

# Analysis of Oak Volatiles by Gas Chromatography-Mass Spectrometry after Ozone Sanitization

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**Abstract:** This research investigates the use of aqueous ozone to sanitize oak wine barrels and the effect it may have on the aroma volatiles from the oak. Toasted, new French oak blocks were treated with 1.0, 5.0, and 10 mg/L aqueous ozone before extraction in model wine solutions. Headspace solid-phase microextraction and gas chromatography coupled with mass spectrometry was used to analyze several oak volatiles. Ozone treatments did not show a significant change in the concentration for each of the volatiles analyzed ( $p = 0.05$ ). In contrast, some volatiles demonstrated significant changes in concentrations within oak blocks treated with 82°C (180°F) water for 5, 10, and 15 min ( $p = 0.05$ ). These results support the use of ozone as a good alternative sanitizing agent for oak wine barrels.

**Key words:** ozone, oak volatiles, SPME, GC-MS, barrel sanitation, enology

Wine is a complex product consisting of many flavors and aromas acting synergistically on consumer senses. These characteristic flavors and aromas make each wine product unique. There are numerous grape varieties found throughout the world, each of which may produce a wine containing flavors or aromas considered to be distinct or characteristic for that varietal. Aside from varietal differences, winemakers may deliberately manipulate the flavors and aromas found in wine through several practices, including grape maturity, duration of fermentation, or choice of yeast strain. Winemakers may choose to use several techniques in creating a style that is significantly different from other wines produced from the same grape variety. One such practice is the maturation and storage of wine in oak barrel containers.

The aromas found in wines may be derived from aging the wine in barrels made from oak wood for several months to sometimes years. The practice of using oak bar-

rels as a storage vessel has made a pronounced impact on the style of wines produced. The oak wood contains volatile compounds that can be extracted into the wines during their storage, producing noticeable aromas. Aromas such as vanilla, spice, toast, and smoke have been reported in wines aged in oak barrels (Mosedale et al. 1999, Singleton 1995). These aromas may vary in intensity and complexity depending upon the age and condition of the barrel.

Given the expense of purchasing new oak barrels, many wineries may repeatedly use them for several years. Barrel sanitation and optimal storage conditions are important for extending a barrel's lifetime. Occasionally, barrels may become contaminated with molds, yeasts, or other spoilage organisms and must be sanitized before further use. Chemical treatments such as sulfur dioxide, sodium peroxycarbonate, and soda ash can leave a residual that may influence the flavor and aroma of wine. Disposal of chemical sanitizers may also pose potential wastewater contamination issues. Chlorine has been used to sanitize barrels; however, this treatment resulted in the production of "cork taint" (trichloroanisol) (Fischer and Fischer 1997). Another treatment commonly used in the winery is hot water (Coggan 2003), which has proven its efficiency as a sanitizer (Wilker and Dharmadhikari 1997). However, hot water is expensive to produce and is suspected to strip flavors from oak barrels.

One alternative for oak barrel sanitization is aqueous ozone. Comparatively, ozone is highly reactive, produces oxygen as a by-product, and is less expensive to produce (Greene et al. 1993). The highly reactive nature and the strong oxidizing power of ozone are lethal to many organisms. In addition, it has a relatively short half-life as it

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Acknowledgments: This work was supported by a grant from the California State University Agricultural Research Initiative (ARI). The authors wish to thank Bob Rogers of Innerstave (Sonoma, CA) who provided the aromatic, toasted French oak blocks. The authors are especially grateful to Joe Mendez of Piper Environmental Group (Castroville, CA) for help and advice in ozone applications and for the donation of the MSR-1 ozone production unit and storage tank.

Manuscript submitted April 2004; revised August 2004

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decomposes back to molecular oxygen, producing very little residue, if any, in sanitized barrels. These benefits make ozone an attractive sanitizing agent.

Although the use of ozone as a barrel sanitizer may continue to gain popularity within the wine industry, it is not completely understood whether the strong oxidizing power of ozone may affect the oak barrel and its performance after treatment. It could be proposed that ozone may not only oxidize unwanted organisms but also react with the organic compounds within the wood that produce desired aromas in wines. Reactions between ozone and these volatile compounds during barrel sanitization may effectively diminish the volatile compounds within the oak and potentially affect the flavor and aroma of wines stored in them.

The purpose of this research was to investigate volatile aroma concentrations extracted from oak wood after ozone sanitization treatment. Volatile concentrations in oak were analyzed before sanitization and compared with final values after treatment. Any decrease in volatile concentration found in the oak was used to represent a reduction in such aroma compound caused by the ozone treatment. In addition, hot water treatments were investigated to see if they would cause a significant reduction in volatile concentrations within the oak wood. Finally, volatile recoveries from both treatments were then compared to see if ozone treatments diminish volatile concentrations greater than do standard hot water treatments.

## Materials and Methods

**Oak source.** New French oak (*Quercus sessilis*) (Allier, France) was provided by Innerstave of Sonoma, CA. The oak wood was seasoned for 10 months in the center of France and then another 14 months in Sonoma, California. It was stored at a length of 305 cm for the first 12 months, with a thickness of 5.4 cm and variable width. Throughout the final 12 months of seasoning the wood was cut to 15 cm in width and 1.3 cm thick; length remained the same. The oak stave was then toasted in an oven using dry forced air held between 177°C and 204°C for 6 hr. This resulted in a medium toast, having a cinnamon to very light chocolate color throughout the entire piece of wood. After toasting, the stave was cut into uniform blocks measuring 2.5 cm x 2.5 cm x 1.3 cm.

**Model wine solution.** As in similar studies, a model wine solution was used throughout the experiment to reduce interferences from the complex chemistry and additional volatiles found in traditional grape wine (Whiton and Zoecklein 2000). The solution was prepared using 10% ethanol (v/v) in deionized water and the addition of tartaric acid so that the total acidity was equal to 6.0 g/L. Sodium hydroxide was titrated into the solution to a final pH of 3.2. The stock solution was stoppered with a cork and stored in a refrigerator to prevent evaporation.

**Ozone treatment.** Ozone gas was produced by a corona discharge generator and bubbled into a tank of recir-

culating water. The water was kept at 20°C using a heat exchanger, and ozone concentration was measured by an in-line dissolved ozone analyzer (Rosemount Inc., Chanhassen, MN). To simulate barrel sanitizing within a winery, the outlet valve beneath the recirculating water tank was opened to produce a continuous flow of 104 to 131 mL water per minute. Two 6.4 mm holes were drilled 2.5 cm from the top of a 400-mL plastic beaker. The beaker was then placed beneath the stream, allowing the beaker to fill with ozonated water and continuously flow out the drain holes near the top.

For each treatment, three random pieces of the toasted oak blocks were selected, having a combined total surface area of 76.5 cm, and the total mass in grams was recorded. The blocks were then placed into the beaker with the continuous flow of ozonated water and submerged with stainless steel tongs for 2 min. Treatments of 1.0, 5.0, or 10 mg/L ozone were performed in triplicate. Oak blocks submerged in water without the addition of ozone were prepared as control treatments, which were also performed in triplicate. After each treatment, the oak wood was then removed and air dried for 2 hr.

**Hot water treatments.** Oak blocks were randomly selected as described above. A 1000-mL glass beaker was filled with 800 mL of deionized water and heated to 82°C on a hot plate. The oak blocks were submerged in the hot water with stainless steel tongs for 5, 10, or 15 min. Each treatment was performed in triplicate. Control treatments were performed by submerging blocks in water held at 20°C for the same 5-, 10-, or 15-min increments. The blocks from all treatments were allowed to air dry for 2 hr following treatment.

**Oak extraction.** After drying, each set of three oak blocks was placed into a ~266-mL glass jar containing 250 mL of model wine solution to mimic barrel maturation after fermentation. The total surface area of wood to volume of wine was estimated to be approximately 3.5 times that found in a standard 227-L barrel. The increased surface area of wood to volume of wine was chosen to amplify the extraction of oak volatiles into the wine solution. One jar was prepared for each treatment-replicate. The small amount of headspace in each jar was then purged with nitrogen gas and sealed with screwtop lid. The sealed jars were placed in a dark incubator and stored at 26°C (80°F) for 14 days. The jars were inverted three times each day to mix the wine solution.

After the 14-day incubation, 20 mL of each jar was pipetted into 40-mL amber glass vials and sealed with open-top polypropylene closures containing 0.01 cm poly(tetrafluoroethylene)/0.3 cm silicone rubber septa and a polyethylene protective cap. All samples were placed in a freezer (-15°C) and stored frozen solid until solid-phase microextraction (SPME) analysis.

**SPME analysis.** After thawing, 4.0 g of sodium chloride, a magnetic stirring bar, and 0.2 mL of a 2-octanol solution, to provide an internal standard, was added to each sample. The 2-octanol solution was prepared by dissolving

0.1 g 2-octanol into 100 mL of model wine. Each vial was placed in a 250-mL beaker of water heated by a hot/stir plate. The temperature was maintained at 40°C and the sample was stirred at 1250 rpm. The sample was allowed to equilibrate for 20 min before the septum of the vial was pierced with a SPME sampler (Supelco, Bellefonte, PA) containing a fiber with a 100- $\mu$ m polydimethylsiloxane coating. The SPME fiber was previously conditioned at 250°C for 30 min in the injection port of a gas chromatograph (GC). With continuous stirring, the fiber was depressed to allow contact with the sample headspace for 30 min. The fiber was then retracted into the sampler and immediately inserted into the port of a gas chromatograph. The SPME sampler remained in the injection port throughout each GC analysis to condition the fiber for the next extraction.

**Gas chromatography-mass spectrometry (GC-MS).** The samples were analyzed using a gas chromatograph equipped with a 30 m X 0.25 mm low bleed (5% phenyl)-methylpolysiloxane capillary column with a 0.25- $\mu$ m coating (Agilent Technologies, Palo Alto, CA) and interfaced to a mass spectrophotometer. The carrier gas was helium at a velocity of 1.0 mL/min with an injector port temperature of 250°C. The column was held initially at 40°C for 6 min, followed by a ramp of 15°C/min to 180°C, and then increasing 20°C/min to 280°C and holding at the final temperature for 15 min. The injection mode was splitless for 2 min and the detector was turned off the first 6 min and again during the final ramp to 280°C. The mass spectrophotometer was operated in the selective ion mode under autotune conditions and the area of each peak was determined by ChemStation software (Agilent Technologies).

**Oak volatile standards.** Furfural, methylfurfural, guaiacol, methylguaiacol, eugenol, isoeugenol, vanillin, and oak lactone standards (Sigma-Aldrich, St. Louis, MO) were used to determine their respective retention times under the above GC parameters. Standards were dissolved into model wine solution and four serial dilutions were prepared from the stock solution. Twenty mL of each dilution was pipetted into a 40-mL vial with 0.4 g NaCl, 0.2 mL 2-octanol solution (internal standard) and a magnetic stir bar. Each dilution was then analyzed using the above SPME and GC methods in triplicate and the peak area for each compound was determined. A calibration curve was generated by plotting the peak area of each compound and its ratio to the peak area of the internal standard for all dilutions.

**Duplicate analysis and spiked recoveries.** Several treatment jars were randomly selected and multiple 20-mL aliquots were taken to prepare duplicate vials. These vials were analyzed following the above SPME extraction and GC methods and compared to results obtained by vials of the same oak treatment. Multiple analyses on the same samples were important to demonstrate the repeatability of the proposed method.

Spiked recoveries also help demonstrate the validity of the method. This process is performed by spiking a sample

with a known standard and then determining the percent of the standard that is recovered by analysis. Random treatment jars were selected and 20 mL of the oak extracted model wine solution was transferred into 40-mL vials. An additional 0.2-mL solution containing a known amount of oak volatile standards was added and analyzed by the above SPME and GC methods. Recoveries were determined by first analyzing the oak wine solution of the given treatment and comparing the results observed after the addition of the known amounts of oak volatiles.

**Statistical analysis.** Correlations for each of the standard calibration curves were confirmed using linear regression analysis with Excel software (Microsoft, Redmond, WA) and  $R^2$  values greater than 0.95 were considered acceptable. Results determined for each oak treatment were calculated in  $\mu$ g analyte per gram of oak wood and an analysis of the variance (ANOVA) was performed using SPSS software (SPSS, Chicago, IL) to determine significant differences ( $p = 0.05$ ). Averages and standard deviations were also performed using Excel and SPSS software.

## Results and Discussion

Oak volatile standards (Sigma-Aldrich, St. Louis, MO) were used to first identify the retention times for each compound on the gas chromatograph. A GC using a flame ionization detector (GC-FID) was first used to analyze the compounds. However, because of its limited sensitivity, concentrations typically found in wine samples were considered too low to detect. By using the MS detector, sensitivity could be greatly increased. In fact, all compounds were easily identified by injecting a 50- $\mu$ L sample of the headspace just above the standard using a gas-tight syringe. However, this provided too concentrated a sample and clear peaks for some of the highly volatile compounds could not be separated. Diluting the samples into ethanol and then sampling the headspace and injecting via the SPME fiber provided better peak shape and better resolution between the peaks in the chromatograms. By injecting one sample at a time onto the GC column, retention times could easily be identified and were confirmed by using the ChemStation software. Using the previously mentioned GC conditions, baseline resolution was obtained for all compounds between 6.0 and 15.0 min in the chromatogram. Once retention times for each compound were determined, the selective ion mode (SIM) was programmed. Three ions were used to identify each peak by mass and relative abundance while avoiding unneeded low molecular weight ion noise. However, only two ions were chosen to select furfural due to the abundance of peaks with similar retention times and common ion fragments. Given the volatility of furfural and ion abundances, the compound was still easily identifiable with just the two ions. Using the SIM program, it was confirmed that each standard could also be identified using the SPME technique and no change in retention times were observed.

It has been previously published that  $\beta$ -methyl- $\gamma$ -octalactone (oak lactone) can be separated into both its *cis* and *trans* isomer (Chatonnet et al. 1999, Feuillat et al. 1997, Perez-Coello et al. 1997). When oak lactone was loaded onto the column, two peaks were found at retention times of 13.1 and 13.4 min. The location of these peaks when compared to published studies indicate that they are similar to other *cis* and *trans* isomer peaks reported by the authors (Gump 1999). Using these published studies, it was determined that the first oak lactone peak at a retention time of 13.1 was the *trans* isomer, and the second peak at 13.4 was the *cis* isomer.

**Calibration curves.** Although many of the peaks could easily be identified when using large headspace samples, it was important to establish calibration curves for each compound using concentrations typically found in oak samples. For some of the very low concentrations, peaks were not easily identifiable by eye, but were still able to be integrated by the ChemStation software. All standards generated curves containing an  $R^2$  value of greater than 0.95, except for isoeugenol. Isoeugenol concentrations appeared to be below the sensitivity threshold under these conditions and produced an  $R^2$  value of only 0.42. However, the curve was used for determining approximate concentrations in oak samples despite the weak correlation.

**Normalization of data.** Although all oak pieces had the same surface area measurements, different densities in the oak produced variable masses. Therefore, some treatment jars contained three oak pieces with a total mass of up to 20 g, while others contained as little as 16 g. In order to normalize the data recovered from all jars, the total analyte recovered was later divided by the total mass of oak used in that jar and recorded in units of  $\mu\text{g}$  analyte per gram of oak. However, no correlation between mass of oak used and amount of analyte recovered was observed.

**Ozone treatments.** Recoveries of oak volatiles after ozone treatments greatly varied between each analyte (Table 1). Some volatiles showed diminished concentra-

tions in the model wines with oak treated with ozone, while others surprisingly increased. When compared to the control wines, furfural, methylfurfural, and eugenol all showed some decrease in concentration after ozone treatment. However, methylguaiacol, vanillin and both *cis*- and *trans*-oak lactone demonstrated an increase in concentration in the wines. However, neither change in concentration from the control treatments was determined statistically significant ( $p = 0.05$ ) by ANOVA analysis. In addition, guaiacol and isoeugenol both showed variable increases and decreases in concentration depending upon the amount of ozone used. Therefore, ozone treatments of the oak did not significantly change the concentration of volatiles recovered in the wines.

**Hot water treatments.** Once the oak was placed into a hot water bath, it began to leach color, turning the water into a dark oak tea. This leaching effect was not observed in the cold water control treatments and seemed to provide an insight that a significant effect in the volatile concentrations would be recorded. In fact, when the model wines were analyzed in the GC, many volatile concentrations were decreased after 5- and 10-min treatments of hot water (Table 2). However, hot water treatments of 15 min showed very little effect on the concentration of many volatiles that were previously affected by the shorter treatment times. In addition, increases in concentration were observed in a few volatiles, including isoeugenol, which demonstrated increased concentrations in all of the hot water treatment samples.

Although changes in almost all volatile concentrations were identified by GC analysis of the wines, only a few were considered statistically significant ( $p = 0.05$ ) by ANOVA analysis. Significant decreases in concentration of furfural, methylfurfural, and eugenol were observed in hot water treatments of 5 min, while only vanillin and eugenol significantly decreased after treatments of 10 min. Any increases in concentration and all of the results observed in hot water treatments of 15 min were not found to be significantly different than the control wines.

**Table 1** Oak volatile recoveries ( $\mu\text{g}/\text{g}$  oak) with relative standard deviations (RSD) from oak blocks in model wine samples after treatments with ozone.

	Control		Ozone treatment					
	0 mg/L	RSD (%)	1 mg/L	RSD (%)	5 mg/L	RSD (%)	10 mg/L	RSD (%)
Furfural	1966a <sup>a</sup>	31	1506a	22	1400a	24	1406a	11
Methylfurfural	132a	39	112a	21	103a	27	117a	10
Vanillin	22.2a	32	28.2a	8	32.1a	10	19.2a	31
Eugenol	2.55a	7	1.32a	18	1.21a	34	1.04a	17
Guaiacol	0.94a	30	1.20a	21	0.51a	21	1.29a	33
Methylguaiacol	0.92a	18	1.33a	14	1.32a	7	1.35a	50
Isoeugenol	2.76a	24	1.65a	30	3.83a	40	2.59a	21
Oak lactone, <i>trans</i>	4.2a	18	9.0a	47	8.5a	8	13.0a	29
Oak lactone, <i>cis</i>	7.0a	9	11.0a	38	9.2a	30	11.0a	23

<sup>a</sup>Mean separation within rows followed by the same letter are not significantly different as determined by ANOVA ( $p = 0.05$ ).

**Table 2** Oak volatile recoveries ( $\mu\text{g/g}$  oak) with relative standard deviation (RSD) from oak blocks in model wine samples after treatment with hot water.

	Control (20°C)						Hot water (82°C)					
	5 min	RSD (%)	10 min	RSD (%)	15 min	RSD (%)	5 min	RSD (%)	10 min	RSD (%)	15 min	RSD (%)
Furfural	2151a <sup>a</sup>	13	1592a	34	2191a	20	1458b	7	1186a	22	1523a	29
Methylfurfural	180.4a	5	114.6a	36	159.7a	20	103.4b	5	78.0a	33	122.5a	34
Vanillin	25.6a	33	20.1a	22	19.2a	17	14.3a	10	9.3b	25	18.4a	34
Eugenol	2.1a	22	1.6a	16	1.4a	17	1.2b	24	1.2b	5	1.9a	25
Guaiacol	1.1a	30	0.9a	57	1.2a	74	1.1a	24	0.9a	20	1.0a	14
Methylguaiacol	1.1a	24	1.2a	38	1.0a	52	1.2a	29	1.6a	31	1.6a	25
Isoeugenol	2.8a	73	2.6a	55	1.7a	55	3.7a	6	3.9a	33	3.75a	37
Oak lactone, <i>trans</i>	10.9a	60	10.7a	67	14.7a	15	10.9a	35	8.2a	22	11.9a	37
Oak lactone, <i>cis</i>	14.0a	27	11.3a	24	8.9a	76	10.6a	30	5.2a	14	11.5a	25

<sup>a</sup>Means within a control or a hot water row followed by the same letter are not significantly different as determined by ANOVA ( $p = 0.05$ ).

**Duplicate analyses and spiked recoveries.** Repeatability of the method was estimated by the relative standard deviation (RSD) of the mean concentration of each volatile (Tables 1 and 2). For each of the ozone treatments these values range from 7 to 50% ( $n = 3$ ). RSDs observed in the hot water treatment ranged from 7 to 37% ( $n = 3$ ); however, the cold water control produced a very wide range from 5 to 76% ( $n = 3$ ). Further analysis into the cold water control discovered that the initial sample analyzed resulted in well below normal concentration values for many of the oak volatiles. If it could be assumed that the initial sample was flawed and was removed from the calculations, then the RSD ranges would then improve to 1.4 to 16%. In addition, duplicate analyses were performed on five treatment samples: the ozone control, ozone treatments of 1.0 mg/L and 10 mg/L, the cold water control of 15 min, and the hot water treatment of 15 min ( $n = 3$ ). The RSD values obtained for this replication fell within the range of RSD values reported in Tables 1 and 2. These results demonstrate the validity of the SPME and GC-MS methods used.

Spiked recovery analyses were performed on two treatment samples. Variable percent recoveries were observed ranging from 57 to 155% ( $n = 3$ ). These results are similar to spiked recoveries observed in previous studies, suggesting that components of wine other than ethanol and pH may affect the extraction of volatiles (Whiton and Zoecklein 2000).

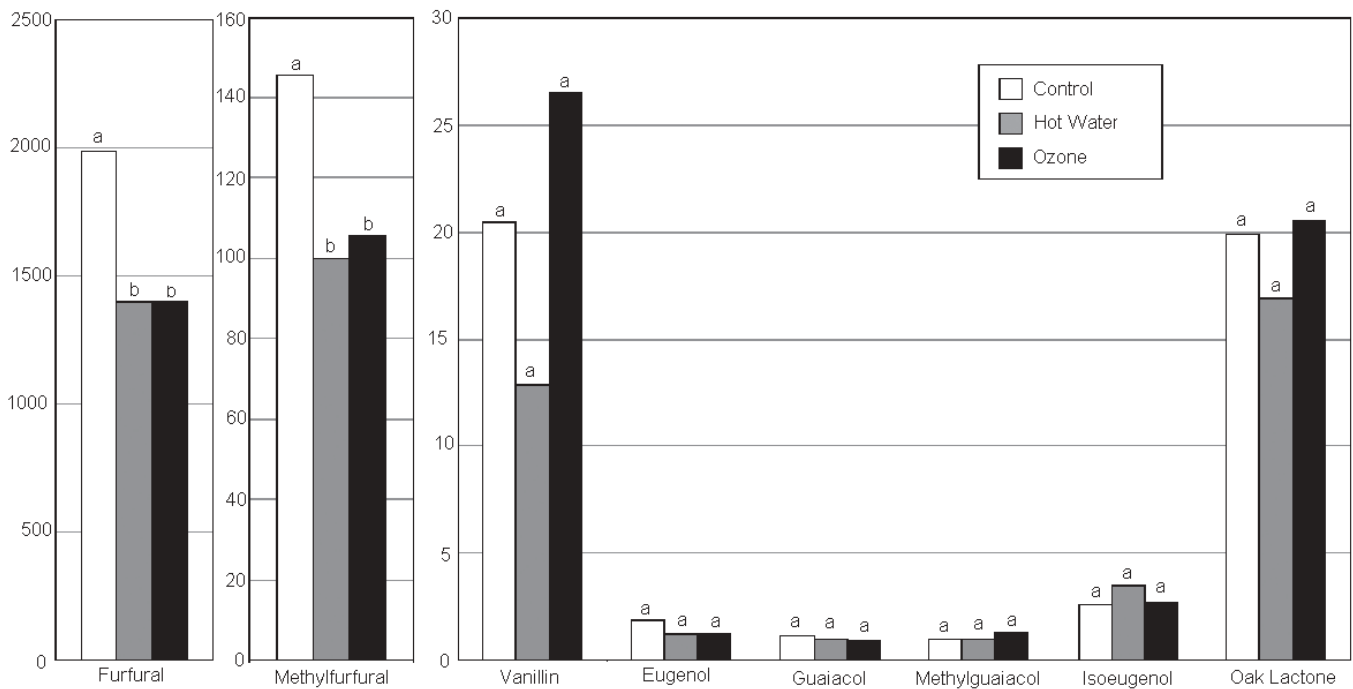
**Ozone and hot water comparisons.** As previously mentioned, many volatile recoveries varied within each treatment, and some individual hot water treatments were determined significantly different than the control. However, it may be possible to directly compare ozone and hot water recoveries by combining the factors into just three treatment groups: control (20°C water), ozone, and hot water (82°C). This was performed by first analyzing the recoveries for all the control treatments from both ozone and hot water. It was determined that for each analyte

neither of the results recovered from 2, 5, 10, and 15 min of cold water (20°C) were significantly different from each other and therefore, could be combined into one treatment factor designated as the control group ( $p = 0.05$ ). The same was then done for the hot water treatments and then the ozone treatments. All hot water recoveries (82°C) of 5, 10, and 20 min for each analyte were also not found to be significantly different from each other and were then combined into one treatment factor. For each analyte, ozone treatments of 1.0, 5.0, and 10 mg/L were found to not be significantly different from each other, with the exception of vanillin. Ozone treatments were also then combined into one treatment factor for each analyte. When analyzing the vanillin recoveries, 10 mg/L was determined significantly different than 5.0 mg/L ( $p = 0.05$ ), but neither was significantly different than 1.0 mg/L. Comparisons of vanillin recoveries between ozone and hot water treatments were still analyzed using the combined data model, and it should be noted that significant differences for the ozone treatment cannot be determined.

Using the combined results in just three treatment factors, the recoveries were then analyzed by ANOVA to determine significant differences from the control group (20°C water). The mean results from each treatment and recoveries are illustrated in Figure 1. Using this model, reduced recoveries were observed by most volatiles for both ozone and hot water treatments. However, a slight increase was observed for isoeugenol from the hot water treatment, while vanillin, oak lactone, and isoeugenol demonstrated increases from the ozone treatment. Only the recoveries for furfural and methylfurfural were determined significantly reduced for both the hot water and the ozone treatments.

## Conclusions

A method is presented to reliably analyze the volatile concentrations within toasted oak pieces as they are ex-



**Figure 1** Mean oak volatile recoveries ( $\mu\text{g/g}$  oak) in combined ozone (1.0, 5.0, and 10 mg/L for 2 min), hot water (5, 10, and 15 min at  $82^\circ\text{C}$ ), and control (2, 5, 10, and 15 min at  $20^\circ\text{C}$ ) treatments of toasted oak blocks. Mean separation within groupings labeled by the same letter are not significantly different as determined by ANOVA ( $p = 0.05$ ).

tracted into model wine solutions. The modified SPME method developed during this research enabled these volatiles to be quantified in very small concentrations and any such changes measured. Although the method followed during this research produced respectable extractions for most of the volatiles, it should be understood that  $40^\circ\text{C}$  for 30 min may not be the optimal parameters for all volatiles. Sensitivity may be increased with specific SPME sampling methods for each analyte.

When compared to control treatments ( $20^\circ\text{C}$  water) of 2 min, no significant effect on volatile recovery was observed ( $p = 0.05$ ) when using ozone at the levels that wineries are presently using. Some of the volatile concentrations within the hot water treatments demonstrated a significant decrease, suggesting that sanitization with  $20^\circ\text{C}$  aqueous ozone will diminish aroma volatiles within the oak wine barrel less than common sanitation methods using hot water ( $82^\circ\text{C}$ ). Sanitation of oak wine barrels with up to 10 mg/L ozone for 2 min does not diminish oak volatiles greater than 15 min of hot water.

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