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Technical Bulletin

# The ETS Oak Aroma Analysis

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Aromas from oak aging are an integral part of many fine wines. Oak species and origin, cooperage practices and winemaking variables all influence the oak aroma compounds present in a given wine. Analyzing the main aroma components released by oak wood is a key to understanding oak contributions to wine flavor.

Until now, analyzing oak aroma belonged only in the research field. ETS Laboratories now offers a tool for the analysis of the primary oak aroma compounds found in wine. Nine compounds are reported, representing a broad range of chemical classes and sensory effects. This panel of analyses has an equally broad range of applications in winery barrel management, research, and quality control.

## OAK AROMA COMPOUNDS:

### Oak Lactones (*cis*- and *trans*-)

The two isomers of oak lactone are the main aroma constituents of raw oak. Associated sensory descriptors are *fresh oak* and *coconut*. The *cis*- isomer is a more powerful aromatic than the *trans*- isomer. American White Oak (*Quercus alba*) is relatively richer in the *cis*- isomer compared to other oak species. Wood seasoning is reported to affect the relative ratios of *cis*- and *trans*-oak lactone. An increase in barrel toast reduces oak lactone amounts as well as their sensory impact in wine.

### Vanillin

Vanillin is the main aroma compound in natural vanilla. Present in raw oak, quantities released in wine are reported to vary with oak species and seasoning. Vanillin increases with medium toast levels, but decreases with very high toast. Vanillin is partially transformed to the non-aromatic vanillyl alcohol by yeast during barrel fermentations.

### Eugenol and Isoeugenol

Eugenol is the main aroma compound in clove. Present in raw oak, eugenol is reported to increase during open-air wood seasoning. Eugenol and isoeugenol possess a very similar *spicy*, *clove-like* aroma. Release in wine is reported to increase with toasting level.

**Measured Compounds:**

| | |
|---|---|
| Oak Lactones | 4-Methylguaiacol |
| Vanillin | Furfural |
| Eugenol | 5-Methylfurfural |
| Isoeugenol | Guaiacol |

### Guaiacol and 4-Methylguaiacol

Wood lignin degradation at very high temperatures (pyrolysis) results in formation of a wide range of volatile phenols. These compounds have *smoky* aromas, and are markers of the *smoky* character imparted by heavily toasted oak. Guaiacol has a *char-like* aroma, while 4-methylguaiacol has a *char-like* and *spicy* character.

### Furfural, 5-Methylfurfural

These compounds result from degradation of sugars and carbohydrates by heat (Maillard reactions). In wood, the carbohydrates, cellulose and hemicellulose are degraded during barrel toasting. Furfural and 5-methylfurfural possess *sweet*, *butterscotch*, *light caramel* and faint *almond-like* aromas. They may contribute such characteristics to oak aged wines.

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# The ETS Oak Aroma Analysis

## *Sensory impacts in wine*

The sensory impact of aroma compounds is commonly evaluated by comparing their concentrations found in wine to their sensory thresholds (determined by adding the pure compound to a model solution or neutral wine). However, wine is complex. Wine matrix effects and chemical interactions strongly influence sensory perception. and sensory thresholds of pure compounds do not always adequately describe a compounds sensory impact.

Oak lactones and vanillin frequently exceed published individual thresholds. They are primarily responsible for occurrence of their associated *fresh oak*, *coconut* and *vanilla* characters in wines.

Other reported compounds seem unlikely to reach published sensory thresholds in wine. This does not mean they do not participate in wine aroma. Substances with chemical and sensory similarities, such as eugenol and isoeugenol, may have additive or cumulative effects even though they are below thresholds individually .

Numerous volatile phenols possessing a *smoky* aroma are always present together. Even though their individual concentrations may be below sensory thresholds, their combination in wine may result in a perceived sensory effect. The compounds guaiacol and 4-methylguaiacol are markers for this complex group.

Dramatic synergistic and masking effects between chemically unrelated volatiles have also been demonstrated. The aroma perception of one compound can be enhanced or repressed by the presence of another compound.

## APPLICATIONS :

Wine and spirit samples, as well as oak material (chips, shavings, cubes…) can be submitted for analysis. In the latter case, oak material is soaked several days in a model solution and the soak solution is analyzed.

The ETS wood aroma analysis is a good tool to evaluate any wine or spirit having contact with oak wood as well as oak materials themselves. Suggested applications are:

- Evaluating barrel trials (wood source, toast, cooper, etc)
- Monitoring wine aging in barrel (evaluation of same wine through time)
- Fine tuning barrel management (effects of barrel age or sanitation programs)
- Evaluating or managing barrel alternatives (staves, chips, beans, etc)
- Comparisons to benchmark or competitor's wines

## REPORTING :

The ETS oak aroma report shows concentrations of each compound, expressed in ì g/L (ppm).

Concentrations are also displayed in relation to a reference sample by means of a “spider “(or “radar”) plot. The information on the spider plot is expressed as a percentage of the reference value. Sensory descriptors associated with reported compounds are indicated on the plots.

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## Analytical Details:

| | |
|---|---|
| **Methodology:** | Headspace/SPME/GC/MS |
| **Measurement Uncertainty:** | Approximately X% |
| **Min Reportable Quantity:** | XXXXX |
| **Equipment Used:** | HP 5973 GC/MS with CTC solid phase micro extraction (SPME) |
| **Sample Size Required:** | Representative 125 mL sample |
| **Target Response Time:** | XX days from receipt of samples |
| **Fee Per Sample:** | 1 Sample: $165 2-10 Samples: $150 >10 Samples: $140 |

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# The ETS Oak Aroma Analysis

- ***Why is there so much variation in oak aromas?***

  The are many causes of variation and many of them interact to form a wide array of potential aroma profiles.

  The source of the oak itself is a substantial source of variation. Oak species differ greatly. To illustrate, the French Pedunculate Oak (*Quercus pedunculata* = *Q. robur*) is known for its relatively faint aroma potential compared to another French Sessile Oak (*Q. sessilis and Q. petraea*). American White Oak (*Q. alba)* can have a strong, distinctive aroma, sometimes considered excessive in some wines. In contrast, Oregon White Oak (*Q. garryana)* seems to have more similarities to the French oaks.

  Geographic origin is linked to botanic species, but different species frequently grow in the same forests, and hybridization does occur.

  Growing conditions, age and genetic variations of individual trees can strongly affect wood structure and composition. Even a stave's position on a trunk has been shown to influence its aroma composition.

  Stave seasoning and drying are important. Kiln drying is likely to result in a different aroma character, air drying and air drying conditions (time spent in open air and humidity level) have a significant influence on wood aroma potential.

  Finally, the cooperage process adds a considerable layer of variability. Definitions of "light" to "heavy" toasts are subjective and vary among coopers. Difficulties in controlling toasting levels generate barrel to barrel variation, and even stave to stave variation in the same barrel as some staves may toast more rapidly than others in the same barrel.

- ***What is the effect of toasting on oak aroma?***

  Toasting oak during barrel processing modifies the structure and chemical properties of the wood. This influences the wood aroma composition and the release of aroma compounds into the wine.

  Increased toasting diminishes the "fresh oak" aromas generally attributed to oak lactones. Simultaneously, "vanilla" and "caramel-like" aromas associated with vanillin, furfural, 5-methylfurfural, and maltol increase. At heavy toast levels these compounds decrease and are replaced by "spicy" (eugenol, isoeugenol, 4-methylguaiacol) and "smoky" characters (4-methylguaiacol, guaiacol, 2-methylphenol).

- ***How does barrel age affect oak aroma?***

  Oak aroma potential decreases rapidly with barrel use. However, different compounds are extracted from oak at different rates. While a one year old barrel will certainly impart less oak character to a wine than a new barrel, the aroma compounds it does contribute are likely to have a different profile than a new barrel.

- ***How do winemaking processes influence the levels of oak aroma compounds actually present in wine?***

  The choice of barrels, the proportion of new oak and the duration of oak-wine contact are the most obvious factors influencing wood aroma in wine. Microbial activity also has an influence. When fermentation is carried out in barrel, aldehydes such as vanillin, furfural and 5-methylfurfural are partially transformed by yeast into non-aromatic alcohols. Absorption of aroma compounds on yeast cells and macromolecules is also possible.

- ***What is a sensory threshold?***

  Sensory thresholds are determined by sensory evaluation of model solutions (or neutral wines) spiked with increasing levels of a pure compound. Sensory impact of aroma compounds is commonly evaluated by comparing their sensory threshold with concentrations actually found in wine. Wine is a very complex matrix, however, and multiple interactions between aroma compounds exist .

- ***How can compounds present in wine below their sensory threshold participate in wine aroma?***

  Most volatiles identified in oak aroma seem unlikely to reach sensory thresholds in wine. However, substances with chemical similarities are often released from oak together (such as eugenol, isoeugenol or other volatile phenols). The combination of similar molecules can result in a perceived sensory effect even when they are below their individual sensory thresholds. These additive and cumulative effects may be quite important in wine.

  Dramatic synergistic effects between unrelated volatiles have also been demonstrated. To illustrate, oak lactone's difference threshold has been found to be 50-fold lower in presence of vanillin (i.e. it is fifty times more potent). Similar effects are likely to occur between oak volatiles and other wine aroma compounds.

- ***What compounds are responsible for "piney", "resin" "cedar" or "dill"- like aromas in oak or wine?***

  Such descriptors, generally associated with American oak *Quercus alba*, seem linked to high levels of *cis*- oak lactone. *Quercus alba* has also been reported to contain relatively high amounts of terpenes. Although terpenes may contribute such aromatic characters, key compounds have not been identified.

# Frequently Asked Questions

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## The ETS Oak Aroma Analysis…

- ***What compounds are responsible for "nutty", "roasted almond" or "roasted hazelnut" – like aromas?***

  Combined sensory effect of known volatiles coming from either wine or oak, such as diaceyl ("fatty", "butter"), free fatty acids ("fatty", "rancid"), furfural, 5-methylfurfural and maltol ("caramelized" tones), may be at least partly responsible for occurrence of such aromas.

  Presence in wine of other compounds possessing "roasted" aromas is currently being investigated by researchers.

- ***What compounds are responsible for "cinnamon" or "nutmeg"- like aroma?***

  Cinnamon and nutmeg possess both "woody" and "spicy" aromas. Such characters in oak aroma can be attributed to the combination of "woody", "coconut-like" oak lactones and "spicy" compounds such as eugenol, isoeugenol, etc.

- ***What compounds are responsible for "breadcrust" or "french toast"- like aromas? For "gingerbread"- like aromas?***

  Very complex sensory impressions can result from combined effect of wine and oak aroma components. "Complex" descriptors, however, can usually be divided in simpler terms, which in turn, can be associated with known wood aroma compounds.

  To illustrate, a "breadcrust" or "french toast" – like character can be divided in "yeasty" flavors (imparted by yeast byproducts in bread as well as in wine), caramelized tones (from carbohydrate byproducts furfural, 5-methylfurfural, maltol) and "smoky" aromas (guaiacol, 2-methylphenol…) contributors.

  The above applies to a "gingerbread" – like aroma, which may be less "yeasty" with additional "spicy" contributors (e.g. eugenol).

- ***A wine lot aged in oak (or in contact with oak chips) developed a disagreeable "dusty", "cardboard" smell. It seems "corked" before bottling. What compounds are responsible?***

  Oak is a possible source of chloroanisoles contamination in bulk wine, but not the only one. Chloroanisoles (TCA, TeCA and PCA) are powerful odorants with a "musty", "moldy", "corked" smell. ETS Laboratories offers determination of chloroanisoles in wine as a routine analysis and oak samples can be submitted for qualitative evaluation. For more information, see our chloroanisole publication.

- ***A red wine lot aged in oak developed a "pharmaceutical", "band aid" or "horsy", "sweaty" aroma. What compounds are responsible?***

  The wine should be tested for 4-ethylphenol (4EP) and 4-ethylguaiacol (4EG). These compounds are byproducts of the yeast *Brettanomyces* and are found together in many wines aged in oak. Periodic screening of wine is recommended to monitor *Brettanomyces* activity during oak aging.

- ***How does ETS analyze oak aromas?***

  "Headspace" above the wine or extract is sampled to specifically analyze compounds from the volatile fraction of a wine sample and to suppress interference from non-volatile compounds, "headspace" above the wine or extract is sampled. Solid phase micro-extraction technology is used (Headspace/SPME) . Analysis of the extract is then performed using gas chromatography with detection by mass spectrometry (GC/MS). This integrated analytical system allows great specificity and sensitivity.

  In the case of oak shavings, chips or cubes, oak material is soaked several days in a model solution and the soak is then analyzed in a method similar to that described above..

- ***How do I submit a sample?***

  Representative wine samples should be submitted in full glass containers or bottles of at least 125 mL. Glass is suggested to avoid any possible interference from plastic polymers.

  Oak shavings, chips or cubes should be submitted in sealed bags or containers (plastic is OK) . Minimum quantity required is 50 g.

- ***What is a representative sample?***

  Considering potential variability of oak aroma composition from barrel to barrel, a sampling plan is very important.

  Single barrel samples can be used to evaluate barrel to barrel variation. For most other applications, composite samples may be a better choice.

- ***What does ETS recommend for oak origin? toasting level? cooperage?***

  Any decisions on what is the desired oak contribution to a wine, quantitatively and qualitatively, belong to winemakers. The ETS wood aroma analysis has been designed as a tool to assist winemakers in their choices, in order to master and optimize oak contribution to wine aroma.

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