AWRI Report

Comparison between electrodialysis and cold treatment as a method to produce potassium tartrate stable wine

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1.0 Executive Summary

During July 2010, The Australian Wine Research Institute (AWRI) in conjunction with Memstar and Orlando Wines undertook an evaluation of an electrodialysis system designed to remove potassium tartrate from wine.

Prior to this field study The Australian Wine Research Institute, in conjunction with Memstar, had developed a comprehensive economic model, comparing electrodialysis to traditional cold treatments. The economic modelling indicated clearly that certain operational parameters were critical to the economic results. These parameters included:

- Power Consumption;
- Water Consumption;
- Waste Water;
- Wine Losses;
- Labour Requirements; and
- Sensory Results.

The results of the trial are summarised in the table below.

	Electrodialysis	**Cold Technique**	**Control (un-stabilised)**
Wine Stability	**Commercially Acceptable***	**Commercially Acceptable***	**Commercial Failure†**
Volume of Wine Processed	29,100	29,100	
Performance Metrics			
Power Consumption (kWh)	77	1,761 – 2,968[1]	
Water Consumption (L)	7,683[2]	3,606	
Wastewater (L)	7,683	1,581	
Waste Water Composition			
K mg/L (from water measurements)	1,170	-	
K mg/L (from wine metal analysis)	1,251	4,381	
K Load on treatment Centre (kg)	5.2	7	
Na mg/L (from water measurements)	112	-	
Na mg/L (from wine metal analysis)	42	42	
Wine Potassium Content (mg/L)	395	335	573
Wine Losses (L)	136	424	
Labour Requirements (hrs)	17	9	
Time Taken to process wine (hrs)	17	384	
Sensory Results	Not significantly different		

*Actual result was a Level 1 failure.
† Actual Result was a Level 2 failure

[1] Depending on when stability is deemed acceptable
[2] Water consumption was higher than expected due to the membranes needing a special clean which is normally required at 3 monthly intervals and coincided with the trial.

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2.0 Introduction

During July 2010, The Australian Wine Research Institute (AWRI) in conjunction with Memstar and Orlando Wines undertook an evaluation of an electrodialysis system designed to remove ionic salts from a wine. The trial was run in order to assess the viability of this system as an alternative technique to traditional cold treatments to remove potassium tartrate from wine.

Prior to this field study The Australian Wine Research Institute, in conjunction with Memstar, had developed a comprehensive economic model, comparing electrodialysis to traditional cold treatments. The economic modelling indicated clearly that certain operational parameters were critical to the economic results. These parameters included:

- Power Consumption;
- Water Consumption;
- Waste Water;
- Wine Losses;
- Labour Requirements; and
- Sensory Results.

The use of electrodialysis (ED) as a wine processing method is not a new concept within the wine industry and there are a number of operational setups both in Australia and internationally. The purpose of this study was not to prove the concept; this is well established, but rather to collect robust information on the operational performance of electrodialysis so that comparisons can be made to traditional cooling methods, analysing both the economic and environmental costs associated with each method.

Wine grapes are high in potassium and when wine is produced potassium tartrate exists in supersaturated or near supersaturated quantities. Untreated wine will often develop potassium tartrate crystals especially if exposed to cold temperatures, such as when refrigerated. For this reason, wineries treat their wine at some stage prior to bottling to remove enough of the potassium tartrate to ensure crystals are not formed at colder temperatures.

2.1 Cold Method

Traditionally this potassium tartrate is removed by chilling the wine to very low temperatures, allowing crystals to form. The crystals are then separated from wine. This process requires the wine to be chilled to temperatures as low as -4 ºC and held at these temperatures, often for many days. Cooling to this temperature is a very energy intensive process. Strategies to reduce the cooling load have generally involved adding seed crystals to the wine. Although this is an effective method, seed crystals are expensive and economic viability requires the effective recovery and reuse of crystals.

2.2 Electrodialysis

The ED process uses ion selective membrane sheets clamped between electrodes. Wine flows on one side of the membrane, with an acidified water on the other side. When the electrodes are switched on an electric field is generated which causes the charged ions to move towards the oppositely changed electrode. This causes the potassium, calcium and tartrate ions to migrate out of the wine, across the membrane and into the water stream.

In order to compare these two processing methods, a trial was setup in which wine was treated simultaneously via electrodialysis and the traditional cold technique. The performance of each technique was then measured using a number of different performance parameters.

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3.0 Materials & Methods

The AWRI observed and monitored the operation of an ED unit under demonstration by Memstar in a commercial winery environment. The ED unit used as part of the investigation was designed to process 3000L of wine per hour. The unit was portable in nature, and consisted of three parts:

- A control cabinet;
- The ED stacks; and
- The holding tanks and valve network system.

3.1 Trial design

The trial was conducted utilising a Pinot Noir sparkling base style wine. Two trials were run simultaneously, one utilised the electrodialysis system the other used the cold method/technique.

The cold technique trial utilised an ammonia refrigeration plant equipped with an evaporative condenser. Key operational parameters for this trial reflect the standard operating practices for the winery. These parameters where known are shown below:

3.1.1 Cold Stabilisation

- Cooling time and tank set-point – -4 ºC for a minimum of 3 days
- Insulation type – Polystyrene
- Brine set-points and dead band adjustment – minus 5 +/- 1º C
- Seeding additives - None used
- Tank sizes – 10kL tanks

The ED trial utilised a mobile ED unit provided by Memstar. This unit has a nominal processing rate of 3000L/hour. Key operational parameters for this trial were specified by Memstar personnel based on their experience and typical industry usage.

3.1.2 Electrodialysis

- Wine clarity required – 1-5 micron filtration
- Additives required – Caustic Soda, Nitric Acid, Citric Acid.
- Processing rates – to be determined by Stabilab analysis
- Membrane replacement frequency –As Memstar quote typical membrane life spans in the order of 1000-2000 hrs for the anions and 2000 – 6000 hrs for the cations.

Cost and quality related outputs were monitored as part of the field assessment and included:

- Labour resource used
- Power use
- Water use
- Wastewater volume and quality
- Process yield (product recovery)
- Wine composition (residual tartrates)
- Sensory attributes
- Additive use.

3.2 Boundary of operations being evaluated

For the purpose of this investigation, unstable wine was drawn from a 680kL tank.

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In order for the electrodialysis system to function effectively wine must be pre-filtered to at least 5 micron. This was achieved using a cross flow filter. The cross flow operation was done during the transfer process from the 680kL tank to the 9700L process tanks. The wine, treated traditionally was cross flow filtered after the cold treatment.

The wine from the 680kL tank was sent to six 9700L tanks, three of which were treated using the traditional cold stabilistaion method, three were treated using the electrodialysis system.

As the process for each treatment method is slightly different, figures 3.1 and 3.2 illustrate the boundary of the investigation for each treatment.

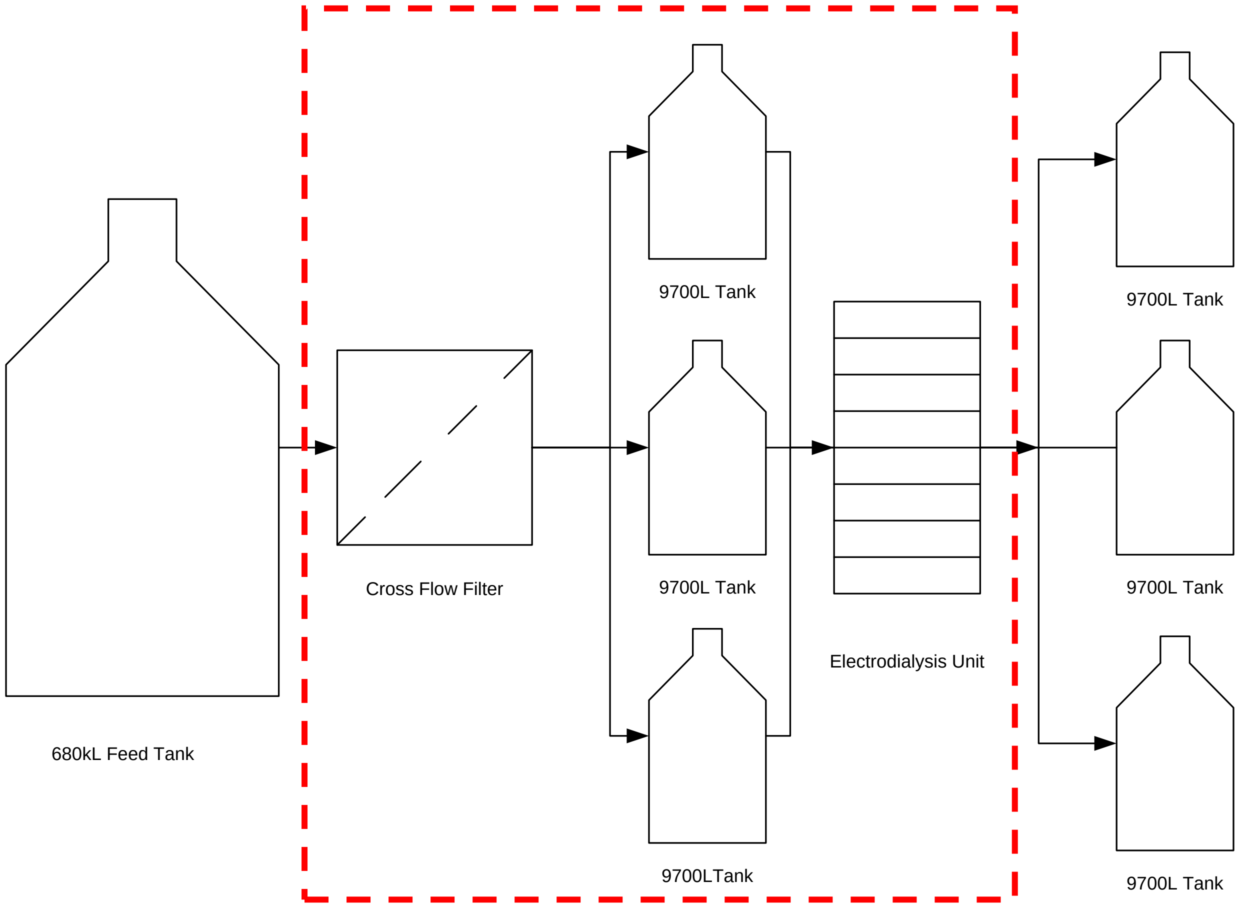

Figure 3.1: ED Process Boundary

Within the process boundary associated with the ED system the following impacts were assessed:

- Wine losses from the cross flow filter, including retentate;
- Wine losses from the ED unit and associated piping;
- Power consumption of the ED unit and associated pumps;
- Water consumption, both as a conductant and for cleaning;
- Waste water composition.
- Cleaning requirements after the 9700L tanks were emptied;
- Cleaning requirements of the ED unit;

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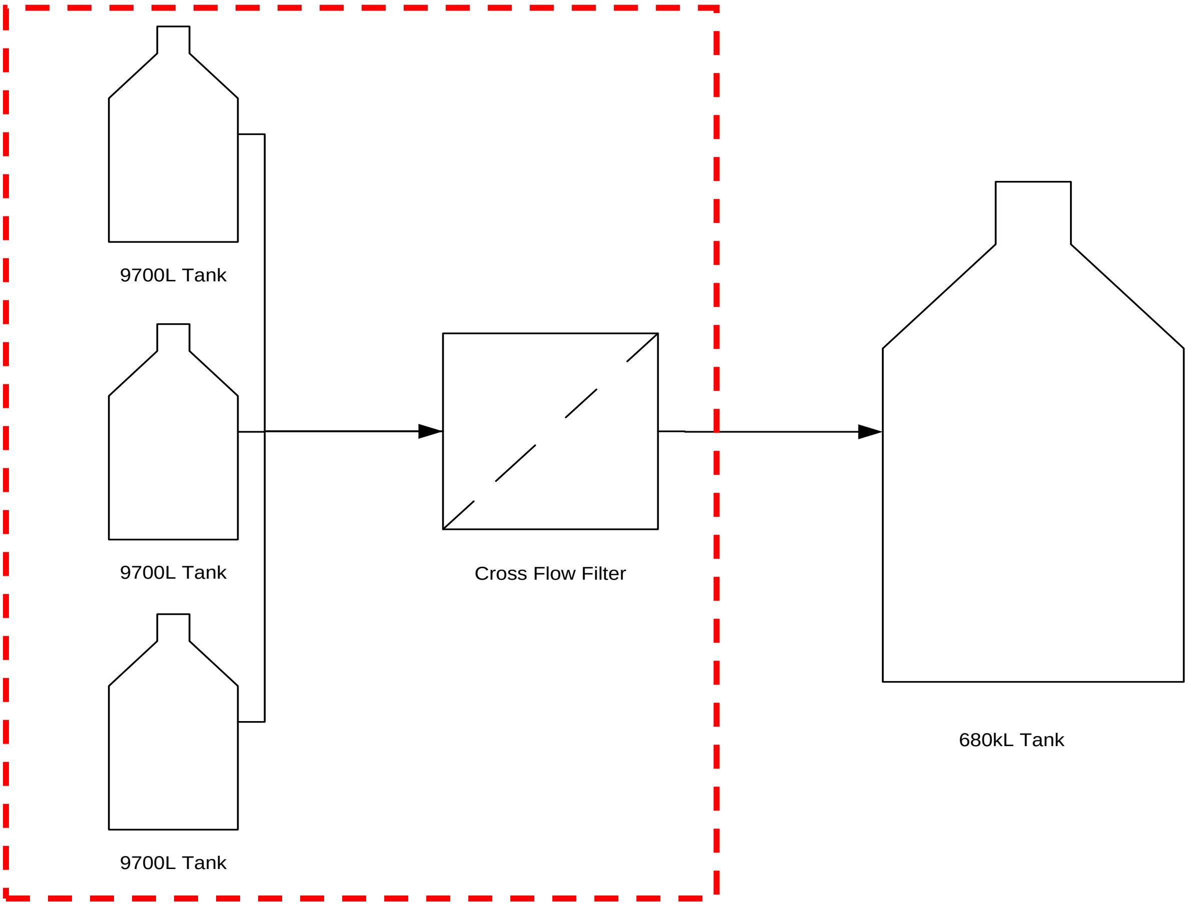

Figure 3.2: Cold Treatment Process Boundary

Within the process boundary associated with the cold treatment the following impacts were assessed:

- Wine losses from the cross flow filter, including retentate;
- Wine losses from the transfer to the cross flow and associated with remaining lees;
- Power consumption of all equipment associated with the cooling system;
- Water consumption associated refrigeration and cleaning;
- Waste water generated;
- Cleaning requirements after the 9700L tanks were emptied.

3.3 Definition of cold stability

Cold stability is defined as per the AWRI Commercial Services Laboratory method. This method is representative of methods used within the wine industry. To test for stability wine is refrigerated for 3 days at -4 degrees C, and inspected for crystallisation. The results are expressed as a 'pass' if no crystalline deposits observed even under an intense light source. Three modes of failure are used, depending on the level of crystallisation observed. These levels are described as:

- **Fail – Level 1:** Borderline fail.
- **Fail – Level 2:** Bad fail.
- **Fail – Level 3:** Crystals visible to the naked eye.

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Although described as a 'fail', wine returning from the cold stability test is usually deemed commercially acceptable if the result is a level 1 failure.

3.4 Measurement method(s) for wine losses
Wine losses associated with this trial were assessed from the following stages.

- For the cold treatment method wine losses included the liquid component of lees left in the tanks and the retentate collected at the cross flow filter post stabilisation.

- For the ED process wine losses included the retentate collected at the cross flow filter and processing wine losses calculated by measuring difference in tank dips.

3.5 Measurement method(s) for waste products
Waste water samples were collected from both the cleaning processes used in this trial and from the ED unit while in operation. Waste water analysis was completed by CSIRO Land and Water.

3.6 Measurement method(s) for power consumption
The measurement of power consumption was completed using a portable power metering devices, fitted to the equipment on-site by winery electricians. The power meter used was a Power Monic PM30, operating with 30sec measurement intervals.

The following equipment was monitored in each scenario.

Metering	**Cold Stabilisation**	**Electrodialysis**
Electricity	Refrigeration Plant Brine pumps Cooling tower, fans and pumps Agitators (as applicable)	Transfer pump(s) Process pump(s) Stack

3.7 Measurement method(s) for water consumption
Water consumption throughout the trial was found to occur in the following events.

Metering	**Cold Stabilisation**	**Electrodialysis**
Water	Cooling tower Tank cleaning	Process waste water Cleaning

In order to measure water consumption associated with the ED unit an in-line flow meter was fitted to the main water feed into the processing area. All water used in the process was fed through this meter. An inline flow meter was installed on the makeup water feed to the cooling tower to measure water consumption in the tower. An additional inline flow meter was fitted to the main feed for the hoses used in tank cleaning.

3.8 Wine quality assessment
To facilitate the wine quality analysis, samples of processed wine were collected as from each of the treatments. These wines were then bottled at the Hickinbotham Roseworthy Wine Science Laboratory under best practice oxygen management conditions. The wines were then stored for six weeks prior to sensory evaluation, to mitigate the impact of "bottle shock."

A sensory difference test (triangle test) was used to determine whether statistically significant differences exist between the treatments.

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4.0 Results & Discussion

4.1 Summary

The results from this experimental work are summarised in the following table.

	Electrodialysis	**Cold Technique**	**Control (un-stabilised)**
Wine Stability	**Commercially Acceptable***	**Commercially Acceptable***	**Commercial Failure†**
Volume of Wine Processed	29,100	29,100	
Performance Metrics			
Power Consumption (kWh)	77	1,761 – 2,968[3]	
Water Consumption (L)	7,683[4]	3,606	
Wastewater (L)	7,683	1,581	
Waste Water Composition			
K mg/L (from water measurements)	1,170	-	
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Sensory Results	Not significantly different		

*Actual result was a Level 1 failure.
† Actual Result was a Level 2 failure

4.2 Wine Analysis

Analyte	**Control**	**Cold Stabilisation Treatment**	**ED Treatment**
Sulphur Dioxide, free (mg/L)	38	41	32
Sulphur Dioxide, total (Mg/L)	87	88	72
Alcohol (% v/v)	11.3	11.4	11.4
Specific gravity	0.99	0.99	0.99
pH	3.09	3.05	3.01
Titratable acid pH 8.2 (g/L)	7.7	7.1	7.3
Titratable acid pH 7.0 (g/L)	7.2	6.8	7
Glucose + Fructose (g/L)	1.1	1.2	1.4

[3] Depending on when stability is deemed acceptable
[4] Water consumption was higher than expected due to the membranes needing a special clean which is normally required at 3 monthly intervals and coincided with the trial.

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Volatile Acidity as Acetic Acid (g/L)	0.37	0.37	0.38

4.3 Power Consumption

Power consumption associated with the ED unit was measured on one phase only, as shown below. Total power consumption was then extrapolated assuming each phase was equal. Each phase of the ED unit consumed 25.6kWhrs of energy to process the three tanks.

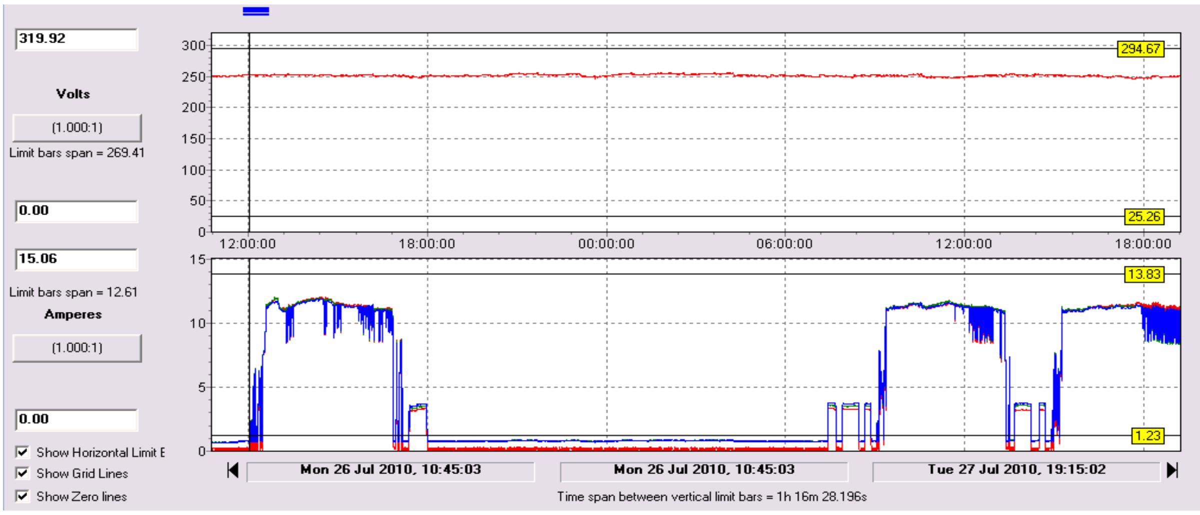

Figure 4.1: ED power Consumption (note the separate peaks corresponding to run time)

The power consumption associated with the refrigeration system was broken into three separate stages:

- Initial pull down- reducing the tank temperature from 14 ºC to -4 ºC.
- Agitation at -4 ºC.
- Settling at -4 ºC.

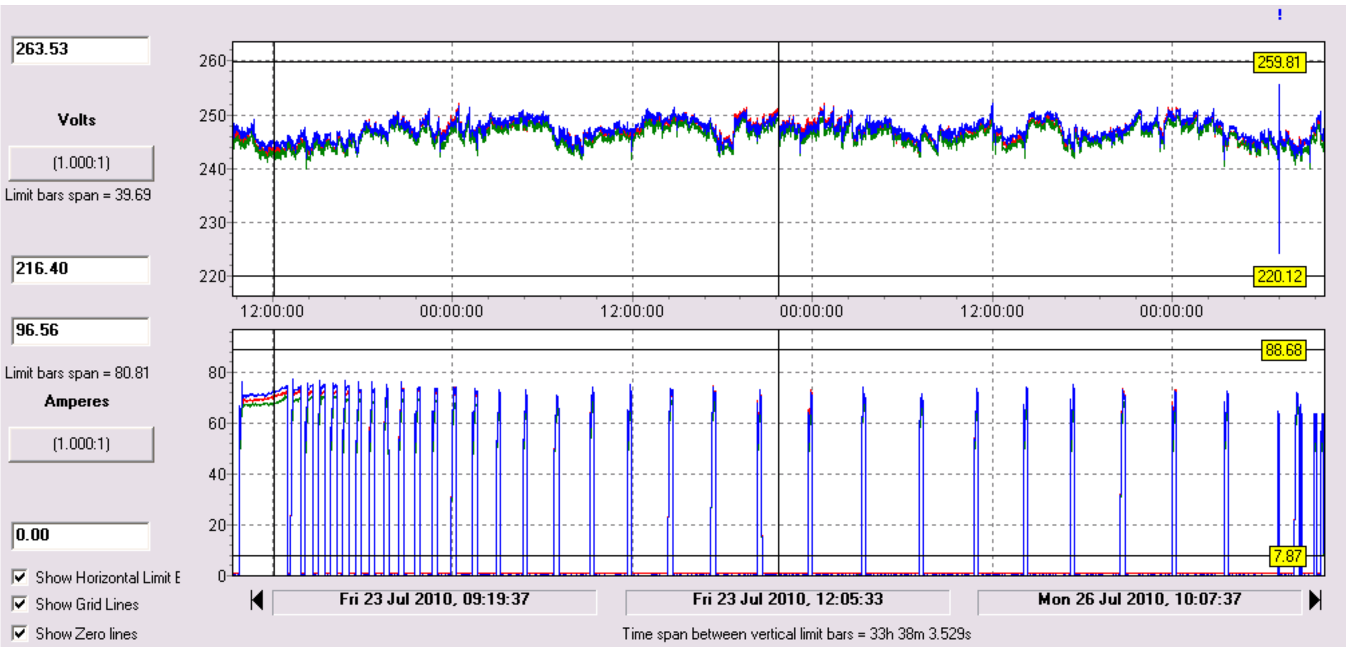

Figure 4.2: Power consumption of refrigeration plant during initial pull down and agitation phases

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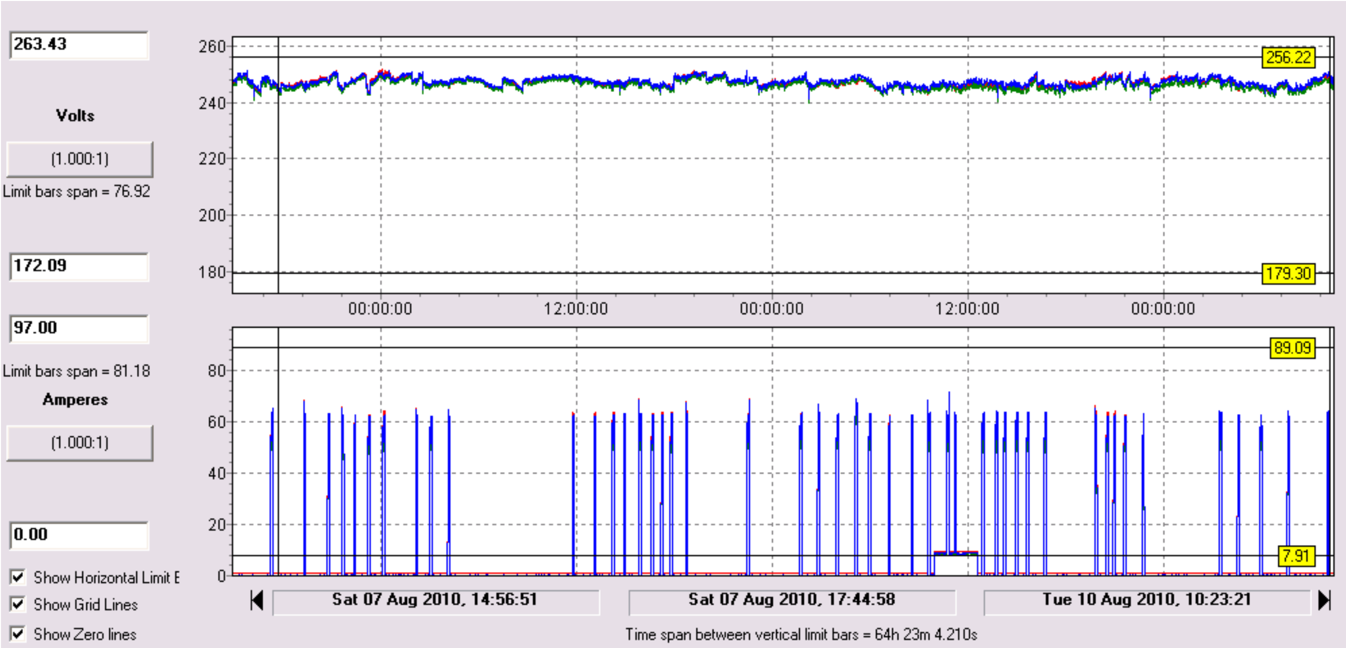

Figure 4.3: Power Consumption - no agitation

These three separate stages each involved different brine pumping and agitation scenarios. Due to the design of the tanks, when agitation was stopped temperatures of -4 ºC were not achieved, leading to continuous circulation of brine.

The following two tables were used to calculate the energy requirements associated with the traditional cold stabilisation method. The first table represents cold agitation for a period of three days, the second represents the actual 10 days that was used as part of this trial.

Cooling Stage	**Brine Pumping (% of time)**	**Agitation (% of time)**	**Time (hrs)**	**Energy (kWhrs)**
Initial Pull Down	100	100	72	785
Agitation at -4ºC	55	100	72[5]	517
Settling at -4ºC	100	0	72	459
Total				1761

Cooling Stage	**Brine Pumping (% of time)**	**Agitation (% of time)**	**Time (hrs)**	**Energy (kWhrs)**
Initial Pull Down	100	100	72	785
Agitation at -4ºC	55	100	240	1724
Settling at -4ºC	100	0	72	459
Total				2968

The biggest uncertainty associated with this trial is defining when a wine is cold stable. The current industry standard for testing cold stability is to observe if crystals have formed after holding the wine for 3 days at -4ºC.

[5] Actual time agitation was used was 240 hours.

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The test is very critical; any observed crystal growth, is deemed a failure, even if the crystals are small enough that they are unlikely to cause a problem in the market place.

In the case of this trial, using the traditional cold techniques we were unable to establish a “pass” even after holding the wine at -4ºC with agitation for 10 days. This was not the case with the wine processed through the ED system which immediately after processing gained a “pass” in the cold stability test although after bottling this wine received a Level 1 Failure.

In our discussions with winemakers we found that practices around cold stabilisation were also varied. For this reason it is difficult to define a standard approach to the practice of cold stability. However, we assumed the following approach which is relatively representative of most practices.

The wine was chilled to -4ºC, and then left on agitation for a period of 3 days. After three days the agitation was switched off and the wine allowed to settle (for 3 days). A sample was then checked for stability, and found to be a level 1 failure. As the results from the ED wine was a clear pass, the tank agitation was then switched on for a further 3 days, before resettling. Again the wine returned a level 1 failure for the cold stability test. The agitation was the switched on for a further 4 days, and the wine allowed to settle and re-checked for stability. The wine again returned a level 1 failure.

Although classified as a failure, the wine is typically accepted by wineries as commercially acceptable if given a level 1 failure. For this reason it is difficult to determine when the cold technique trial should be defined as complete. For this reason the results for power consumption have been shown in two separate ways.

Although some winemakers are likely to cold treat their wine for more than three days, there are very few who are likely to treat the wine for less than three days, therefore the lower value presented in this report is a good indication of a minimum value that might be expected as a result of the traditional cold treatment.

4.4 Water Consumption

Water Consumption was measured during the ED process with a flow meter. The following results were obtained.

Process	Per Tank (averaged)	Total (3 tanks)
Wine Processed (L)	9,700	29,100
Water used during ED processing only (L)	1,379	4,139
Caustic Rinse (L)	519	1,559
Nitric Rinse (L)	236	708
Tank Clean Post Treatment (L)	241	723
Total (L)	2,376	7,129

When running the ED unit the processing speed appeared to be less than that stated by the manufacturer. For each tank processed, the unit was able to function effectively at 3,000L per hour for the first 2 hours, but then would begin to run at much reduced processing rates. This was thought to be due to the membranes requiring a special clean which is normally required at 3 monthly intervals and coincided with the trial. The ED was cleaned at the end of each 9,700L tank. Theoretically the unit should have only needed cleaning after running for 12 hours or after processing 36,000L of wine. The reality in this investigation was that the unit was cleaned after every 9,700L tank.

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This contributed to additional water consumption and additional caustic and nitric acid consumption that should not be theoretically needed.

Water consumption associated with the traditional Cold treatment method was associated with both cleaning and the evaporative condenser.

Process	Total (for all three tanks)
Wine Processed (L)	29,100
Water for evaporative condenser (L)	2,024
Water for solids removal per tank (L)	723
Caustic / Citric Rinse per tank (L)	858
Total Water Consumption for cleaning (L)	3,605

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4.5 Waste Water

The waste water from the ED unit was analysed and found to have the following composition.

	Cl^- (mg/L)	Ca (mg/L)	K (mg/L)	Mg (mg/L)	Na (mg/L)	S (mg/L)	Fe (mg/L)	Mn (mg/L)	P (mg/l)
ED Mains Water	69	17.1	8.65	7.23	42.4	12.2	<0.1	<0.05	0.449
ED Waste Water	407.5	133.75	1105.5	182.5	102.475	160.5	0.6325	2.8875	125.5

The waste water associated with the traditional tank cleaning was assessed by deduction using metal analysis of the wine. The wine before and after each treatment method was analysed using atomic adsorption spectroscopy. The results are as follows.

Treatment	Element	mg/L
Unstable (Control Wine)	Potassium	573
ED Wine	Potassium	395
Cold Treatment wine	Potassium	335

The amount of potassium salts present in the waste water associated with the cold treatment is estimated by mass balance from the information above.

4.6 Wine Losses

Wine Losses were calculated by tank dips and the sum of wine held as retentate post the cross flow filtration system. Wine losses play an important role in the economic evaluation of cold stability systems.

4.7 Labour Requirements

Labour requirements were measured by observing each process.

ED Unit based on 3x9700L tanks	**Hrs**	**Cold Treatment based on 3x9700L tanks**	**Hrs**
Set up hoses, pumps etc	3	Set up hoses, pumps etc	1.5
Cleaning Cycle day 1	1		
Start up Process	0.5	Check, monitor, perform additions	2
Check and Monitor Day 1	3		
Check Monitor Day 2	5	Clean up at completion	1
Cleaning Cycle day 2 - 2 cycles	3	Post stab process (Racking)	3
Clean up at completion	0.45	Clean tank post use (Tartrate removal)	3
Clean tanks post use	1.5		
Total	17.45[6]		10.5

[6] The labour requirements observed in this trial, for the ED system, may overestimate the labour requirements associated with a permanently fixed machine. Additionally if the system was functioning as per its design specification then only 1 cleaning cycle would have been necessary reducing the labour requirements associated with cleaning.

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The labour requirements associated with the ED unit could be thought of similarly to that of a cross flow filtration unit. In order to operate the unit, a specific set of new skills must be acquired and the unit typically needs some assistance and management particularly at the start and end of batches and for cleaning cycles.

4.8 Sensory Results

The following table provides the results from the triangle test.

Table 1. Results of the triangle tests, n=30 responses.

Comparison	Correct Answers	Significance
Control vs Treatment	13	n.s.

n.s. – not significant at 5% ($P < 0.05$) level

From table above it can be seen that there was no significant difference between the Control and Treatment wines. Comments from the tasters did not yield any additional information.

The test had a good statistical power, as calculated after the test, of greater than 0.98 of finding a difference if one existed, given 25% of correct answers above chance. In other words, if 25% of assessors could perceive an effect of the treatment, the test would have had greater than 98% likelihood of finding the difference.

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5.0 Summary

The key findings from this trial summarised below:

- There was no sensorial difference in the wines treated by electrodialysis compared to the cold technique;
- Electrodialysis offers significant advantage in the power consumption;
- Electrodialysis offers significant advantage in minimising wine losses;
- Waste water volume is higher for electrodialysis, however the total salt loading in the waste water is lower; and
- The labour requirements to operate the electrodialysis unit are higher than conducting the cold technique.

Based on the results presented in this report, electrodialysis appears to offer a viable alternative method to tartrate stabilise wines. The specific benefits of electrodialysis will depend on the specific operational requirements of individual wineries, however there are clear opportunities to realise improved economic and environmental performance in the tartrate stabilisation of wine.

The consumption of water associated with the ED system should be considered in light of the life cycle of wine production and represents only a minor addition to the overall water footprint of a bottle of wine. Life cycle assessment could be used to further illustrate this. Life cycle assessment could also be used to develop a generic statement about the environmental credentials of both ED and traditional cold stability using a common metric.

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