

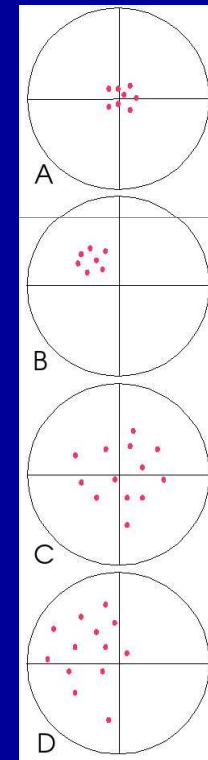


WHY DO LABS DIFFER?

2010 SAWLA

Back to basics

- What does a winemaker expect from a lab?
- Accurate analyses
- Free of charge!
- Yesterday!!



What is expected from a lab?

- **Accurate analyses for decisions/predictions**
 - Prevent spoilage (VA, SO₂, micro)
 - Completion of processes (fermentation, mlf, stabilisation)
 - Adjustments (TA, RS, SO₂)

- Protection of equipment (filterability)
- Legal specs (alcohol, RS, metals, additives)
- Shelf life
- Research / product development

- What do labs need to achieve that?

- Suitable
 - Area
 - People
 - Methods
 - Instruments
 - Specifications
- Policies, procedures, work instructions to manage the people and processes

- Control

- Check samples
- Standard reference material
- Proficiency testing (Interlab comparison)
- Audits
- Continuous improvement

HOW IS COMPETENCY
PROVEN?

- Accreditation (ISO 17025)
- And if not accredited?
- Need to show evidence of competency (records)
 - Audited by client
 - Internal Quality control (control charts)
 - SAWLA Proficiency testing scheme 1x/ month?? (Interlab)

Why do even accredited
labs still differ from each
other?

Influences on analysis results

- **METHOD**
 - Primary/Reference
- **MACHINE**
 - different instruments (pH electrodes, burettes, automatic titrators)
- **MAN**
 - Qualifications/ Skills/ Training/Experience
Morale(pressure/ relaxed/ well payed)
 - Career jumpers / loyal
- **MATERIAL**
 - Chemicals / Filters / Fining agents

- ENVIRONMENT

- Draughts

- Temperature

- Food

- Good housekeeping

- QUALITY OF THE SAMPLE

- Representative (of tank as well as process)
- Clean
- Full
- Fresh
- Temperature

Sapmonsters

	pH			Totale suur	
	Opgeskud	bostand		Opgeskud	Bostand
sap	3.42	3.43		6.65	5.07
sap	3.49	3.50		7.14	6.02
sap	3.47	3.46		5.9	5.52
sap	3.71	3.72		4.81	4.21

Sapmonsters

Herontledings		
	pH	TA
sap 1	3.77	3.61
	3.75	4.01
	3.75	5.42
	3.73	5.57
	3.72	6.51
sap 2	3.75	4.38
	3.73	4.66
	3.72	5.91
	3.71	4.94

	pH	TA
pulp	3.74	8.13
sentrifugeer	3.66	5.01

4 METHODS

- TOTAL ACIDITY
- VA
- ALCOHOL
- (COLD STABILITY)

TOTAL ACIDITY

- Titration
- FTIR (Winescan)
- Segmented/continuous flow
- HPLC
- Enzymatic



pH + TA

LAB A

METTLER

RADIOMETER

LOCAL

ULTRASONIC

25ML

Titrator

Electrode

Buffers

Degas

**Sample
volume**

LAB B

METROHM

METROHM

METROHM

VACUUM

50ML

pH + TA

LAB C

LAB D

WINESCAN

WINESCAN

HAND METHODS

**INTERNATIONAL
WINE STANDARDS**

SAWLA INTERLAB RESULTS

Total Acidity All values

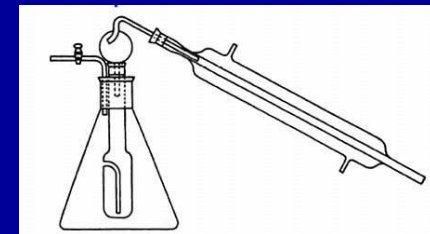
	Result g/L	Z-Value	Performance	Method		Result g/L	Z-Value	Performance	Method
1	3.72	-10.438	E	Titration	19	5.27	-0.158	A	Titration
2	5.05	-1.617	B	Titration	20	5.29	-0.025	A	Titration
3	5.07	-1.484	B	Titration	21	5.3	0.041	A	Titration
4	5.07	-1.484	B	FTIR	22	5.31	0.107	A	FTIR
5	5.12	-1.153	B	Titration	23	5.31	0.107	A	Titration
6	5.13	-1.086	B	Titration	24	5.34	0.306	A	FTIR
7	5.14	-1.02	B	Titration	25	5.36	0.439	A	Titration
8	5.17	-0.821	A	Titration	26	5.39	0.638	A	Titration
9	5.17	-0.821	A	FTIR	27	5.4	0.704	A	Titration
10	5.19	-0.689	A	FTIR	28	5.44	0.969	A	FTIR
11	5.2	-0.622	A	FTIR	29	5.44	0.969	A	Titration
12	5.22	-0.49	A	Titration	30	5.45	1.036	B	FTIR
13	5.22	-0.49	A	FTIR	31	5.46	1.102	B	Titration
14	5.23	-0.423	A	FTIR	32	5.49	1.301	B	Titration
15	5.24	-0.357	A	FTIR	33	5.5	1.367	B	Titration
16	5.25	-0.291	A	Titration	34	5.53	1.566	B	Titration
17	5.25	-0.291	A	Titration	35	5.74	2.959	C	FTIR
18	5.25	-0.291	A	Titration	36	7.8	16.621	E	FTIR

INTERLAB RESULTS

		<u>TOTAL ACIDITY (G/L)</u>					
		Month 1	Month 2	Month 3	Average	Std dev per method	Std dev per group
1	<u>LATE HARVEST</u>						
	FTIR	6.30	6.37	6.83	6.50	0.29	0.18
	Titration	6.70	6.58	6.59	6.62	0.07	
	Segmented Flow		6.60				
2	<u>DRY RED</u>						
	FTIR	5.49	5.32	5.31	5.37	0.10	0.09
	Titration	5.22	5.31	5.29	5.27	0.05	
3	<u>SEMI SWEET</u>						
	FTIR	6.09	5.78	6.19	6.02	0.21	0.20
	Titration	5.89	5.88	6.07	5.95	0.11	
	Segmented flow		5.62				

VOLATILE ACIDITY

- Distillation
 - Differently designed apparatus
 - Methods being applied differently
 - Reagents (fresh/standardised)
- FTIR
 - Calibration ?
- Segmented flow (auto analyzer)
 - Calibrated with acetic acid standards

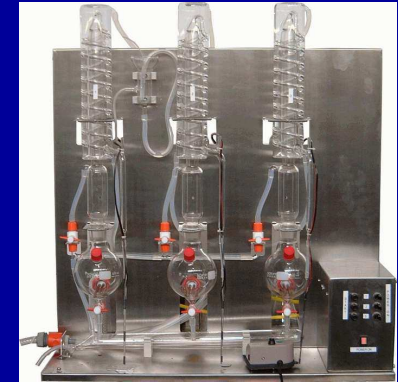


INTERLAB RESULTS

		VOLATILE ACIDITY (G/L)					
		Month 1	Month 2	Month 3	Average	Std dev per method	Std dev per group
1	LATE HARVEST						
	Distillation	0.33	0.32	0.34	0.33	0.01	0.04
	FTIR	0.27	0.27	0.26	0.27	0.01	
	Segmented Flow	0.29		0.3			
2	DRY RED						
	Distillation	0.53	0.49	0.52	0.51	0.02	0.02
	FTIR	0.49	0.49	0.51	0.50	0.01	
	Segmented Flow	0.52		0.50			
3	SEMI SWEET						
	Distillation	0.37	0.32	0.35	0.35	0.03	0.02
	FTIR	0.38	0.36	0.38	0.37	0.01	
	Segmented flow	0.34					

ALCOHOL

- Distillation - different distilling apparatus
 - Pycnometer
 - Vol:vol \rightarrow density (oscillation)
 - Gibertini Densimat
- NIR (Near Infra Red)
 - Alcoalyzer
 - Infralyzer



ALCOHOL

- FTIR (Fourier Transform Infrared Spectroscopy)
 - Winescan
 - Oenofoss
- Boiling point
 - Ebulliometer
 - Glasschem's 3-in-1
- Enzymatic



ALCOHOL All values

	Result % vol	Z-Value	Perfor mance	Method		Result % vol	Z- Value	Perfor mance	Method
1	12.57	-4.723	E	Distillation	19	12.91	0.175	A	Distillation
									Near Infra
2	12.74	-2.274	C	Distillation	20	12.93	0.464	A	Red
3	12.75	-2.13	C	Distillation	21	12.97	1.04	B	FTIR
4	12.77	-1.842	B	Distillation	22	12.97	1.04	B	Distillation
5	12.81	-1.265	B	Distillation	23	12.98	1.184	B	Distillation
				Near Infra					
6	12.81	-1.265	B	Red	24	12.99	1.328	B	Distillation
7	12.83	-0.977	A	Distillation	25	13.01	1.616	B	Distillation
8	12.83	-0.977	A	FTIR	26	13.01	1.616	B	Distillation
9	12.83	-0.977	A	FTIR	27	13.02	1.76	B	Distillation
10	12.83	-0.977	A	Distillation	28	13.06	2.337	C	Distillation
11	12.85	-0.689	A	FTIR	29	13.07	2.481	C	FTIR
12	12.85	-0.689	A	FTIR	30	13.07	2.481	C	Distillation
13	12.85	-0.689	A	Distillation	31	13.1	2.913	C	FTIR
14	12.86	-0.545	A	Distillation	32	13.1	2.913	C	Distillation
15	12.87	-0.401	A	Distillation	33	13.1	2.913	C	Ebulliometer
16	12.88	-0.257	A	Distillation	34	13.12	3.201	D	FTIR
17	12.88	-0.257	A	Distillation	35	13.15	3.633	D	Distillation
18	12.88	-0.257	A	Distillation					

Do labs differ from each other?

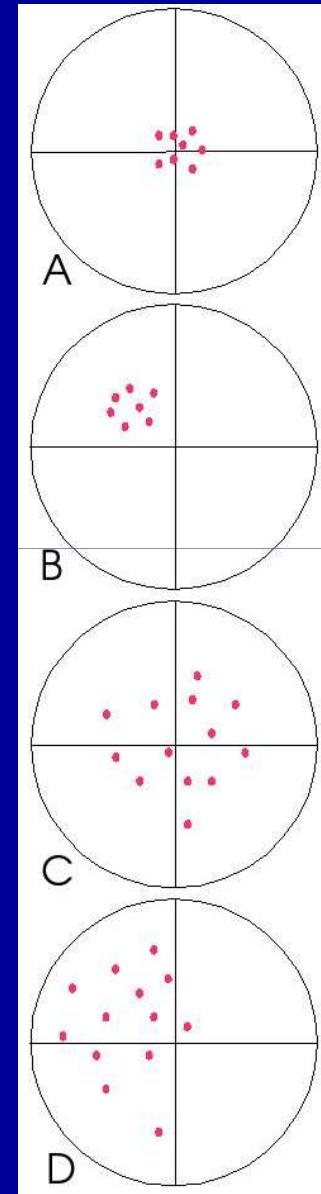
- *Yes! For sure!*
- *And they will forever.....*

LABS

- Get your house in order
- Standardised Methods (References)
- Know the analysis process + influences
- Optimize the process (get the influences under control)
- Validate and determine uncertainties

VALIDATION

- Accuracy
- Precision
- Ruggedness
- Range
- Robustness
- Uncertainty
- Traceability
- Specificity



LABS

- Maintain validation (through control samples)
- Use information gathered
- Talk to your client
- Show him your results
- Explain the uncertainties
- Offer to repeat the analysis (keep your samples for a certain period)

GENERAL ACCEPTABLE TOLERANCES

- Alcohol (4 – 16%) 0.2%
- VA (0.1-1.2g/L) 0.1g/L
- TA (3.0 – 8.0g/L) 0.2g/L
- pH (3.0 – 4.5) 0.1
- RS (1.0 – 10.0g/L) 0.5g/L
- SO₂-Asp (5 – 60mg/L) 3mg/L

– Every Lab should state their own


WINEMAKERS

What should you do?

FIT FOR PURPOSE

- Alcohol determination
 - after fermentation (turbid / gas)
 - labeling purposes (final sample?)
 - export (after labeling)
 - bottling line

 - Pycnometer
 - NIR (centrifuged)
 - FTIR
 - Replicates?



Don't kill the
Lab!

Interpretation

- Alcohol: $12,1\% \pm 0.2\%$ (95%)
- If label is 12.5%?
 - Re-analyse
 - Re-sample and analyse
 - Reference method
 - Take more samples and determine average

FIT FOR PURPOSE

- VA - determination
 - monthly control samples
 - Low VA samples
 - Borderline cases (0.8 and 1.2g/L)
 - Distillation
 - FTIR
 - Segmented flow

FIT FOR PURPOSE

- Cold Stability (tartrate + colour)
 - After blending
 - After / during stabilisation
 - Before bottling
 - Local / overseas
 - Small / large volume
- Conductivity (temperature/contact time)
- Rebelein
- Quick freeze
- Fridge test (period)

Frequent problems

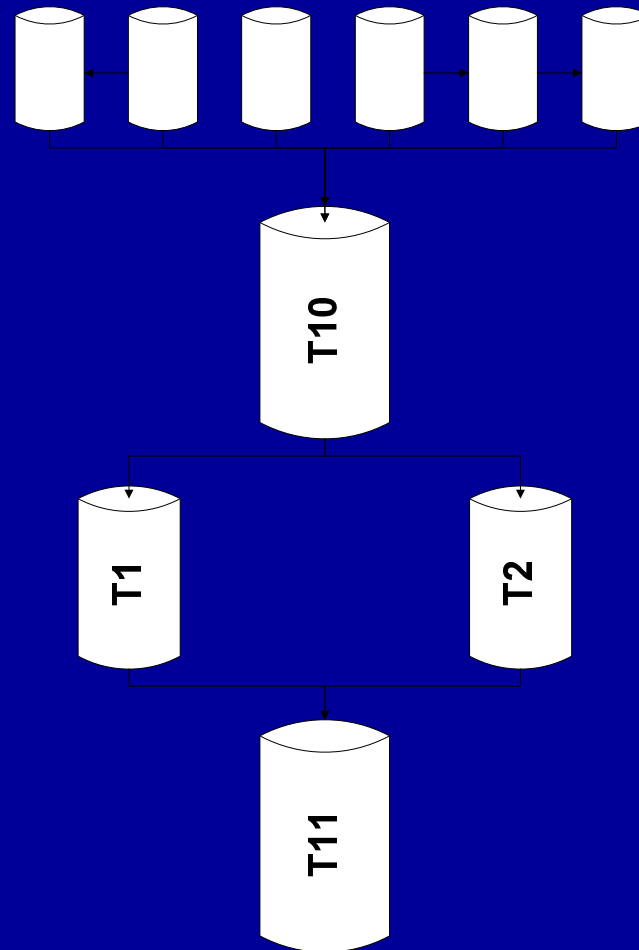
- The FSO₂ was 15mg/L in my tank. We lifted it to 45 mg/L and after bottling it is now 65. The 15mg/L couldn't have been right.
- We lifted the FSO₂ to 50mg/L and 2 days after bottling it fell to 29mg/L
 - Test after additions
 - SO₂ stability / DO

Frequent problem cases

- You said the wine needed 40g/HL bentonite - I added 80g to make sure! (Na/Ca bent?)
- T1 and T2 were protein stable and now you find it unstable - But it is the same wine!

Stable + Stable = Unstable???

BLENDING



Alc 12.5
TA 5.7
Stable

Alc 12.8
TA 5.9
Stable

Frequent problems

- P&QC got this and you got that
 - history
 - repeat
 - 3 labs

SO₂

- Ripper
- Aspirasie
- Fiastar

SO2 vergelykings

Rooi				Jong Rooi				Wit			
Asp FSO2	Asp TSO2	FIA FSO2	FIA TSO2	Asp FSO2	Asp TSO2	FIA FSO2	FIA TSO2	Asp FSO2	Asp TSO2	FIA FSO2	FIA TSO2
42	109	42	110	2	4	2	11	31	92	32	102
44	105	42	105	1	4	2	13	46	128	44	130
35	113	35	112	22	41	23	53	31	117	29	138
28	96	29	96	1	3	0	14	2	57	3	66
24	108	25	105	9	15	10	22	12	206	13	180
38	127	35	125	11	14	12	20	3	54	4	68
35	62	34	61	5	38	4	43	25	89	23	90
31	90	31	90	3	5	2	14	24	108	23	110
22	71	23	70	4	7	5	14	38	127	37	150
25	51	23	52	3	23	4	33	22	106	19	126
28	78	27	78	2	11	1	25	43	136	43	147
34	59	33	58	7	16	6	33	5	37	5	48
33	71	30	71	3	4	6	11	37	127	37	143
33	65	33	66	2	6	1	17	68	147	65	158
13	55	15	52	35	46	32	54	25	105	25	117
30	64	31	64	10	40	9	52	3	74	4	76
31	94	31	92	5	31	1	41	3	53	3	62
18	114	18	117	2	5	3	14	11	57	13	70
4	35	6	36	32	52	29	71	38	107	40	128
42	109	42	110	3	3	2	12	31	149	29	172
60	138	60	141	6	17	5	30	45	155	44	172

Where to from here?

- Choose your lab of preference or stick to your assigned one - don't do lab hopping!!
- Find out what they do and how they do it (it's your right to audit a lab)
- Check them regularly and talk to them about it
- Educate yourself to interpret the results

Where to from here?

- You know (or are supposed to...) what the expected result should be
- If you don't - you probably don't test enough
- If it is not - ask them to repeat, draw another sample, share the process with them.

CONCLUSION

- LABS will always differ
- The winemaker can NOT make (good) wine without proper analysis results and knowledge about the results
- The Lab can NOT do proper analyses without good samples and knowledge about the samples

WE NEED EACH OTHER

SO

PLEASE TAKE HANDS!

If you need a copy of the presentation,
please contact me at
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