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Prevalence of Wildfire Smoke Exposure Markers in Oaked Commercial Wine

Mango Parker [1]* WenWen Jiang [1,2] Adrian D. Coulter [1] Tracey E. Siebert [1] Eleanor Bilogrevic [1] I. Leigh Francis [1] and Markus J. Herderich [1,2]

Abstract

Background and goals

Grapes exposed to wildfire smoke and wine produced from contaminated grapes can be robustly identified through quantitative analysis of smoke exposure markers, volatile phenols, and phenolic glycosides (PGs). This assessment is based on comparison of data from suspect samples to concentrations of phenolic compounds typically found in non-smoke-exposed grapes and unoaked wines.

Oak products for winemaking are typically heat treated and represent a major source of guaiacol and other volatile phenols in wine. Although contact with oak products is thought to contribute negligible concentrations of PGs, the lack of data from oaked wines confounds the identification of a potential risk of smoke taint development in wine when assessing commercially produced, oaked wine. Therefore, this study aimed to determine the typical concentrations of smoke exposure markers in commercially produced, oaked wine.

Methods and key findings

Commercially produced wines (20 to 30 each) of Cabernet Sauvignon, Chardonnay, Pinot noir, and Shiraz cultivars were sourced from Australian regions and vintages free from known wildfire smoke exposure. Gas chromatography-mass spectrometry and high-performance liquid chromatography-mass spectrometry demonstrated that syringol and guaiacol were relatively abundant in oaked wine, reaching concentrations of 200 μg/L. In contrast, most PGs were <10 μg/L, and trace concentrations of cresols were infrequently found.

Conclusions and significance

The concentrations of established wildfire smoke marker compounds (guaiacol, 4-methylguaiacol, syringol, 4-methylsyringol, *o*-cresol, *m*-cresol, *p*-cresol, syringol gentiobioside, 4-methylsyringol gentiobioside, cresol rutinoside, phenol rutinoside, guaiacol rutinoside, and 4-methylguaiacol rutinoside) were determined in oaked Australian Cabernet Sauvignon, Chardonnay, Pinot noir, and Shiraz wines. The data enable confident identification of smoke-affected wine that has been in contact with oak.

Key words: oak, phenolic glycosides, smoke, volatile phenols, wine

Introduction

Since it was first reported in 2003, smoke taint caused by wildfire smoke has resulted in many millions of dollars in losses for wine producers worldwide, including in Australia, Canada, Chile, Greece and other Mediterranean countries, South Africa, and California (AWRI 2003, Krstic et al. 2021). Wine made from smoke-exposed grapes has been described as "smoky, burnt, ash, ashtray, salami, smoked salmon," and notably, "lingering retro-nasal ash character" (AWRI 2003). Research over the last decade has established that smoke-exposed grapes and wines made from smoke-exposed vineyards can be reliably identified by measuring volatile phenols (VPs) and phenolic glycosides (PGs), and comparing these exposure markers to known concentrations typically found in non-smoke-exposed grapes and wines (Coulter et al. 2022). The concentration of smoke markers in grapes has recently been shown to predictively model smoke flavor intensity in wine (Parker et al. 2023). The compounds utilized for identifying smoke-exposed grapes and wine include VPs: guaiacol; 4-methylguaiacol (MeGu); *o*-, *m*-, and *p*-cresol; syringol; and 4-methylsyringol (MeSyr), and glycosides: syringol gentiobioside (SyGG), methylsyringol gentiobioside (MSyGG), cresol rutinosides (CrRG), guaiacol rutinoside (GuRG), methylguaiacol rutinoside (MGuRG), and phenol rutinoside (PhRG).

VPs are formed from thermal lignin degradation during combustion, which can occur during toasting of oak products and barrels prior to their use in winemaking, and more generally, from generation of smoke by burning woody materials (Wittkowski et al. 1992,

[1]The Australian Wine Research Institute, PO Box 197, Glen Osmond, SA, 5064, Australia; and [2]The University of Adelaide, PMB 1, Glen Osmond, SA 5064, Australia.

*Corresponding author (mango.parker@awri.com.au; tel: +61 8 8313 6600)

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Simoneit et al. 1993, Chatonnet et al. 1999). Smoke is a highly complex mixture composed of solid and liquid aerosols and hundreds of volatile organic compounds, and it can vary significantly in composition (Simoneit et al. 1993, Rogge et al. 1998, Sekimoto et al. 2018, MacSween et al. 2020). When vineyards are exposed to smoke, VPs (namely, guaiacols, cresols, and syringols) are taken up by grape berries and leaves and rapidly metabolized to form PGs (Hayasaka et al. 2010a, 2010b). PGs can be detected in grape berries soon after smoke exposure and can accumulate during ripening, following initial exposure (Wilkinson et al. 2012, Caffrey et al. 2019). During winemaking, VPs and PGs are readily extracted into must and wine (Ristic et al. 2011, Caffrey et al. 2019).

Therefore, wine made from heavily smoke-exposed grapes may contain high concentrations of VPs and PGs (Hayasaka et al. 2010a), whereas the concentrations of individual VPs and PGs in non-smoke-exposed wine rarely exceed 15 μg/L (Coulter et al. 2022). VPs impart smoky and medicinal odors, and PGs contribute to the "lingering ashy aftertaste" by releasing VPs in-mouth during tasting (Parker et al. 2012, Mayr et al. 2014). Recently, some thiophenols have been implicated in evoking ashy flavor when spiked with VPs together into non-smoked red wine, although the exact relationship between thiophenols and their precursors in grapes and wine remains to be established (Tomasino et al. 2023).

Smoky odors and flavors in wine can be derived from oak contact during wine production (Pollnitz et al. 2004, Spillman et al. 2004, Ribéreau-Gayon et al. 2006), along with many desirable flavor attributes such as vanilla, coconut, toast, spice, and coffee (Garde-Cerdán and Ancín-Azpilicueta 2006). Guaiacol, syringols, and cresols are typically detectable in oaked wine (Ribéreau-Gayon et al. 2006), with their concentrations varying by species and origin of oak wood and by the degree of toasting (Cerdan et al. 2002, Fernández de Simón et al. 2010). Concentrations of guaiacol and syringol up to 140 μg/L and 500 μg/L, respectively, have been reported in wine produced with heavily toasted oak. As an aside, the formation of guaiacol as an artifact during gas chromatography-mass spectrometry analysis may have contributed to such high values, and true concentrations may have been much lower (Perez-Prieto et al. 2002, Pollnitz et al. 2004). Reported concentrations of cresols have generally been <5 μg/L (Prida and Chatonnet 2010, Chira and Teissedre 2013). Only one study of 79 wines reported higher values of *m*-cresol, with an average concentration of 7 μg/L and a maximum value of 158 μg/L (Prida and Chatonnet 2010). By contrast, Chatonnet et al. (1999) found the toasting of oak produced only trace levels of cresols, and Cadahía et al. (2003) did not detect any cresols in extracts of toasted oak. Although contact with oak products is thought to contribute negligible concentrations of PGs, smoke marker data from oaked wine are currently lacking.

In summary, oak treatment complicates the interpretation of analytical data for VPs when assessing wine made from grapes suspected of smoke exposure, and no data are available for concentrations of PGs in oaked wine made in years without smoke exposure of vineyards. Therefore, this study was initiated to determine the concentrations of VPs and PGs in commercial oaked wine, with the aim of providing information critical for identifying smoke-affected wine that has been in contact with oak. The concentrations of key smoke marker compounds, VPs, and PGs were determined in 88 commercial oaked wines from four cultivars and compared to concentrations found in unoaked, non-smoke-exposed wine and unoaked, smoke-affected wine. Overall, the results establish which markers are suitable indicators of smoke exposure when assessing oaked wine.

Materials and Methods

Oaked wine selection

A total of 88 wines from four different cultivars (Cabernet Sauvignon, Chardonnay, Pinot noir, and Shiraz [n = 28, 20, 20, and 20, respectively]) were purchased from local wine stores in Adelaide, Australia, in 2022. All wine was selected from Australian wine regions. Due to the occurrence of multiple wildfire events across Australia in the 2019 to 2020 growing season, wines from vintage 2020 and 2021 were avoided, considering that up to 15% of wine from the 2020 vintage may have been included in the final wine blend, under current label integrity rules (Wine Australia 2020). Three Shiraz wines from vintage 2020 had been made from grapes from the McLaren Vale and Barossa wine regions, which were not exposed to wildfire smoke (Wine Australia 2020). The aim of the wine selection was to achieve a broad distribution of samples across price points, regions, and vintages. Details of the wine selection can be found in Supplemental Table 1.

The winemaking details of all selected wine were found on producers' websites, and to the authors' knowledge, all wine was oak treated. The oak treatments included grapes fermented in French barrels, maturation in French or American barrels (old or new), and wine in contact with oak chips or staves after fermentation, for various periods of time. In summary, all wine was selected from various subregions in Australia and made from grapes without apparent exposure to wildfire smoke, but had undergone varied oak treatment during vinification.

Smoke-affected wine

Unoaked but smoke-affected wine was made in 2020 from grapes exposed to smoke prior to veraison (Jiang et al. 2022) or from grapes that had experienced a range of smoke events during the 2019 to 2020 ripening season (Parker et al. 2023). A total of 49 smoke-exposed wines were used from the cultivars Chardonnay, Pinot noir, and Shiraz (n = 16, 14, and 19, respectively), with a broad range of VP and PG concentrations, as reported previously.

Unoaked small-scale wine made from non-smoke-exposed grapes

Small-scale fermentations were conducted on non-smoke-exposed grape berries collected from multiple regions across Australia over four vintages to produce 192 unoaked wines. Non-smoke-exposed and unoaked Cabernet Sauvignon (n = 32) wines were made over two vintages (2010 and 2011), Chardonnay and Shiraz (n = 52 and 66, respectively) wines were produced over three vintages (2010, 2011, and 2016), and Pinot noir (n = 42) wines were produced over four vintages (2010, 2011, 2016, and 2017). Details of sample collection, winemaking, and analysis results are described and presented by Coulter et al. (2022).

Chemical analysis

The concentrations of guaiacol; MeGu; *m*-, *o*-, and *p*-cresols; syringol; MeSyr; 5-methylfurfural; (*cis*)-oak lactone; eugenol; furfural; (*trans*)-oak lactone; and vanillin in oaked wine samples were determined using an Agilent 6890 gas chromatograph coupled to an Agilent 5973 mass selective detector, as reported previously (Pollnitz et al. 2004). The analysis was performed by Affinity Labs, a commercial unit of the Australian Wine Research Institute (Urrbrae, SA, Australia). The limit of quantification (LoQ) was 1 µg/L for guaiacol; MeGu; *m*-, *o*-, and *p*-cresols; syringol; and MeSyr, and the LoQ was 10 µg/L for 5-methylfurfural, (*cis*)-oak lactone, eugenol, furfural, (*trans*)-oak lactone, and vanillin. The PG compounds in all wines, namely SyGG, CrRG, GuRG, MGuRG, MSyGG, and PhRG, were determined according to the liquid chromatography-mass spectrometry method published by Hayasaka et al. (2013). The LoQ for PGs was 1 µg/L.

Results and Discussion

Concentrations of oak-specific volatiles (*cis*-oak lactone, *trans*-oak lactone, vanillin, 5-methylfurfural, eugenol, and furfural) are summarized (Table 1). As the *cis*- and *trans*-oak lactones found in wine are known to be derived only from oak wood (Masuda and Nishimura 1971, Otsuka et al. 1974), the data in Table 1 confirm that one or more oak products had been used in the production of all wine used in the present study. A wide range of concentrations were observed, reflecting different oak treatments in commercial wine styles.

The concentrations of smoke exposure markers, VPs, and PGs in the commercial oaked wines are summarized by cultivar in Table 2. To illustrate how key smoke marker compounds distinguish smoke-exposed wine from oaked wine, the results from oaked wine were compared with data from wine made from smoke-exposed grapes and from unoaked small-lot wine made from non-smoke-exposed grapes (Figure 1).

Syringol and MeSyr were the most abundant compounds in the oaked wine, with median values in Shiraz wine ranging from 3 to 47 µg/L and reaching maximum concentrations of 187 µg/L and 96 µg/L, respectively (Table 2). These values are higher than those observed in smoke-affected wine (Parker et al. 2023). Syringol and MeSyr were rarely detected in smoke-affected unoaked Chardonnay. Median values in smoke-affected red wine ranged from 6 to 12 µg/L for syringol and <1.0 to 4 µg/L for MeSyr; maximum concentrations were 65 µg/L and 25 µg/L, respectively. MeGu was also present in many of the oaked wines, reaching a maximum of 35 µg/L, which is in line with previously reported values (Ribéreau-Gayon et al. 2006, Prida and Chatonnet 2010) and higher than the maximum value of 25 µg/L observed in smoke-affected wine (Parker et al. 2023). Overall, MeGu, syringol, and MeSyr had similar abundance in oaked and smoke-affected wine and are not suitable markers to distinguish oaked from smoke-affected wine.

Guaiacol concentrations in the oaked wine had median values ranging from 2 to 24 µg/L and a maximum concentration of 47 µg/L (Table 2). These values are in line with those previously reported for oaked wine (Spillman et al. 2004, Ribéreau-Gayon et al. 2006, Prida and Chatonnet 2010). The guaiacol concentrations in the oaked wine were similar but generally lower than values observed in smoke-affected wine, which had median values ranging from 2 to 55 µg/L and maximum concentrations up to 125 µg/L, clearly demonstrating that guaiacol concentration alone cannot be used to distinguish between oaked and smoke-affected wines (Figure 1). The values are higher than those found in wine made from non-smoke-exposed grapes, in which the reported 99th percentile value is typically <5

Table 1 Concentrations of oak-derived volatiles in commercial oaked wine. Limit of quantification (LoQ) for all compounds is 10 µg/L.

		5-Methylfurfural (µg/L)	*cis*-Oak lactone (µg/L)	Eugenol (µg/L)	Furfural (µg/L)	*trans*-Oak lactone (µg/L)	Vanillin (µg/L)
Cabernet Sauvignon	Min	<LoQ	77	<LoQ	67	39	18
	Max	18	294	34	329	208	132
	Median	<LoQ	134	20	109	119	62
Chardonnay	Min	<LoQ	15	<LoQ	31	10	13
	Max	326	172	19	1709	105	289
	Median	98	88	11	473	56	84
Pinot noir	Min	<LoQ	<LoQ	<LoQ	62	<LoQ	13
	Max	60	155	23	369	135	151
	Median	13	52	<LoQ	135	45	49
Shiraz	Min	<LoQ	<LoQ	<LoQ	79	<LoQ	<LoQ
	Max	106	488	37	508	88	296
	Median	18	54	<LoQ	170	30	56

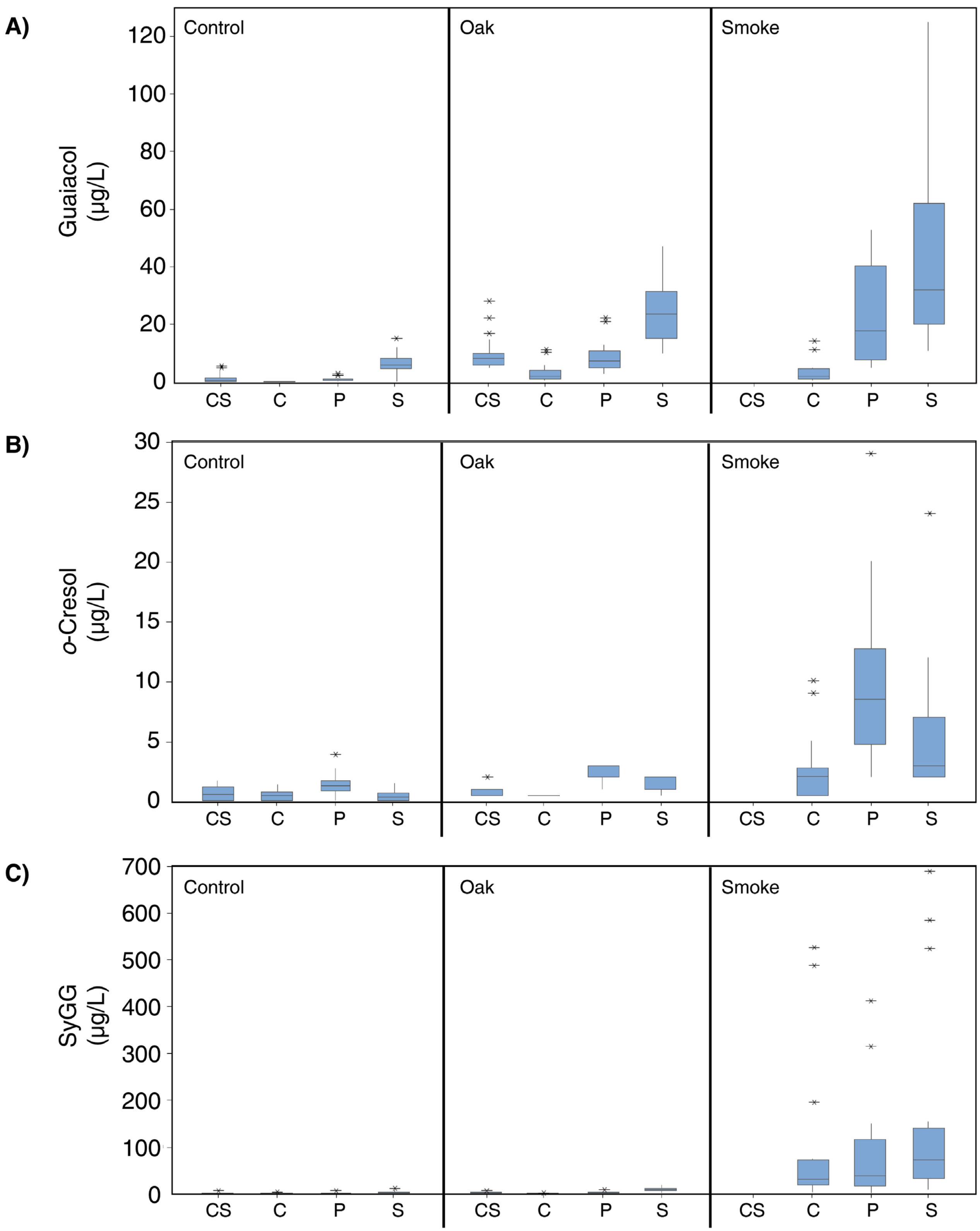

Figure 1 Smoke exposure markers guaiacol (**A**), *o*-cresol (**B**), and syringol gentiobioside (**C**; SyGG) in commercial oaked, smoke-affected, and control unoaked small-scale wines made from non-smoke-exposed grapes. CS, Cabernet Sauvignon; C, Chardonnay; P, Pinot noir; S, Shiraz. Control, unoaked small-lot wines from grapes with no smoke exposure (n = 192), published previously (Coulter et al. 2022); Oak, wines described in this study; Smoke, wines made from Chardonnay, Pinot noir, and Shiraz grapes exposed to smoke while ripening in the vineyard (n = 49), published previously (Jiang et al. 2022, Parker et al. 2023). No data on smoke-affected Cabernet Sauvignon wine were available for comparison.

μg/L for most varieties and 13 μg/L for Shiraz. The values are also higher than the maximum value (2.3 μg/L) reported by Merrell et al. (2021) for Pinot noir wine from the 2019 vintage.

Concentrations of each of the cresols (*o*-, *m*-, and *p*-cresol) were <5 μg/L in all oaked wine and below the LoQ of 1 μg/L in many of the wines (Table 2 and Figure 1). The maximum concentration of any isomer was 3 μg/L of *o*-cresol in six of the Pinot noir wines. These values are comparable to those found in non-smoke-exposed small-scale ferments (controls) (Coulter et al. 2022), the maximum value (2.8 μg/L) reported by Merrell et al. (2021) for non-smoke-exposed Pinot noir wine from the 2019 vintage, and previous reported concentrations in oaked wine (Ribéreau-Gayon et al. 2006, Prida and Chatonnet 2010). By contrast, values in smoke-affected wine reached 29 μg/L (*o*-cresol). Therefore, cresol concentrations >3 μg/L in wine could potentially indicate smoke exposure.

PGs were all <20 μg/L in the oaked wine. All PGs were below the LoQ (1.0 μg/L) in all oaked Chardonnay wine, and many PGs were below the LoQ (1.0 μg/L) in most of the Pinot noir wine. The oaked Shiraz and Cabernet Sauvignon wines had higher concentrations of PGs, a trend also seen in the non-smoke-exposed wine. GuRG and SyGG were the most abundant PGs in the oaked wine and the only PGs that exceeded 10 μg/L. Shiraz wine generally had higher concentrations of SyGG and GuRG than the other cultivars, with median values of 8 μg/L and 7 μg/L and maximum concentrations of 18 μg/L and 20 μg/L, respectively.

Surprisingly, some of the red wine had concentrations of SyGG and GuRG that exceeded those typically observed in small-scale wine made from non-smoke-exposed grapes under controlled conditions, which are generally <13 μg/L (Coulter et al. 2022). Nonetheless, the concentrations of PGs in both control and oaked wine were very low compared to the values observed in wine made from smoke-affected grapes (Figure 1). In smoke-exposed Shiraz wine, SyGG was commonly detected in the range of 13 to 123 μg/L and reached a maximum of 690 μg/L, and GuRG was detected in the range of 11 to 85 μg/L (Parker et al. 2023).

To our knowledge, this is the most comprehensive survey of PGs in commercially produced wine. In many cases, concentrations of PGs allow for reliable differentiation between wine made from grapes with and without smoke exposure, even after the wine was in contact with oak products. Still, certain limitations should be considered. The smoke-exposed wines in this study were all sourced from one vintage in Australia and generally had similar patterns of smoke exposure markers, with SyGG being the most abundant smoke marker. However, other patterns are possible, such as the recently reported higher relative abundance of PhRG in California wine (Wilkinson and Ristic 2020, Crews et al. 2022). Variations in the relative abundance of smoke exposure markers likely reflect differences in fire behavior, environmental conditions, and/or the type of fuel from which smoke was generated; for example, pyrolysis of angiosperms such as hardwood Eucalyptus trees yields syringols, guaiacols, and cresols, whereas syringol is absent in smoke from burning gymnosperms such as Pinus woods (Simoneit et al. 1993, Kelly et al. 2012). Therefore, the authors recommend considering the whole suite of VPs and PGs when attempting to identify wine made from smoke-exposed grapes after future smoke events.

Table 2 Concentrations of volatile phenols and phenolic glycosides in commercial oaked wine. Limit of quantification (LoQ) for all compounds is 1 μg/L. Gu, guaiacol; MeGu, 4-methylguaiacol; *o*-, *m*-, and *p*-cres, *o*-, *m*-, and *p*-cresol, respectively; Syr, syringol; MeSyr, 4-methylsyringol; SyGG, syringol gentiobioside; MSyGG, methylsyringol gentiobioside; PhRG, phenol rutinoside; CrRG, cresol rutinosides; GuRG, guaiacol rutinoside; MGuRG, methylguaiacol rutinoside.

		Gu (μg/L)	MeGu (μg/L)	*o*-Cres (μg/L)	*m*-Cres (μg/L)	*p*-Cres (μg/L)	Syr (μg/L)	MeSyr (μg/L)	SyGG (μg/L)	MSyGG (μg/L)	PhRG (μg/L)	CrRG (μg/L)	GuRG (μg/L)	MGuRG (μg/L)
Cabernet Sauvignon	Min	5	<LoQ	<LoQ	<LoQ	<LoQ	22	1	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ
	Max	28	13	2	2	1	109	44	6	<LoQ	3	5	16	3
	Median	8	2	<LoQ	<LoQ	<LoQ	39	5	2	<LoQ	1	2	2	1
Chardonnay	Min	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	1	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ
	Max	11	16	<LoQ	<LoQ	<LoQ	57	54	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ
	Median	2	2	<LoQ	<LoQ	<LoQ	6	5	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ
Pinot noir	Min	3	<LoQ	<LoQ	<LoQ	<LoQ	7	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ
	Max	22	6	3	<LoQ	2	48	18	7	<LoQ	2	4	4	2
	Median	8	2	<LoQ	<LoQ	<LoQ	19	<LoQ	2	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ
Shiraz	Min	10	<LoQ	<LoQ	<LoQ	<LoQ	24	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ	<LoQ
	Max	47	35	2	2	2	187	96	18	2	3	6	20	7
	Median	24	3	<LoQ	<LoQ	<LoQ	48	6	8	<LoQ	1	3	7	2

Conclusion

This study determined the concentrations of known smoke marker compounds in commercial oaked wines of Cabernet Sauvignon, Chardonnay, Pinot noir, and Shiraz cultivars. Comparing smoke marker data from commercial oaked wine with concentrations previously reported in unoaked small-scale wine made from non-smoke-exposed grapes and with unoaked wine made from grapes exposed to wildfire smoke enabled a selection of phenolic compounds that can be used to identify smoke exposure when evaluating a suspect sample. Specifically, SyGG; other phenolic glycosides; and the VPs *o*-, *m*-, and *p*-cresol are suitable to distinguish smoke-affected wine from oaked and unoaked wines made from non-smoke-exposed grapes. By contrast, VPs guaiacol, MeGu, syringol, and MeSyr are clearly not suitable for distinguishing smoke-affected wine from oaked wine because their concentrations found in oaked wine were similar and, in some cases, exceeded concentrations in smoke-affected wine.

Further research is needed to establish the concentrations of smoke markers in oaked wine made from other cultivars and to include oaked and smoke-affected wines from other vintages, regions, and countries. Further validation of these results can be gained by testing the effect of known amounts and duration of oak treatments on smoke-related marker compounds. Despite these limitations, this study allows producers and researchers to assess smoke marker concentrations in wine to indicate wildfire smoke exposure of grapes used for commercial wine.

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Supplemental Data

The following supplemental materials are available for this article at ajevonline.org:

Supplemental Table 1 Commercial wine included in the study.

ORCID

Mango Parker 0000-0002-7200-601X
WenWen Jiang 0000-0003-2085-7848
Adrian D. Coulter 0000-0002-9300-2544
Tracey E. Siebert 0000-0002-4878-839X
Eleanor Bilogrevic 0000-0003-1896-1507
I. Leigh Francis 0000-0003-4081-3578
Markus J. Herderich 0000-0002-4136-8886

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Data Availability

All data underlying this study are included in the article and its supplemental information.

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