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# Discrepancies in analytical results for volatile acidity

The AWRI has recently received queries about differences between results of volatile acidity (VA) analyses conducted overseas and those conducted in Australia. This column answers some of the questions raised when this issue arises.

## By Adrian Coulter



#### Has the wine spoiled during transport?

Volatile acidity was probably the wine industry's first measure of wine quality and is routinely used as an indicator of wine spoilage due to acetic acid. Consequently, if a wine that arrives in another country is analysed for VA and the result is significantly higher than indicated on the accompanying export certificate, it might be assumed that spoilage has occurred during transport. While spoilage could be a possibility, typically, the discrepancy between the VA results is due to differences in the methods used to measure it.

#### What methods are used for VA analysis?

The VA level can be determined in a number of ways, including the traditional method of steam distillation followed by titration with a base, gas chromatography, enzymatic assays, high performance liquid chromatography and Fourier transform infrared spectroscopy (FTIR). It is estimated that about half of Australian wineries use either enzyme or FTIR methods, with the balance typically using a distillation method. No matter what method is used, the results are usually expressed in grams per litre 'as acetic acid', whether or not the method directly measures acetic acid concentration.

## Why are the results for acetic acid and VA sometimes different?

Generally, VA analysis results are fairly consistent between methods (within

 $\pm$  0.05 g/L). However, it's not unusual for a distillation method result to be higher than, say, an enzyme method result. This is because the enzyme method measures acetic acid only, whereas the distillation method measures other volatile acids as well as acetic acid. Often, FTIR methods are calibrated with the results of enzymatic analysis, so FTIR methods also typically just measure acetic acid.

## Why does the distillation method give higher results?

A number of factors can render the results of VA analysis by steam distillation higher, including:

- the presence of carbonic acid (from carbon dioxide)
- the presence of acids other than acetic acid (e.g. lactic acid, formic acid, butyric acid, propionic acid, succinic acid and sorbic acid (if added))
- the presence of high levels of sulfur dioxide (SO<sub>2</sub>)
- the presence of excess hydrogen peroxide (which can be used during the analysis to oxidise SO<sub>2</sub>).

Carbon dioxide  $(CO_2)$ , which is titrated in the distillate as carbonic acid, is a potential major source of error due to insufficient degassing of the wine sample prior to analysis. The water used to generate steam can also contain  $CO_2$ , so this water also needs to be degassed, which can be achieved by venting the steam for about 15 seconds once the water is boiling.

If added to a wine, sorbic acid can also be a major source of error in VA analysis and needs to be accounted for by subtracting a factor from the VA result: 1 g sorbic acid = 0.535 g acetic acid (Zoecklein et al. 1996).

High levels of  $SO_2$  can cause erroneously high results due to carry-over of  $SO_2$ with the distillate, so hydrogen peroxide  $(H_2O_2)$  is often added to a wine sample to oxidise the SO<sub>2</sub>. However, excess  $H_2O_2$ can lead to the formation of formic and isovaleric acids, which can result in a higher level of VA being measured. Generally, 0.5 mL of 0.3%  $H_2O_2$  added to 5 mL of degassed wine provides enough  $H_2O_2$  to oxidise any SO<sub>2</sub> present, minimising the potential for error.

### Which method should I use then?

Acetic acid is the spoilage compound produced in the largest quantities by oenologocial microorganisms and which imparts the sensory aspect generally perceived as the odour of vinegar. Hence, acetic acid is the compound of interest for winemakers and methods that specifically target this compound, such as enzymatic methods, are preferable. In addition, enzymatic methods have been shown to be more precise and more accurate than distillation methods (Bruer et al. 1985, McCloskey 1976). Consequently, an enzyme method, or a method calibrated against an enzyme method, that targets the compound of interest, will give more consistent results and is therefore recommended over a distillation method.

For further information on volatile acidity analysis, contact the AWRI helpdesk on helpdesk@awri.com.au or 08 8313 6600.

## References

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