

## **Which alcohol analysis method should you use?**

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### **Introduction**

In this article we review the methods available for determining alcohol, advantages and disadvantages of each method, and strategies for ensuring your label meets the requirements of an importing country.

### **Types of test available**

In the Australian industry, there are a number of methods currently used for determining alcohol. They are:

- Distillation/Densitometry
- Distillation/Hydrometry
- Distillation/Pycnometry
- Ebulliometry
- Gas Chromatography (GC)
- Near Infra-red Reflectance (NIR)
- High Performance Liquid Chromatography (HPLC)

Of the above tests, automated methods such as GC, HPLC and NIR are usually only found in the larger laboratories. Small to medium enterprises tend to utilise either ebulliometry or distillation/hydrometry. Distillation/Pycnometry is the European Union's (EU) reference method<sup>1</sup> and is typically used only in dispute resolution.

### **Advantages and disadvantages of each method**

A summary of the advantages, disadvantages and applicability of each method is summarised in Table 1 below.

Given that automated methods such as GC and HPLC are typically only used in the largest and more advanced winery laboratories, little more will be discussed here, except to say that in theory, they are more accurate than the reference method or methods that rely on distillation. This is because the distillation method relies on the assumption that only water and ethanol is collected during the distillation process. This is not entirely correct since fusel oils and other volatile components will also be distilled, potentially leading to a slightly higher apparent alcohol content<sup>2</sup>. The advantage of GC and HPLC is that these instruments can be calibrated against pure water/alcohol standards, and provided that the alcohol peak is fully resolved from other wine sample components, should give a more accurate result.

Table 1: Comparison of Alcohol Analysis Methods

Method	Advantages	Disadvantages	Applicability to Winery Laboratories
Distillation/ Densitometry	<ul style="list-style-type: none"> <li>• Density measurement is automated and accurate</li> <li>• Can also be used for determining density of wine</li> </ul>	<ul style="list-style-type: none"> <li>• High capital cost</li> <li>• Distillation takes time</li> </ul>	Large
Distillation/ Hydrometry	<ul style="list-style-type: none"> <li>• Low capital cost</li> <li>• Accurate</li> <li>• No standard required</li> </ul>	<ul style="list-style-type: none"> <li>• Distillation takes time</li> <li>• Hydrometers are delicate</li> <li>• Dearer if cooled water bath used</li> </ul>	Small
Distillation/ Pycnometry	<ul style="list-style-type: none"> <li>• Reference method</li> <li>• Accurate</li> <li>• No standard required</li> </ul>	<ul style="list-style-type: none"> <li>• Labour and time intensive</li> <li>• Precision balance required</li> </ul>	Medium/Large
Ebulliometry	<ul style="list-style-type: none"> <li>• Relatively low capital cost</li> <li>• Fast</li> <li>• No standard required</li> </ul>	<ul style="list-style-type: none"> <li>• Residual sugar and other dissolved solids leads to inaccuracies</li> <li>• Not suitable for export labelling</li> </ul>	Small
GC	<ul style="list-style-type: none"> <li>• Accurate</li> <li>• Automated</li> <li>• Can be used for other analyses</li> </ul>	<ul style="list-style-type: none"> <li>• High capital and operational costs</li> <li>• Skilled operator required</li> </ul>	Large
NIR	<ul style="list-style-type: none"> <li>• Accurate for table wines</li> <li>• Fast</li> <li>• Can be automated</li> <li>• Low operator skill</li> <li>• Low maintenance</li> </ul>	<ul style="list-style-type: none"> <li>• High capital cost</li> <li>• Not suitable for fortified and botrytised wines</li> </ul>	Medium/Large
HPLC	<ul style="list-style-type: none"> <li>• Accurate</li> <li>• Automated</li> <li>• Can be used for other analyses</li> </ul>	<ul style="list-style-type: none"> <li>• High capital and operational costs</li> <li>• Skilled operator required</li> </ul>	Large

### Distillation methods

In terms of ease of use, capital cost and accuracy, distillation/hydrometry is the method most suited to small to medium sized wineries. In theory, this method should give results similar to the distillation/pycnometry method, but generally tends to give lower results<sup>3</sup>. The reasons for this may be;

- Density is significantly affected by temperature variations. For better accuracy, samples should be equilibrated to 20.0°C before volume adjustments are made, or density measurements are taken. However, constant temperature baths are relatively expensive, costing several thousand dollars. Such baths can also be used for stability tests (heat and cold).
- Hydrometers/alcoholometers must be kept very clean. Any material adhering to the hydrometer will cause it to sink deeper in the liquid, resulting in an apparent lower density/alcoholic strength reading. Iso-propyl alcohol is a very useful cleaning agent.
- Before use or on a yearly basis, always check a new/used hydrometer against a certified reference standard. Also, check the scale to ensure it has not moved. Good quality hydrometers will have a mark on the scale that looks like this: ">----<". As long as this mark is aligned to an etched line on the stem, the scale is satisfactory. If the scale has moved, the hydrometer should be discarded.

Distillation/Pycnometry could also be used by a small/medium winery, but it is very time consuming. It also requires a precision 4-figure balance, which can cost a few thousand dollars. One advantage of owning a precision balance is that reasonably accurate alcohol/water standards can be prepared gravimetrically using pure alcohol and distilled water and reference to published alcohol/water tables. To ensure better accuracy, weighing bottles/pycnometers and constant temperature baths will also be required.

### **Ebulliometry**

Ebulliometry is a very simple, rapid and reasonably accurate method for analysing most dry wines. Unfortunately, its accuracy is compromised by presence of residual sugar or other dissolved solids. Experience within our laboratory indicates that for dry wines ebulliometry gives lower results compared to distillation/pycnometry by approximately 0.2 % with a range of 0.0 to 0.6% volume/volume (v/v). Despite this range, ebulliometry is still suitable as a QC tool for at least providing a good estimate of alcohol. However, it cannot be relied upon for labelling purposes, particularly for the EU.

Brand-name ebulliometers can be relatively expensive, and wineries can make their own ebulliometers using standard glassware and readily available thermometers.

### **NIR**

NIR technology has been around for over a decade. With more accurate systems now available, it is becoming the industry standard amongst larger

wineries. Trials within our laboratory indicate that the latest models are of comparable accuracy to HPLC and Distillation/Pycnometry for table wines. However, deviations of up to 0.3% v/v can be experienced for botrytis or some fortified wines and are not recommended for these wines. Some NIR systems require calibration against a number of wine samples, but newer models require a simple two-point calibration using distilled water and an accurately prepared alcohol/water mixture.

### **Strategies for label alcohol statements**

Fortunately, most countries allow a reasonable variation between actual and label alcohol values. For Australia, the alcohol must be accurate to within 1.5 % v/v. However, the EU requirement is very strict, allowing a variation of up to 0.5% v/v only. Furthermore, the alcohol value must be stated in terms of 0.5% intervals, eg, 12.0, 12.5, 13.0 % v/v. So, if a winery obtains a value of 12.7%, they may label the wine as either 12.5 or 13.0% v/v.

NATA accredited laboratories only can issue EU certificates of analysis, and these laboratories may use a different method to the winery. It is therefore quite common for differences in alcohol values to be reported even if both parties have performed the analysis diligently. For example, the reference method, distillation/pycnometry, allows for a variation between laboratories of up to 0.2% v/v!

Therefore, what is the best way to get accurate alcohol contents and avoiding having to re-label wines for the EU?

- If possible test a finished sample after bottling, ideally one taken from the middle of a bottling run.
- Use a NATA accredited Laboratory to perform the alcohol test, and then use the same laboratory for the Certificate testing after packaging is complete. (Note! Export analysis can only be performed on finished product).

### **References**

1. EEC Regulation No. 000/90, 1990, Official Journal No 3. 10. 90, 35-49
2. Antonelli, A; Ethanol determination by packed GC: a quick method with small sample amount and high sensitivity, *Vitic. Enol. Sci.* 49 (4), 165-167 1994
3. Sneyd, N; Analysis of alcoholic strength in wine, *AWRI Technical Review*, (64), 2-6, 1990