

# Bourbon Barrel Aging Optimization

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## ABSTRACT

Bourbon barrel aging is a common and increasingly popular brewing technique. Oak sensory impact and bourbon spirit flavors are desirable properties of bourbon barrel aging, and are additive properties of this technique. Known issues with this aging technique include higher than normal infection rates for both *Lactobacillus* and *Pediococcus* beer spoilage organisms. The question of how to maximize desirable additive properties while minimizing harmful spoilage and evaporative loss was the trigger for this study. Oak analytical markers for desirable sensory attributes were monitored on a routine basis using a gas chromatography/mass spectrometry method, and individual barrels were evaluated for sensory spoilers, pH, and microbiological stability over time.

**Keywords:** barrel, bourbon, odor activity values (OAV)

## SÍNTESIS

La maduración en barricas de “bourbon” es una técnica común y muy popular de elaborar cerveza artesanal. El impacto sensorial del roble y del licor “bourbon” le imparten propiedades deseables a la maduración en barricas de “bourbon,” y son propiedades aditivas de esta técnica. Una desventaja de esta técnica es una alta contaminación tanto para *Lactobacillus* como para *Pediococcus*. Este estudio tuvo el objetivo de determinar como se podría maximizar las propiedades deseables de la técnica a la vez de minimizar el daño perjudicial de la contaminación y de minimizar las pérdidas por evaporación. Se cuantificaron los “marcadores” analíticos del roble para atributos sensoriales deseables usando métodos de cromatografía de gas y de espectrometría de masa, evaluando barricas individuales para pH, estabilidad microbiológica y bacterias dañinas.

**Palabras claves:** barricas, “bourbon,” valor de actividad de olor (OAV)

## Introduction

Stone Brewing Company has made a number of large-scale barrel aged beers, a popular brewing technique. Bourbon barrels are widely available and are used only once by bourbon producers.

Using first-use bourbon barrels, i.e., bourbon barrels that had previously aged bourbon, results in an alcohol increase in the aged beer from remaining bourbon soaked into the staves of the barrel, extraction of bourbon flavors, and oak flavors. Use of second-use barrels, bourbon barrels that aged bourbon, then aged beer, and then are refilled with another beer for further aging, results in extraction of additive oak flavor compounds, and slow oxidation without alcohol pickup.

Both of these types of barrel aging techniques are used at the Stone Brewing Co., some beers aged in first use only, some second use only, and some a blend of these two types of barrels depending on the desired result.

In 2012, a unique opportunity to age the same strong amber beer in both first and second use barrels from the same bourbon

producer presented itself. All of these barrels had aged a 10-year-old bourbon prior to filling with beer for aging. The second use barrels were previously filled with a strong black beer and aged for 6 months. The same beer was also aged in some charred bourbon barrels that had not been used to age bourbon.

These barrels were sampled for gas chromatography/mass spectrometry (GC/MS) analysis for oak compound analysis on a routine basis. The goal of the project was to tie this analysis to sensory analysis. High levels of oak impact are detected in barrel aged beers in as little as 2 months, and extended aging is often a trade-off between increasing the additive barrel aging flavor impact and decreasing microbiological stability. Using this analytical method provided useful data that correlated well with our sensory analysis.

## Methods

GC/MS was used to analyze a standard oak analysis profile on common analytical markers for barrel aging on one style of beer in three different barrel types. The barrels used were:

1. Virgin barrels, charred standard barrels for aging bourbon that had not stored bourbon
2. First use barrels that had previously stored bourbon and were then filled with beer
3. Second use barrels that had stored bourbon, then beer for 6 months, and then filled with the same beer as the other two barrel types and aged for 13 months.

Barrels were sampled periodically and analyzed using the oak profile method. A statistically significant number of barrels were sampled for each of the three lots of barrels using a military sampling standard MIL STD 105 and combined into a composite sample for each barrel type. Sampling was originally intended to occur every 3 months, but was not achievable due to construction in the barrel warehouse. Table 1 shows the sampling plan.

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**Table 1.** Sampling plan for three experimental barrel lots

Barrel type	Number of barrels	Number of samples in composite	Sampled at 3 months	Sampled at 7 months	Sampled at 10 months	Sampled at 13 months
Virgin barrels	6	2	Yes	Yes	Yes	No
First use barrels	30	8	Yes	Yes	Yes	No
Second use barrels	38	8	No	Yes	Yes	Yes

**Table 2.** Compounds analyzed in oak profiling, common sensory descriptors, and flavor thresholds

Compound	Sensory descriptors	Flavor threshold
5-methyl furfural	Toast/butterscotch/caramel; acrid in very high concentrations	1,000
5-hydroxymethyl furfural	Toast/butterscotch/butter/caramel	1,000
Furfural	Bread/toast/butterscotch/caramel; acrid in very high concentrations	3,000
Furfuryl alcohol	Bready/burnt; acrid in very high concentrations	8,000
Vanillin	Natural vanilla	50
<i>trans</i> -lactone	Fresh oak/coconut	20
<i>cis</i> -lactone	Fresh oak/coconut (stronger isomer?)	20
Eugenol	Spice/clove	10
Guaiacol	Smoke	10
<i>trans</i> -iso eugenol	Spice/clove/carnation	10
<i>cis</i> -iso eugenol	Spice/clove	10

**Figure 1.** Gas chromatograph/mass spectrometer used for analysis in this study.

Once sampled, oak compounds were extracted prior to analysis. The extraction was performed by measuring 30 mL of sample into a 40 mL vial using a graduated cylinder. A 2 mL aliquot of dichloromethane was then added with a volumetric pipet, and the samples were capped with a TFE lined cap. The samples were then placed in an Envirogenie (Scientific Industries, Inc.), and tumbled, end over end, for 24 h at 8 rpm and 20°C. The dichloromethane extracts were centrifuged at 1,500 rpm for 5 min.

The supernatant was decanted off of the sample and placed into injection vials with internal standard, 50 ppm Vanillin-d<sub>3</sub>, then placed in the auto sampler tray of an Agilent 7890A/5975C GC/MS system. One microliter of sample was injected directly onto a Restek RTX-Wax column, and analyzed. Figure 1 shows the GC/MS machine.

Numerical results are converted to odor activity values (OAV); the concentration of each compound is then divided by the flavor threshold. Table 2 shows the flavor thresholds of the compounds that are commonly found in bourbon barrel aged beers.

Quality control checks were performed for all barrels just prior to completing aging and racking into a blending tank. The quality control check consists of pulling a sanitary sample of beer, tasting for any indicators of spoilage, and a pH check. All pH checks should be within 0.02 of the median for this set of barrels with the same beer lot and age. Lower pH may be indicative of spoilers.

This quality control technique has proven more effective than traditional microbiological stability checks and is more sustainable. Using traditional microbiological analysis proved ineffective in detecting spoilers in the barrels, producing false negative results and spoilers appearing in the final blend in the tank.

## Results and Discussion

Raw data was divided by the flavor threshold, calculating the OAV of each composite sample. Six compounds were above the flavor threshold on one or more sets of barrels (OAV  $\geq 1$ ). The compounds present in significant levels above the flavor threshold were: *cis*-lactone, vanillin, 5-hydroxymethyl furfural, and to a lesser extent guaiacol, eugenol, and *trans*-isoeugenol.

Note that flavor thresholds for these oak aging flavor compound markers have never been experimentally derived for beer, but flavor thresholds used in this study are instead those for water. Flavor thresholds for these carrier beverages are very close to those derived for wine (David Hay, *personal communication*) at  $\pm 15\%$  of those flavor thresholds. These flavor thresholds in turn are thought to be slightly higher than or identical to beer (R. Newman, *personal communication*).

Levels of *cis*-lactone were comparable after 10 months of aging in both the brand new virgin bourbon barrels and first use bourbon barrels (Fig. 2). Concentrations were  $>18$  and  $>17$  OAVs, respectively, while the second use barrels were depleted in *cis*-lactone at  $>6$  and  $>10$  OAVs after 10 and 13 months.

Vanillin levels were well above the flavor threshold in all three barrel types (Fig. 3). Levels were as high as  $>14$  OAVs in the brand new virgin bourbon barrels after only 3 months in barrel. Levels increased or decreased over time. Note that the 7 month brand new bourbon barrels, 7 month first use bourbon barrels, and 10 month second use bourbon barrels, all of which

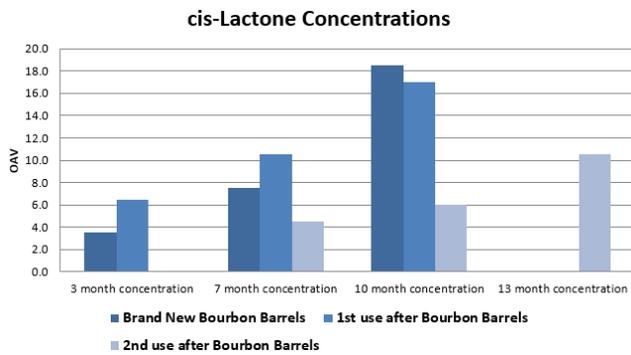


Figure 2. *cis*-lactone OAV concentrations over time.

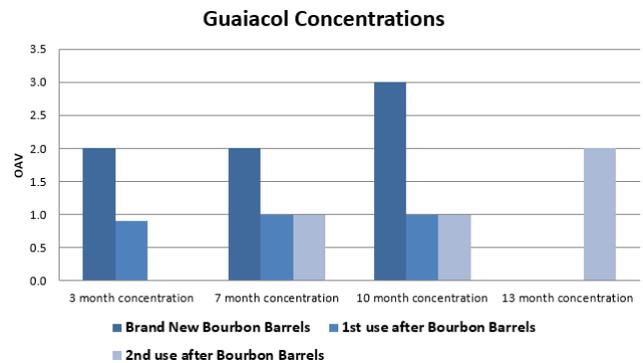


Figure 5. Guaiacol OAV concentrations over time.

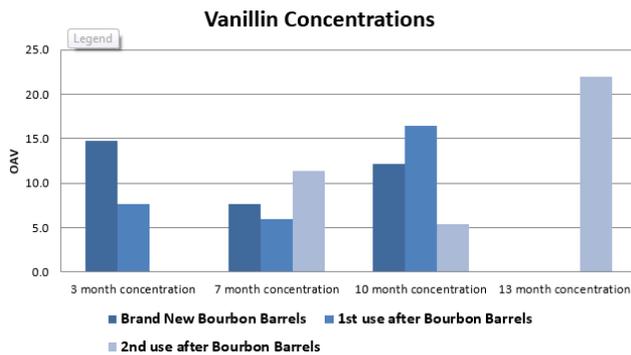


Figure 3. Vanillin OAV concentrations over time.

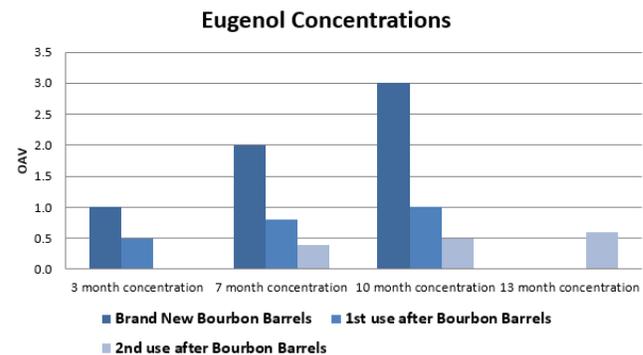


Figure 6. Eugenol OAV concentrations over time.

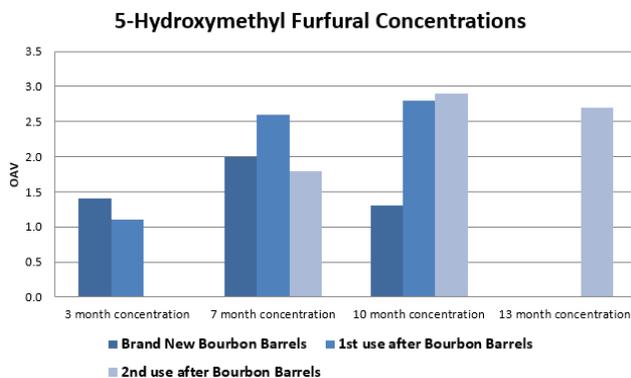


Figure 4. 5-hydroxymethyl furfural OAV concentrations over time.

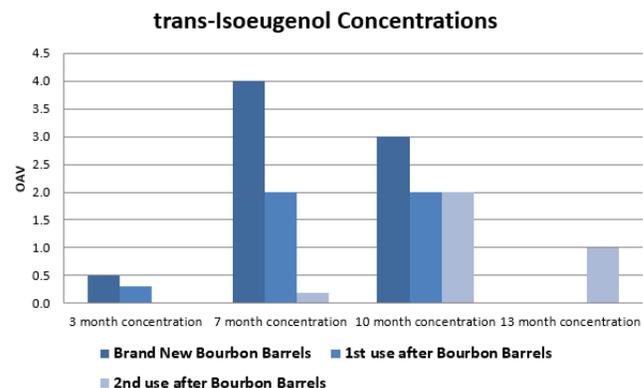


Figure 7. *trans*-isoeugenol OAV concentrations over time.

were sampled at the same time, were the only samples that decreased in concentration over time. This could indicate a possible analytical error.

Levels of 5-hydroxymethyl furfural increased over time (Fig. 4). Levels at 10 months barrel age were 2.7 and 2.8 times the flavor threshold in first and second use barrels, respectively, and lower in virgin/brand new barrels. Levels do not seem to deplete with repeated use of the barrel.

Guaiacol and eugenol levels were at or above flavor thresholds and increased over time in new virgin bourbon barrels (Figs. 5 and 6). Levels in first and second use bourbon barrels were at or below flavor threshold and increased or were flat over time. Levels of *trans*-isoeugenol increased over time in all sets of barrels but did not go above flavor threshold until 7 months of age (Fig. 7). Levels of 5-hydroxymethyl furfural

were higher in new virgin bourbon barrels than in first and second use barrels.

Overall smoke and spice compounds were much lower in first and second use bourbon barrels than in new virgin barrels. The bourbon aging prior to filling with beer in first and second use bourbon barrels depleted these guaiacol, eugenol, and *trans*-isoeugenol compounds.

Using Stone Brewing Company’s internal procedure for detecting spoilers with pH and sensory, and confirming these results with tank sampling, significant microbiological spoilage organisms were detected in bourbon barrels at 10 months. Microbiological stability was intact at up to 6 months barrel age (Table 3).

**Table 3.** Microbiological stability by percentage of total barrels over time.

Time in barrel	2 months	3 months	4 months	6 months	10 months	13 months	20 months
Number of barrels	159	126	8	38	68	6	74
Failure rate	0%	0%	0%	0%	18%	100%	49%

## Conclusions

In as little as 3 months aging in new or used bourbon barrels results in significant extraction of vanillin and *cis*-lactone, well above the flavor threshold. Concentrations of 5-hydroxymethyl furfural were also above the flavor threshold after 3 months, but just above that threshold. Concentrations of *cis*-lactone and 5-hydroxymethyl furfural continued to increase over time, while it was difficult to conclude that vanillin concentrations increased or decreased after the initial 3 month monitoring time.

Flavor compounds associated with smoke and spice such as *trans*-isoeugenol, eugenol, and guaiacol were at far higher levels in unused barrels that had not previously stored beer, bourbon, or any other liquid. These compounds were either lower concentration or below the flavor threshold in used barrels.

Second use bourbon barrels were depleted in *cis*-lactone compared to first use barrels, but both were still above the flavor threshold. Second use barrels still had very high levels of vanillin that was further extracted during barrel aging.

At the Stone Brewing Company, our barrel program does not always rely on extended barrel aging, with some releases aging for 3–6 months. Desirable sensory attributes are at suitable levels in many cases after these relatively short aging times. Microbiological stability is very good at 6 months of age, and our barrel aged beers that are microbiologically stable at the time of racking from barrel are still stable years after being packaged.

Extended barrel aging of a year or more is still practiced at Stone Brewing Company and has its place, but is not always required to achieve a desirable result.

The use of second use barrels, those that housed bourbon, then beer, and then a second use filled with beer, is an aging technique we often use to achieve desirable flavor attributes without significant alcohol increase. The beer is often de-

scribed as less “woody,” a descriptor often associated with oak lactones (Fig. 2). It is typically also more tannic, less alcohol heat, and less bourbon flavor than first use barrels.

The use of newly charred bourbon barrels that have not previously stored any other liquid has its place, and is a useful way of incorporating spice and smoke notes to a blend.

Overall analytical and microbiological analysis correlated well with sensory data, and was useful in confirming sensory results.

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