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Composition and sensory properties of Cabernet Sauvignon wine aged in French versus American oak barrels

by

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Zusammensetzung und sensorische Eigenschaften von Cabernet-Sauvignon-Wein nach Lagerung in französischen und amerikanischen Eichenfässern

Zusammenfassung. — Kalifornischer Cabernet-Sauvignon-Wein des Jahrgangs 1980 wurde 115, 239 und 338 d lang in neuen Eichenfässern (228 l) gelagert, die in den USA (Missouri) und Frankreich (Nevers) nach ähnlichen Verfahren gefertigt worden waren. Wein, der in 19-l-Glasballons unter denselben Bedingungen gelagert wurde, diente als Kontrolle. Die Eichenfaß-gelagerten Weine besaßen signifikant höhere Gehalte an titrierbarer Säure (TA), flüchtiger Säure (VA), Äthanol, Gesamtphenolen (TP), polymeren Phenolen (PP) und nicht-flavonoiden Phenolen (NFP) als die Kontrolle. In französischen Eichenfässern gelagerter Wein zeigte einen signifikant niedrigeren Kaliumspiegel und signifikant höhere Gehalte an TA, VA, TP und NFP als Wein aus amerikanischen Fässern. Während die erhöhte Äthanolkonzentration durch die Verdunstung von Wasser zustande kam, stieg der Gehalt an TA, VA, TP und NFP hauptsächlich infolge der Extraktion aus dem Eichenholz an. Die signifikant höheren Werte der PP waren das Ergebnis einer schnelleren Polymerisierung von flavonoiden Weinphenolen in den Eichengebinden. Im sensorischen Triangeltest unterschied sich der Kontrollwein signifikant von beiden faßgelagerten Weinen; ein Vergleich der Weine, die gleich lang in französischen und amerikanischen Eichenfässern lagerten, zeigte nur bei den 115 d lang gelagerten Weinen einen signifikanten Unterschied.

Introduction

Despite the importance of the effects of storage in French versus American oak barrels on wine flavor and composition, very little quantitative research has been published on this topic. Although the composition of oak wood and its extracts have been studied extensively, there has been only a limited amount of research comparing the French and American species of white oak used for cooperage. Further, most of the investigations of beverages stored in French and American barrels have involved distilled spirits.

Although barrels are coopered from white oak in both France and the U.S., different species are utilized. Of the eight species used in America, 45 % of the oak suitable for cooperage is *Quercus alba*. In France, only two species are utilized for barrel building, *Q. sessilis* and *Q. robur*, neither of which is found in the U.S. In both locations, the species are quite similar and often hybridize (SINGLETON 1974). Although the differences between French and American oaks may be attributed in part to the different species of white oak employed, other factors affect the composition of oak wood as well. Differences in soil, climate, and growth habit account for regional differences in France (GRAFF and TCHELISTCHEFF 1969). The age of a tree and its growth rate further affect the level of phenols in the wood.

Possibly the most important influence on the properties of French and American oak barrels is the difference in coopering methods. The way in which the stave wood is dried (MARKMAN 1974, PONTALIER *et al.* 1982), the use of steam versus an open fire for

bending the staves, and the level of "toasting" or charring on the inside of the barrel (VALAER and FAZIER 1936, LIEBMANN and SCHERL 1949, BALDWIN *et al.* 1967, GUYMON and CROWELL 1968, REAZIN 1981) affect the extraction of components from the barrel.

In comparing material extracted from oak chips with 55 % (v/v) ethanol, SINGLETON *et al.* (1971) reported that nearly 2.5 times more phenols were extracted from French oak than from American. In both types of oak, 87.7 % of the extracted phenols were nonflavonoid. Smaller amounts of extract from American oak chips were required to produce a detectable aroma change in wines (SINGLETON 1974). Specific compounds which have been reported to contribute to the aroma of oak-aged beverages include vanillin (SINGLETON and NOBLE 1976), furfural (GUYMON and CROWELL 1972), and oak lactone (SIMPSON 1980), all of which were found in higher levels in brandies aged in American oak than in French (GUYMON and CROWELL 1968, 1972, ONISHI *et al.* 1977). In brandy, fixed and total acidity increased and pH decreased with time in the barrel due to the extraction of fixed and volatile acids from the wood (VALAER and FAZIER 1936, LIEBMANN and ROSENBLATT 1943, GUYMON and CROWELL 1968, JAROSZ and JAROSZ 1969, ONISHI *et al.* 1977). A greater decrease in pH occurred in brandy stored in French (Limousin) oak than in American (GUYMON and CROWELL 1970).

In this study, the chemical composition and sensory properties of Cabernet Sauvignon wines which were aged in French and American oak barrels coopered by the same methods were investigated.

Materials and methods

Materials

Wine was produced commercially by standard winery practices from 1980 Cabernet Sauvignon (87.5 %) and Merlot (12.5 %) grapes from Alexander Valley, California. The wine was fined with 0.5 lb gelatin/1000 gal and filtered using diatomaceous earth as a filter aid before racking into barrels.

The 228-l (60 gal) French and American oak barrels were coopered by the same methods. The stave wood was air-dried outdoors for at least 3 years and bent over a small fire, leaving the inside of the barrels lightly charred. The American oak barrels were coopered by Tonnellerie Francaise (Calistoga, CA) from oak wood from Missouri. The French barrels were built by Tonnellerie Ludonaise (Ludon, France) from oak wood grown in the Nevers area of France.

15 French and 15 American oak barrels each were filled with the wine as well as 2 5-gal glass carboys, which served as the control wine with no barrel aging. The carboys were laid on their sides, and barrels rolled $\frac{1}{8}$ turn to ensure contact between the wine and the bungs as ullage developed. After 115 d, 1-gal samples were removed from 5 of the French oak barrels and combined in a 5-gal glass carboy to reduce barrel to barrel variability and stored under conditions identical to the control until the end of the storage study. The remaining 10 French oak barrels were topped up, bunged and rolled as before. Samples were taken from 5 American oak barrels in the same manner. Once the barrels were sampled, they were not used again. After 239 and 338 d, samples were taken using the same procedures. The wines were then adjusted to 25 ppm free sulfur dioxide, as determined by the aeration-oxidation method (AMERINE and OUGH 1984). The wine was then bottled into 750-ml bottles and transferred to the University of California winery cellar, where they were stored at 15.5 °C until analyzed.

Chemical Analyses

Absorbance at 420 nm and 520 nm were determined on all wines and at 520 nm on wines bleached with sulfur dioxide. The contribution of polymeric anthocyanins to absorbance at 520 nm was estimated as described previously (BERG 1953, RIBÉREAU-GAYON and STONESTREET 1965). Titratable acidity, pH, volatile acidity, ethanol, color density and color hue were determined by procedures described by AMERINE and OUGH (1980). Potassium was determined on samples diluted 1 : 200 with distilled water by atomic emission spectroscopy at 769.9 nm using an air-acetylene flame.

Total phenols were determined by the method of SINGLETON and ROSSI (1965) as modified by SLINKARD and SINGLETON (1977). Nonflavonoid phenols were analyzed by the procedure described by SINGLETON *et al.* (1971). Polymeric phenols were separated by dialysis using Spectrapor 3 semi-permeable tubing (Spectrum Medical Industries, Los Angeles, CA), which retains compounds with a molecular weight larger than 3500 as described by WATSON (1975). The total and nonflavonoid phenols in the undialyzed fraction were quantified as described above.

Sensory Analysis

To determine if the wines differed significantly in flavor, the French and American oak-aged wines were compared at each storage time in duo-trio difference tests. In addition, the control wine was compared to all of the oak treated wines in separate duo-trio tests. For each comparison, which was presented twice in daily sessions, judges were instructed to both taste and smell the wines. In two subsequent sessions, for each type of oak the aromas of wines aged for different storage times were compared, with three sets being evaluated in each session.

All of the tests were performed in individual booths at 23–25 °C under red lights. Aroma (and taste) were evaluated on 25-ml samples in coded clear 6.5-oz wine glasses, which were covered with watch glasses. The presentation order of the samples was randomized. Distilled water was provided for rinsing in between samples in sessions in which the wines were tasted.

Table 1
Means¹⁾ and least significant differences (LSD) of standard wine analysis
Mittelwerte und Signifikanzschwellen (LSD) der Standard-Weinanalyse

	Storage time in barrel (d) and type of barrel ²⁾							LSD
	0	115Am	115 Fr	239Am	239 Fr	338Am	338 Fr	
pH	3.410	3.395	3.381	3.390	3.367	3.383	3.357	— (NS)
Titratable acidity (g tartaric acid/l)	6.10 ^a	6.26 ^b	6.45 ^d	6.36 ^c	6.60 ^f	6.50 ^e	6.74 ^g	0.04 (P < 0.001)
Volatile acidity (g acetic acid/l)	0.39 ^{a)}	0.52 ^{ab}	0.54 ^{ab}	0.53 ^{ab}	0.58 ^b	0.49 ^{ab}	0.61 ^b	0.0154 (P < 0.05)
Potassium (mg/l)	910 ^{ab}	922 ^{ab}	917 ^{ab}	928 ^b	906 ^a	929 ^b	906 ^a	21.92 (P < 0.01)
Ethanol (% v/v)	13.44 ^a	13.46 ^a	13.51 ^{ab}	13.58 ^{bc}	13.70 ^d	13.70 ^d	13.69 ^{cd}	0.1161 (P < 0.001)

¹⁾ Means within each row with the same letter are not significantly different at the level of significance indicated. Means are the average of 4 determinations except ethanol where n = 6.

²⁾ Am = American oak; Fr = French oak.

NS = Not significant.

13 students in the Department of Viticulture and Enology who had participated in a variety of sensory tests of wine and model systems prior to this experiment were selected as judges based on their availability and interest. One day of training was used to familiarize them with the wines and testing procedures employed in the study, prior to the formal testing.

Data Analysis

Compositional data were analyzed by one-way analysis of variance, fixed model. Data for the 7 wines were evaluated (using the analytical replications as multiple observations per cell) as specified by the contrasts footnoted in Table 2 to permit comparison of the control versus the mean of the 6 wood treatments, of the mean of the American oak-aged wines versus the mean of the French, and across time within each time of wood. Bonferroni confidence intervals were calculated for the above contrasts (NETER and WASSERMAN 1974), in addition to Fisher's least significant differences (LSD) to permit pairwise comparison of wine means. Analyses were performed using the Rummage program (Scott, D.T., N.W. Carter, and G.R. Bryce, Brigham Young University, Provo, Utah). The significance of the duo-trio difference test results were interpreted from a cumulative binomial probability table, where $P=0.5$, one-tailed (AMERINE and ROESSLER 1976).

Table 2

Degrees of freedom (df), mean squares (MS), F-ratios, significance of differences among treatments, and significance of contrasts (L_i)¹ among treatments as calculated from Bonferroni confidence intervals for standard wine analysis

Statistische Auswertung der Standard-Weinanalyse: Freiheitsgrade (df), Mittlere Abweichungsquadrate (MS), F-Werte, Signifikanz der Unterschiede zwischen den Behandlungen, Signifikanz der Kontraste (L_i) zwischen den Behandlungen (Berechnung aus den Konfidenzintervallen nach Bonferroni)

	Source of variation	df	MS	F	Sig.	L_1	L_2	L_3	L_4	L_5	L_6
pH	Wines	6	0.0155	1.0391	NS						
	Error	7	0.0149			NS	NS	NS	NS	NS	NS
Titratable acidity	Wines	6	0.0915	1429.8	***						
	Error	7	0.000064			C < R	A < F	$A_1 < A_3$	NS	$F_1 < F_3$	NS
Volatile acidity	Wines	6	0.000097	6.47	* ²)						
	Error	7	0.000015			—	—	—	—	—	—
Potassium	Wines	6	185.81	11.41	**						
	Error	7	16.29			NS	F < A	NS	NS	NS	NS
Ethanol	Wines	6	0.0255	55.73	***						
	Error	7	0.000457			C < R	NS	$A_1 < A_3$	NS	$F_1 < F_3$	D

¹) L_1 = Control (C) vs. mean of oak aged samples (R). L_2 = mean of American oak samples (A) vs. mean of French oak samples (F). L_3 = value of 115-d American oak samples (A_1) vs. 338-d American oak sample (A_3). L_5 = similar contrast for French oak (F_1 vs. F_3). L_4 = value of 239-d American oak sample vs. mean of 115 and 338-d American oak samples. L_6 = similar contrast for French oak. D = significant deviation from linearity.

²) Bonferroni contrasts not performed for $P < 0.05$.

NS, *, **, *** = Not significant, significance at $P < 0.05$, $P < 0.01$ and $P < 0.001$, respectively.

Results and Discussion

The mean values for pH, titratable acidity (TA), volatile acidity (VA), potassium and ethanol are shown in Table 1. For the same data, the analyses of variance are summarized with the significance of calculated contrasts in Table 2. Although the change in pH over time was not significant, the pH decreased as barrel aging time increased, with a consistently greater decrease for the French oak-aged wines. Titratable acidity increased over time, with the French oak-aged wine having significantly higher values than the American oak samples at all storage times. Both the drop in pH and rise in TA with time in oak were reported in previous studies of barrel aged brandy and whiskey (VALAER and FAZIER 1936, LIEBMAN and SCHERL 1949, GUYMON and CROWELL 1970, ONISHI *et al.* 1977).

For both French and American oak-aged wines, part of the increase in TA may be attributed to concentration of acids as a result of evaporation from the barrels, which ranged from 5 to 8 l/barrel at 338 d, a 2.2–3.5 % concentration. However, the large increase in TA of 6.6 and 10.5 % after 338 d of aging in American and French oak barrels, respectively, indicates that extraction of acid from the wood has occurred.

The potassium content of the American oak-aged wines was significantly higher than that of the French and the control wines. While the increase in potassium indicates its extraction from oak, which has not been previously reported, the pH and concentration of potassium and titratable acidity represent a complex equilibrium. Carboxylic, phenolic and volatile acids extracted from wood decrease the pH and increase the TA. However, precipitation of potassium bitartrate from a wine in which tartaric is the major acid and the pH is below 3.6 will result in lower values for titratable acidity, potassium and pH. If the pH were greater than 3.6 initially, on precipitation of potassium bitartrate, a pH rise would be expected (BIEDERMANN 1953, BOULTON 1980).

Table 3
Absorbance of wines^{1,2)} at 420 nm and 520 nm and calculated values for color density and hue
Extinktion der Weine bei 420 und 520 nm sowie die daraus errechneten Werte der Farbintensität und -tönung

	Storage time in barrel (d) and type of barrel ³⁾						
	0	115 Am	115 Fr	239 Am	239 Fr	338 Am	338 Fr
A _{420 nm}	3.86	4.01	4.13	4.21	4.19	4.29	4.88
A _{520 nm}	5.52	5.79	6.05	6.02	5.99	6.19	6.33
A _{520 nm}	3.94	4.35	4.49	4.61	4.66	4.46	4.53
bleached with SO ₂							
A _{420 nm} + A _{520 nm} (color density)	9.38	9.80	10.18	10.23	10.18	10.48	11.21
A _{420 nm} + A _{520 nm} (color hue)	0.699	0.693	0.683	0.699	0.699	0.693	0.771

1) Results are given for a 10 mm path length, corrected for dilution.

2) Statistical analyses not performed because duplicate samples were not prepared.

3) Am = American oak; Fr = French oak.

The change in volatile acidity with barrel aging followed the same pattern as TA, with a greater increase for the French oak samples. For the 338 d French and American oak-aged wines, the increase in volatile acidity (expressed as g tartaric acid/l) represented 27.5% and 22.5 %, respectively, of the increase in titratable acidity. These values, which are consistent with previous data in which 15—35 % of the total acid extracted from oak was volatile acid (JAROSZ and JAROSZ 1969), suggest that the increase in VA is due primarily to extraction from the oak.

Table 4

Means¹⁾ (n = 2) and least significant differences (LSD) of the phenolic content of wines (mg GAE/l)
Mittelwerte und Signifikanzschwellen (LSD) des Phenolgehaltes der Weine (mg GAE/l)

	Storage time in barrel (d) and type of barrel ²⁾							LSD
	0	115 Am	115 Fr	239 Am	239 Fr	338 Am	338 Fr	
Total	2050 ^a	2050 ^a	2087 ^{ab}	2081 ^{ab}	2150 ^{bc}	2090 ^{ab}	2165 ^c	70.27 (P < 0.001)
Polymeric	710 ^a	744 ^{ab}	762 ^{bc}	800 ^{cd}	756 ^b	835 ^{de}	860 ^e	42.49 (P < 0.001)
Nonflavonoid	464 ^a	513 ^{ab}	525 ^{ab}	510 ^{ab}	542 ^b	498 ^{ab}	540 ^b	71.47 (P < 0.001)
Polymeric nonflavonoid	43 ^a	50 ^{ab}	55 ^{bc}	55 ^{bc}	60 ^c	50 ^{ab}	60 ^c	7.26 (P < 0.001)

¹⁾ Means within each row with the same letter superscript are not significantly different at the level of significance indicated.

²⁾ Am = American oak; Fr = French oak.

Table 5

Degrees of freedom (df), mean squares (MS), F-ratios, significance of differences among treatments, and significance of contrasts (L_i) among treatments as calculated from Bonferroni confidence intervals for phenolic analyses

Statistische Auswertung der Phenolanalysen: Freiheitsgrade (df), Mittlere Abweichungsquadrate (MS), F-Werte, Signifikanz der Unterschiede zwischen den Behandlungen, Signifikanz der Kontraste (L_i) zwischen den Behandlungen (Berechnung aus den Konfidenzintervallen nach Bonferroni)

	Source of variation	df	MS	F	Sig.	L ₁	L ₂	L ₃	L ₄	L ₅	L ₆
Total phenols	Wines	6	4083.61	24.39	***						
	Error	7	187.42			C < R	A < F	NS	NS	F ₁ < F ₃	NS
Polymeric phenols	Wines	6	5650.40	92.31	***						
	Error	7	61.21			C < R	NS	A ₁ < A ₃	NS	F ₁ < F ₃	D
Nonflavonoid phenols	Wines	6	1449.73	8.37	**						
	Error	7	173.21			C < R	A < F	NS	NS	NS	NS
Polymeric nonflavonoid phenols	Wines	6	77.97	43.67	***						
	Error	7	1.78			C < R	A < F	NS	NS	NS	NS

¹⁾ Refer to Table 2 for explanation of contrasts.

NS, **, *** = Not significant, significance at P < 0.01 and P < 0.001, respectively.

The significant 0.26 % (v/v) increase in ethanol with 338 d of barrel aging is primarily the result of evaporative loss of water during storage, as this corresponds to the increase in concentration predicted from the ullage. There was no difference in ethanol concentration between the two oaks.

As shown in Table 3, with the exception of the 239-d French oak-aged wine, there was a consistent increase in color density with barrel aging, with a greater increase in the French oak samples. The increase in 420 nm and 520 nm absorbances was nearly proportional, consequently, for both series of wines the color hue remained nearly unchanged. The increase in the absorbance at 420 nm with barrel aging was greater in the French oak-aged wines, consistent with the results reported previously in brandy studies (GUYMON and CROWELL 1970). Whereas the increase in the absorbance at 420 nm largely reflected the extraction of colored material from oak over time (REAZIN 1981), after 338 d of barrel aging in French and American oak, respectively, 75 and 78 % of the increase in absorbance at 520 nm were the result of polymerization of anthocyanins. The rest of the increase in 520 nm absorbance was due to the drop in wine pH which increased the proportion of anthocyanins in the red form, the flavylium ion (RIBÉREAU-GAYON 1974).

The mean values for phenolic data are shown in Table 4, with the analyses of variance and significance of contrasts listed in Table 5. All phenolic values increased significantly with barrel aging, with a greater extraction of total and nonflavonoid phenols and of polymeric (or large molecular weight) nonflavonoid phenols occurring in French barrels. The highly significant increase in total phenolic content of the wines with barrel aging was primarily due to nonflavonoid phenols, as observed previously in oak extracts (SINGLETON 1974). As indicated by the very small increase in polymeric nonflavonoid content over time and the primarily nonflavonoid composition of oak phenols,

Table 6

Comparison of control vs. oak aged wines and of wines of the same storage time in American vs. French oak · Results of duo-trio difference tests for aroma and taste of wines (n = 13 judges × 2 reps)

Beurteilung des Weinaromas und -geschmacks mit Hilfe des Triangeltests (n = 13 Prüfpersonen × 2 Wiederholungen) · Vergleich von Kontrollweinen (Glasballon) mit Eichenfaß-gelagerten Weinen sowie von Weinen aus amerikanischen Eichenfässern mit Weinen aus französischen Eichenfässern bei gleicher Lagerungsdauer

Comparison ¹⁾	Correct responses	Significance
Control vs. 115 Am	22	***
Control vs. 115 Fr	24	***
Control vs. 239 Am	23	***
Control vs. 239 Fr	19	*
Control vs. 338 Am	24	***
Control vs. 338 Fr	22	***
115 Am vs. 115 Fr	19	*
239 Am vs. 239 Fr	15	NS
338 Am vs. 338 Fr	15	NS

¹⁾ Am = American oak; Fr = French oak.

NS, *, *** = Not significant, significance at $P < 0.05$, $P < 0.001$, respectively.

the increase in polymeric phenols is primarily due to the more rapid polymerization of wine flavanoid phenols in oak than in glass.

Results for the difference tests are shown in Tables 6 and 7. The glass-aged control wine was very highly significantly different from all of the oak-aged wines ($P < 0.001$), with the exception of the 239-d French oak sample ($P < 0.05$). In comparing French and American oak-aged wines at each storage time, only the 115-d wines were significantly different from each other ($P < 0.05$). For wines stored for different times in the same kind of oak the only significant difference was between the wines aged in American oak for 115 and 338 d ($P < 0.05$).

The differences between the samples aged for 115 d in French and American oak were minor and too small to be described, whereas the difference between the control and the oak-aged wines were much more noticeable. The glass-aged control was dominated by a vegetative note, which was greatly reduced with oak aging. In addition, the oak-aged wines had aromas imparted by the barrel aging, which were absent in the control. In another publication, comparison of these aromas is discussed at greater length (AIKEN and NOBLE 1983). Although the wines were not compared for astringency or bitterness intensity, this was probably not a factor in the discrimination between the control and the barrel aged wines because of the high phenolic content in the wine initially (2050 mg gallic acid equivalents (GAE)/l) and the small increase of 115 mg GAE/l produced by storage in oak for 338 d. In a previous study (ARNOLD and NOBLE 1979), differences in astringency or bitterness were not detected when the phenolic content was increased by 110 mg GAE/l in a base wine with an initial concentration of 232 mg GAE/l.

The lack of difference in wine flavor between wines aged in French and American oak barrels may be attributed to several factors. Most probably, the strong varietal character of the very astringent Cabernet Sauvignon wine when assessed at 20 months of age masked the oak flavor differences. Further, differences between American and French oak were minimized by utilizing barrels coopered by the same practices. Although the sources of the air-dried oaks differed here, PONTALIER *et al.* (1982) found

Table 7

Comparison of wines stored in the same type of oak for different storage times · Results of duo-trio difference tests for wine aroma ($n = 13$ judges)

Beurteilung des Weinaromas mit Hilfe des Triangeltests ($n = 13$ Prüfpersonen) · Vergleich von Weinen, die im selben Eichenfaßtyp unterschiedlich lang lagerten

Comparison ¹⁾	Correct responses	Significance
115 Am vs. 239 Am	9	NS
115 Am vs. 338 Am	10	*
239 Am vs. 338 Am	8	NS
115 Fr vs. 239 Fr	6	NS
115 Fr vs. 338 Fr	9	NS
239 Fr vs. 338 Fr	7	NS

¹⁾ Am = American oak; Fr = French oak.

NS, * = Not significant, significance at $P < 0.05$, respectively.

for Allier oak that the method by which the staves were dried affected wine flavor more than other cooperage factors. Because of the unpredictable nature of flavor reactions, these results should not be generalized to all wines, without further experimentation. Future research investigating the effects of similarly coopered French and American barrels on the flavor of other red wines, and certainly of white wines is essential.

Summary

Cabernet Sauvignon wine aged in similarly coopered, lightly charred French and American oak barrels had increased titratable acidity and decreased pH values over the control glass-aged wine, which was primarily the result of extraction of acid from the wood. Extracted volatile acids accounted for 22.5 and 27.5 % of the increase in titratable acidity in American and French barrels, respectively. As with the pH and titratable acidity changes, the increase in total and nonflavonoid phenols extracted from the barrels was greater in the French than in the American oak-aged wines. Potassium increased significantly with barrel aging in the American oak samples, with no significant difference occurring between the control and the French. In sensory difference tests, all oak-aged wines could be discriminated from the glass-aged control. However, in comparing wines aged in French and American oak for the same time, only those aged for 115 d were significantly different ($P < 0.05$).

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