Proficiency testing for wine analysis

By Luke Warner, Chairperson, Interwinery Analysis Group. Email: chairperson@interwinery.com

Luke provides some background on the Interwinery Analysis Group, which provides quality management to enable confidence in winery laboratory results, demonstrates how the group’s statistical reports identify questionable results, and shares some of its findings on recent alcohol analyses.

The Interwinery Analysis Group (IWAG) is a not-for-profit proficiency testing provider that supports wineries and wine testing facilities in Australia and the rest of the world. Run by a volunteer committee of laboratory staff from wineries throughout Australia, IWAG formally commenced in 1983 and this year enters its 30th year of operation.

Currently, IWAG has more than 200 members from every winemaking region in Australia as well as several from New Zealand, France, Israel, Denmark, UK, South Africa and America, making it the world’s largest proficiency testing provider of its type.

Fundamentally, IWAG’s proficiency program is a quality management tool that gives technicians, winemakers and customers a level of confidence in laboratory results. The program involves up to 200 wineries and wine testing laboratories around the world that analyse duplicate wine samples concurrently a total of six times per year. In each round, laboratories are free to test the wine samples for up to 19 wine analytes, meaning IWAG is therefore able to accommodate smaller operations that may not be interested in analysing the full suite of 19 analytes.

Rounds of testing always run for seven consecutive days (Thursday to the following Wednesday) during which time laboratories can analyse the wine samples and submit results via a portal on the IWAG website. About a week following submission, a confidential statistical report is made available to members allowing them to compare their analytical results to those submitted by other laboratories. These reports allow laboratories to identify questionable results, investigate all sources of possible error and immediately initiate corrective action.

Membership of IWAG costs $350 per year which includes samples for six rounds of testing (12 bottles), access to statistical reports, the forum and past seminar presentations, and free attendance to two seminars per year.

Interpreting the Statistical Reports

In the statistical reports, each analysis is graphed in a similar fashion to the one shown in Figure 1 (in fact, this graph was taken from the latest IWAG round of proficiency testing and depicts alcohol). The results for sample A are plotted on the x-axis against the results for sample B on the y-axis. Each pair of results is symbolised as a dot, or a sunflower symbol where more than one lab has identical results for both samples. As can be seen in Figure 1, result pairs can fall within one of three distinct areas: within the ellipse, outside the ellipse but on the 45° line and neither within the ellipse or on the 45° line.

Interpretation of the graph is simple and critical to participation in proficiency testing. It is important to review the graph in conjunction with the results that were submitted to IWAG and the numerical statistical data included with each graph. That way, members are able to determine whether their results fall within the 95% confidence ellipse (blue dot) or not (green and red dots).

Scorpions Wine Spoilage Microbe Diagnostic

Despite best practice methods, microbial contamination can still occur during wine production. However, rapid and early detection of spoilage microbes before they negatively affect wine is now possible through the Scorpions™ detection system available through ETS Laboratories. Allowing winemakers the opportunity to intervene and prevent spoilage, the Scorpions™ system utilises a quantitative PCR method to reliably detect and quantify specific wine spoilage microbes at levels down to 10 cells/ml. Suitable for wineries of all sizes, the Scorpions™ assays have been available in Australia since 2009 and remain the most advanced, real-time, PCR-based analyses for the detection of wine spoilage organisms. The latest version utilises true multiplex capabilities to detect Brettanomyces, Zygosaccharomyces and Saccharomyces in a single analysis. In addition, six different categories of bacteria, including twelve Lactobacillus, five Pediococcus and six Acetic Acid bacteria species, are detected with the new bacteria assay.

Advanced Analytical Australia Pty. Ltd.
11 Julius Avenue, North Ryde NSW 2113
ph.: +61 2 9888 9077
fax.: +61 2 9888 9577
mobile: 0411213191
contact@advancedanalytical.com.au
www.advancedanalytical.com.au
# Ideal Result

Ideal results are symbolised as blue dots or sunflower symbols, which lie within the 95% confidence ellipse for that analysis. Results within this area are close to the group mean.

# Random Errors

A random error often indicates a source of human error with typically one result usually being correct and the other erroneous. This could be due to sample preparation or some sort of oversight during the analysis (e.g., evaporation of ethanol from leaving a sample on the bench for a long period of time). 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 instance.

# Systematic Errors (or Bias in Measurement)

A systematic error indicates that the results differ significantly from the group mean and are most likely due to 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 false sense of security. Standards and, more importantly, spikes are useful in determining the source of these problems which are usually reagent, equipment, method, calibration or training based. 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.

---

**Figure 1.** An example of a graph from a statistical report demonstrating the different areas where results might lie, and their interpretation (note that graphs in statistical reports consist of only blue dots – other colours were used here to demonstrate and provide interpretative information about the different areas of the graph).

---

**ADELAB SCIENTIFIC**

Direct importers of GLASSCHEM distillation systems for the determination of Volatile Acids, Sulphur Dioxide and Alcohol in wines.

Available in single or multiples of each parameter or in different combinations.

**Shown:**

Combined VA, SO₂ and OH in one unit

---

**Residue Analysis**

We specialize in residue analysis and offer a wide range of standard and specialist residues, both at our own ISO 17025 accredited facility and using GCMS and LCMS technology.

We offer a range of services for grapes and wine including the following:

- Phosphorous acid
- Dithiocarbamates
- Phenoxyacetic herbicides (e.g. 2,4-D)
- Glyphosate, Parquat / Diquat
- Scorpion DNA detection for spoilage organisms including Brettanomyces
- ‘Brett’ (taint) - 4-EP & 4-EG
- Smoke taint
- Yeasts & Moulds
- Metals analysis
- Bisphenol A (BPA)

We also test soil, water and air.

Advanced Analytical Australia is an independent contract laboratory offering:

- Chemical, tri-biological and microbiological testing
- ISO/IEC 17025 accredited and OECD GLP recognised by RATA

Modern and emerging technologies

Experienced staff

Reliable customer service

For further information on all our services and pricing, please contact your nearest office.

---

**Adelab Scientific**

Email info@adelab.com.au

ALCOHOL ANALYSES

One of the most important analytes in wine and by far one of the most reported among IWAG members during testing rounds is alcohol. Alcohol analysis is important for many reasons including taxation, labelling requirements and sensory characteristics. The Australian Food Standards Code (Standard 2.7.1) says that in Australia, for alcoholic beverages containing 1.15-6.49%v/v alcohol, the maximum allowable tolerance for the label alcohol is ±0.5%v/v. For alcoholic beverages with more than 6.50%v/v alcohol, the tolerance from the label can be a maximum of ±1.5%v/v. For wine producers exporting to other countries, these tolerances are often a lot tighter – in some cases down to 0.8%v/v for wines containing up to 20%v/v alcohol (Europe).

From the last seven years of IWAG testing (2006 to April 2013), considerable information regarding alcohol analysis has been observed including the adoption of new methods by the industry, trends in methods of analysis and the accuracy of those methods. This article aims to bring to light some of those observations and how a proficiency testing program may be able to assist wineries to measure analytes as accurately as possible to conform to legal and labelling requirements.

For the first IWAG round of 2013 (February), the range of reported results for alcohol from 148 members was 1.68%v/v. Given that range is greater than the maximum allowable tolerance for wine alcohol content in Australia, at least one or more participating members from that round of testing was able to easily identify and rectify any questionable results.

NIR technology, such as the Alcolyzer from Anton Paar, has always been a popular choice for wineries to analyse the alcohol content of their wines. For IWAG members who report results for alcohol, the NIR method of determination has jumped from 42% of total submissions in 2006 to 63% of total submissions in April 2013. As NIR has become an increasingly popular method of determining the alcohol content in wine, the relative error (coefficient of variation) over the years has decreased to reflect this. Conversely, in 2006, 40% of the members who submitted results for alcohol stated their method as being distillation (distillation followed by hydrometry, density meter, refractometry or pycnometry). In April 2013, this number had dropped by more than half to only 17% of total submissions.

NIR technology allows laboratories to measure the alcohol content of their products quickly and accurately, hence the adoption of this method by the industry. Examination of the relative errors of the various methods of alcohol analysis since 2009 showed, unsurprisingly, that NIR delivers the lowest relative error: NIR (1%CV), FTIR (1.2%CV), distillation-hydrometry (1.7%CV), distillation-density meter (1.9%CV), ebulliometer (2.3%CV).

In Australia, it is a requirement for NATA accredited laboratories to participate, as often as practicably possible, in a range of proficiency testing programs. Though not NATA accredited itself, the IWAG committee is working to achieve ISO 17043 Proficiency Testing accreditation. Participation in proficiency testing programs alone does not guarantee improved lab performance. Only by combining regular proficiency testing rounds with a sound lab quality management system can a winery laboratory be confident that it is making correct production decisions.

For further information about IWAG, visit www.interwinery.com.au. Join now and receive a free copy of ‘Microbiological analysis of grapes and wine: techniques and concepts’, by Patrick Iland, Paul Grbin, Martin Grinbergs, Leigh Schmidtke and Alison Soden in conjunction with IWAG.