

# A Comparative Study on the Evolution of Wine Aged for 12 Months in a Flextank vs. a Two-Year-Old Oak Barrel

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**Abstract:** The objective of this study was to compare the performance of an ECO15 standard maturation weight HDPE (high density polyethylene) Flextank to that of a similarly sized two-year-old barrel, both equipped with an oak adjunct. Performance was assessed both quantitatively by measuring and analyzing pertinent enological parameters and qualitatively by tasting and evaluating the wine throughout the duration of the study.

This study demonstrates that, for the first 6 months, the ECO15 Flextank mimics a 55-L (14.5-gal) two-year-old oak barrel from an oxygen transfer rate (OTR) perspective provided that the lid is tightly sealed. As the barrel imparted more tannins, the two wines exhibited slightly different behaviors after 6 months based on free sulfur dioxide (SO<sub>2</sub>) and colorimetric analyses. The extra tannins may have contributed to greater binding of free SO<sub>2</sub> and less reacting with other wine compounds, more pronounced red color and color intensity as measured quantitatively but not detectable by visual inspection, and greater fining action. The barrel shape is also believed to have contributed to the greater measured clarity although, again, not detectable by visual inspection of the wine in a glass. Surprisingly, there were much heavier tartrates in the Flextank wine after 6 months although temperature and alcohol concentration were similar in both wines. The lower phenol content and acidity made the Flextank wine taste smoother after 6 months. There were no organoleptic differences that stood out even though a concentration effect was expected in the barrel wine due to evaporative losses; both wines recorded similar alcohol concentrations throughout the study. As expected, due to the headspace that forms in barrels, the barrel wine produced slightly more volatile acidity (VA), but well below detection threshold.

It is recommended that a little olive oil be applied to the gasket for a tighter fit to avoid oxygen ingress; a suitably designed wrench is also recommended to ease removal of the lid. A Flextank dry airlock is recommended in lieu of a rubber bung seated in the threaded port until the wine is sufficiently degassed, and then replaced with a solid Flextank plug. Teflon tape should be applied on threaded parts. And given the large surface area and potentially large headspace at the top of the Flextank, the tank should be filled to the very top via the small port with the large lid in place.

**Key words:** Flextank, ECO15, HDPE tank, oak barrel, oxygen transfer rate (OTR), WineStix

**Introduction:** Oak barrels have long been used in winemaking. There exists a natural harmony among oak wood compounds, wine constituents, and the oxygen transferred into barrels that, in general, results in greater wines compared to their non-oak-aged counterparts. The main wine constituents of interest here are polyphenols, and more specifically, tannins, the substances responsible for the drying, puckery sensation in the mouth, and anthocyanins, the red-color pigment molecules.

Aside from the plethora of aromas and flavors that oak and particularly toasted oak impart to wine, the infinitesimally small amount of oxygen entering barrels from the outside environment, a phenomenon known as micro-oxidation, improves and stabilizes

color, smoothens tannins for a softer “mouthfeel,” i.e., the combined sensation of tannins, acids, ethanol, and polysaccharides, and improves aging potential. Those aromas and flavors also become more concentrated due the slow evaporative loss of ethanol and water — the “angel’s share.”

But oak barrels are a significant investment; acquisition costs are high, they require care and maintenance, and they have a limited lifespan in that they become “neutral” after several uses and no longer impart those much-desired aromas and flavors.

Oak powder, chips, staves, cubes, balls and spirals — collectively referred to as oak adjuncts — are inexpensive alternatives to oak barrels, driving the cost of “oak-aged” wines down significantly.

Oak adjuncts can be used either in inert vessels, such as stainless steel tanks, or in neutral oak barrels. There is no oxygen ingress into inert vessels and therefore oak adjuncts are only used for imparting oak aromas and flavors. In neutral barrels, micro-

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oxidation still occurs, albeit at a slower rate, and can therefore better replicate newer barrels when used with adjuncts.

There are other technologies and products that can replicate the benefits of oak-barrel aging. Micro-oxygenation, known as MOX, uses a specially designed control unit to inject minuscule amounts of oxygen into stainless steel tanks fitted with oak adjuncts, typically long oak staves.

HDPE (high density polyethylene) vessels can be manufactured to similar oxygen permeation rates, or oxygen transfer rates (OTR), as oak barrels, and can therefore be used with oak adjuncts to mimic micro-oxidation in barrels. HDPE vessels are manufactured from resin into extremely durable polyethylene that can last significantly longer than the useful life of an oak barrel and which do not affect the aromas and flavors of wine, unlike the first generation of HDPE tanks (Carey 2009).

Some studies have examined the effects of oxygen transfer through HDPE material (Nguyen et al. 2010) and concluded that HDPE tanks in combination with oak adjuncts (Cronje 2020; del Alamo-Sanza et al. 2015) are a viable alternative for maturing wines similarly as in oak barrels. Cronje (2020) interviewed and reported that two South African winemakers found that new-barrel matured wines have sweeter tannins and a long finish but that red wines matured in HDPE tanks have better color extraction.

Flex tanks, manufactured by Smak Plastics, Inc. (flextank.com), is one such line of HDPE tank products and the focus of this study. Their standard maturation weight (SMW) HDPE vessels are designed to have an oxygen permeation rate or OTR similar to that of a typical second-year barrel when used at a nominal cellar temperature of approximately 13°C (55°F) (Flex tank 2020). Based on verbal communication with Flex tank, “second-year barrel” is interpreted to mean “2-year-old barrel.” Their heavyweight tanks are heavier vessels with thicker walls with oxygen permeation rates about half that of SMW vessels, comparable to “neutral” barrels.

The objective of this study is to compare the performance of a SMW Flex tank tank to that of a similarly sized two-year-old barrel, both equipped with an oak adjunct. Performance is assessed both quantitatively by measuring and analyzing pertinent enological parameters and qualitatively by tasting and evaluating the wine throughout the duration of the study.

## Materials and Methods

**Enological Parameters:** Several enological parameters that could provide clues as to any potential differences in wine evolution between the barrel and the Flex tank batches were measured and monitored throughout this study.

Total Acidity (TA), expressed in g/L as tartaric acid equivalents, is a measure of the concentration of all fixed and volatile acids. Changes in TA would primarily be due to potassium bitartrate (KHT) formation and precipitation during aging, more so if the wines are subjected to colder temperatures, and possibly from any changes in volatile acidity (VA).

Volatile Acidity (VA), expressed in g/L or mg/L as acetic acid equivalents, is a measure of the concentration of all volatile (steam-distillable) acids. Increasing VA levels would point to increasing acetic acid amounts resulting from the activity of acetic acid bacteria, which thrive in the presence of oxygen, or from chemical oxidation of ethanol into acetaldehyde then into acetic acid, which would point to excessive oxygen ingress and exposure. Acetaldehyde concentrations could not be measured in this study; special instrumentation and analytical techniques are required.

Free sulfur dioxide (SO<sub>2</sub>), or FSO<sub>2</sub>, is a measure of the concentration of molecular SO<sub>2</sub> (MSO<sub>2</sub>) and bisulfite ions (HSO<sub>3</sub><sup>-</sup>) collectively protecting wine against microbial and chemical spoilages, respectively. Bisulfite ions react with oxygen, more specifically with its radicals, e.g., hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), and with oxidized polyphenols, such as brown-colored *o*-quinones, causing them to revert to their colorless forms. These reactions cause FSO<sub>2</sub> to be consumed and to drop. FSO<sub>2</sub> drops further as bisulfite ions also bind with certain wine compounds, such as acetaldehyde and polyphenols, and cause an increase in bound SO<sub>2</sub> (BSO<sub>2</sub>). Tannins and anthocyanins are strong SO<sub>2</sub> binders, though acetaldehyde is a much stronger binder (Blouin 2014). Since similar wines are expected to have bisulfite ions bind similarly, any differences in FSO<sub>2</sub> measurements between batches could point to differences in oxygen consumption, which would mean oxygen is permeating into vessels at different rates. FSO<sub>2</sub> could drop further in barrels if not topped up promptly and regularly as molecular SO<sub>2</sub> fills the headspace until equilibrium is re-established. That MSO<sub>2</sub> is lost every time barrel bungs are pulled.

Total sulfur dioxide (TSO<sub>2</sub>) is a measure of the total amount of SO<sub>2</sub>; it is the sum of FSO<sub>2</sub> and BSO<sub>2</sub>. By measuring TSO<sub>2</sub> and calculating BSO<sub>2</sub>, the latter can give important clues as to any differences in binding between the two batches of wine. For example, if oak wood from the barrel is still contributing tannins, BSO<sub>2</sub> in the barrel batch may be higher than the Flex tank batch. Similarly, BSO<sub>2</sub> may increase if there is oxidative spoilage with the formation of acetaldehyde.

FSO<sub>2</sub>, MSO<sub>2</sub>, BSO<sub>2</sub> and TSO<sub>2</sub> are all expressed in mg/L (equivalent to ppm).

Dissolved Oxygen (DO), expressed in mg/L, is a measure of the instantaneous amount of oxygen dissolved in wine. Any spikes in DO would point to an oxygen ingress problem. And a low DO coupled with a low FSO<sub>2</sub> could mean that oxygen has been consuming FSO<sub>2</sub>, again pointing to an oxygen ingress problem. The total amount of oxygen consumed in each batch over the course of the study could not be measured; special instrumentation and analytical techniques are required.

pH is a measure of a wine’s microbiological stability; the higher the pH, the greater the risk of microbial deviations. The optimal amount of FSO<sub>2</sub> needed to protect wine with sufficient MSO<sub>2</sub> is a function of pH. Greater amounts of FSO<sub>2</sub> are needed at higher pH since the molecular fraction, i.e., MSO<sub>2</sub>, is smaller at higher pH. pH changes may also point to changes in acidity, either TA or VA, due to, for example, tartrates or acetic acid

forming, respectively. A drop in TA due to KHT forming and dropping as tartrates can cause pH to increase or decrease depending on whether wine pH is above or below 3.65 — the approximate pH at which bitartrate ions ( $\text{HT}^-$ ) are at maximum concentration. Color is also affected by pH: as pH increases, there is a drop in red color and intensity as anthocyanins shift towards their colorless forms.

Ethanol content, expressed as a percentage of ethanol to wine volume (% ABV), is a measure of the amount of ethanol in wine. A decrease in % ABV can result from microbial or chemical transformations to acetaldehyde and acetic acid in the presence of oxygen. There are also evaporative losses of ethanol from the barrel to the environment, but this is difficult to measure as water too evaporates to the outside of the barrel.

Turbidity, expressed in Nephelometric Turbidity Units (NTU), is a measure of the degree of turbidity, or clarity. Since the barrel and Flextank have different physical properties — a “bulging” cylindrical shape on the horizontal axis versus a completely cylindrical shape on the vertical axis — it can be expected that precipitable matter can sediment at different rates.

Color evolution is used to assess aging in wines. Young red wines are characterized by a reddish, somewhat purple color. As wine ages, polyphenols oxidize into their brown-colored forms, or *o*-quinones, which, in the absence of FSO<sub>2</sub>, can be seen by an orange-hued tint at the rim, then to brownish hues as the wine ages and exceeds its ability to preserve red color. A very low FSO<sub>2</sub> (below 10 mg/L) or large FSO<sub>2</sub> drops are signs of impending oxidative spoilage, but several color parameters are used to better gauge color evolution and assess a potential browning problem.

Red wines are characterized by three absorbance peaks ( $A_\lambda$ ) in the visible spectrum at wavelengths ( $\lambda$ ) of 420, 520 and 620 nm, which correspond to the yellow, red and purple/blue components of color, respectively. Absorbance measurements are expressed in absorbance units (a.u.). Young red wines display a red color with some purple hues; quantitatively, that translates into low  $A_{420}$  and high  $A_{520}$ ;  $A_{620}$  is relatively much smaller than  $A_{420}$  and  $A_{520}$ . As wine ages, it slowly sheds some of its red color and its purple hues ( $A_{520}$  and  $A_{620}$  decrease), and moves progressively towards yellow, orange and brown colors ( $A_{420}$  increases). These absorbance measurements are used to calculate several other key parameters for assessing red wines: color intensity, hue, blue index and brilliance of red.

Color Intensity (IC), expressed in absorbance units (a.u.), is a measure of the intensity of color and is calculated here as the sum of  $A_{420}$ ,  $A_{520}$  and  $A_{620}$ ; there is a variation of this parameter that is the sum of  $A_{420}$  and  $A_{520}$ , but it is mainly used for older wines. Light-colored reds will have IC values in the range 3–5; medium-colored reds will have IC values in the range 5–8; and deep-colored reds will have IC values in the range 8–12 and higher. Since  $A_{420}$  and  $A_{520}$  change in opposite directions (and that  $A_{620}$  is small) during wine aging, IC can change either way; color intensity does not necessarily decrease with aging.

Hue (H) is a measure of the degree of color evolution and is calculated as the ratio of the yellow component of color to the red component, i.e.,  $A_{420}/A_{520}$ . Young wines will have H values below

0.8, and increasing H values, especially beyond 1.0, are indicative of oxidation and increasing browning.

Blue Index is a calculation of the ratio of the purple/blue component of color to the red component, i.e.,  $A_{620}/A_{520}$ , and provides an indication of changes in purple/bluish color during wine aging.

Brilliance of Red (dA), expressed as a percentage, is a measure of red color “brightness,” and is calculated as a ratio of yellow and purple to red as  $[1 - ((A_{420} + A_{620}) / (2 \times A_{520}))] \times 100$  (Ribéreau-Gayon et al. 2012). Young wines have dA values in the range 40–60%.

Total Phenol Index (TPI) is a measure of the amount of polyphenols in wine, and can point to differences in tannin reactions, and possibly further tannin extraction from oak wood of the barrel or oak adjuncts. Light-bodied, low-tannin wines will have TPI values in the range 25–30; medium-bodied wines with good tannic structure will have TPI values in the range 30–50; and full-bodied, tannin-loaded wines will have TPI values over 50.

**Study Methodology:** Wine from Touriga Nacional from the 2019 vintage from Lodi (CA) was vinified from frozen must purchased from Musto Wine Grape Company (Hartford, CT). Following completion of both the alcoholic and malolactic fermentations, the wine was stabilized with potassium metabisulfite ( $\text{K}_2\text{S}_2\text{O}_5$ ) according to pH with 0.5 mg/L MSO<sub>2</sub> and a 100% adjustment to account for binding. The base wine was analyzed for the enological parameters described previously: 13.9% ABV, pH 3.65 with a TA of 6.86 g/L, of which 0.227 g/L (226.9 mg/L) is VA, FSO<sub>2</sub> of 25.3 mg/L, TSO<sub>2</sub> of 54.0 mg/L, turbidity of 13.0 NTUs,  $A_{420}$  of 2.240 a.u.,  $A_{520}$  of 3.080 a.u.,  $A_{620}$  of 0.640 a.u., IC of 5.96 a.u. with H of 0.73, Blue Index of 0.21 and dA of 53.2%, and TPI of 44.1.

The wine was then re-adjusted for FSO<sub>2</sub> according to pH and DO, using a binding adjustment factor of 33%, to 54.2 mg/L and then transferred to a 15-gal (56.7-L) SMW ECO15 Flextank purchased from Flextank (Vancouver, WA), and a 2-year-old 55-L (14.5-gal) Boutes oak barrel, which had previously and continuously held two batches of the same wine (Sangiovese) for approximately 24 months.

To compensate for possibly different oxygen uptake and FSO<sub>2</sub> consumption during the vessel-filling operations, FSO<sub>2</sub> and DO levels were re-measured after the transfers, and FSO<sub>2</sub> adjusted to target levels based on pH and DO of the now separate batches. This method of managing FSO<sub>2</sub> — as opposed to adjusting both batches to the same FSO<sub>2</sub> level — was to allow maintaining the same MSO<sub>2</sub> in both wines. As pH and DO may change in each vessel, wines may quite possibly require different FSO<sub>2</sub> levels. Therefore, comparisons of FSO<sub>2</sub> consumption are based on relative changes — not absolute changes.

A demijohn-sized Allier French Oak, Medium Plus Toast WineStix purchased from Peter DeVivi Productions (Waterloo, NY) was introduced into each vessel. Each WineStix was trimmed by 5 cm (2 in.) so that the one for the barrel could be completely inserted.

This point was the start of the study, referred henceforth to as T0. Each successive month is labeled as T0+i, where  $i=1, 2, \dots, 12$ .

WINE	Date	Time + months	TA (g/L)	VA (mg/L)	pH	%ABV	Adjusted		TSO2 (mg/L)	DO (µg/L)	NTU	A <sub>420</sub> (a.u.)	A <sub>520</sub> (a.u.)	A <sub>620</sub> (a.u.)	IC	H	Blue Index	dA (%)	TPI
							FSO2 (mg/L)	FSO2 (mg/L)											
Base Wine	3-Dec-19	T0	6.86	226.9	3.65	13.9	25.3	54.2	54.0	1420	13.0	2.240	3.080	0.640	5.96	0.73	0.21	53.2	44.1
Barrel	3-Dec-19	T0			3.65		36.3	55.7		1810									
Barrel	10-Jan-20	T0+1			3.66		34.8	38.7		85									
Barrel	10-Feb-20	T0+2			3.70		32.2	42.3		85									
<b>Barrel</b>	<b>9-Mar-20</b>	<b>T0+3</b>	<b>6.18</b>	<b>314.9</b>	<b>3.64</b>	<b>14.0</b>	<b>32.2</b>	<b>36.7</b>	<b>87.7</b>	<b>80</b>	<b>1.63</b>	<b>2.160</b>	<b>2.760</b>	<b>0.530</b>	<b>5.45</b>	<b>0.78</b>	<b>0.19</b>	<b>51.3</b>	<b>42.3</b>
Barrel	10-Apr-20	T0+4			3.56		29.5	30.6		70									
Barrel	10-May-20	T0+5			3.71		27.1	42.7		65									
<b>Barrel</b>	<b>10-Jun-20</b>	<b>T0+6</b>	<b>5.62</b>	<b>343.1</b>	<b>3.61</b>	<b>14.0</b>	<b>28.0</b>	<b>36.2</b>	<b>92.7</b>	<b>65</b>	<b>1.27</b>	<b>2.280</b>	<b>2.860</b>	<b>0.572</b>	<b>5.71</b>	<b>0.80</b>	<b>0.20</b>	<b>50.1</b>	<b>43.3</b>
Barrel	10-Jul-20	T0+7			3.68		27.0	40.1		70									
Barrel	9-Aug-20	T0+8			3.61		27.2	34.2		70									
<b>Barrel</b>	<b>7-Sep-20</b>	<b>T0+9</b>	<b>5.74</b>	<b>341.0</b>	<b>3.68</b>	<b>14.2</b>	<b>28.4</b>	<b>39.8</b>	<b>106.5</b>	<b>85</b>	<b>1.41</b>	<b>2.370</b>	<b>2.890</b>	<b>0.582</b>	<b>5.84</b>	<b>0.82</b>	<b>0.20</b>	<b>48.9</b>	<b>50.0</b>
Barrel	10-Oct-20	T0+10			3.64		27.1	36.3		80									
Barrel	10-Nov-20	T0+11			3.65		25.9	37.2		80									
<b>Barrel</b>	<b>10-Dec-20</b>	<b>T0+12</b>	<b>5.42</b>	<b>364.7</b>	<b>3.63</b>	<b>14.3</b>	<b>27.1</b>	<b>39.0</b>	<b>128.3</b>	<b>80</b>	<b>1.18</b>	<b>2.450</b>	<b>2.920</b>	<b>0.576</b>	<b>5.95</b>	<b>0.84</b>	<b>0.20</b>	<b>48.2</b>	<b>45.6</b>
Flextank	3-Dec-19	T0			3.65		37.3	55.6		1770									
Flextank	10-Jan-20	T0+1			3.67		34.5	39.6		144									
Flextank	10-Feb-20	T0+2			3.70		29.8	42.3		85									
<b>Flextank</b>	<b>9-Mar-20</b>	<b>T0+3</b>	<b>6.21</b>	<b>269.8</b>	<b>3.63</b>	<b>14.0</b>	<b>31.1</b>	<b>35.9</b>	<b>86.8</b>	<b>80</b>	<b>1.93</b>	<b>2.130</b>	<b>2.720</b>	<b>0.506</b>	<b>5.36</b>	<b>0.78</b>	<b>0.19</b>	<b>51.5</b>	<b>41.3</b>
Flextank	10-Apr-20	T0+4			3.56		27.1	30.7		95									
Flextank	10-May-20	T0+5			3.73		27.9	44.7		65									
<b>Flextank</b>	<b>10-Jun-20</b>	<b>T0+6</b>	<b>5.33</b>	<b>278.5</b>	<b>3.63</b>	<b>14.0</b>	<b>33.5</b>	<b>37.8</b>	<b>100.4</b>	<b>65</b>	<b>1.42</b>	<b>2.110</b>	<b>2.610</b>	<b>0.518</b>	<b>5.24</b>	<b>0.81</b>	<b>0.20</b>	<b>49.7</b>	<b>42.3</b>
Flextank	10-Jul-20	T0+7			3.69		28.4	41.1		80									
Flextank	9-Aug-20	T0+8			3.64		30.9	36.6		70									
<b>Flextank</b>	<b>7-Sep-20</b>	<b>T0+9</b>	<b>5.37</b>	<b>264.2</b>	<b>3.70</b>	<b>14.2</b>	<b>28.4</b>	<b>41.6</b>	<b>111.8</b>	<b>75</b>	<b>1.50</b>	<b>2.280</b>	<b>2.780</b>	<b>0.524</b>	<b>5.58</b>	<b>0.82</b>	<b>0.19</b>	<b>49.6</b>	<b>45.3</b>
Flextank	10-Oct-20	T0+10			3.67		30.9	38.9		70									
Flextank	10-Nov-20	T0+11			3.69		28.5	40.7		70									
<b>Flextank</b>	<b>10-Dec-20</b>	<b>T0+12</b>	<b>5.02</b>	<b>258.3</b>	<b>3.69</b>	<b>14.2</b>	<b>31.0</b>	<b>44.4</b>	<b>120.3</b>	<b>70</b>	<b>1.59</b>	<b>2.180</b>	<b>2.580</b>	<b>0.518</b>	<b>5.28</b>	<b>0.84</b>	<b>0.20</b>	<b>47.7</b>	<b>43.6</b>

**Table 1** Data collected over a 12-month period for a similar wine aged in a 2-year-old oak barrel and in a standard maturation weight (SMW) HDPE ECO15 Flextank.

Until T0+5, FSO2 was adjusted for DO based on a stoichiometric factor of 4, i.e., theoretically, 4 mg FSO2 are consumed for every 1 mg DO. A factor of 2.5 was used starting at T0+6 to reflect the average FSO2 consumption reported in studies and the literature (Pascal et al. 2019; Schneider 2008).

The dry airlock supplied with the Flextank was initially used instead of fitting a rubber bung with a wet airlock on the threaded port on the large lid to minimize the possibility of air ingress from around the threads. The lid is equipped with a gasket for sealing the tank, and must be tightened to 25 foot pounds for an airtight seal. A Flextank-supplied solid (green) cap was then installed midway between T0+1 and T0+2 to replace the dry airlock. Teflon tape was applied to all threaded parts to minimize air ingress and the possibility of leakage through the top cap should wine expansion occur. This Flextank model is equipped with a threaded ½-inch ball valve and hose barb at the bottom for racking.

Both batches were stored in a temperature-controlled cellar at 13°C (55°F) with relative humidity between 55% and 75%.

The barrel was topped up every two weeks and after taking samples for analysis. The same wine reserved in a separate vessel was used for topping. It is estimated that approximately 2.5 L, or 4.5% of the total wine volume in barrel, was used for topping. The Flextank was topped up every month after taking samples for analysis, and here too, using the same wine reserved in a separate vessel. It is estimated that approximately 1.8 L, or 3.2% of the total wine volume in the Flextank, was used for topping. These

topping volumes were not used to make any adjustments to enological parameters measured in this study.

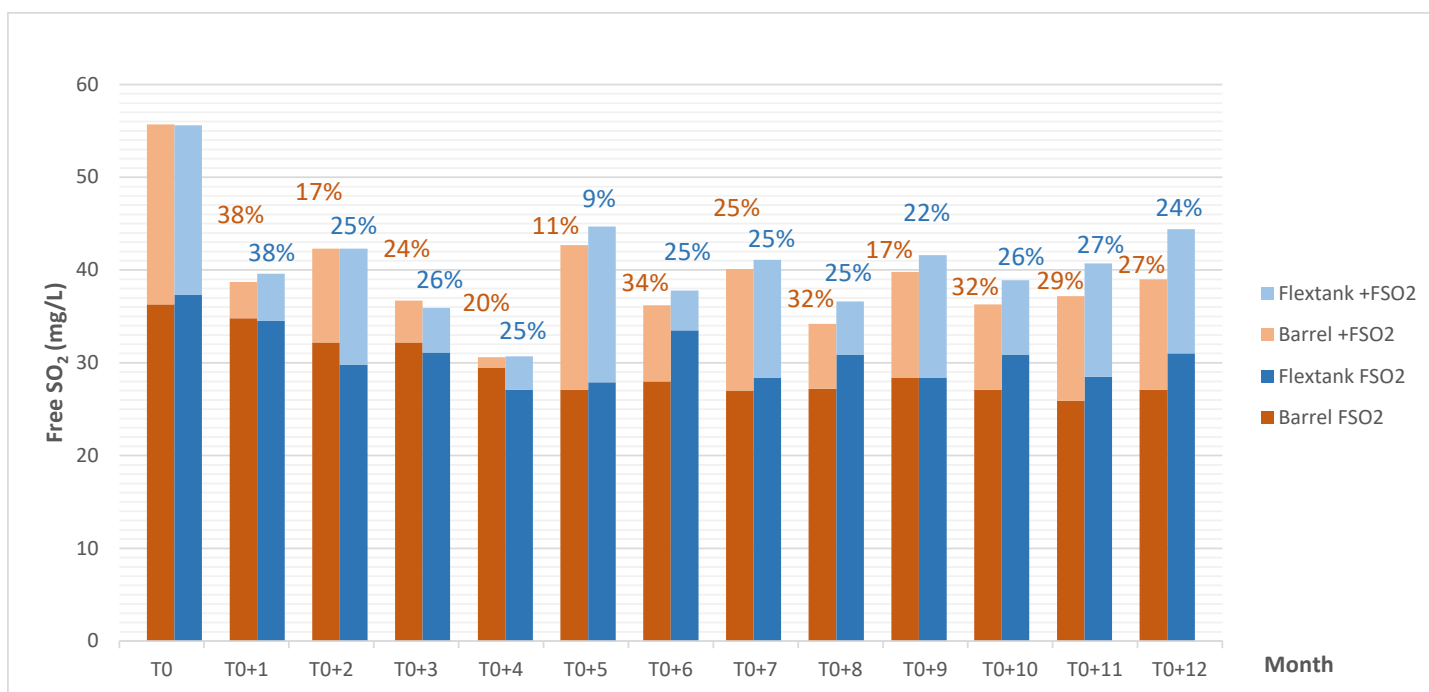
At one-month intervals (T0+1, T0+2, T0+3, etc.), pH, FSO2 and DO were measured in each batch to determine FSO2 consumption and losses and instantaneous oxygen levels, then FSO2 was adjusted back to the proper level based on pH and DO.

At three-month intervals (T0+3, T0+6, etc.), in addition to the parameters measured at one-month intervals, TA, VA, TSO2, % ABV, turbidity, color parameters and phenol index were measured. Sensory evaluations were also performed to assess evolution of each wine using qualifiers of appearance, aroma and bouquet, taste, aftertaste and overall impression. The tastings were not conducted blind.

All parameters were measured once only, and repeated only to confirm what may possibly have been perceived as an erroneous measurement. Data for the measured parameters for the base wine and the barrel and Flextank batches at T0 and over the course of 12 months are presented in Table 1.

**Test Equipment:** TA, pH, FSO2 and TSO2 were measured using a Hanna HI 902 Automatic Potentiometric Titration System. The pH electrode was calibrated at 3.00, 4.01, 7.01 and 10.01 using Hanna buffer solutions. TA was measured using a fixed endpoint of pH 8.2; samples were degassed prior to analysis.

FSO2 and TSO2 were measured using the Ripper method with an ORP electrode. BSO2 was calculated as the difference between TSO2 and FSO2 measurements.



**Figure 1** FSO<sub>2</sub> changes in wines aged in barrel and Flextank over a 12-month period. The solid bars represent FSO<sub>2</sub> measured; lighter-colored bars on top of solid bars represent the amount of FSO<sub>2</sub> added to bring levels back up to the required levels according to pH and DO.

For VA, samples were steam-distilled into 100 mL of distillate using a Cash Still to collect volatile acids. The distillate was then titrated similarly as TA using the Hanna HI 902 unit.

Potassium (K<sup>+</sup>) was measured using the Hanna HI 902 unit with a potassium ISE (Ion Selective Electrode). This was not part of the parameters planned to be measured; it was only measured at T0+6 when considerable tartrates were noticed in the Flextank batch at racking.

% ABV was measured using a Dujardin-Salleron ebulliometer with calibration against distilled water.

DO was measured using a NomaSense O<sub>2</sub> P300 Analyzer and dipping probe equipped with an optical sensor calibrated to ambient air.

NTU was measured using a Hach 2100P Turbidimeter calibrated using Formazin standards.

Color and polyphenol analyses were carried out using a Thermo Scientific Genesys 10S UV-Vis spectrophotometer with all calibrations done against distilled water. A<sub>420</sub> and A<sub>520</sub> measurements were made without dilution using 1-mm (pathlength) quartz cuvettes; A<sub>620</sub> measurements were made without dilution using 5-mm quartz cuvettes. All reported measurements are normalized as if taken with 10-mm cuvettes. TPI was measured using the Folin-Ciocalteu method at 750 nm as per Method OIV-MA-AS2-10 using 10-mm quartz cuvettes (OIV 2013). All samples were degassed and filtered down to 0.45 μm using 25-mm syringe filters.

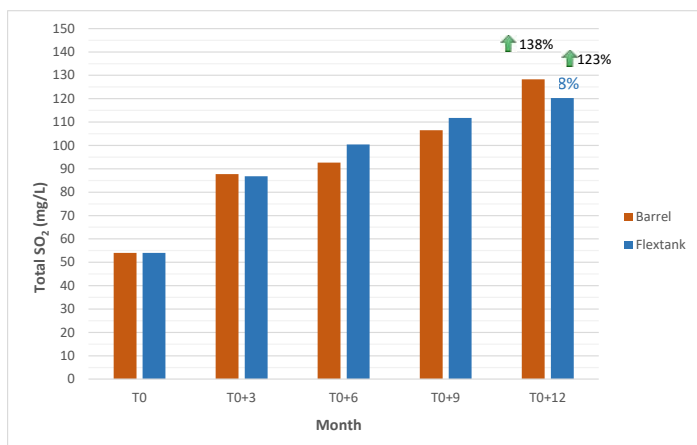
Barrel and Flextank samples were retrieved using a glass wine thief from as close to the center of wine volume as possible from each vessel. This was to avoid taking samples too close to the edge of vessels or closures.

## Results and Discussion

**SO<sub>2</sub> and DO Analyses:** Refer to Figures 1, 2 and 3, and Tables 2 and 3.

In Figure 1, the solid-colored bars represent the measured FSO<sub>2</sub> for a month; the lighter-colored bars represent the amount of FSO<sub>2</sub> added to achieve the required target based on pH and DO for each batch. For example, at T0, the barrel wine measured 36.3 mg/L FSO<sub>2</sub>, and sulfite was added to bring that up to 55.7 mg/L — the target determined based on pH 3.65 and 1810 mg/L DO. The drop in FSO<sub>2</sub> is then calculated based on the FSO<sub>2</sub> measured in the following month, i.e., solid-colored bars of the following month. For example, at T0+1, FSO<sub>2</sub> in the barrel wine dropped by 20.9 mg/L, which is the difference between the adjusted FSO<sub>2</sub> of 55.7 mg/L at T0 and the measured FSO<sub>2</sub> of 34.8 mg/L at T0+1. Since FSO<sub>2</sub> was adjusted based on pH to maintain the same MSO<sub>2</sub> level in each batch and not the same FSO<sub>2</sub> level, the FSO<sub>2</sub> analysis here is based on percent change.

After the first month, at T0+1, FSO<sub>2</sub> dropped by 38% to about the same level in each batch. This significant drop represents a similar binding in both batches, as would be expected at this early stage. Then, FSO<sub>2</sub> drops are between 11–34% and 9–27% for the barrel wine and the Flextank wine, respectively. FSO<sub>2</sub> in the barrel wine was measured at relatively the same level, for all practical purposes and when instrumentation error is taken into consideration, from T0+4 to T0+12, whereas there were greater variations in the Flextank wine, possibly pointing to differences in oxygen consumption in the two wines.



**Figure 2** TSO<sub>2</sub> changes in wines aged in barrel and Flextank over a 12-month period.

Figure 2 compares measured TSO<sub>2</sub> in each batch at 3-month intervals. Each batch experienced the same level of binding by T0+3 and therefore the same increase in TSO<sub>2</sub>. From there on, the two batches behaved slightly differently based on percent increase at T0+6, T0+9 and T0+12. Both batches ended at about the same TSO<sub>2</sub> level, 128 mg/L in the barrel vs. 120 mg/L in the Flextank, by T0+12. But examining the amount of FSO<sub>2</sub> added each month and the extent of binding in each batch, Tables 2 and 3 show that after 12 months the barrel wine had 21% FSO<sub>2</sub> and 79% BSO<sub>2</sub> with 48% FSO<sub>2</sub> loss compared to 26% FSO<sub>2</sub> and 74% BSO<sub>2</sub> with 55% FSO<sub>2</sub> loss in the Flextank batch. This behavior could result from oak wood in the barrel still releasing tannins into the wine.

	BARREL								
	MEASURED			EXPECTED					SO <sub>2</sub> LOSS
	FSO <sub>2</sub>	BSO <sub>2</sub>	TSO <sub>2</sub>	+FSO <sub>2</sub>	FSO <sub>2</sub>	BSO <sub>2</sub>	TSO <sub>2</sub>		
BASE	25.3	28.7	54.0	28.9	54.2	28.7	82.9		
T0	36.3			19.4	55.7	46.6	102.3		
T0+1	34.8			3.9	38.7	67.5	106.2		
T0+2	32.2			10.1	42.3	74.0	116.3		
T0+3	32.2	55.5	87.7	4.5	36.7	55.5	92.2	28.6	
T0+4	29.5			1.1	30.6	62.7	93.3		
T0+5	27.1			15.6	42.7	66.2	108.9		
T0+6	28.0	64.7	92.7	8.2	36.2	64.7	100.9	16.2	
<b>TOTAL T0 to T0+6</b>				<b>91.7</b>				<b>44.8</b>	
%	<b>30%</b>	<b>70%</b>							
T0+7	27.0			13.1	40.1	73.9	114.0		
T0+8	27.2			7.0	34.2	86.8	121.0		
T0+9	28.4	78.1	106.5	11.4	39.8	78.1	117.9	14.5	
T0+10	27.1			9.2	36.3	90.8	127.1		
T0+11	25.9			11.3	37.2	101.2	138.4		
T0+12	27.1	101.2	128.3					10.1	
<b>TOTAL T0 to T0+12</b>				<b>52.0</b>				<b>24.6</b>	
%	<b>21%</b>	<b>79%</b>						<b>48%</b>	

**Table 2** 12-month analysis of FSO<sub>2</sub>, BSO<sub>2</sub> and TSO<sub>2</sub> of the barrel wine.

Investigating this further and looking at FSO<sub>2</sub> loss/consumption after 6 months, i.e., 44.8 mg/L in the barrel wine vs. 43.6 mg/L in the Flextank wine, it can be assumed that both

the barrel wine and Flextank wine exhibited similar consumptions by oxygen given that TPI was relatively the same until T0+6. TPI test data is presented further down.

Looking at consumption between T0+6 and T0+12, the barrel wine consumed 24.6 mg/L FSO<sub>2</sub> vs. 36.2 mg/L in the Flextank wine, which is now a significant difference. Since TPI increased in the barrel wine between T0+6 and T0+9, a plausible conclusion here is that some FSO<sub>2</sub> in the barrel wine became bound, and there was a greater rate of FSO<sub>2</sub> consumption than binding in the Flextank wine compared to the barrel wine.

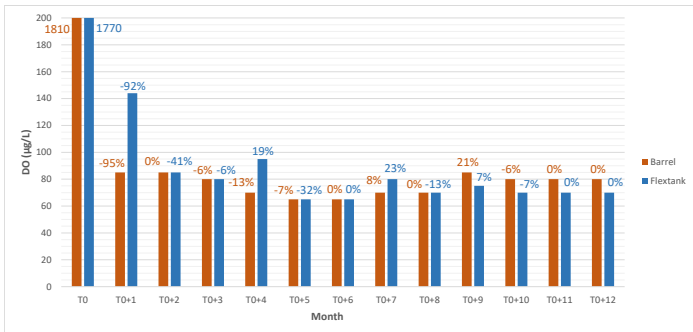
The DO analysis in Figure 3 does not show any irregularities that could explain any correlation between FSO<sub>2</sub> consumption by oxygen. There are however two small anomalies at T0+1 (144 µg DO/L) and T0+4 (95 µg DO/L); these are indeed small but beyond instrumentation error. The absolute amounts of DO are not significant at those levels around 100 µg/L, but anything over 80–

	FLEXTANK								
	MEASURED			EXPECTED					SO <sub>2</sub> LOSS
	FSO <sub>2</sub>	BSO <sub>2</sub>	TSO <sub>2</sub>	+FSO <sub>2</sub>	FSO <sub>2</sub>	BSO <sub>2</sub>	TSO <sub>2</sub>		
BASE	25.3	28.7	54.0	28.9	54.2	28.7	82.9		
T0	37.3			18.3	55.6	45.6	101.2		
T0+1	34.5			5.1	39.6	66.7	106.3		
T0+2	29.8			12.5	42.3	76.5	118.8		
T0+3	31.1	55.7	86.8	4.8	35.9	55.7	91.6	32.0	
T0+4	27.1			3.6	30.7	64.5	95.2		
T0+5	27.9			16.8	44.7	67.3	112.0		
T0+6	33.5	66.9	100.4	4.3	37.8	66.9	104.7	11.6	
<b>TOTAL T0 to T0+6</b>				<b>94.3</b>				<b>43.6</b>	
%	<b>33%</b>	<b>67%</b>							
T0+7	28.4			12.7	41.1	76.3	117.4		
T0+8	30.9			5.7	36.6	86.5	123.1		
T0+9	28.4	83.4	111.8	13.2	41.6	83.4	125.0	11.3	
T0+10	30.9			8.0	38.9	94.1	133.0		
T0+11	28.5			12.2	40.7	104.5	145.2		
T0+12	31.0	89.3	120.3					24.9	
<b>TOTAL T0 to T0+12</b>				<b>51.8</b>				<b>36.2</b>	
%	<b>26%</b>	<b>74%</b>						<b>55%</b>	

**Table 3** 12-month analysis of FSO<sub>2</sub>, BSO<sub>2</sub> and TSO<sub>2</sub> of the Flextank wine.

85 µg/L, as is typically measured in other wines, may be suspect. It is suspected that these are related to the difficulty in sealing the large lid tightly. Up until T0+4, the lid was removed to take samples for analysis, then the lid was replaced, but apparently not tight enough, particularly at T0+1. Starting at T0+4, the lid gasket was dabbed with a little olive oil to get a better seal; samples were then taken from the port on the lid. The port had been sealed with a solid (green) cap. DO was below 80 µg/L for the remainder of the study period. The lid can be difficult to remove and particularly a challenge once the gasket is dabbed with olive oil. An adjustable strap wrench had to be used to remove the lid.

The high DO values of 1810 µg/L and 1770 µg/L at T0 reflect oxygen uptake resulting from the racking into the barrel and Flextank, respectively.

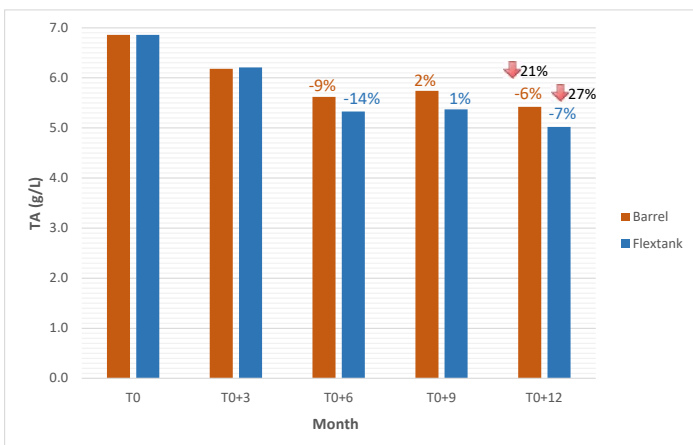


**Figure 3** DO changes in wines aged in barrel and Flextank over a 12-month period.

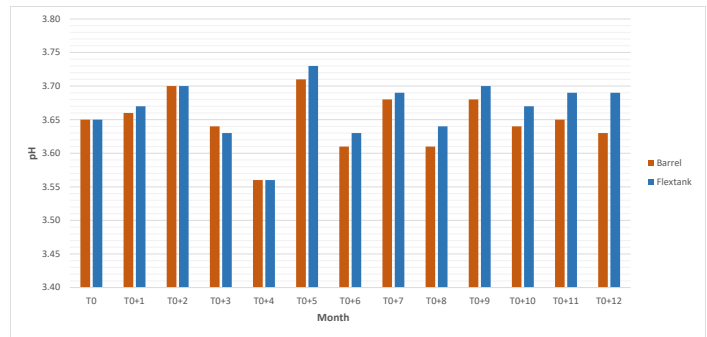
### TA, VA and pH Analyses: Figures 4, 5 and 6.

From Figure 4, TA dropped by the same amount (and percentage) by T0+3 but then by a larger amount in the Flextank by T0+6, with an overall drop of 21% in the barrel wine vs. 27% in the Flextank wine by T0+12. The greater drop at T0+6 in the Flextank wine is concluded to be due to more significant KHT formation and precipitation as witnessed at racking and by the larger drop in potassium ion ( $K^+$ ) concentration, 1923 mg/L vs. 1723 mg/L from 2102 mg/L in the base wine. The wine was not racked at T0+12, and therefore, it is not yet confirmed if there was more tartrate precipitation. TA continued to drop until T0+12, albeit at a slower rate in the barrel. These differences in KHT behavior and TA need to be investigated as the % ABV and temperature were similar in the barrel and Flextank.

In Figure 5, pH measurements are seen to vary from month to month, likely due to calibration issues. Those issues notwithstanding, pH values are similar in both batches until T0+4, but it is then slightly higher in the Flextank wine for the remainder of the study. The greater KHT precipitation and TA drop in the Flextank wine has caused wine pH to increase since pH was just around or just above the 3.65 threshold of maximum bitartrate ( $HT^-$ ) concentration.

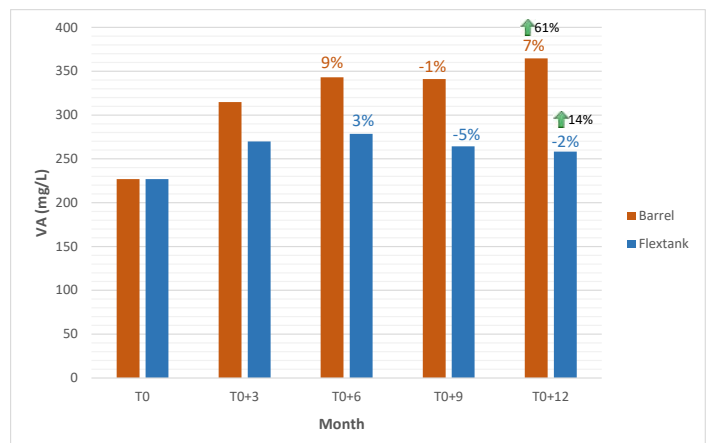


**Figure 4** TA changes in wines aged in barrel and Flextank over a 12-month period.



**Figure 5** pH changes in wines aged in barrel and Flextank over a 12-month period.

Figure 6 shows an increase in VA in the barrel wine, as would be expected, due to the headspace formed resulting from wine being absorbed into the wood and some lost to evaporation, but well below the detection threshold of 600–700 mg/L. The increase was greatest at T0+3 with only small changes from thereon. After 12 months, VA was up by 61% in the barrel wine compared to 14% in the Flextank wine. The VA increase in the Flextank wine at T0+3 would not be expected, but this was likely due to not securing the lid tightly in the first month.



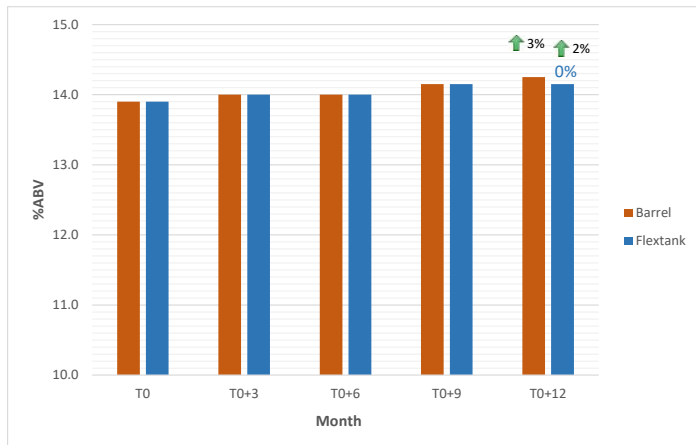
**Figure 6** VA changes in wines aged in barrel and Flextank over a 12-month period.

### % ABV Analysis: Figure 7.

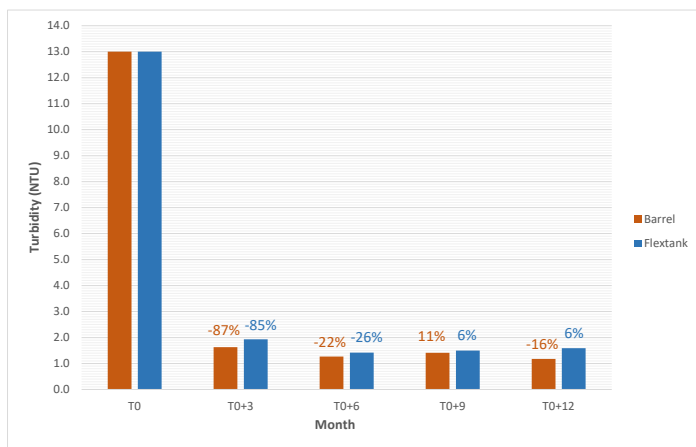
There were small increases of 2–3% in % ABV after 12 months in both wines. A concentrating effect was expected in the barrel wine, but since there were no evaporative losses in the Flextank and that both wines had similar % ABV throughout the study period suggest that any changes may be due to instrumentation error.

### Turbidity Analysis: Figure 8.

Turbidity changes measured at 3-month intervals demonstrate that the barrel wine is clearing slightly faster than the Flextank wine. Both wines dropped in the order of 85–87% by T0+3, however, given the initial turbidity of 13.0 NTU at T0, turbidity was already 16% lower in the barrel wine at T0+3 and 26% lower at T0+12. At turbidity levels below 2 NTU, a 10% difference is already significant although not necessarily visible by visual examination in a glass. The barrel wine is clearing faster likely due to its shape; it is shorter and longer than the Flextank.



**Figure 7** %ABV changes in wines aged in barrel and Flextank over a 12-month period.



**Figure 8** Turbidity (NTU) changes in wines aged in barrel and Flextank over a 12-month period.

The increase in turbidity in both wines at T0+9 is due to the wines having been racked (carefully) following analysis at T0+6. The barrel wine dropped back by T0+12 to just below the T0+6 level while the Flextank wine has increased slightly. This demonstrates that even a careful racking can cause some sediment to be picked up and turbidity to increase, and that another racking after 3 months, as many winemakers do, does not accomplish any improvements. It is also likely that the increase in tannins between T0+6 and T0+12, according to TPI, contributed to a faster precipitation and clearing of the barrel wine; tannins are known to have a fining action.

**Color and Polyphenol Analyses:** Figures 9, 10, 11, 12, 13, 14, 15 and 16.

The yellow component ( $A_{420}$ ) (Figure 9) in the Flextank wine remained fairly constant throughout the duration of the study but dropped overall by 3% since T0.  $A_{420}$  has progressively increased in the barrel wine and has increased by 9% overall since T0, and was 12% higher than the Flextank wine by T0+12. An increase in  $A_{420}$  typically results from the oxidation of polyphenols into their brown-colored *o*-quinone forms; however, based on FSO<sub>2</sub> and DO analysis, in that both wines exhibited similar changes until T0+6 and that the barrel wine had more binding and less FSO<sub>2</sub>

consumed until T0+12, it is surmised that oak tannins and other polyphenols may still be extracted from the barrel wood, and which are likely playing a role in forming yellow-pigmented polymers.

The extraction of more tannins would also support the hypothesis that they are contributing to a more stable and greater red color based on  $A_{520}$  measurements (Figure 10). Following a similar drop (10–12%) in both wines as measured at T0+3, likely due to anthocyanins being absorbed and dropped by tartrates as well as from complexation and sedimentation,  $A_{520}$  and hence the red color in the barrel wine continued to increase slightly, though not visible in the glass, and dropped by 5% after 12 months compared to 16% in the Flextank wine. The barrel wine was 13% “more red” than the Flextank wine by T0+12. The brilliance of red (dA) (Figure 15) was however similar in both wines and decreased at every measurement interval, as would be expected when red wines age. But it is interesting to note that the barrel wine still had a slightly richer red color.

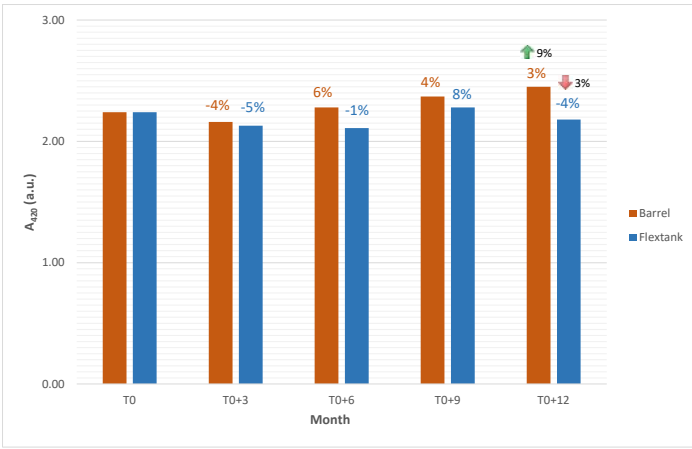
After the initial drop at T0+3,  $A_{620}$  measurements (Figure 11) remained relatively unchanged in both wines over the 12-month period although the barrel wine was marginally higher. In relation to the red color, i.e.,  $A_{520}$ , the Blue Index (Figure 14) too remained relatively unchanged in both wines over the 12-month period. These data suggest that both wines are still similarly very young.

Looking at it collectively by calculating color intensity (IC) from  $A_{420}$ ,  $A_{520}$  and  $A_{620}$ , IC (Figure 12) in the barrel wine was back up to the T0 level after 12 months while the Flextank wine had dropped by 11%, with the barrel wine having 13% greater intensity at T0+12, but again, these differences in IC were not yet visible in the glass.

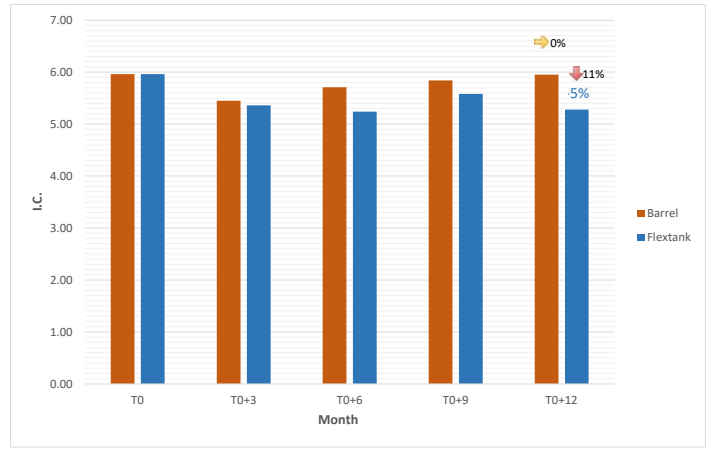
Given the higher  $A_{420}$  and  $A_{520}$  in the barrel wine, hue (H) (Figure 13) was similar in both wines. By T0+6, both wines reached the 0.8 threshold, indicative that both wines have started their oxidative evolution and at a similar rate. Over 12 months, hue has increased similarly by 15% in both wines.

Total phenols analysis (Figure 16) shows relatively constant TPI measurements in both wines until T0+6, and relatively large jumps at T0+9, though considerably greater in the barrel wine, which, instrumentation error notwithstanding, suggests that more tannins are still being extracted from the barrel wood. The differences in TPI in the barrel and Flextank wines were 4.7 and 2.0, in favor of the barrel wine, at T0+9 and T0+12, respectively. A TPI difference greater than 2.0 can be significant and quite possibly detectable in a taste test. The drop in TPI in both wines between T0+9 and T0+12 suggests a higher rate of tannins binding or oxidation than extraction from the WineStix and also oak wood in the barrel wine. After 12 months, the barrel wine had a TPI 3% higher than at T0 while it was 1% lower in the Flextank wine. Given the sensitivity of TPI measurements, these small changes are significant.

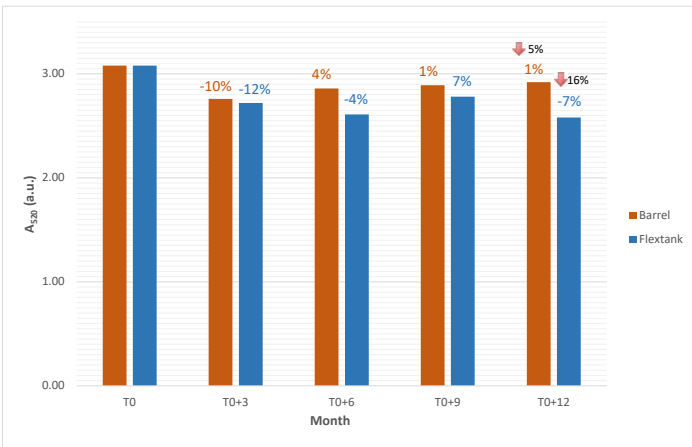




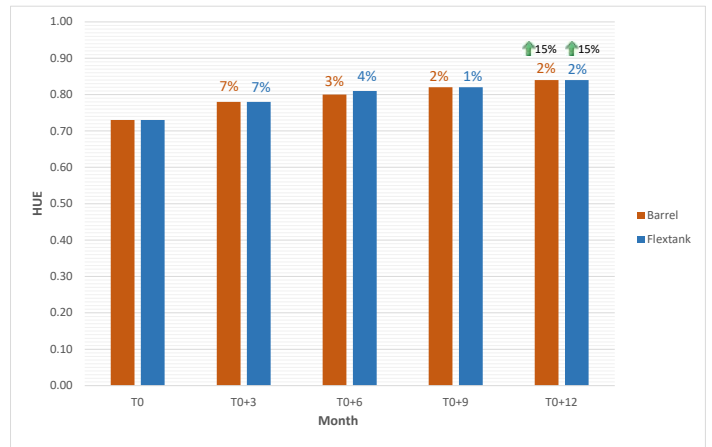
**Figure 9**  $A_{420}$  changes in wines aged in barrel and Flextank over a 12-month period.



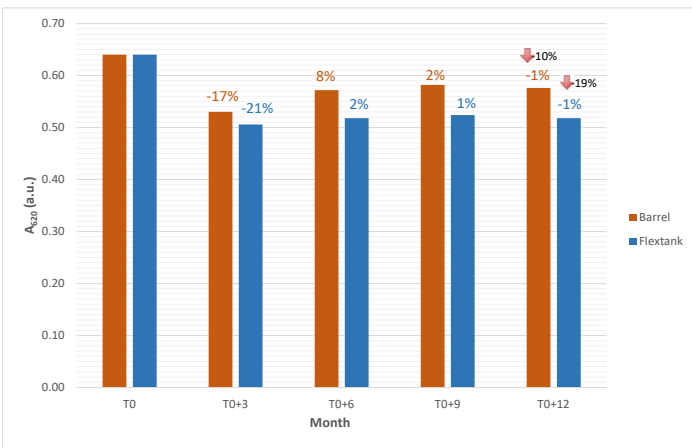
**Figure 12** Color intensity (IC) changes in wines aged in barrel and Flextank over a 12-month period.



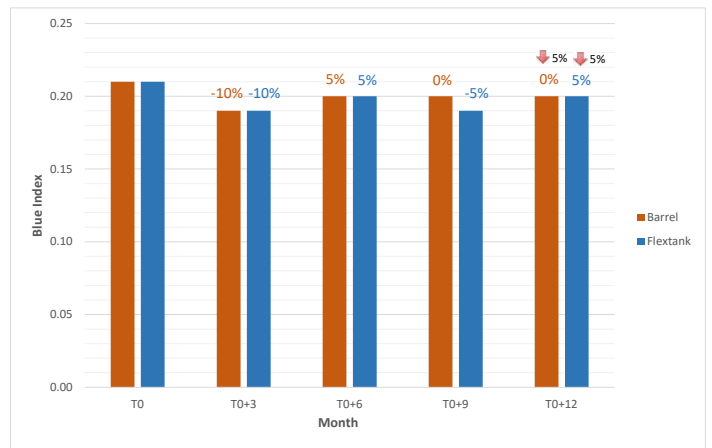
**Figure 10**  $A_{520}$  changes in wines aged in barrel and Flextank over a 12-month period.



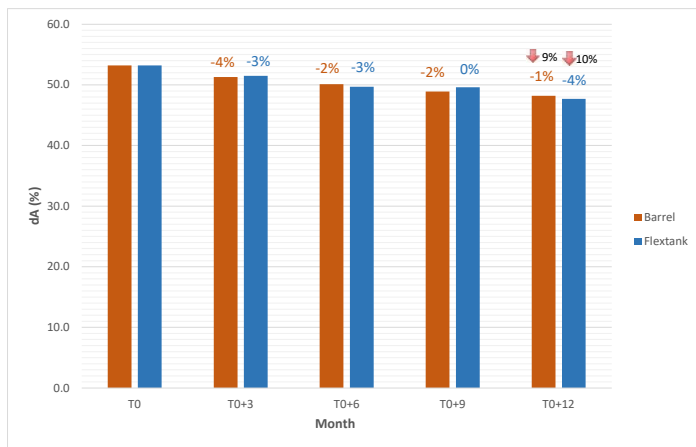
**Figure 13** Hue (H) changes in wines aged in barrel and Flextank over a 12-month period.



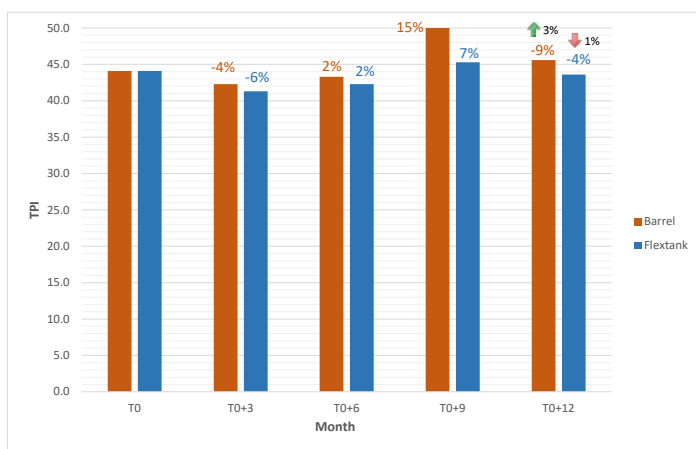
**Figure 11**  $A_{620}$  changes in wines aged in barrel and Flextank over a 12-month period.



**Figure 14** Blue Index changes in wines aged in barrel and Flextank over a 12-month period.



**Figure 15** Brilliance of red (dA) changes in wines aged in barrel and Flextank over a 12-month period.



**Figure 16** Total Phenol Index (TPI) changes in wines aged in barrel and Flextank over a 12-month period.

**Sensory Evaluations:** The barrel and Flextank wines appeared and tasted identical at T0+3 and T0+6, displaying a limpid, medium-light red color with a purplish ring, pleasant fruity aromas with a hint of leather though with a touch of green character, but having shed the slight barnyard aroma detected in the base wine at T0. Both wines tasted medium-bodied with improving structure and greater tannins over time although both tasted very young still. The Flextank wine did taste somewhat smoother at T0+6 likely due to the drop in acidity from KHT precipitation. At T0+9, the Flextank wine was tasting smoother still, coincidentally with the greater TPI (and hence more tannins) measured in the barrel wine. A lower TPI coupled with a lower TA will make a wine taste smoother. Tasting notes at T0+12 were consistent with those at T0+9, with the Flextank wine still tasting a touch smoother. The slightly higher VA in the barrel wine has not had any perceptible impact yet on aromas and flavors. Both wines only had hints of oak aromas at T0+12. The WineStix have slow and subtle impacts in the first 12 months.

## Conclusions

This study demonstrates that, for the first 6 months, the standard maturation weight (SMW) ECO15 Flextank mimics a 55-L (14.5-gal) two-year-old oak barrel from an oxygen transfer rate (OTR) perspective provided that the lid is tightly sealed to 25 foot pounds for an airtight seal according to Flextank product literature. Average users are however not able to measure this.

As the barrel imparted more tannins, the two wines exhibited slightly different behaviors after 6 months based on free sulfur dioxide (SO<sub>2</sub>) and colorimetric analyses. The extra tannins may have contributed to greater binding of free SO<sub>2</sub> and less reacting with other wine compounds, more pronounced red color and color intensity as measured quantitatively but not detectable by visual inspection, and greater fining action. The barrel shape is also believed to have contributed to the greater measured clarity although, again, not detectable by visual inspection of the wine in a glass.

Surprisingly, there were much heavier tartrates in the Flextank wine after 6 months although temperature and alcohol concentration were similar in both wines. The lower phenol content and acidity made the Flextank wine taste smoother after 6 months. There were no organoleptic differences that stood out even though a concentration effect was expected in the barrel wine due to evaporative losses; both wines recorded similar alcohol concentrations throughout the study.

As expected, due to the headspace that forms in barrels, the barrel wine produced slightly more volatile acidity (VA), but well below detection threshold.

It is recommended that a little olive oil be applied to the gasket for a tighter fit to avoid oxygen ingress; a suitably designed wrench is also recommended to ease removal of the lid. A Flextank dry airlock is recommended in lieu of a rubber bung seated in the threaded port until the wine is sufficiently degassed, and then replaced with a solid Flextank plug. Teflon tape should be applied on threaded parts. And given the large surface area and potentially large headspace at the top of the Flextank, the tank should be filled to the very top via the small port with the large lid in place.

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