the other on the x and y axis. Each pair of results forms a dot on the graph that lies within a certain area.

The statistical information also includes a calculation of the mean or average of all the results. This mean is used to calculate the 95% confidence interval or the ellipse that surrounds the accurate results.

The aim of participation is to get all your laboratory results within this 95% "ring" of confidence (where the red dots lie). The more participants in the program, the tighter this confidence interval should be. The results are also provided in numeric form so that simple comparison to the mean and the average can also be performed.



This graph demonstrates the different areas where results might lie, and their interpretation. A result falling within the circle is ideal, while results falling on the 45 degree line, but not within the circle, indicate a systematic or bias error. Results that fall neither on the 45 degree line nor within the circle are usually random in nature.

● **Ideal** — A result in this area means that you have submitted a result that lies within the 95% confidence circle (or ellipse in



this case) for that analysis. This means that your result is very close to the mean result for the whole group.

● Random errors — A random error often indicates human error, as one result is usually correct and the other erroneous. This could be due to sample preparation error or some sort of oversight during the analysis (i.e., not adding a critical reagent). It is usually difficult to determine what happened after the event and is therefore important to have systems in place that prevent it from happening in the first place.

Systematic or bias errors —

A systematic error indicates that the errors have occurred across multiple analyses and are likely to be errors somewhere in the "system." These errors are the most dangerous because analysis of a duplicate sample will give the same result leading to a sense of false security.

Standards, and more importantly spikes, are useful in determining the source of these problems, which are usually based on errors involving reagents, equipment, method, or training. Spikes are a more useful tool in this instance because sometimes the problem is matrix-related and analysis of an aqueous standard solution will not always isolate the problem.

How do we determine where our results are on the graph?

In order to determine where your result lies in comparison to others, review the data with your submitted results in hand. By using the grid on the graph with your results you can easily determine which dot you are — whether it is in the red, blue, or green region.

(Please note that on the true graphs, the dots are all black. Colors were used in this case to demonstrate and provide interpretative information about the different areas of the graph).

Evaluation of these graphs and the position of your laboratory on them is critical in proficiency testing. Alternatively, you can compare your results to the numeric results also provided.

To neglect this task is like failing to get your results after sitting an exam or having a blood test! The second most important task is to review those analyses that did not provide "good" results and attempt to determine where any deficiencies might lie. A review of the method, some additional standards, duplicates or spikes, and inspection of the equipment and method would be some areas to look at. The group also offers corrective action samples so that you can repeat the analysis after determining and fixing the problem.

Disadvantages of this type of proficiency testing program

While this statistical analysis provides comparative information about the results, it does not provide any information about the absolute accuracy of any one result. Proficiency testing based on accuracy depends on two assumptions.

The first is that one particular laboratory will be accurate in its analysis every time and is therefore classed as a reference laboratory. Some laboratories are more likely to be accurate, such as those with quality accreditation and sophisticated equipment. However, while there are humans involved in the process, there will always be a risk of error.

The second assumption is that the samples submitted to every participant are homogenous. To make sure that this indeed occurs, exhaustive homogeneity testing is performed. The Interwinery Analysis Group has recently achieved ISO guide 17043 Proficiency Testing accreditation.

Samples are taken ex-bottling line and, for the most part, experience few problems with bottle variation. The statistical analysis is somewhat dependent on the sample size; therefore the more participants, the more useful the results.

How much does it cost to participate?

Membership in the Interwinery Analysis Group costs AUD\$350 per year for six rounds. This fee includes samples, results, access to a trouble-shooting forum and seminar presentations, and free seminar attendance to two seminars per year. However these membership fees do not include freight to overseas destinations. The 2013 fees for shipping to the U.S. are AUD\$350, bringing the total cost to AUD\$700 or just over AUD\$100 per round.

Now that you have learned about proficiency testing programs, you need to take advantage of this wonderful support mechanism for wine industry laboratories.

The program offers complete confidentiality of individual laboratory results, while at the same time providing a network of contacts within the industry that can be a valuable resource should you have a problem. Most often, if you have identified a problem with one of your analyses, you can be sure that several other wineries have experienced the same problem at some time.

If you join before end of January 2013, you will be able to participate in five of the six yearly rounds and will also be able to check your current analysis against the results available for round one!

Sue Caloghiris (formerly Sue Weeks wrote 16 Winery Laboratory Quality columns in PWV 2000/2001/2002) has been active in the Australian wine industry for more than 25 years including 14 years at The Australian Wine Research Institute. She now runs Bibber International which offers several services to the wine industry world-wide and is secretariat of the Interwinery Analysis Group. She is CoAuthor of Chemical Analysis of Grapes and Wine: Techniques and Concepts. For more information, please visit www.interwinery.com.