

Rapid Induction of Ageing Character in Brandy Products – Part II. Influence of Type of Oak

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As part of a broader study that investigated techniques for the rapid induction of the needed ageing character in brandy products, the effect of oak type on quality and chemical composition of oak wood extracts and matured and unmatured pot-still brandy, is reported on. Extracts, prepared from American and French oak chips supplied by a South African cooper, and from commercially obtained oak, and representing different levels of toasting, were added to 70% (v/v) unmatured pot-still brandy and stored for eight months in glass containers (Schott bottles) at room temperature, or in the case of controls, below 0°C. Matured and unmatured (control) pot-still brandy samples were analysed for wood-derived congeners by means of HPLC and GC. Although French oak initially yielded better quality products, these effects lost prominence and, after eight months maturation, yielded similar sensory quality to American oak. French oak samples had higher concentrations of wood-derived congeners (including eugenol, the furan derivatives and aromatic aldehydes). However, the American oak generally contained higher concentrations of oak lactones than their French counterparts, with higher proportions of the more sensorially potent *cis*-form of lactone than its *trans*-isomer.

Over the years wood from many different tree species has been used to manufacture barrels. These species include red oak, chestnut, red or sweet gum, sugar maple, beech, black cherry, acacia karri and mulberry. However, oak (genus *Quercus*) soon proved to be the most suitable for wine and spirit maturation (Chatonnet, 1999). The oak species most commonly used in barrel-making for brandy and wine maturation are *Quercus alba*, also known as American oak, which grows in different areas in the United States, *Q. petraea* (Sessile oak) and *Q. robur* (pedunculate oak), which grow in Europe (Walker, 1996). The most popular wood for barrels is French oak, although oak wood from Eastern Europe and Russia is becoming increasingly available. Wine and spirit producers often prefer certain oak types over others for the maturation of different kinds of alcoholic beverage. The French “appellation d’origine contrôlée” (AOC) *eaux-de-vies*, for example, are traditionally matured in barrels fabricated from oak from nearby regions, with cognac almost exclusively matured in oak from the Limousin and Tronçais regions (Guymon & Crowell, 1970). French oak, primarily Limousin, have been used in large quantities for ageing brandy in several countries. These include South Africa, Germany, Mexico and Australia (Guymon & Crowell, 1970). American whiskies are naturally matured in American oak barrels, while the Scotch whiskey industry has gradually shifted from using predominantly European to American oak barrels (Clyne *et al.*, 1993).

The methods by which barrels are manufactured are not the same, the greatest differences being observed between European and American cooperages. The degree of heating also varies, being mild to moderate in most European cooperages. In contrast,

in American cooperages where the barrels are to be used for the production of bourbon or other grain whiskies, heating is usually intense, leading to intense charring (Mosedale & Puech, 1998). Compared to French (i.e. Tronçais oak, Limousin oak, Gascony oak), Russian and Bulgarian oaks, American oak has the least amount of tannin (Puech, 1984).

Since wood for barrel making varies in price, and because barrels can only be used for maturation purposes a limited number of times, it is evident that the development of alternatives to traditional barrel maturation, such as the use of wood extracts, could have significant economic implications for the wine- and spirit industries. To this effect a number of workers have carried out studies on the different extractable compounds in different types of oak woods (Guymon & Crowell, 1968; Singleton, 1974; Hacker, 1991; Pérez-Coello *et al.*, 1999; Masson *et al.*, 1995, 2000).

American and French oak chips obtained from a cooper (specially prepared), and from a commercial supplier, and representing different levels of toasting, were compared.

This is the second in a series of articles (Van Jaarsveld *et al.*, 2009). The first article concerned the importance of extracts in brandy maturation, the preparation of oak extracts, the influence of different extraction conditions and mediums, and suppliers of oak chips on chemical and sensory profiles. This paper discusses the effect of oak type on the sensory and chemical profiles of fortified extracts and unmatured and matured pot-still brandy. Toasting is discussed in a separate paper. Interactive aspects of the collected data will be discussed in a subsequent review paper.

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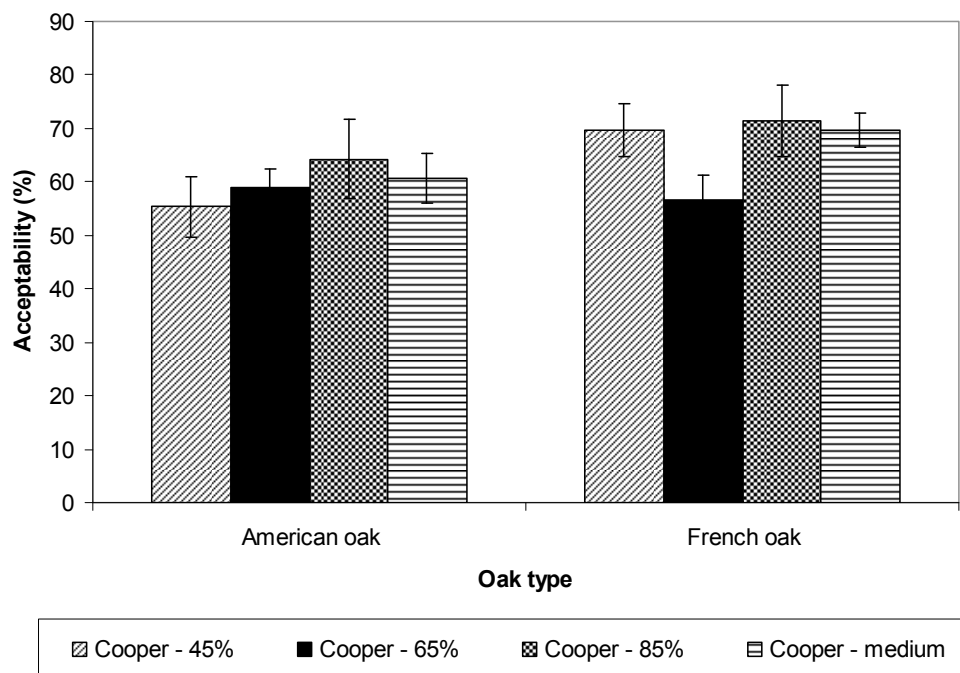


FIGURE 1

Percentage sensory acceptability of oak extracts prepared from chips of different types (American and French) of differently toasted (untoasted, medium and heavy) oak obtained from a cooper and subjected to various treatments. Legend, i.e. cooper 45%, indicate the group subjected to statistical treatment, i.e. only cooper and the 45% (v/v) concentration level, with all other treatments nested. Refer to part I in this series for more details regarding various treatments. Error bars represent the standard error of the mean.

MATERIALS AND METHODS

Materials and methods have been described previously (Van Jaarsveld *et al.*, 2009). Briefly, American and French oak chips, representing different levels of toasting and obtained from a cooper and a commercial supplier, were placed in a water- or a 55% (v/v) ethanol medium and boiled under reflux with backflow cooling for 5 h. Open concentration or concentration under vacuum using a Buchi rotavapour followed. Extracts were fortified to 40% (v/v). Fortified extracts were added to 70% (v/v) unaged pot-still brandy (procured as one batch) at 60 mL/L and stored for eight months in glass containers (Schott bottles) at room temperature, or in the case of controls, below 0°C. Samples were subjected to sensory and chemical analysis. Fortified (40% v/v) extracts were sensorially evaluated, in duplicate, by a panel of seven judges for acceptability for brandy production and expressed in terms of a simple “yes” or “no response”. The number of “yes” scores for each extract evaluated were expressed as a percentage of the total number of evaluations per extract. Pot-still brandy samples were also sensorially evaluated for overall quality by a panel of seven experienced judges. A line method was used, i.e. evaluating the brandy characteristics by marking an unstructured, straight 10 cm line. The left-hand and right-hand ends of the line were indicated by the terms, “not detectable” and “prominent”, respectively.

RESULTS AND DISCUSSION

From the analysis of variance (ANOVA) of the treatments, oak type (American or French), level of toasting, supplier (i.e. cooper or commercial), type of medium (ethanol or water), concentration type (open or reduced pressure) and concentration level (45, 65 or 85%), with results given in Tables 1 to 5 and Figs 1 to 4, the

significance of the effects of the different treatments and their interactions were determined. Type of oak as a treatment is discussed in this article.

Extracts

American and French oak have sufficiently different extractables so that it is often possible for an experienced taster to identify which oak was used for brandy or wine ageing (Singleton, 1974). In this study, sensory evaluation (for acceptability only) of fortified oak extracts showed, for both commercial and specially prepared chips from a cooper, that French oak yielded equally or more acceptable extracts than American oak (Fig. 1). Oak species, therefore, certainly influenced the observed differences in the character of wood extracts prepared from them. This agrees with the studies of Singleton (1995) and Francis *et al.* (1992), who reported that American oak was generally less intense in aroma properties than French oak.

Unmatured pot-still brandy

Except for *cis*-oak lactone, French oak generally yielded higher concentrations of volatile wood-derived congeners than American oak (Table 1). The concentrations of less volatile wood-derived congeners in unaged pot-still brandy prepared from American oak extracts are either similar to ($p \geq 0.05$) or significantly lower ($p \leq 0.05$) than those from French oak (Table 2).

Eight-month matured pot-still brandy

Little difference in overall sensory quality ($p \geq 0.05$) was observed between eight-month matured pot-still brandies prepared with extracts using American oak chips and those prepared using French oak chips (Fig. 2).

TABLE 1

Gas Chromatographic determination of wood-derived congeners in unmatured pot-still brandy prepared from oak (cooper only) extracts.

Type/Treatment	n*	Mean (mg/L)						
		Furfural	5-Methyl-furfural	Guaiacol	<i>trans</i> -Oak lactone	<i>cis</i> -Oak lactone	<i>o</i> -Cresol	Phenol
American oak	12	17.618 ^b (1.893)**	0.798 ^a (0.342)	0.950 ^a (0.185)	nd***	0.161 ^a (0.076)	nd	nd
French oak	24	30.238 ^a (2.535)	0.895 ^a (0.265)	1.143 ^a (0.121)	nd	0.000 ^b (0.000)	nd	nd
		Ethyl guaiacol	<i>p</i> -Cresol	Eugenol	2,6-Dimethoxy-phenol	5-Hydroxy-methyl furfural	Vanillin	
American oak	12	0.000 ^a (0.000)	nd	0.000 ^a (0.000)	29.470 ^a (1.392)	2.640 ^a (0.557)	2.580 ^a (0.460)	
French oak	24	0.004 ^a (0.004)	nd	0.015 ^a (0.015)	33.114 ^a (0.975)	8.281 ^a (1.728)	9.616 ^a (3.178)	

*, Number of evaluations of samples; **, standard error of the mean; ***, not detected. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of extracts concentrated by 65% (v/v).

TABLE 2

High-performance Liquid Chromatographic analysis of unmatured pot-still brandy prepared from oak (cooper only) extracts.

Type/Treatment	n*	Mean (mg/L)				
		Gallic acid	Catechin	Vanillic acid	Syringic acid	<i>p</i> -Coumaric acid
American oak	12	1.230 ^b (0.102)**	0.389 ^b (0.389)	1.006 ^b (0.290)	2.614 ^b (0.562)	0.000 ^a (0.000)
French oak	24	8.005 ^a (0.412)	4.163 ^a (1.108)	1.460 ^a (0.246)	3.726 ^a (0.511)	0.494 ^a (0.120)
		Syringaldehyde	<i>m</i> -Coumaric acid	Ellagic acid	Coniferaldehyde	Sinapaldehyde
American oak	12	5.844 ^a (1.310)	0.167 ^a (0.048)	15.637 ^b (3.031)	7.989 ^a (2.011)	25.832 ^a (6.887)
French oak	24	5.880 ^a (0.971)	0.271 ^a (0.065)	44.187 ^a (6.969)	8.023 ^a (1.374)	31.000 ^a (4.605)

*, Number of evaluations of samples; **, standard error of the mean. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of extracts concentrated by 65% (v/v).

French oak was described as more “woody” and “toasted” than American oak (Fig. 2). As reported by Sefton *et al.* (1993), the differences in sensory quality between American and French oak reflected in the concentrations of oak components in the corresponding oak extracts and pot-still brandies (Tables 3 and 4).

Except for *trans*- and *cis*-oak lactones, the concentrations of volatile and less volatile components were generally higher in eight-month matured pot-still brandies prepared from French oak as compared to American oak (Tables 3 and 4). European oak is known to be higher in extractables than American oak,

but with the same substances (Singleton, 1974, 1995). The total impact of higher concentrations of wood-derived congeners, but lower concentrations of the important flavour compounds *cis*- and *trans*-oak lactones in French samples, would probably explain the minor differences ($p \geq 0.05$) in quality between French and American samples aged in glass containers for eight months. Unmatured pot-still brandies prepared with French oak extracts were initially sensorially more favourable compared to their American oak counterparts. Differences in eight-month matured pot-still brandies, however, were less conspicuous. The most

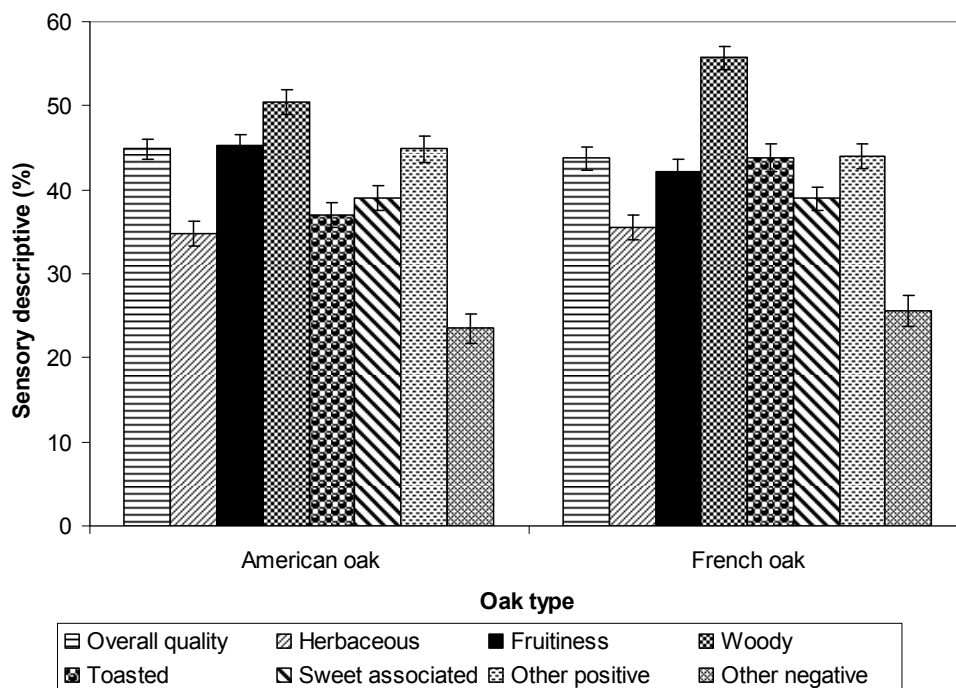


FIGURE 2

Overall sensory quality and other sensory descriptives of eight-month matured pot-still brandy from oak extracts prepared with chips of different types (American and French) of differently toasted oak obtained from a cooper and subjected to various treatments. Only the 65% (v/v) concentration level was considered in the statistical evaluation. Refer to part I in this series for more detail regarding the various treatments. Error bars represent the standard error of the mean.

TABLE 3

Gas Chromatographic analysis of eight month matured pot-still brandy prepared using oak extracts from different species of oak chips specially prepared by a cooper.

Type/Treatment	n*	Mean (mg/L)						
		Furfural	5-Methyl-furfural	Guaiacol	trans-Oak lactone	cis-Oak lactone	Phenol	o-Cresol
American oak	48	17.816 ^b (0.926)**	0.827 ^b (0.174)	0.530 ^a (0.045)	0.029 ^a (0.022)	0.183 ^a (0.047)	0.000 ^a (0.000)	0.000 ^a (0.000)
French oak	48	22.857 ^a (1.738)	1.108 ^a (0.226)	0.540 ^a (0.045)	0.020 ^a (0.020)	0.024 ^b (0.024)	0.012 ^a (0.012)	0.005 ^a (0.005)
		Ethyl guaiacol	p-Cresol	Eugenol	2,6-Dimethoxy-phenol	5-Hydroxy-methyl furfural	Vanillin	
American oak	48	2.328 ^b (0.066)	0.000 ^a (0.000)	0.142 ^b (0.029)	34.371 ^b (0.384)	3.222 ^b (0.346)	2.888 ^b (0.292)	
French oak	48	2.585 ^a (0.048)	0.011 ^a (0.011)	0.250 ^a (0.043)	36.945 ^a (0.490)	4.616 ^a (0.408)	3.857 ^a (0.446)	

*, Number of evaluations of samples; **, standard error of the mean. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of extracts concentrated by 65% (v/v).

plausible explanation for this phenomenon is the increase in, or rather, appearance, of *trans*-oak lactone in eight-month matured pot-still brandies (not observed in unmatured preparations) and significantly higher quantities of *cis*-oak lactone in American oak preparations as compared to their French oak counterparts. Although analysis revealed differences in concentrations of wood-derived congeners, tasters were unable to detect significant

differences between eight-month old pot-still brandies prepared with American oak from those prepared using French oak extracts in this study. In addition to the concentration and detection threshold limits of the individual wood-imparted compounds, their synergistic actions and long term stability are important factors impacting on oak aroma and taste (Singleton, 1995). Other authors have also reported sensory panel difference tests showing

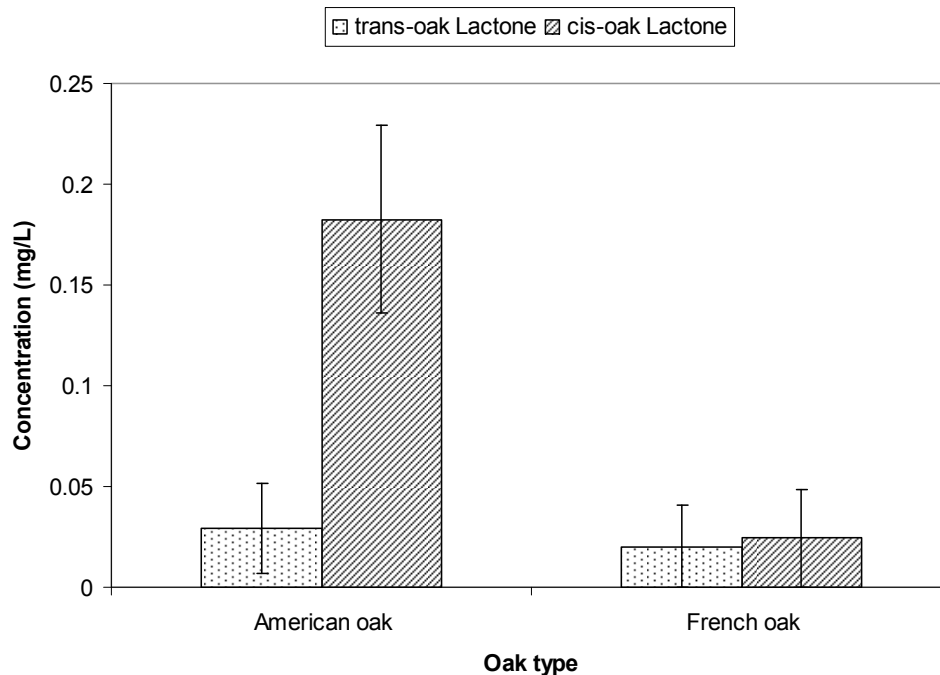


FIGURE 3

GC-determined concentration of *trans*-oak lactone and *cis*-oak lactone in eight-month matured pot-still brandy samples prepared with extracts representing chips of different types (American and French) of differently toasted oak obtained from cooper and subjected to various treatments. Only the 65% (v/v) concentration level was considered in the statistical evaluation. Refer to part I in this series for more detail regarding the various treatments. Error bars represent the standard error of the mean.

TABLE 4

High-performance Liquid Chromatographic analysis of eight-month matured pot-still brandy prepared from oak (cooper only) extracts.

Type/Treatment	n*	Mean (mg/L)						
		Gallic acid	Proto-catachuic acid	<i>p</i> -Hydroxybenzoic acid	Catechin	Vanillic acid	Syringic acid	<i>p</i> -Coumaric acid
American oak	48	0.863 ^b (0.075)**	0.574 ^a (0.157)	0.055 ^b (0.027)	1.655 ^a (0.909)	0.978 ^b (0.159)	2.398 ^b (0.284)	0.000 ^a (0.000)
French oak	48	7.380 ^a (0.320)	0.410 ^b (0.130)	0.736 ^a (0.159)	0.750 ^b (0.218)	1.864 ^a (0.196)	3.081 ^a (0.376)	0.014 ^a (0.010)
		Syringaldehyde	<i>m</i> -Coumaric acid	Ellagic acid	Coniferaldehyde	Sinapaldehyde		
American oak	48	7.753 ^b (0.854)	0.000 ^a (0.000)	4.743 ^a (0.625)	14.161 ^b (1.366)	14.167 ^b (1.921)		
French oak	48	10.610 ^a (1.118)	0.043 ^a (0.015)	4.859 ^a (0.606)	42.188 ^a (4.774)	16.493 ^a (2.313)		

*, number of evaluations of samples; **, standard error of the mean. Treatments with the same superscript within columns do not differ significantly ($p \geq 0.05$). Data representative of extracts concentrated by 65% (v/v).

little to no difference between the two types of oak (Francis *et al.*, 1992), notwithstanding differences in the concentrations of important oak compounds (Gawel *et al.*, 2002). Most studies, however, report on the ability to sensorially differentiate between American and French oak (Guymon & Crowell, 1970; Francis *et al.*, 1992; Singleton, 1995) with tasters being able to sensorially differentiate between the use of American from French barrels up to the third filling. Guymon & Crowell (1970) reported Limousin-

aged brandy to be milder in oak odour and aroma, more agreeable and subtle, and softer in taste than the American oak counterpart.

General

The concentrations of sensorially important compounds vary between French and American oak. American oak contains higher concentrations of oak lactones than French oak (Otsuka *et al.*, 1974; Anonymous, 1995; Chatonnet & Dubourdieu, 1998;

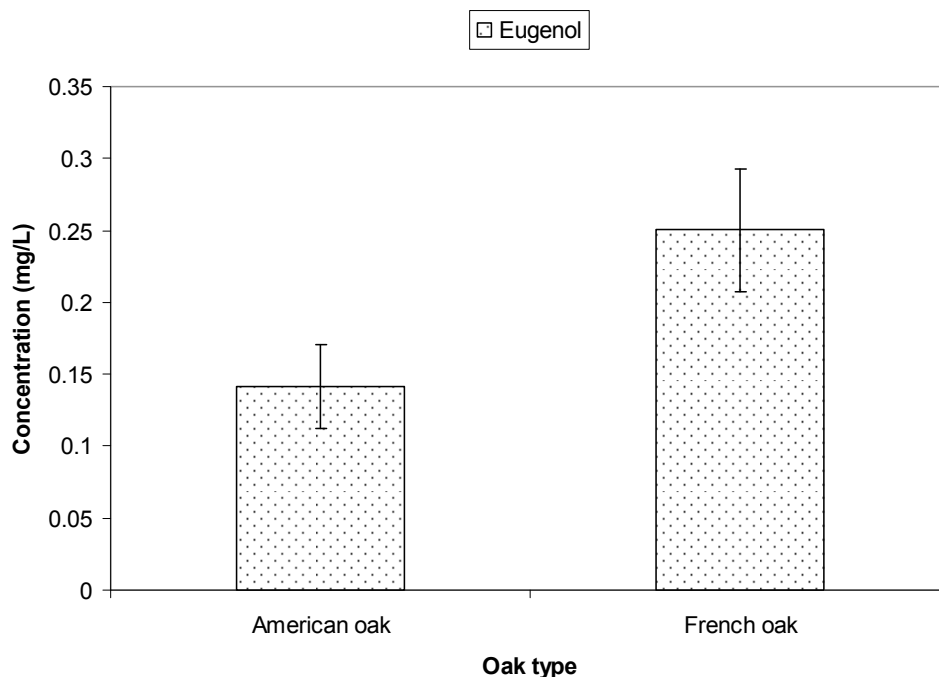


FIGURE 4

GC-determined concentration of eugenol in eight-month matured brandy samples prepared with extracts from chips of different types (American and French) of differently toasted oak obtained from a cooper and subjected to various treatments. Only the 65% (v/v) concentration level was considered in the statistical evaluation. Refer to part I in this series for more detail regarding the various treatments. Error bars represent the standard error of the mean.

Chatonnet, 1999; Gawel *et al.*, 2002; Spillman *et al.*, 2004). This difference is reflected in unmaturing and matured pot-still brandies in this study, where those prepared with American oak were generally characterised by higher concentrations of oak lactones than those prepared using French oak (Tables 1, 3; Fig. 3). In addition, and similar to reports in literature (Spillman *et al.*, 2004), higher proportions of the sensorially more potent *cis*- form of oak lactone are generally present in American and French oak-prepared pot-still brandies compared to its *trans*-isomer (Tables 1, 3; Fig. 3). Singleton (1995) reported a 77% *cis* to 23% *trans* ratio for European oak. Otsuka *et al.* (1974) reported greater amounts of *trans*- than *cis*-oak lactone. Also, as reported in literature (Singleton, 1995; Chatonnet & Dubourdieu, 1998; Hale *et al.*, 1999), American white oak was found to be richer in *cis*-methyl-octalactone (*cis*-oak lactone) than its European counterparts (Tables 1 and 3; Fig. 3). American oak, due to its higher content of *cis/trans* isomers of β -methyl- γ -oactalactone (oak lactone), has greater aromatic potential than European oak. This factor, together with its lower content of extractable polyphenols, makes American oak easier to identify (Chatonnet & Dubourdieu, 1998; Mosedale & Puech, 1998). Studies (Godden *et al.*, 1999) have shown that the source of the oak is more important and determining than the nature of the fermentation treatment. Variation in oak lactone, however, is not only influenced by the source of the oak, but also by other factors such as location/origin, with some authors (Sefton & Spillman, 1995) reporting opposite trends, i.e. finding American oak to be poor and French oak to be rich in *cis*-oak lactone. In previous studies performed at the ARC Infruitec-Nietvoorbij, using a different method of extraction, higher concentrations of *cis*-oak lactone, and lower concentrations of *trans*-oak lactone were found in French oak toasted at 230°C as compared to American oak toasted at 220°C. The aroma associated with oak

TABLE 5

Aromatic acid/aldehyde quotients in matured pot-still brandies.

Supplier	Type of oak	n*	Syringaldehyde: Vanillin ratio		
			Minimum	Maximum	Mean
Cooper	American oak	36	0.30	4.19	2.47 (0.15)**
	French oak	37	0.00	6.39	2.76 (0.22)
			Gallic acid: Vanillin ratio		
	American oak	36	0.01	0.77	0.25 (0.03)
	French oak	37	0.03	4.04	1.65 (0.15)
			Vanillic acid: Vanillin ratio		
	American oak	36	0.00	1.01	0.32 (0.04)
	French oak	37	0.00	1.83	0.38 (0.06)
			Syringic: Vanillic acid ratio		
	American oak	32	0.96	9.25	3.47 (0.45)
	French oak	43	0.00	5.52	2.26 (0.26)

*, Number of evaluations of samples; **, standard error of the mean. Data representative of brandies prepared with extracts from either French or American oak chips of different toast levels obtained from a cooper and concentrated by 65% (v/v).

lactone has been considered to be desirable by most assessors (Suomalainen *et al.*, 1974; Sefton *et al.*, 1993). Nevertheless, the quantity of extractable methyl-octalactones, particularly the *cis*-isomer, can sometimes be excessive in American oak. Excessive concentrations of lactones may give rise to a resinous odour that

masks the fruity character, with possible negative effects on aroma (Chatonnet & Dubourdieu, 1998; Sauvageot & Feuillat, 1999). This may partly explain why, in this study, American oak preparations, in spite of higher concentrations of methyl-octalactone, fared similarly to matured pot-still brandies prepared with French oak (Fig. 2). Although the concentrations of *trans*-oak lactone in this study (Tables 1 & 3) were generally below the reported thresholds of 0.8 mg/L, 0.067 mg/L and traces to 0.68 mg/L (Otsuka *et al.*, 1974; Singleton, 1995), *cis*-oak lactone on the other hand, generally in the case of American oak-prepared and/or commercial samples, was found at concentrations above the reported thresholds of 0.07 mg/L, 0.79 mg/L, 0.092 mg/L and 0.13 to 1.54 mg/L in brandies and commercial products of different ages (Otsuka *et al.*, 1974; Singleton, 1995; Mosedale & Puech, 1998). In addition, European cooperage oak samples are reported to have 161% of the extractable solids of North American samples and 154% of the phenol per unit of extractable solids, whereas American oak contributed more oak flavour to wine per unit of extract (Singleton, 1974).

As was found by Sefton *et al.* (1993), Pérez-Coello *et al.* (1999) and Spillman *et al.* (2004), higher concentrations of eugenol were generally found in samples prepared with French oak than in those prepared with American oak (Tables 1 and 3; Fig. 4). Singleton (1995) reported American oak to contain higher concentrations of eugenol than French oak. The furan derivatives, i.e. furfural were present in lower quantities in American than French oak extracts (Tables 1 and 3), which is in agreement with the results reported by other authors. Guymon & Crowell (1970), however, reported lower concentrations of furfural in Limousin barrel-aged brandy. In this study, quantities of aromatic aldehydes in pot-still brandies prepared from American and French oak, coniferaldehyde, synapaldehyde and syringaldehyde are the most abundant, with vanillin present in lesser quantities. Higher syringaldehyde than vanillin contents in liquid and powder oak extracts, wine spirit extracts, and spirits aged in oak barrels were also reported by other authors (Puech, 1988; Giménez Martínez *et al.*, 2001). All compounds were present in higher concentrations in French than American oak-prepared samples, including the aromatic acids syringic acid and vanillic acid (Tables 1 to 4). Guymon & Crowell (1970) reported that American oak contains more of these compounds than French oaks. Suomalainen & Nykänen (1972) reported syringaldehyde and vanillin to be generally the most abundant, with coniferaldehyde often equally abundant, but sinapaldehyde present in very small amounts, especially in French oak. Consideration must always be given to the fact that furan concentrations, and furan derivative concentrations, may increase considerably during heat treatment, and that observed differences between American and French oak may also be due to possible differences in heat treatments. In this study the syringic acid:vanillic aromatic acid ratios of unaged pot-still brandies from different suppliers prepared with American and French oak chips ranged from 2.4 to 2.6 and from 2.0 to 2.3, respectively. The ratios of these lignin-derived aromatic acids in eight-month matured pot-still brandies are shown in Table 5. Generally the maximum and average ratios were higher in French oak as opposed to American oak samples, except for the syringic acid:vanillic acid ratio (Table 5). The type of wood is known to influence the gallic acid:vanillin ratio in brandies; the gallic acid:vanillin ratio has been used in studying the quality of brandies (Giménez Martínez

et al., 2001). Puech (1988) reported syringic acid:vanillic acid ratios of 1.5 for Armagnac brandies, 1.1 to 7.17 for liquid wood extracts, and 1.6 to 3.5 for extracts in powder form.

CONCLUSIONS

Although French oak initially yielded better quality products, these effects lost prominence, yielding similar sensory quality to American oak after eight months maturation. The differences in sensory quality between American and French oak reflected in the concentrations of oak components in the corresponding oak extracts and pot-still brandies. French oak samples generally had higher concentrations of wood-derived congeners (including eugenol, the furan derivatives and aromatic aldehydes). American oak, however, generally contained higher concentrations of oak lactones than their French counterparts. The sensorially more potent *cis*-form was generally present in higher proportions than the *trans*-isomer. Although recommendations regarding single treatments can be made, it must be borne in mind that it is not always a single treatment, but a combination of various practices, that yield the best quality products.

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