

## *QA Systems, making sure it all works...*

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Quality Systems and Laboratory Manager  
AWRI Commercial Services*



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# What is quality?



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Quality assurance is the underpinning framework that promotes the delivery of reliable analytical measurements.

It is a **system** that focuses on:

Management  
Documentation  
Proficiency testing

Quality control  
Methods  
Internal audits

Staff training  
Record keeping  
Equipment



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# Why bother with a quality system?



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Laboratory provides analyses which are considered important to monitor various stages of the winemaking process and form part of the quality assurance program associated with good winemaking.

## Keep winemakers focused on winemaking!

Capacity to troubleshoot when there are errors

Provide evidence results are fit for purpose

Avoid litigation

Avoid expensive product recalls



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# Quality control



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- ❖ Training staff to perform well documented procedures using regularly calibrated equipment with checks and audits in every batch
- ❖ Typical analytical checks include:
  - Duplicates
  - Standards...usually non-matrix
  - Spiked samples...usually matrix and alternative to CRM's
  - Control samples...with known parameters
  - **How do we determine known parameters?**
  - Frequency dependent on many things.....
- ❖ corrective action procedures



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- ❖ Implement corrective action!

**These are a waste of time if you don't... !**

- ❖ Simple control charts

- visible, instant means of monitoring trends
- statistical approach to setting the decision rules and evaluating test data.

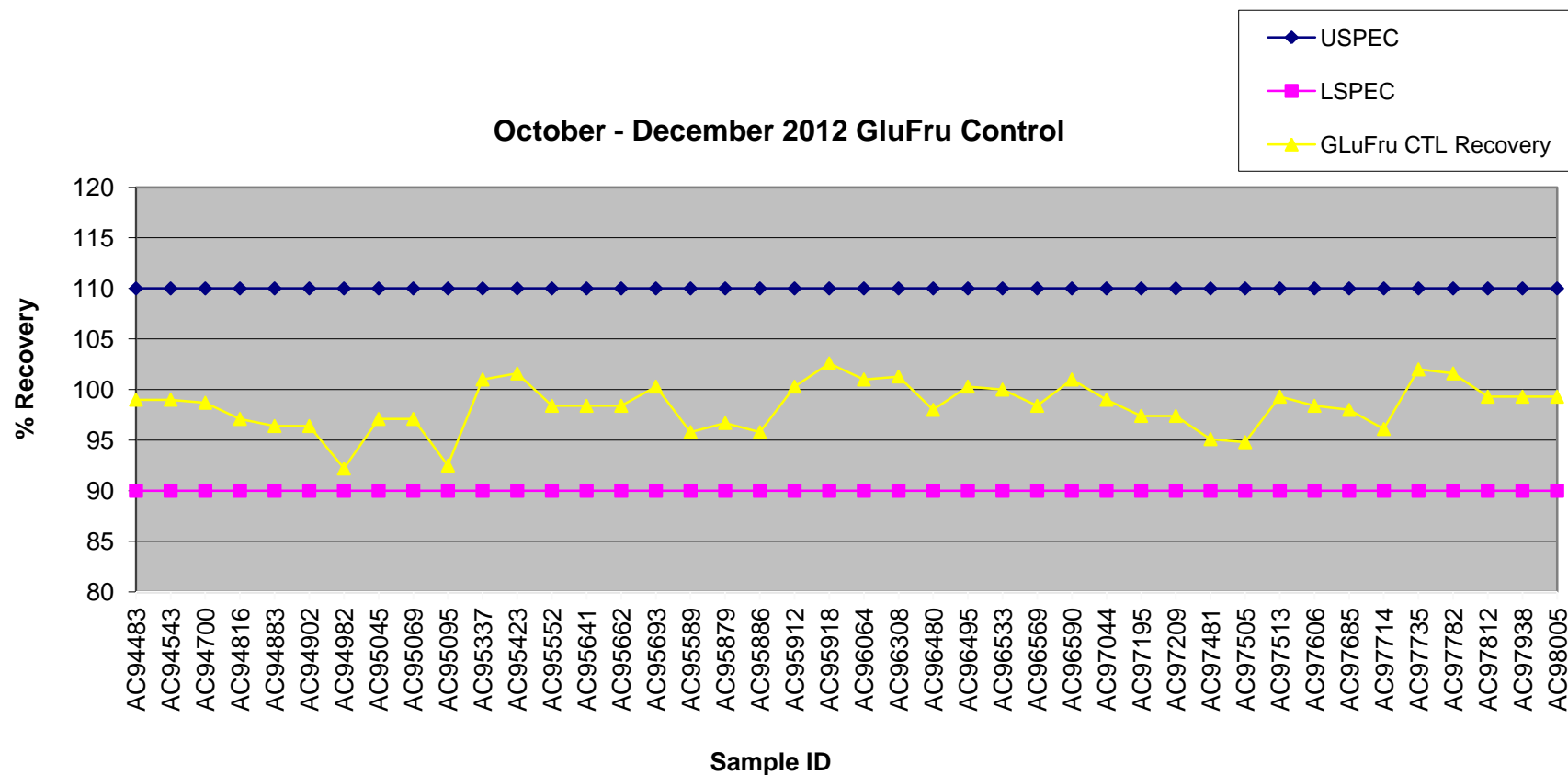


# Quality control - trending



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October - December 2012 GluFru Control



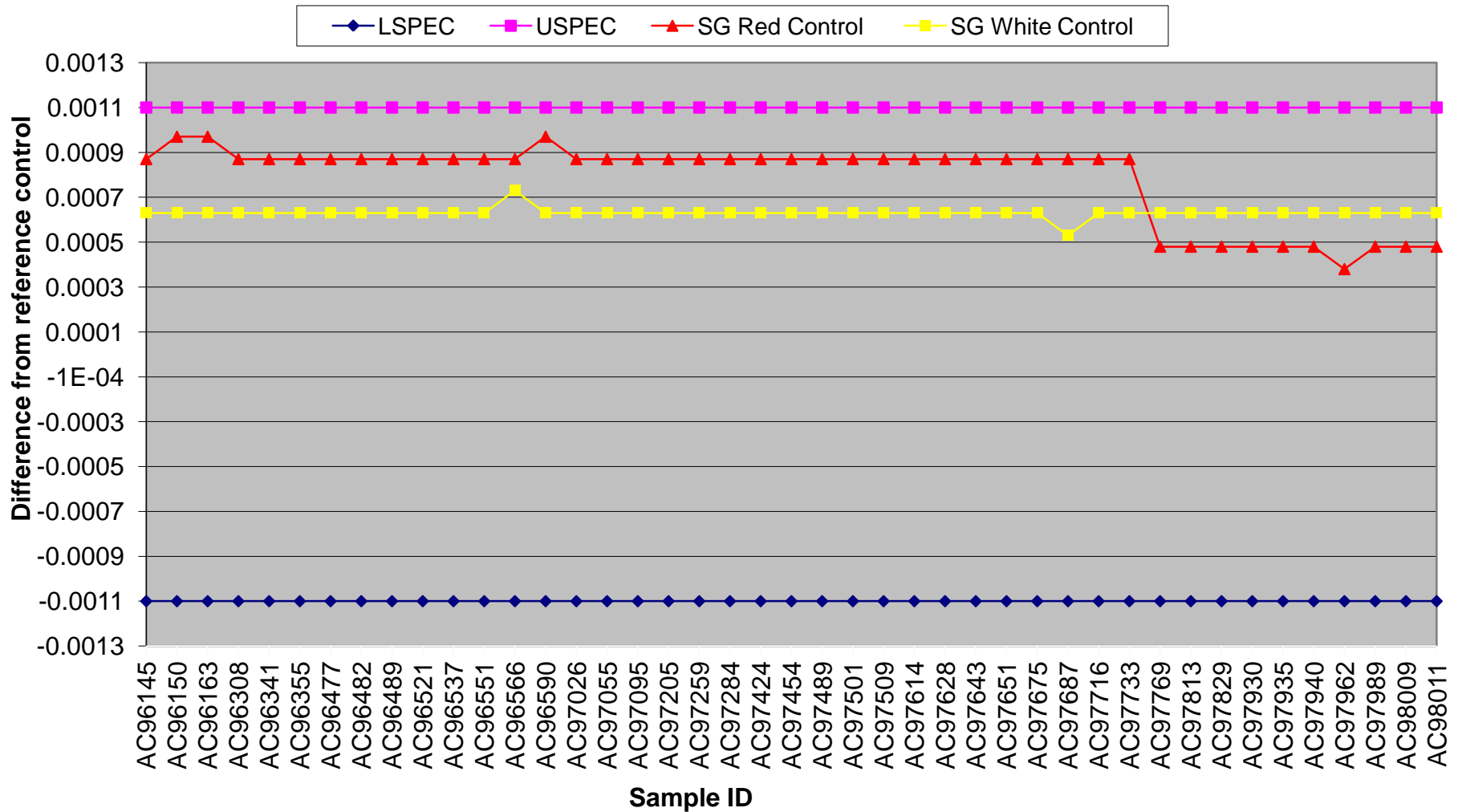
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# Quality control - trending



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## November 2012 SG Control WineScan



# Documentation



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Established and maintained procedures and policies to control and review all documents including:

- Methods
- Regulations
- Standards
- Software etc.

Position descriptions / qualifications etc.

Personal development plans

Corrective action procedures...Confidence in troubleshooting



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6. After analysis, rinse the cuvette with iso-octane and dispose of all samples in the labelled iso-octane waste container located in the fume cupboard.
7. Absorbance readings of 0.025 or less are recorded as **zero**. These readings are the average background readings for white and red wines which contain no sorbic acid, and equate to a concentration of 5 mg/L. In certain wine types, e.g. fortified and botrytised wines, the background absorbance reading may be higher than 0.025. In such cases a senior staff member should be consulted prior to data being accepted.
8. Some opaque beverages, usually not wine, require distillation as a preparatory step to avoid the possibility of false positive results (see AOAC methods).

#### Reference

Ziemelis, G.: Somers, T.C. Rapid determination of Sorbic Acid in Wine. Am. J. Enol. Vitic. 29(3): 217-219; 1978.

#### Uncertainty of Measurement (UOM)

Uncertainty of measurement was calculated by determining the average, standard deviation and the coefficient of variation of reproducibility data of 7 replicates. From this it could then be estimated that the UOM at the 95% confidence interval was equal to 2 x SD for samples with levels < 80 mg/L and 2 x CV (%) for samples > 80 mg/L. The UOM was determined to be  $\pm 4$  mg/L if < 80 mg/L and  $\pm 10\%$  if > 80 mg/L.

Data for the above calculations can be found at F:\Analytical Service\Service Delivery\Implementation\Laboratory\Method Development and Validation\Measurements of Uncertainty 2009 -2010\Uncertainty calculations 2009 -2010.xls

Further validation and investigation was performed on the method in late 2012 to early 2013 and can be found at F:\Analytical Service\Service Delivery\Lean\LGPI\LGPI202.doc. Adjustments to the method were made as a result of the findings.

#### Appendix 1. Quality Assurance

The reliability of the method is monitored using the following procedure:

1. A standard of appropriate sorbic acid concentration is included with every batch of analyses performed. The standard recovery is calculated from the result as follows:  
$$\text{Standard recovery} = \frac{\text{Result obtained}}{\text{Result expected}} \times 100\%$$

Standard recovery should be within the range 90 – 110%.
2. Every tenth determination is performed in duplicate. Duplicates should agree to  $\pm 4$  mg/L if the value is less than 80 mg/L and  $\pm 10\%$  if greater than 80 mg/L.

Authoriser: Quality Manager

Version: 1.5 Current

Date last modified: 10/05/2013

Doc ID: 3832

# Methods



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## A picture is worth a thousand words...

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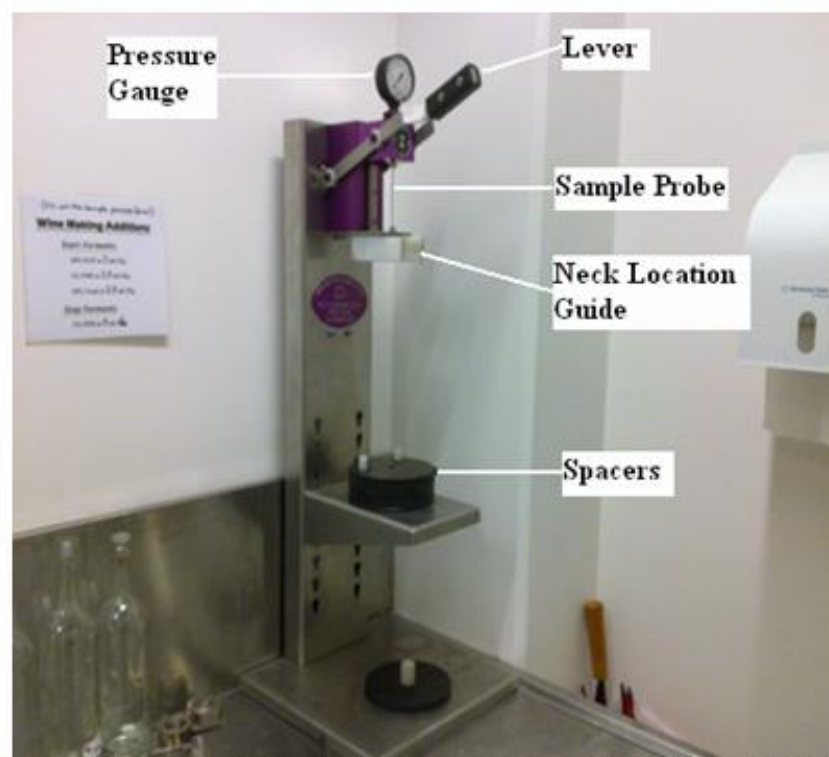


Figure 1: Henstock Technologies Bottle Pressure Tester (Model 15-22/1)

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# Training



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	A	B	C	D	E	F	G	H	I	J	K
1	<b>Commercial Services</b>										
2	This is a controlled document.										
3	<b>PF01 Training Record Form - Analytical Lab</b>										
4											
5	Name:			Position:			Commencement Date:				
6											
7	Competence Level										
8	Analysis/Procedure										
1=Unskilled		2=Limited skills		3=Competent		4=NATA-proficient					
9	LM02	Batch		Batch		Batch		Batch		Batch	
10	Total Dry Extract	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
11											
12	LM04	Batch		Batch		Batch		Batch		Batch	
13	pH/TA Titrameter	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
14											
15	LM06	Batch		Batch		Batch		Batch		Batch	
16	Copper by AA	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
17											
18	LM06	Batch		Batch		Batch		Batch		Batch	
19	Iron by AA	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
20											
21	LM06	Batch		Batch		Batch		Batch		Batch	
22	Sodium by AA	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
23											
24	LM06	Batch		Batch		Batch		Batch		Batch	
25	Potassium by AA	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
26											
27	LM06	Batch		Batch		Batch		Batch		Batch	
28	Calcium by AA	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
29											
30	LM09	Batch		Batch		Batch		Batch		Batch	
31	Free & Total SO <sub>2</sub> by stills	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
32											
33	LM10	Batch		Batch		Batch		Batch		Batch	
34	Volatile Acidity by stills	Date	Initials	Date	Initials	Date	Initials	Date	Initials	Date	Initials
35											
36	LM13	Batch		Batch		Batch		Batch		Batch	

Page 1

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# Training



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No point having your lab techs trained with world cup skills, if they do not understand the game plan!!

## Approved Signatory Record

<b>Approved Signatory</b>	<u>Larry Labtech</u>	<b>Laboratory</b>	<u>Analytical Laboratory</u>
<b>Assessed by:</b>	<u>Larrys Boss</u>	<b>Date of Evaluation</b>	<u>22/6/2011</u>
<b>Criteria</b>	<b>Approval</b>	<b>Comments</b>	
Principles of the tests they perform or supervise	<input type="radio"/> No <input checked="" type="radio"/> Yes	Is familiar with all LM's having been proficient in them for more than a year.	
Competency in reporting procedure	<input type="radio"/> No <input checked="" type="radio"/> Yes	Has fulfilled all requirements and reported all different reports possible.	
Knowledge of ISO/IEC 17025-2005 Standard	<input type="radio"/> No <input checked="" type="radio"/> Yes	Has the required knowledge and understanding of ISO/IEC 17025-2005.	
Knowledge of the management system	<input type="radio"/> No <input checked="" type="radio"/> Yes	Has read the LQM earlier in the year (January) in preparation for the NATA audit.	
Knowledge of NATA rules, FAD & relevant technical / policy circulars	<input type="radio"/> No <input checked="" type="radio"/> Yes	Has the required knowledge and understanding of NATA rules, FAD and relevant technical/policy circulars.	
Estimation of uncertainty of measurement	<input type="radio"/> No <input checked="" type="radio"/> Yes	Has completed UOM on LM32 (Rebelein method) and completely understands SD, CV% and error.	

Scope of approval for signatory:

All methods covered under the scope

# Traceability



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- ❖ LIMS an advantages
- ❖ Manual systems can achieve the same
- ❖ Beneficial for corrective actions / litigation purposes

**Multisample Validation - Sample Header Fields**

Save Cancel

Sample	Validate	Previously	Analyses	Any	Audit	QA/QC	Sample Code	Sample Colctr	Col Date	ColTime	Sub Date	SubTime	P.O. Num	Project	Log Rec	Awri_no	Replicates
ID	Now	Validated	Validated	Viols	Trail	Data											
AD09110	<input checked="" type="checkbox"/>	Yes	Yes	Yes	Yes	Yes	INTERNAL		10/07/2013	00:00	10/07/2013	12:19		12.1.2.430	130710122138	130710007	1

Sample header info    Comments    Special information    Analysis order    Setup headers

Validate samples

# Traceability



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- ❖ Valuable for training
- ❖ Can help with interpretation of results
- ❖ Trending of procedural errors

Audit Trail Entries for Sample: AD09110							Print	Close
Changed Item	Previous Value	New Value	Change Date	Time	Changed By	Reason		
SO2_REF_STILL - Sulfur Dioxide Still result	A,B	A/B	10/07/2013	14:40	Newell, Bryan	Data entry error.		
SO2T_FIA -	Pending	Analysis removed	11/07/2013	09:53	Hoxey, Leanne	juice sample therefore reference SO2 required not FIA LH		
T_SO2T_FIA -	Pending	Analysis removed	11/07/2013	09:53	Hoxey, Leanne	juice sample therefore reference SO2 required not FIA LH		
P_SO2T_FIA -	Pending	Analysis removed	11/07/2013	09:53	Hoxey, Leanne	juice sample therefore reference SO2 required not FIA LH		
D_SO2T_FIA -	Pending	Analysis removed	11/07/2013	09:53	Hoxey, Leanne	juice sample therefore reference SO2 required not FIA LH		



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# Proficiency testing



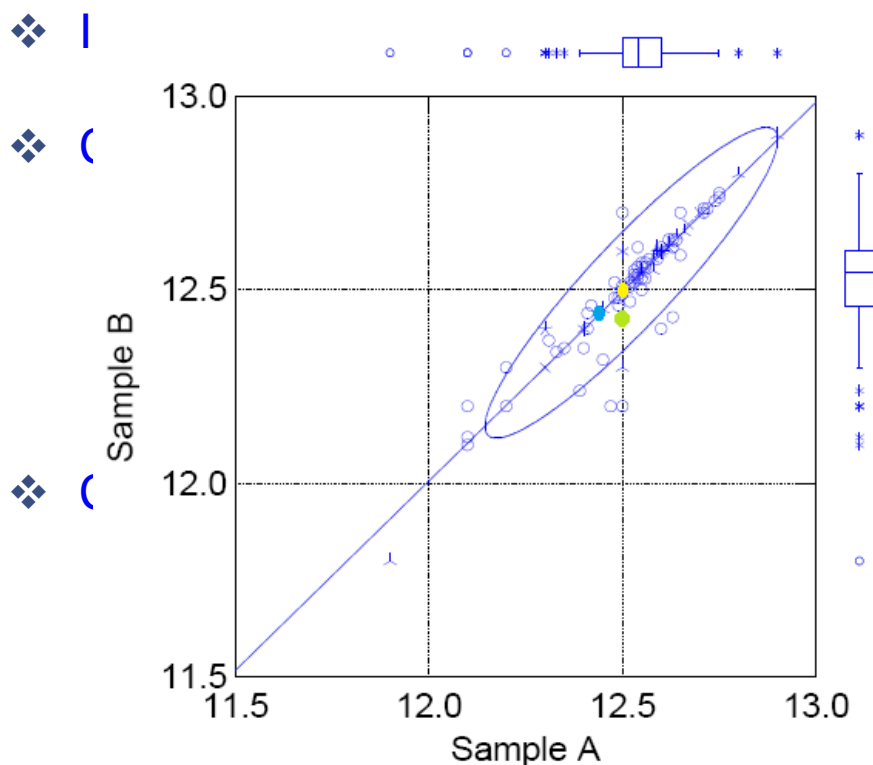
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Interwinery Analysis Group

Statistical Report Round 10-03

May 2010

## Alcohol



Results for Z-numbers 296 were removed as extreme outliers.

	Alcohol A	Alcohol B
Reported at 20°C	% v/v	% v/v
N of cases	146	146
Minimum	11.9	11.8
Maximum	12.9	12.9
Range	1.0	1.1
Median	12.5	12.5
Mean	12.5	12.5
95% CI Upper	12.5	12.5
95% CI Lower	12.5	12.5
Standard Dev	0.2	0.2
C.V. %	1.6	1.6
Lab Result		
Lab Z-Score Result		

AWRI Method	Sample A	Sample B
NIR	12.4	12.4
Distillation	12.5	12.4
Winescan	12.5	12.5

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## Quality Review (Internal Audit) – 2012

Subject: VA (LM10)

: SG (LM24)

: Alcohol by distillation (LM23) & by NIR (LM13)



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❖ Important point The Interwinery red samples AC82202 and AC82203 analysed for volatile acid, specific gravity, alcohol by distillation and by NIR during January 2012 were assessed for compliance with tolerances against samples analysed in May 2012 AC88307 and AC88308.

❖ Can help identify Summary of the results:

VA - (LM10) - Distillation

Components	AC82202 g/L	AC82203 g/L	AC88307 g/L	AC88308 g/L	Uncertainty g/L	Within Specs
VA	0.27	0.27	0.27	0.27	± 0.04	Y

❖ Can use hold SG - (LM24) - Densitometry

Components	AC82202	AC82203	AC88307	AC88308	Uncertainty g/cm3	Within Specs
SG	0.9959	0.9960	0.9958	0.9959	± 0.0002	Y

❖ Good follow up Alcohol - (LM23) - Distillation

Components	AC82202 % v/v	AC82203 % v/v	AC88307 % v/v	AC88308 % v/v	Uncertainty % v/v	Within Specs
Alcohol	11.6	11.6	11.6	11.5	± 0.1	Y

Alcohol - (LM13) - NIR

Components	AC82202 % v/v	AC82203 % v/v	AC88307 % v/v	AC88308 % v/v	Uncertainty % v/v	Within Specs
Alcohol	11.7	11.8	11.7	11.6	± 0.1	Y

Hard copies of the reports for all samples can be found in Office 2.73 filed in the folder 'Interwinery Reports/Reviews 2012'.

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16<sup>th</sup> July, 2012

e.g. IWAG



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The retain sample for AC80344 was located in cellar position Rk11:R:7 and resubmitted for Certificate of Analysis with a new sample ID AC87252.

## Summary of the results:

### ❖ Can combine documentation and analysis audits

Components	AC80344	AC87252	Uncertainty	Within Specifications
Alcohol	13.0%	13.1%	± 0.1%v/v	Y
TA- pH 8.2	5.8g/L	5.8g/L	± 0.1g/L	Y
pH	3.59	3.59	± 0.05	Y
Glu/Fru	0.4g/L	0.6g/L	± 0.3g/L	Y
VA as AA	0.66g/L	0.65g/L	± 0.05g/L	Y
SG	0.9934	0.9934	±0.0002	Y

## FIA (LM29)

Components	AC80344 mg/L	AC87252 mg/L	Uncertainty mg/L	Within Specifications
Free SO <sub>2</sub>	18	16	± 4.0	Y
Total SO <sub>2</sub>	51	45	± 4.0	Y

The PDF's of the reports for AC80344 and AC87252 are linked below.

AC80344 report located at <\\Rimmer\labworks\LWDATA\Reports\2011\November\CofA\cofa-111111c.pdf>

AC87252 report located at <\\Rimmer\labworks\LWDATA\Reports\2012\April\CofA\cofa-120413a.pdf>

Heather Tosen

16<sup>th</sup> April, 2012



# Equipment



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**Equipment**

New Save As Save Apply formula Attachment Export Para-Link® Send Roles Event Date History Close

Name:  ID/OrigID : 599 / 599 Status: Complete (1.0) (Leanne Hoxey)

Label:

Select Step: **1**

**Page: Equipment**

Type

Model

Serial #

Date

Location

Description / Notes

Supplier

Serviced By



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***Thankyou!***



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