

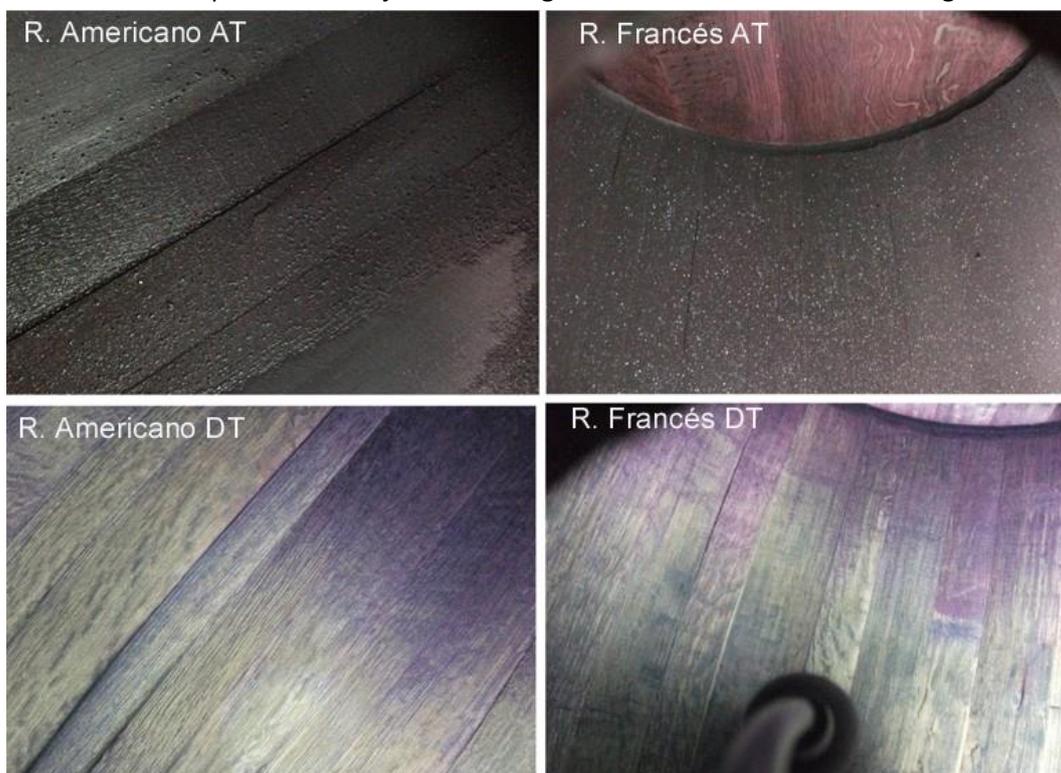
## EFFECT ON 3 AND 4 YEAR OLD BARRELS REGENERATED BY THE BARENA® METHOD ON THE RELEASE OF TANNINS AND AROMAS AND AT A MICROBIOLOGICAL LEVEL

Antonio Palacios (Laboratorios Excell Ibérica S.L.) and Nathalie Beaucourt (Laboratorios Clean Biotec S.L. L. )

### 1-. INTRODUCTION

The Barena® regeneration of 3 and 4-year-old used barrels is the only regeneration method that manages to reactivate their main oenological functions and consists of the following steps:

- Initial physical-mechanical pickling by projection of quartz particles. The pressure is regulated according to the state of the barrel. The pickling is carried out on a thickness between 0.2 and 0.4 millimetres.
- Removal of pickling residues by rinsing with previously dechlorinated water.
- Application of dry steam heated at above 100° for a few minutes in order to obtain a good microbiological sanitation.
- Rinsing, drying and bunging of barrels.
- Gaseous sulphur is later injected for its good conservation while transiting to the cellar.



*Figure 1. Photograph of the interior of the untreated barrels (AT) and after the Barena® treatment (DT)*

### 2-. MATERIALS AND METHODS: TESTS CARRIED OUT AT AN INDUSTRIAL LEVEL IN WINERIES:

## 2.1-. Test on the Cabernet Sauvignon variety (Bordeaux); effect on tannins and aromas:

This test comes from France and is made with the variety Cabernet Sauvignon. It compares 3-year-old barrels from the same winery and all containing the same wine, the first with a maintenance wash according to the traditional technology of the winery, the second with the same system but implemented with steam, the third is a Barena® regenerated barrel and the fourth is a new barrel.

Figure 2 shows the level of tannins released by the different types of barrels into the wine from the same test. The most remarkable thing is that the greatest amount of tannin release is observed on the Barena® regenerated barrel. This was predictable due to the small renewal of the internal layer of the wood enabled by the pickling system with the projection of quartz particles. The level of the new barrel comes after, followed later down by the level observed in the barrels maintained with the winery classic system, also 3 year-old.

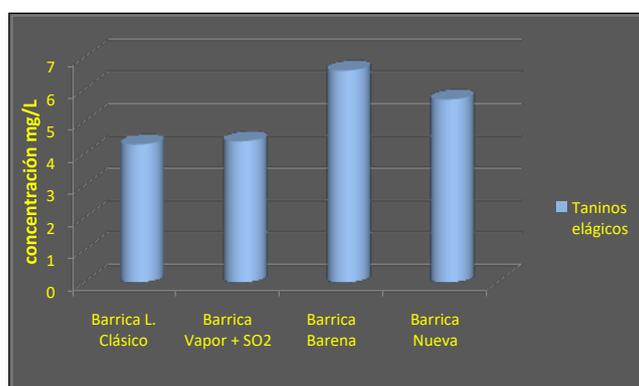


Figure 2: representation of the concentration of ellagic tannins in the different barrels

Concerning the aromas results, we can also observe that the new barrel is the one that stands out the most in the release of wood volatiles, as expected. However, the second place is occupied by the Barena® barrel. The most outstanding results are observed in terms of cismethyloctolactone, vanillin, hydroxylactone and syringol, see table 1.

Table 1: results of the analysis of wine aromas in different barrels

Tipo de lavado µg/L de vino	Barrica L. Clásico	Barrica Vapor +Barrica Barena	Barrica Nueva	
	SO2			
Furfural	89	88	88	129
Metil-5-furfural	4	5	5	86
Alcohol furfurílico	23	29	17	24
Guaiacol	8	10	10	14
Trans-metilolactona	1	2	0	2
Cis-Metilolactona	137	154	158	321
Metil-4-guaiacol	1	1	1	7
Fenol	5	5	5	6
Etil-4-guaiacol	7	7	7	3
O-cresol	1	1	1	1
m-p-cresol	2	2	2	2
Eugenol	28	24	27	41
Etil-4-fenol	46	40	41	9
Isoeugenol	1	1	1	11
Siringol	31	50	53	63
5-hidroximetilfurfural	42	41	52	84

Alil-4-siringol	8	9	13	45
Vainillina	55	72	96	231
Siringaldehido	104	141	175	731

Figure 3, from the same test, shows the same results as in the previous case, but grouping the chemical compounds into olfactory families, where it can be seen that the new barrel is the most outstanding from an aromatic point of view, and in second place is the 3-year-old barrel regenerated with the Barena® method, where the sum of lactones and phenolic aldehydes are the ones that show the most differences compared to systems with classic barrel maintenance in the cellar. This new status, to which the treatment leads to the used barrels, allows an aromatic reactivation, but without being as intense as that of a new barrel, have a lower impact on the wine. A very interesting effect when minimum barrel aging times must be met due to regulations.

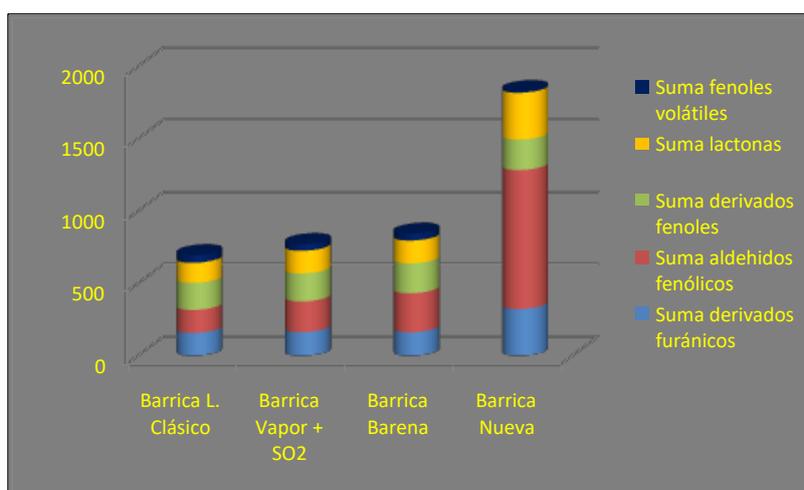
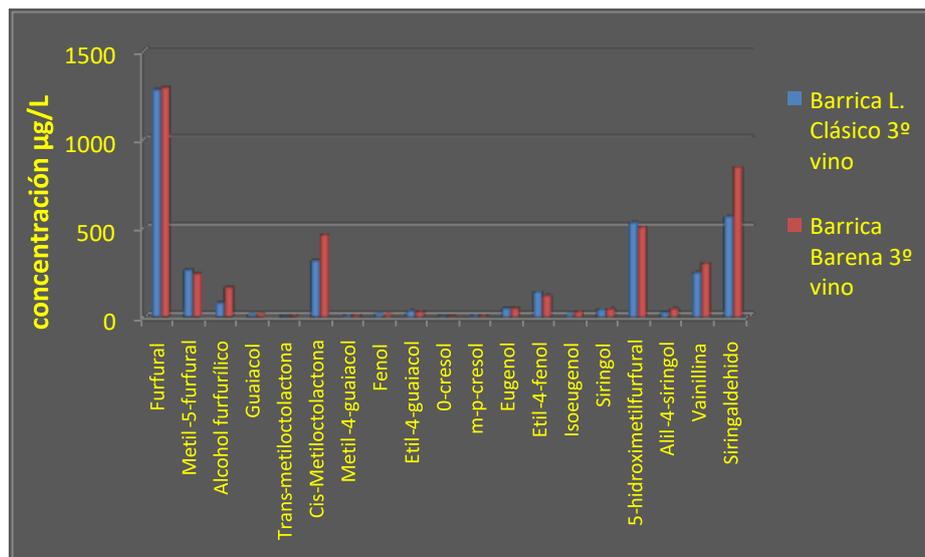


Figure 3: representation of the aromas grouped by families, (concentrations in µg/L).

## 2.2-. Test on a wine of the Cabernet Sauvignon variety (Bordeaux); effect on the aromas

Figure 4 compares the same wine of the Cabernet Sauvignon variety kept in classic-cellar-washed barrels in its third year against the same wine aged in a barrel with Barena® regeneration, both three years old.



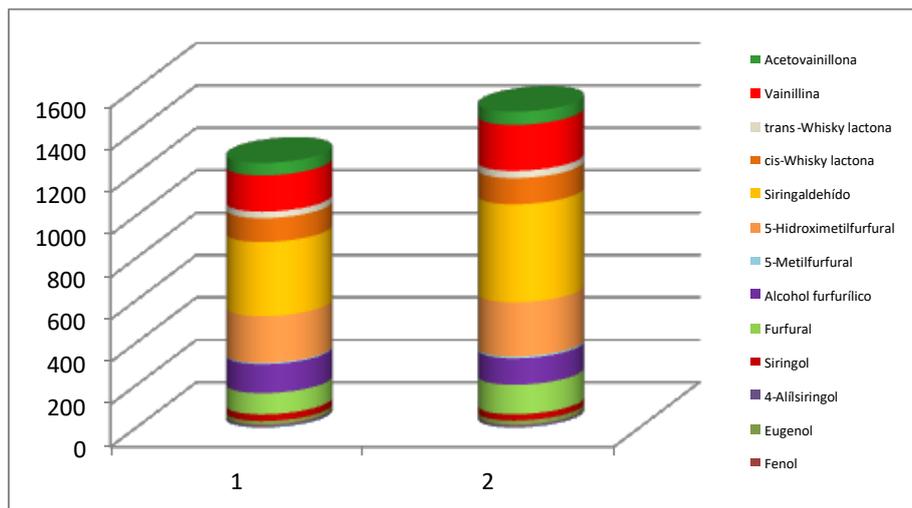
**Figure 4:** representation of the aromas of the wood in two different barrels, one of them with Barena regeneration.

The most remarkable element about the results is that, in terms of differences, it is the Barena® regenerated barrel that releases more methylactone and syringaldehyde than the other test control barrel. This is an indicator of the regeneration method's respect of the toast layer.

### 2.3-. Tests on Tempranillo Crianza and Reserva wines (D.O.Ca. Rioja); effect on aromas:

Two parallel tests are presented below, one with Tempranillo wine vintage 2012 aged for 9 months in standard cellar barrels, versus a regenerated barrel in its 4th year. In this test we intend to study the effect on the aged wine. In the second test the same type of monitoring is done, but using a 2011 wine, destined to the Reserve type, but in this case, the same wine has 24 months of aging in standard winery barrels and one of them 18 months in standard barrels plus, 6 months later and at the end of the process, in a regenerated barrel also in its 4th year, looking for the final refinement without marking the wood aromas much.

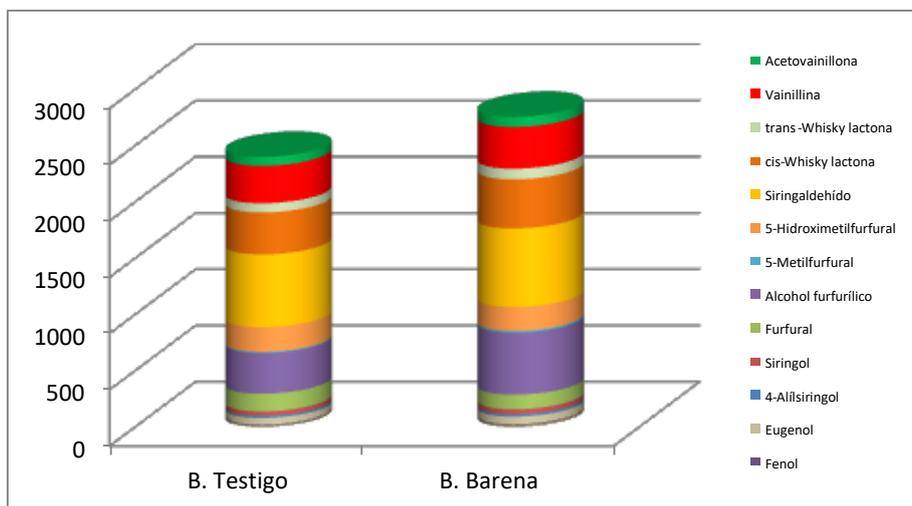
The results of the first Crianza type wine can be seen in figure 5, where we see increases of 16.5% in eugenol, 39% in furfural, 43% in 5-methylfurfural, 32% in syringaldehyde, 9.7% in ciswhiskylactone, 26.9% in vanillin and 4.7% in acetovainillone in the wines aged in regenerated barrels.



**Figure 5:** Analysis of aromatic components of wood for the same wine 2012 Aging with 9 months in standard cellar barrels versus regenerated barrels in its 4th year.

Concerning the second Reserva wine (Figure 6), the increases observed on the wine with 18 months in standard barrels and 6 months in regenerated barrels, compared to the same sample with 24 months in standard barrels, were 24,2% for guaiacol, 14% for eugenol, 51.8% for furfuryl alcohol, 60.4% for 5-methylfurfural, 17% for cis-whiskylactone, 11.7% for vanillin and 12.5% for acetovainillone.

These results are very interesting. Considering the average prices per liter of wine from new French oak barrels (0.75 euros/litre) and American oak barrels (0.38 euros/litre) with 4 years of active use, the prices of regenerated barrels (0.06 euros/litre) and the average prices of chips (0.03-0.02 euros/litre), the ageing in regenerated barrels gives an added value to the wine, with very profitable investments at low costs.



**Figure 6:** Analysis of wood aromatic components for the same 2011 Reserva wine, but in this case, the same wine has 24 months of aging in standard barrels in the winery and one of them 18 months in standard barrels plus 6 months in regenerated barrels in its 4th year.

#### 2.4-. Test on Tempranillo aged in French oak barrels (D.O.Ca. Rioja); sensory effect:

The wine used is a Reserva 2014 that has been aged in French oak barrels of different ages and has been treated in different ways: regenerated with the Barena® method at its 4th, 6th, 8th

and 10th year, blue and test control barrels maintained with the usual winery cleaning treatment. The wines were analyzed on the aromas coming from the wood, by means of gas chromatography and mass spectrometry after 13 months of aging.

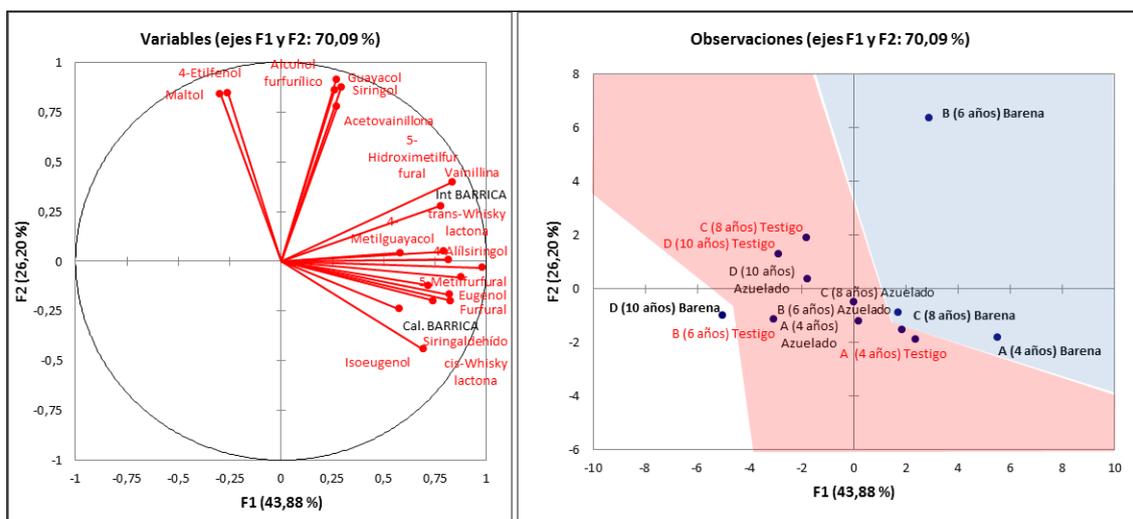


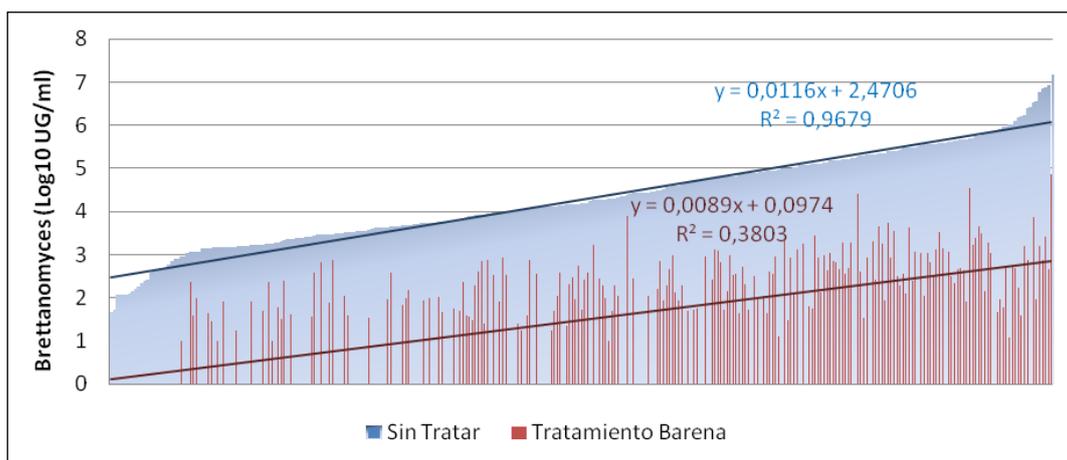
Figure 7: ACP according to wood aromatics compounds at 13 months.

When the representation of the analyzed samples is made in the factorial plane, a graph is obtained (figure 7) that is capable of explaining 70.09 % of the variance. In the ACP representation at 13 months of ageing, two coloured areas are observable. The first (upper right quadrant in blue) includes Barena® barrels 4 and 6. In this case, the sensory attributes of aromatic intensity and quality also go in the same direction as the vectors representing the most appropriate aromatic compounds in oak. The barrels that stand out the most in this respect are the 4 and 6 year old Barena® treated barrels. Later, in an intermediate region (red color), we find the wines representing the blue barrels and the test control ones, among others.

### 3-. Microbiological effect of the Barena® treatment

A total of 325 barrels analysed before and after the Barena® treatment were compared, corresponding to different wineries, different cooperages and with wines from 1998 to 2010. The barrels were sampled by adding a constant volume of water and then collected.

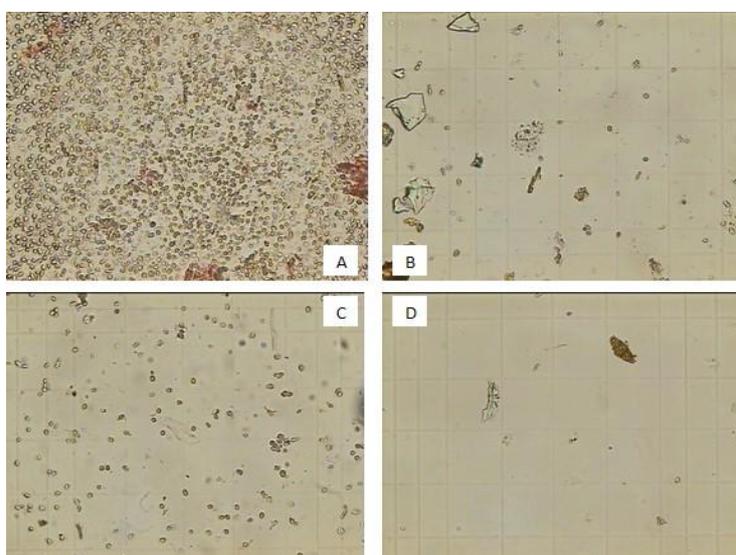
The analysis technique used is time PCR. The expression of the results is measured in Genomic Units (GU/ml). Before its treatment only 12.31% had an initial contamination of *Brettanomyces* lower than  $10^3$  UG/ml (3 Logarithmic units), the rest exceeded this figure, and 53.54% had an initial contamination higher than  $10^4$  UG/ml (4 Log units) even reaching in some cases to exceed one million UG/ml (6 Log units). Once treated by the Barena® method, the initial contamination tends to be reduced between 2.5 and 3 log units on average (see Figure 8). This means that if the initial contamination was  $10^3$  UG/ml it is reduced to less than 10 UG/ml, and if the initial contamination is greater than  $10^6$  UG/ml it is reduced to  $10^3$  (3 Log).



**Figure 8:** Brettanomyces concentration, expressed in logarithmic units, in 325 barrels analyzed before and after the Barena treatment. The trend line and its correlation coefficient are added for each group.

However, with the PCR technique, the DNA of the target species is detected, which implies that the results are obtained from live, dead and viable non-cultivable colonies. To check the disinfection status, controls were carried out by means of traditional culture. In none of the cases did *Brettanomyces* grow, so there were no viable cultural cells.

Additionally, controls were made for the presence of indicator microbial flora (mesophilic aerobes at 30°C), an indicator of the state of disinfection of the barrel after treatment. In 99.9% of the cases the microbial count was null.



**Figure 8:** Total cell count in Newbauer's chamber Optical microscopy image, x400 magnification  
A- Untreated barrel, initial contamination above 107 CFU/ml.  
B- Previous treated barrel.  
C- Untreated barrel, initial contamination of  $10^5$  CFU/ml  
D- Treated anterior barrel

#### 4-. GENERAL CONCLUSIONS:

- The Barena® method regenerates 3 and 4 year old used barrels by completely eliminating the tartrate film, thus reactivating their main oenological functions,

- respecting the toasting layer and allowing the wine to be enriched with its own wood aromas, giving it a sensory profile very close to that of the new barrel.
- Wines aged in regenerated barrels in their third and fourth year are capable of increasing their aromatic complexity, both in terms of typical oak wood aromas and toast.
  - The kinetics of the transfer of wood aromas is proportional to the time the wine remains in the regenerated barrels.
  - Sensorially speaking, wines aged in barrels treated by the Barena® method are wines with sharper fruit aromas, more evident wood reminiscences and more rounded and balanced mouths than the same wine in used barrels.
  - Barrel regeneration revalues used barrels, mainly in their third and fourth year, increasing their time of use in the winery as an active continent for the qualitative improvement of the wine.
  - From a microbiological point of view, the Barena treatment disinfects the interior of the oak barrels in 99.9% of the cases in the extensive sampling carried out. The microbiological improvement of the wood of the barrels and therefore of the wine contained in them, allows for softer oenological techniques with respect to protection against contaminating microorganisms.

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