Stabilab™ - the new Standard in Tartrate Stability testing

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Introduction

In recent years many advances in tartrate stabilization have occurred giving winemakers more options than the expensive cold contact process. These include electrodialysis, the use of mannoproteins (Mannostab™, Laffort Australia) and shortly CMC carboxymethylcellulose which is currently with ANZFA for approval in Australia. It would seem that there might also be need to change and upgrade our testing procedures from the old refrigeration/freezer test.

At Winechek we have found this to be true and in February 2010 we installed the latest technology, a Stabilab™ (Figure 1) machine form France.

Figure 1. The Stabilab™ with conductivity probes & agitator assembly in place

Background

At Winechek we gave up on the traditional and widely used refrigeration or freezer tests in 2005 as we found them neither reliable, nor easily repeatable. Our findings are confirmed by P.Ribereau-Gayon et al (2006) & B.W. Zoecklien et al (1997) and many others in the documented literature as the test is based on the phenomenon of spontaneous non-induced crystallization, a slow undependable/unpredictable process.

We have mainly used the Saturation Temperature (Tsat) conductivity test established by Erbsloh to estimate Stabilization Temperature (Tcs) for tartrate stability testing. The Tsat test has the advantage of being rapid, 5-10 minutes test time and is therefore very useful when a winery is utilizing the Cold contact process to tartrate stabilize their wine. We found that it was excellent for tank whites, which is the majority of samples but possible gave false failing results with some dark Roses, most reds and extended lees contact whites. We investigated other means of determining tartrate stability including Potassium measurement pre & post Potassium Hydrogen Tartrate (KHT) seeding and chilling, also the -4ºC for 3 & 6 days but found all these methods either costly, hard to interpret or too long to be useful for our clients. We determined to find another rapid method that would fill this whole in our testing ability and after about a year of investigation finally found the Stabilab™.

The Stabilab™ was researched and developed by INRA (one of the largest agricultural research institutes in Europe) & Eurodia (producers of the Electrodialysis technology) to model tartrate stability of wine at -4°C for 6 days, the OIV/European reference test. It is basically a strictly temperature controlled cold bath with agitators to constantly stir the wine under test & conductivity probes (Figure 2) to constantly measure changes in electrical conductivity (µS/cm). The conductivity of a wine is an objective measure of the concentration of ions in solution, Potassium (K) is the major cation found in wine normally more than ten fold the ion concentrations of Sodium & Calcium. When the precipitation of Potassium Hydrogen Tartrate (KHT) crystals occurs we observe a decrease in Potassium & therefore conductivity. By supersaturating the wine with KHT crystals, holding the wine at -4°C and continuous agitating we optimize & facilitate the precipitation of tartrate crystals.

Figure 2. The conductivity probe & agitator assembly, 8 sets for 8 tests.

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The Stabilab™ Tests

The Stabilab™ can perform two different tests, **DIT%** ("Degré d'Instabilité Tartrique" or Degree of Tartrate Instability test) and the **ISTC 50** (*Indice de Stabilite Tartaric Critique*, or Critical Tartaric Stability Index with a 50g/hL KHT addition).

DIT%

The purpose of the test is to answer the following questions:
-Is the wine stable? (Does not require treatment)
-If the wine is unstable, what level of treatment is required?

	Procedure	What is happening
1.	Temperature of the wine sample is measured and must be between 15° & 25°C	Getting a conductivity reading at room temperature for comparison to -4ºC
2.	Wine sample brought to -4ºC & then another conductivity measurement	Preparing sample for actual determination of stability & getting an initial reading
3.	Add a quantity equivalent to 4g/L of Potassium Hydrogen Tartrate (KHT) to the wine	Supersaturating the wine with KHT & have a large amount of seeding crystals available to induce precipitation of KHT
4.	The wine is agitated at -4°C for 4 hrs with	Create a perfect environment for

	conductivity measurements made every 10 minutes	the formation of KHT crystals & their precipitation. The monitoring of conductivity indicates changes in the concentration of Potassium (K) caused by the precipitation of KHT.
5.	The resulting conductivities are transferred to a computer software package that calculates the DIT% and gives many other measures	Assessment of wine stablility

The software generates a report showing the results (Figure 3)

Figure 3 Example of a DIT% report

Test nouveau logiciel the 01/07/2008 at 4:26 pm

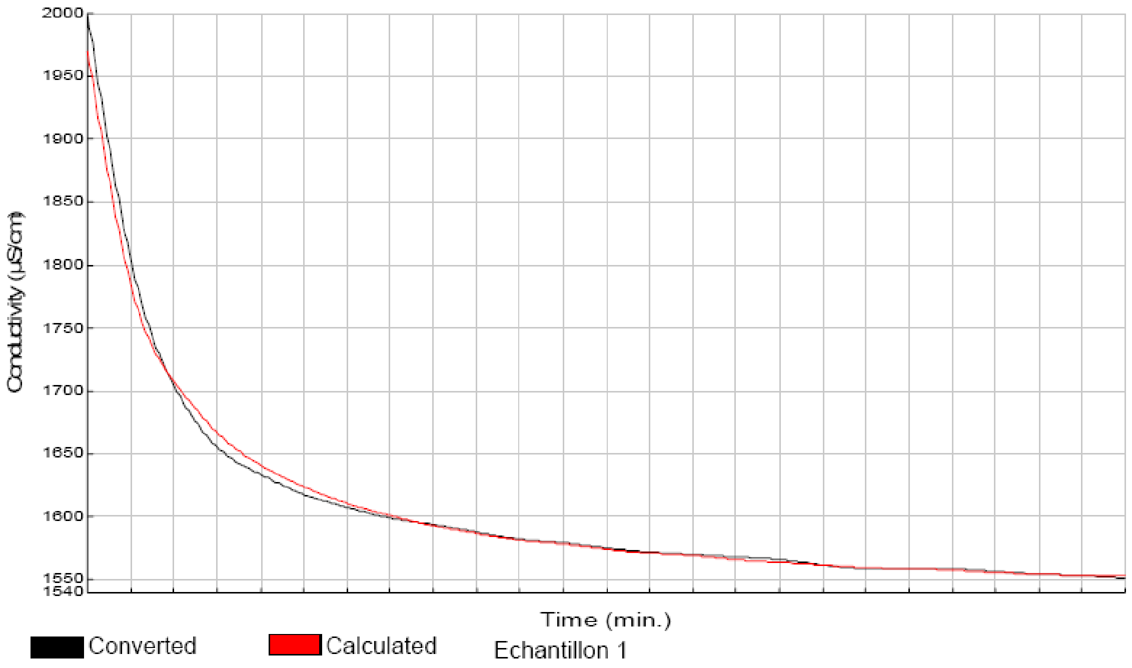
DIT Echantillon 1

Wine presumed UNSTABLE

01/02/2008

°C	min.	C meas.	C conv.	C calc.
-4.0	0	918	1999	1970
-4.0	10	828	1803	1783
-4.0	20	783	1705	1708
-4.0	30	760	1655	1667
-4.0	40	750	1633	1641
-4.0	50	743	1618	1624
-4.0	60	738	1607	1611
-4.0	70	734	1599	1601
-4.0	80	732	1594	1593
-4.0	90	729	1588	1587
-4.0	100	727	1583	1582
-4.0	110	725	1579	1578
-4.0	120	723	1575	1574
-4.0	130	722	1572	1571
-4.0	140	721	1570	1569
-4.0	150	720	1568	1566
-4.0	160	719	1566	1564
-4.0	170	717	1562	1562
-4.0	180	716	1559	1560
-4.0	190	716	1559	1559
-4.0	200	716	1559	1558
-4.0	210	715	1557	1556
-4.0	220	714	1555	1555
-4.0	230	713	1553	1554
-4.0	240	712	1551	1553

Initial Conductivity (µS/cm)	1999
Coefficient of conversion	2.178
Coefficient of correlation	0.9984
Conductivity after 4h (µS/cm)	1553
Conductivity drop after 4h	22.3%
Conductivity after 24h (µS/cm)	1534
Conductivity drop after 24h	23.3%
Infinite Conductivity (µS/cm)	1530
Rate of electrodialyse	23.5%
Inital temperature (°C)	20.0



This report shows us on the left side a table with 5 columns:

- the 1st column presents the temperature in the wine during the test (the set point),

- the 2nd column presents the time of the test,
- the 3rd column presents the measured conductivity at the temperature of the test,
- the 4th column presents the converted conductivity by the coefficient of conversion (conductivity converted to 20°C),
- the 5th column presents the calculated conductivity from the mathematical model.

On the right side, calculated results are presented:
- The initial measured conductivity at room temperature (**Co**). This conductivity is compensated at 20°C
- The coefficient of conversion Conductivity20°C/Conductivity-4°C.
- The coefficient of correlation between the converted curve and the calculated curve (r2),
- The conductivities from the calculated curve after 4, 24 hours and infinite time (**C∞**),
- The DIT ("Degré d'Instabilité Tartrique" = conductiviy drop) after 4, 24 hours and infinite time. The DIT% is the Rate of Electrodialysis.
- The initial temperature of the wine

The graph is a representation of the change in conductivity vs time of the test

ISTC 50

The purpose of the test is to answer the following questions:
- Is the wine stable after some stabilization treatment

	Procedure	What is happening
1.	To a quantity of wine the equivalent of 0.5g/L (50g/hL) KHT is added	Slightly destabilize the wine
2.	Place wine in a 36°C waterbath for 30 minutes	Completely dissolve the KHT
3.	Measure the conductivity at this temperature	Getting a conductivity reading at 36°C temperature for comparison to -4ºC
4.	Wine sample brought to -4ºC & then another conductivity measurement	Preparing sample for actual determination of stability & getting an initial reading
5.	Glass beads added	To induce precipitation of KHT by vibration
6.	The wine is agitated at -4°C for 2 hrs for whites & Rose & 4hrs for Red wines with conductivity measurements made every 10 minutes.	Create a perfect environment for the formation of KHT crystals & their precipitation. The monitoring of conductivity indicates changes in the concentration of Potassium (K) caused by the precipitation of KHT.
7.	The resulting conductivities are transferred to a computer software package that calculates the ISTC50 result	Assessment of wine stablility

The software generates a report showing the ISTC50 results (Figure 4)

Figure 4 Example of a ISTC50 report

200210 ISTC2 the 02/20/2010 at 5:12 pm

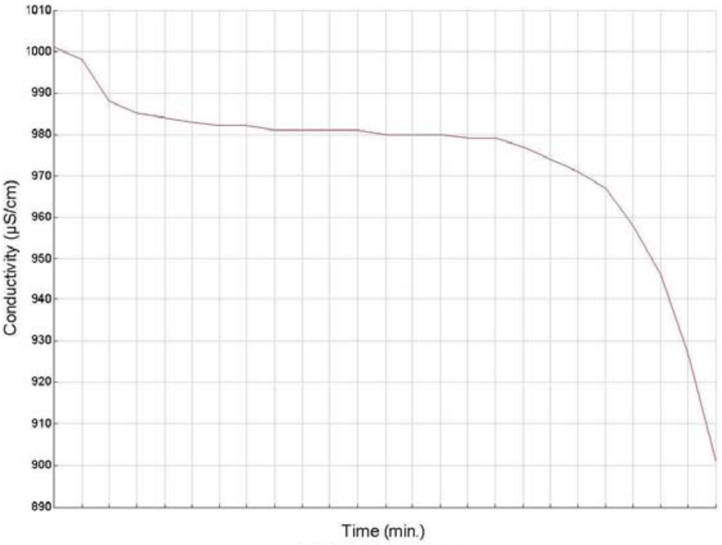
ISTC 50

20/02/2010

°C	min.	C meas.
-4.0	0	1001
-4.0	10	998
-4.0	20	988
-4.0	30	985
-4.0	40	984
-4.0	50	983
-4.0	60	982
-4.0	70	982
-4.0	80	981
-4.0	90	981
-4.0	100	981
-4.0	110	981
-4.0	120	980
-4.0	130	980
-4.0	140	980
-4.0	150	979
-4.0	160	979
-4.0	170	977
-4.0	180	974
-4.0	190	971
-4.0	200	967
-4.0	210	958
-4.0	220	946
-4.0	230	927
-4.0	240	901

Kind of wine : Red

Wine presumed UNSTABLE



09EDCW 400ADD

In the left hand table;
First column is temperature in °C
Second column is time in minutes
Third column is conductivity readings in uS/cm

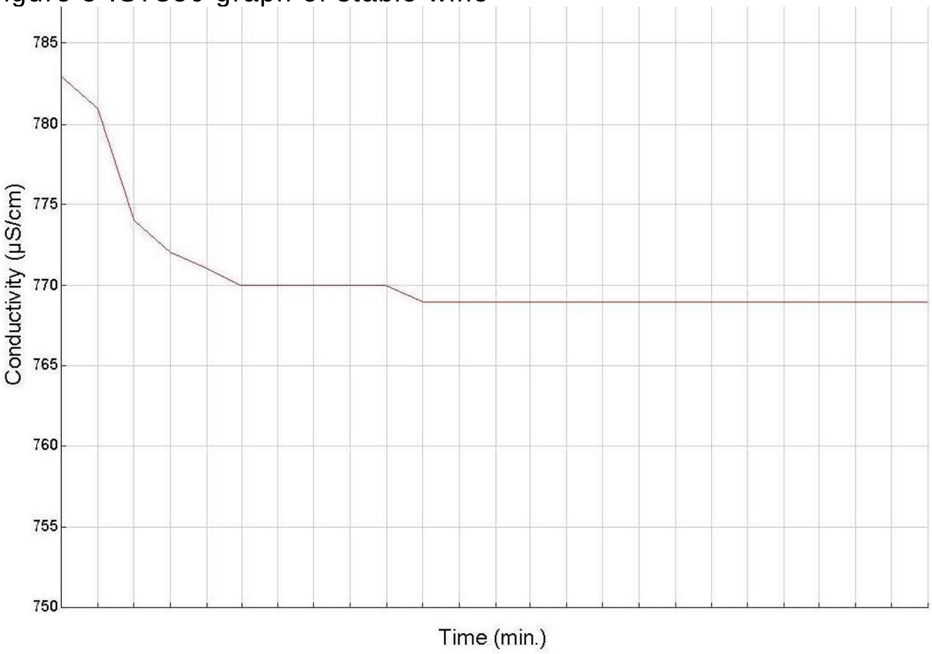
The graph is a representation of the change in conductivity vs the time of the test.

Results

The wine is STABLE if the conductivity drop is < 3 µS/cm between 40 and 120 minutes for White and Rosé wines.
The wine is STABLE if the conductivity drop is < 5 µS/cm between 40 minutes and 240 minutes for Red wine.

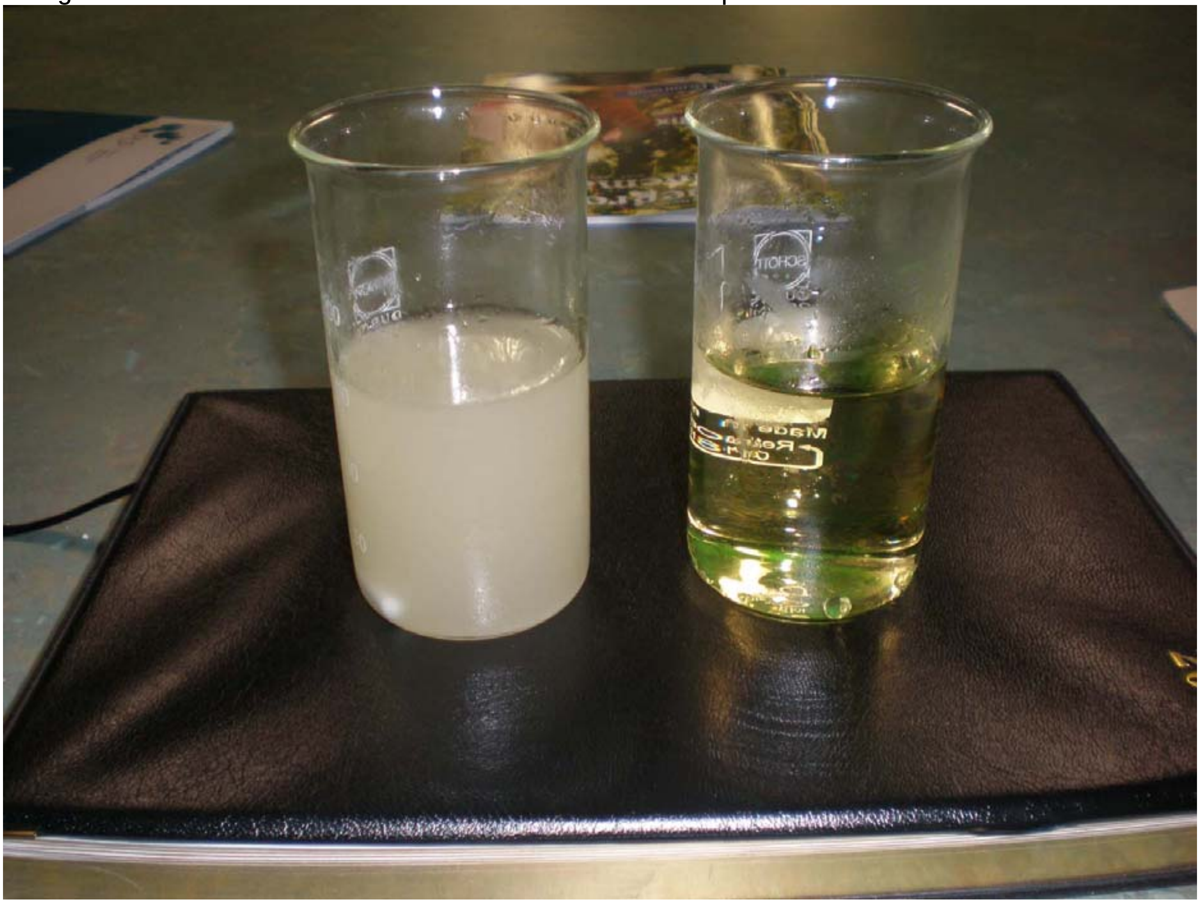
Obviously the wine in Figure 4 was found unstable with a conductivity drop of 83µS/cm between 40 and 240 minutes. Stable wine graphs appear like Figure 5. Note the lack of conductivity change for most of the time of testing.

Figure 5 ISTC50 graph of stable wine



Interestingly at the end if the ISTC50 test when cleaning up the machine and disposing of the sample it is very comforting to observe the tartrate precipitates or lack thereof which correlates with the results see Image 1. The sample on the left is very unstable whilst the one on the right is stable.

Image 1 ISTC50 test Unstable vs Stable – visual comparison



Several journal articles, posters & product promotional literature have been written regarding the new technologies available for tartrate stabilization and have quoted the Stablilab™ and it's DIT% & ISTC50 results as confirmation of how effective they products work, P.Bowyer et al (2009 & 2010), D.Wollan (2010). We have found that as well as this many British retail chains like Tesco's accept very few tartrate stability tests for confirming stability and the ISTC50 is just one of the few they do accept!

Summary

We believe this to be the new standard for Tartrate stability testing as it can be used for all wine styles and gives accurate determinations no matter what stabilization process or product is used to stabilize the wine. It is relatively fast taking 5-6 hours to complete a test and provides qualitative indication of the wines instability unlike the qualitative results from the -4ºC for 3 or 6 days tests. The Stabilab™ is very useful for the determination of Electrodialysis requirements, mannoprotein (Mannostab™, Laffort Australia) and CMC dosage determination via the DIT% test. Also for confirming the stability of all wines including those post treatment by the aforementioned technologies via the ISTC50 test giving a degree of comfort to those winemakers using these new technologies.

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