Food Research International 87 (2016) 168–179

Contents lists available at ScienceDirect

Food Research International

journal homepage: www.elsevier.com/locate/foodres

Perceived minerality in sauvignon blanc wine: Chemical reality or cultural construct?

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ARTICLE INFO

Article history:
Received 29 April 2016
Received in revised form 9 June 2016
Accepted 29 June 2016
Available online 1 July 2016

Keywords:
Wine
Minerality
Sensory
Chemical
Cross-cultural

ABSTRACT

The study aimed to determine the relationship between perceived mineral character in wine and wine chemical composition. We investigated the sensory properties and chemical composition of sauvignon blanc wines from two major sauvignon-producing countries, New Zealand and France. Sensory experiments employing 16 wines (8 French, 8 New Zealand) were conducted in Marlborough, New Zealand and in three regions of France, namely Bordeaux, Burgundy, and the Sancerre/Loire region. Wine professionals (31 New Zealanders and 32 French professionals) sensorially characterised the 16 wines under three conditions, bouquet only (ortho-nasal olfaction), palate only (nose clip condition), and full tasting (global condition: ortho-nasal olfaction, retronasal olfaction, taste, trigeminal stimulation). Sensory data from the global condition only are reported in this article. Physical and chemical analyses conducted on all wines included wine standard parameters, elemental composition, volatile aroma composition, and measures of organic acids. Major results demonstrate that (i) on average French and New Zealand wines were perceived similarly in intensity of mineral character, although judgments to individual wines differed as a function of participant culture; (ii) French and NZ participants drew on different information to make their sensory judgments; and (iii) several aspects of wine composition associated positively with perception of mineral character while others associated negatively, the significant associations differing as a function of participant culture.

1. Introduction

Mineral characteristics are frequently reported when white wines are described sensorially (Ballester, Mihnea, Peyron, & Valentin, 2013; Heymann, Hopfer, & Bershaw, 2014; Parr, Ballester, Peyron, Grose, & Valentin, 2015). Despite this, our knowledge and understanding of the role played by a wine's composition in producing characteristics perceived as *mineral* remains limited. Combining both sensory and chemical data has become an established method for examining a range of wine phenomena including regional characterisation of wines (Green, Parr, Breitmeyer, Valentin, & Sherlock, 2011), perceived quality (Sáenz-Navajas et al., 2015), and influence of yeast (King et al., 2010). We aimed to combine sensory data from wine professionals of two diverse cultures (France, NZ) with chemical data to investigate the relation between wine composition and perception of mineral character in sauvignon blanc wines.

In previous articles, we reported sensory data concerning qualitative and quantitative aspects of perceived minerality in chardonnay wines (Ballester et al., 2013) and sauvignon blanc wines (Parr et al., 2015). The present study extends this work by investigating the sensorial reality of perceived minerality in wine from a wine-composition perspective. More specifically, we associate key aspects of the sensory data concerning the sauvignon wines described in Parr et al. (2015) with physical and chemical characteristics of the same sixteen wines to investigate which, if any, aspects of wine composition are potentially relevant to perception of mineral character in sauvignon wines. Analysing both sensory and chemical data together is essential to elucidating the nature of minerality in wine, as chemical data alone cannot predict how an experienced taster will evaluate a wine's character (Noble & Ebeler, 2002).

1.1. Potential sources of perceived mineral character in wine

1.1.1. Wine origin and elemental composition

Historically, wine origin has been an important factor when considering perceived minerality in wine, not least because of an assumed link

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http://dx.doi.org/10.1016/j.foodres.2016.06.026

between perception of mineral character in wine and aspects of vineyard *terroir* or grape provenance (see Maltman, 2013 for a review). A number of wine writers defend the literal definition of minerality, namely the sensory impact of elements such as salts (Vignon, Pillet, Kessler, Lhotellier, & Le Bras, 2012). However, Maltman (2013) propounded that there is no evidence for a direct link between minerals in a soil and elements in a wine. The current study does not purport to address this issue directly. On the other hand, we considered the issue indirectly in two ways. First, we investigated the relation between concentration of wine elements and perceived minerality to determine the relationship between elemental composition and wine flavour, including perception of mineral character (Yang, Duan, Du, Tian, & Pan, 2010). In doing this, we note that concentrations of Ca, Mg, K and other elements measured in the wines are as likely to have their source in oenological processing (e.g., deacidification; use of bentonite for fining) as in vineyard soil composition (Maltman, 2013). Our interest in the current study was in their influence on wine perception rather than their source. Second, we investigated the influence of wine origin, with sauvignon wines from French regions where the wines have a long history of being described in terms of stony or soil notes (e.g., “flinty”, “silex”, “chalky”, “wet stones”) by their producers compared with New Zealand sauvignons. The latter wines have historically been described as fruit-driven, with the interest in their perceived minerality a more recent fashion (Parr, Green, White, & Sherlock, 2007; Parr et al., 2015). Hence, we measured a suite of biologically relevant elements that may affect wine perceived attributes, either as a result of plant uptake (Tyler & Olsson, 2001) or oenological processing.

1.1.2. Acidity

Mineral character in wine, in particular in wines from cooler climates, is often associated with wine acidity (Coutier & Marchand, 2011; Ross, 2012) by wine writers and wine producers. To date, this remains a hypothesis in that data from the few studies that have investigated perception of minerality in wine in relation to wine acidity have produced equivocal results. Heymann et al. (2014) reported minerality to be positively correlated with acid taste, and with measures of malic acid, tartaric acid, and titratable acidity. On the other hand, Ballester et al. (2013) found one sub-group only of their participants to associate minerality with perceived acidity, and Parr et al. (2015) reported no association or an inverse association of perceived minerality with perceived sourness. One conceivable reason for the lack of consistency in previously reported data is that the relation between individual aspects of acidity such as pH and titratable acidity may be more relevant to perceived wine qualities than the individual components themselves. In the present study we explored how the quantitative relation between wine pH and titratable acidity (e.g., low pH and high TA, low pH and low TA) related to qualitative and quantitative aspects of perceived minerality in wines by employing an acidity index (Plane, Mattick, & Weirs, 1980) as a derived variable.

1.1.3. Association of minerality with sulphur compounds

Concurrent increased usage of the descriptor *mineral* and increased usage of anoxic bottle closures, in particular screw-cap closures, has not gone unnoticed by wine writers (e.g., Goode & Harrop, 2011). Various hypotheses have been put forward in wine industry media, often indirectly, to suggest that increased perception of minerality in wines bottled under closures other than cork could have its basis in factors related to sulphide reduction and/or the implication of odoriferous thiol compounds. We investigated wine compositional differences in the volatile sulphur compounds typically associated with the pungent aromas of perceived reduced character (e.g., hydrogen sulphide) with perception of mineral character in the wines. As with acidity, recent empirical results concerning this hypothesis are not definitive. Although Parr et al. (2015) found no association, Ballester et al. (2013) and Heymann et al. (2014) both reported sensory data demonstrating a positive relationship between reductive notes and perceived minerality. However, this perceptual relationship was not confirmed conceptually with Ballester et al. (2013) and Le Fur and Gautier (2013) both reporting few citations of reductive or sulphur notes when wine experts provided linguistic data in the form of definitions of minerality.

Other sulphur compounds found in sauvignon wine, notably the varietal thiols, are generally considered to contribute positively to a wine's aroma (Darriet, Tominaga, Lavigne, Boidron, & Dubourdieu, 1995; Green et al., 2011; Tominaga, Furrer, Henry, & Dubourdieu, 1998). Further, several thiol compounds reported in sauvignon wine and in chardonnay wine have been linked with mineral notes in prior research. For example, Tominaga, Guimbertau and Dubourdieu (2003a, b) identified and assayed a thiol compound benzenemethanethiol (BMT), the descriptors of which are gun flint, in various wines including chardonnay, sauvignon blanc and sémillon, with wine tasters reporting empyreumatic, spicy and mineral characters in sauvignon blanc wines as BMT concentration increased (Tominaga, Baltenweck-Guyot, Peyrot des Gachons, & Dubourdieu, 2003). In their sensory study, Parr et al. (2015) reported that citrus and passion fruit, two characters often associated with 3-sulfanylhexan-1-ol (3MH) and 3-sulfanylhexyl acetate (3MHA), respectively, significantly contributed but in opposite ways to the perception of minerality in New Zealand sauvignon wines (Parr et al., 2015), with citrus a positive predictor and passion fruit a negative predictor of minerality. Hence, in the current study, we determined concentrations of varietal thiols in the wines including BMT and a fermentative thiol with reductive notes, ethyl-2 sulfanylacetate (E2MA) (Nikolantonaki & Darriet, 2011).

1.1.4. Absence of flavour in wine

The final hypothesis we considered concerns an association between perceived minerality in white wine and the sensory context created by relative absence of flavour in a wine, in particular, the absence of fruity characteristics (Goode, 2012). It has been hypothesised by some wine writers that a low intensity of varietal flavours (e.g., low concentrations of varietal thiols, esters, and/or methoxypyrazine compounds in sauvignon wine) may give rise to a sensory context where a wine is judged as *mineral* by a process of elimination, that is, merely because no other descriptor comes to mind. To investigate this notion, the current study related measures of perceived minerality with determination of concentrations of chemical compounds typically associated with the fruity and vegetal aspects of sauvignon blanc wines (Green et al., 2011).

1.2. Sauvignon blanc

Sauvignon blanc, *Vitis vinifera* L. var. sauvignon blanc, has been described as a variety presenting characteristic nuances from herbaceous characters associated with various alkyl 2-methoxypyrazines to more fruity nuances related to passion fruit, grapefruit and boxwood nuances associated largely to the presence of powerful varietal thiols. These latter compounds can evolute during wine aging to produce empyreumatic, smoked, flinty/stone nuances depending on vine provenance (Dubourdieu, Tominaga, Masneuf-Pomarede, Peyrot des Gachons, & Murat, 2006). The implicated chemical compounds, along with the sensory qualities of sauvignon wine, have been the subject of much research (e.g., Allen, Lacey, Harris, & Brown, 1991; Darriet et al., 1995; Tominaga et al., 1998; Parr et al., 2007; Parr, Valentin, Green, & Dacremont, 2010; King et al., 2010; Capone & Jeffery, 2011; Green et al., 2011; Pena-Gallego, Hernandez-Orte, Cacho, & Ferreira, 2012). For current purposes, sauvignon blanc is an ideal varietal upon which to investigate perceived minerality as a function of grape provenance. The major reason for this is that the classic variants from central France are frequently described in terms of mineral characteristics (Easton, 2009; Parr et al., 2010) whereas New Zealand sauvignon is judged typically as exhibiting intense fruity and vegetal characters rather than obvious minerality (e.g., Parr et al., 2007, 2010). Hence, in terms of our directional hypothesis linking wine flavour and perceived minerality, the classic French sauvignons and the NZ wines may have distinct sensory and compositional profiles in terms of wine impact compounds.

1.3. Summary and hypotheses

Over recent years, multivariate statistical techniques have been developed to elucidate the relationships between sensory and physicochemical data. We employed these techniques to correlate sets of sensory data from two cultures with chemical data concerning composition of French and NZ sauvignon wines. Based on the limited scientific literature on the topic of minerality in wine, along with anecdotal evidence from wine industry professionals, we hypothesised that there would be an inverse relation between perceived minerality and concentration of volatile aroma compounds considered the source of sauvignon blanc wine's characteristic varietal character, primarily the volatile thiols and 3-isobutyl-2-methoxypyrazine (IBMP). We also predicted that qualitative and quantitative aspects of wine acidity, volatile sulphur composition, and elemental composition would each associate statistically with perceived minerality, the precise nature of such relations to be determined. Finally, on the basis of data reported in Parr et al. (2015) where French participants relied on olfaction to a greater degree in their global wine judgments than NZ participants, we hypothesised that cross-cultural differences would be evidenced in terms of physico-chemical sources of perceived minerality.

2. Materials and methods

2.1. Sensory methods

2.1.1. Participants

Thirty-one NZ (11 female, 20 male) and 32 French (4 female, 28 male) wine professionals participated in the sensory study. All were experienced with production and tasting of sauvignon wines. Mean age of the NZ participants was 38.6 years (age range = 30–61 years). Participants included oenologists, winemakers, and wine producers, fourteen of whom were formally designated wine judges. Three participants reported that they were smokers. Mean number of years of wine industry experience was 13.5 years (range = 6–32 years). The French wine professionals (mean age = 43.8 years, range = 26–61 years) participated in one of the three major sauvignon-producing regions of France, either in Bordeaux ($N = 10$), Sancerre ($N = 13$), or Chablis in Burgundy ($N = 9$). They included oenologists ($N = 16$), oenology researchers ($N = 5$), vignerons/viticulturists ($N = 10$), and negociants ($N = 1$) who were involved in sauvignon blanc production and sales. Smoking data were not recorded. The experiment was performed in the official language of the respective countries, namely in French in France and in English in NZ, and in keeping with ethical requirements of the Lincoln University Human Ethics Committee, NZ, and University of Burgundy, France.

2.1.2. Wines

Sixteen sauvignon blanc wines from the 2010 vintage were selected for the experiment. The wines, listed in Table 1, comprised eight sauvignon wines from Marlborough, NZ, and eight sauvignon wines from France. All wines were 100% sauvignon blanc. The French wines were from the major sauvignon regions of France, namely Loire, Saint Bris, and Bordeaux. The Marlborough, NZ sauvignons were from four sub-regions of Marlborough: Rapaura, Wairau Lowlands, Southern Valleys, and Awatere Valley. The wines were selected by senior wine professionals in each country on the basis of three main criteria. These were (i) that each wine was judged by its producers as reflecting well its provenance and its vintage (2010), (ii) that the eight wines from each country spanned a range in terms of a priori judgments of perceived minerality (e.g., as judged by available wine reviews by reputable wine critics/reviewers), and (iii) that the wines were non-oaky. A fourth consideration concerned employing French and NZ wines produced from both hand-harvested and machine-harvested fruit. This factor was included due to recent reports (Parr, Schlich, Theobald, & Harsch, 2013) that grape processing operations at harvest may influence concentrations of key impact compounds that underlie the varietal expression of sauvignon blanc wine.

Table 1
Sauvignon blanc wines employed in the experiment. Marlb = Marlborough, New Zealand. All wines were from the 2010 vintage.

Wine code	Wine origin	Alc % *v*/*v*
NZSVA	New Zealand, Southern Valleys, Marlb	13
NZLWWH	New Zealand, Lower Wairau, Marlb	14
NZAVV	New Zealand, Awatere Valley, Marlb	13.5
FBCLM	France, Bordeaux	13
FSBI	France, Saint Bris	12.5
FLPC	France, Loire	12.5
NZLWB	New Zealand, Lower Wairau, Marlb	13
FSBAGA	France, Saint Bris	12.5
NZSVCH	New Zealand, Southern Valleys, Marlb	13.5
FSHB	France, Sancerre	13
FSFC	France, Sancerre	13
NZAVFB	New Zealand, Awatere Valley, Marlb	13
FLCH	France, Loire	12.5
NZRSL	New Zealand, Rapaura, Marlb	13.5
FBCBB	France, Bordeaux	13.5
NZRS	New Zealand, Rapaura, Marlb	13
Warm-up	New Zealand, Marlborough	12.5

2.1.3. Procedure

The NZ component of the study was conducted at the sensory facilities of the Marlborough Wine Research Centre (MWRC), Blenheim, NZ, in two sessions, each lasting approximately two hours, and separated by 1 week (7–9 days). Due to geographical distance between the French wine regions, the sensory sessions for the French component took place in three facilities: the sensory lab of the Institut des Sciences de la Vigne et du Vin in Bordeaux, the meeting room of the Centre Technique Interprofessionnel des Vins du Centre-Loire in Sancerre, and the meeting room of the Bureau Interprofessionel des Vins de Bourgogne (BIVB) in Chablis. All facilities were suitable in terms of requirements for conducting sensory analysis (ASTM, 1986), providing a quiet, odourless environment that was large enough to avoid communication between panelists.

The fully within-subject design required every participant to evaluate every wine via every task, the order of the tasks being the same for each participant. Participants were advised that they would taste and make judgments about sixteen wines and that all wines were sauvignon blanc. They were not given any other information about the study. Wines were served in standardised tasting glasses (ISO, 1977) that were opaque (black) to eliminate visual cues and were coded with 3-digit numbers. In order to limit carryover effects and memory biases, all wine samples were presented in a different order specific to each participant within each session according to a Williams Latin square arrangement generated by FIZZ software (Biosystemes, Courtenon, France).

In their first session, participants undertook by full global evaluation an extended, free-sorting task followed by a descriptive rating task. Data from the descriptive rating task only are reported in the current article. Table 2 shows that the 20 descriptors employed comprised five sauvignon aromatic characteristics (herbaceous, boxwood, citrus, green, passion fruit), three tastes (sweetness, bitterness, sourness), five classes considered as potential descriptions of types of perceived minerality and/or reductive characteristics (flinty/stony/smoky, chalky, iodine/oyster shell, pencil/graphite, matchstick/burnt rubber/sulphide), and six other characteristics, the latter three in particular considered to require ensemble (i.e., overall) judgments (astringency, freshness, concentration, complexity, familiarity, liking). Thus, the rated characteristics comprised those related to sauvignon varietal character (Parr et al., 2007, 2010) and those relevant to perception of minerality and

Table 2
Descriptors and ensemble-judgment attributes employed in the sensory experiment.

English terms	French terms	Experimental conditions		
		Olfactory	Nose clip	Global
Mineral	Minéral	Yes	Yes	Yes
Passion fruit	Fruit de la passion	Yes	No	Yes
Herbaceous	Herbe coupée	Yes	No	Yes
Boxwood/cat's urine	Buis/pipi de chat	Yes	No	Yes
Citrus (lemon, grapefruit)	Agrumes (citron, pamplemousse)	Yes	No	Yes
Green (vegetal/green capsicum)	Végétal/poivron vert	Yes	No	Yes
Sweetness	Sucré	No	Yes	Yes
Bitterness	Amertume	No	Yes	Yes
Acidity/sourness	Acidité/Vivacité	No	Yes	Yes
Astringency	Astringent/sécheresse	No	Yes	Yes
Concentration	Concentration	Yes	No	No
Concentration/palate weight	Corps/volume en bouche	No	Yes	Yes
Freshness/zingy	Fraîcheur	Yes	Yes	Yes
Flinty/stony/smoky/gun flint	Pierre a fusil/silex frotté/fumé	Yes	Yes	Yes
Chalky/calcareous	Craie/calcaire	Yes	Yes	Yes
Iodine/oyster shell	Coquillages/iodé	Yes	No	Yes
Pencil/lead/graphite	Mine de crayon/graphite	Yes	Yes	Yes
Matchstick/struck match/ burnt rubber/sulphide	Allumette/allumette grattée/phosphore/Caoutchouc brule/souffre/réduit	Yes	No	Yes
Complexity	Complexité	Yes	No	Yes
Familiarity	Familiarité	Yes	No	Yes
Liking	Aimez ce vin?	Yes	No	Yes

reductive characteristics (Ballester et al., 2013). The descriptors were rated in the same order by all participants with the descriptor "mineral" always the first descriptor to be rated and "liking" was the final wine attribute to be rated. Flavour intensities were rated via a 100 mm, horizontal visual analogue scale (VAS: see Parr et al., 2007) anchored by the words "absent" on the left-hand side and "very strong" on the right-hand side. The scales for the ensemble wine characteristics were anchored as follows: concentration/palate-weight and complexity scales were anchored with "low" at the left-hand end and "high" at the right-hand end, the familiarity scale was anchored with "unfamiliar" at the left-hand end and "familiar" at the right-hand end, and the liking scale with "strongly dislike" to the left and "strongly like" to the right.

In a second session, participants evaluated the same wines via two other tasks and these data are reported elsewhere (Parr et al., 2015).

2.2. Chemical and instrumental analyses

At the time of the sensory study in NZ, samples (180 mL) from each of the 16 wines were collected, placed into three 40-mL SPME sample bottles and two 30-mL specimen vials, immediately frozen, and sent to Lincoln University for selected chemical analyses. Five volatile organic acids, sixteen fermentation-derived volatile aroma compounds (Table 3), one methoxypyrazine compound (IBMP), five non-volatile organic acids, and eighteen elements were measured in all wines at Lincoln University. A further 500 mL of each wine was taken for replicate testing (triplicate) of physical wine parameters (e.g., pH, sugars, TA, Alc. *v*/*v*) at MWRC. These latter samples were not frozen.

Just after the sensory study's conduction in Bordeaux, wine samples were taken for analysis of five volatile thiol compounds and six volatile sulphur compounds. This meant that the NZ and French wine samples for chemical analysis were all taken within a 1-month time period. The analyses at the University of Bordeaux were conducted in duplicate. Sample volumes taken from the 16 wines were kept under nitrogen at 4 °C in glass bottles until required for analysis. Subsamples of 2 × 10 mL and 2 × 50 mL were taken on the same day for the analysis of volatile sulphur compounds and volatile thiols, respectively.

2.2.1. Wine standard parameters

Wine sugars, pH, titratable acidity (TA), ethanol, free and total sulphur dioxide (SO_2), and dry extract were determined at MWRC. All analyses were undertaken in triplicate. Titratable acidity in g L^{-1} was analysed by the titration of 5 mL of juice diluted with 30 mL of distilled water to pH 8.2 using a Mettler Toledo DL50 autotitrator and Mettler Toledo pH electrode (Mettler Toledo Gmbtt Analytical, Switzerland), with endpoint calculations performed by LabX Pro titration software (Mettler Toledo Gmbtt Analytical, Switzerland). A Metrohm 744 pH meter and Metrohm Solitrode electrode (Metrohm AG, Switzerland) were used to determine pH values. Determinations of wine pH and total acidity (g L^{-1}) were used to compute an Acidity Index for each wine as per Plane et al. (1980):

Acidity index = total acid (g L^{-1}) − pH

Reducing sugars in gL^{-1} were determined by the Rebelein method as described in Iland (2004). Free, bound, and total sulphur dioxide concentrations in mg/L were determined by the aspiration method described in Iland (2004). Ethanol was determined by NIR using an Anton Paar alcoholyser. Dry extract was calculated in g L^{-1} from density and alcohol.

Table 3
The 16 fermentation-derived aroma compounds measured and sensory descriptors associated to the compounds not including the volatile acids (see Table 4) and the sulphur compounds which are listed in the Methods section of the text.

Chemical compound	Odour/flavour notes
Isoamyl acetate	Banana, fruity, sweet[a,b,c]
Isoamyl alcohol	Fusel, solvent[a,b,d]
Ethyl hexanoate	Fruity, anise, green apple, banana, brandy, wine-like[a,b,c]
Hexyl acetate	Apple, cherry, pear, sweet, floral[b,g]
Hexanol	Grass, herbaceous, woody[a,b,e]
trans-3-Hexen-1-ol	Green, bitter, fatty[g,d,h]
cis-3-Hexen-1-ol	Grass, green, bitter, fatty[a,b,e]
Phenylethyl alcohol	Rose, honey[a,b,d]
Ethyl butanoate	Fruity, strawberry, apple, banana[a,b,c]
Ethyl isobutyrate	Fruity, strawberry[a,c,e]
Ethyl acetate	Varnish, pineapple, sweet, fruity, solvent, balsamic[b,f,g]
Ethyl decanoate	Fruity, brandy, grape, soap[a,b,f]
Benzaldehyde	Almond, fragrant, nutty, cherry[b,g]
Ethyl octanoate	Sweet, floral, fruity, fresh, banana, pear, brandy[a,b,c]
Isobutanol	Bitter, fusel, solvent, alcohol, nail polish[b,d,f]
Diethyl succinate	Fruity, melon, winelike[a,b,h]

Notes: [a](Sáenz-Navajas, Campo, Fernández-Zurbano, Valentin, & Ferreira, 2010)
[b] (Peinado et al., 2004)
[c] (Botelho, Mendes-Faia, & Climaco, 2008)
[d] (Campo, Ferreira, Escudero, Marqués, & Cacho, 2006)
[e] (Escudero, Campo, Fariña, Cacho, & Ferreira, 2007)
[f] (Lukić, Plavša, Sladonja, Radeka, & Peršurić, 2008)
[g] (Fang & Qian, 2005)
[h] (Moyano, Zea, Moreno, & Medina, 2002)

2.2.2. Solid phase micro-extraction (SPME) fiber selection and conditioning

A 2-cm-long Stableflex DVB/CAR/PDMS combination SPME fiber (p/n 57,348-U, Supelco Bellefonte, PA, USA, through Sigma Aldrich Australia) was selected for non-sulphur SPME methods used in this current work. Prior to use, the SPME fiber was conditioned at 270 °C in the gas chromatograph mass spectrometer (GC-MS) injection port for 1 h. Before each sample was analysed, the SPME fiber was conditioned in helium for 10 min at 270 °C in a fiber conditioning station attached to a CTC Combi-Pal auto-sampler (CTC Analytics AG, Zwingen, Switzerland) used with the Shimadzu GC-MS (Shimadzu Corporation, Kyoto. Japan).

2.2.3. Volatile organic acids—HS-SPME GC-MS

Five volatile organic acids were determined utilising a procedure reported in full elsewhere (Tomasino et al., 2015) with their quantification parameters listed in Table 4. Details of the procedure employed are as follows. Automated headspace (HS) SPME-GC-MS analysis was carried out on a Shimadzu GC-MS-QP2010 GC-MS equipped with a CTC Combi-Pal auto-sampler using GC-MS solution version 2.5 as the data acquisition software. The chromatography was performed using two GC columns in series, namely a Rtx-Wax 30.0 m × 0.25 mm ID × 0.5 μm film thickness (polyethylene glycol; Restek, Bellefonte, PA, USA) and a Rxi-1 ms 15 m × 0.25 mm ID × 0.5 μm (100% dimethyl polysiloxane; Restek, Bellefonte, PA, USA). The helium carrier gas was set to a constant linear velocity of 46.8 cm s^{-1}. The injector was operated in splitless mode for 3 min then switched to a 20:1 split ratio.

The column oven was initially held at 50 °C for 3 min (during desorption of the SPME fiber), then heated to 240 °C at 10 °C min^{-1}, then further increased to 250 °C at 30 °C min^{-1} and held at this temperature for 5 min. The total run time was 27.33 min. The interface and MS source temperatures were set at 250 °C and 200 °C, respectively. The MS was operated in electron impact mode (EI) at an ionisation energy of 70 eV.

All analytic data were acquired in full scan mode. Selected ions (m/z in scan mode, Table 4) were used for the quantification of these analytes, by comparing the peak area of selected ions for each compound with those of the selected internal standards. The peak area ratios were then plotted against the concentration of prepared standards (Table 4).

All five standards used to generate calibration curves for quantitative analysis were obtained from commercial suppliers Sigma-Aldrich (Auckland, New Zealand), Merck (Auckland, New Zealand) and AppliChem GmbH (Lab Supply Ltd., Dunedin, New Zealand). The 3 deuterated standards used were purchased from Sigma-Aldrich and CDN isotopes (SciVac PTY. Ltd., Hornsby NSW, Australia).

Primary standard solutions were prepared in 10% ethanol (Scharlau Chemie SA, HPLC Grade ACS ISO UV–vis) for 4 of the 5 volatile organic acid compounds and 2 of the 3 deuterated compounds. Primary standards were made in 100% ethanol for octanoic acid and d_2-octanoic acid due to their low aqueous solubility. All primary standard solutions were stored in amber bottles at −20 °C.

For GC-MS analysis, secondary standard solutions were prepared from these primary standards by dilution with either 100% ethanol (for octanoic acid and d_2-octanoic acid) or 10% ethanol for the rest. The concentration of each standard solution was individually selected so that when combined into a composite standard could be diluted to working strength. This created a range of concentrations that fully spanned the anticipated concentrations of the volatile organic acids found in the wine. Six diluted standards were used to generate the calibration curve for each aroma compound. The diluent for these standards was acidified deionised water (pH 3.5) with the ethanol content standardised to 1.4% (10-fold dilution).

Sample preparation involved pipetting 0.9 mL of wine (thawed 40 mL SPME sample bottle) and 8.06 mL of acidified deionised water (pH 3.5) into 20 mL SPME sample vials (a 10-fold dilution of the wine), followed by 40 μL of the internal standard solution (a mix of 3 internal standards, see Table 4). Crystalline sodium chloride (4.5 g) was then added to the SPME vial just prior to capping. Samples were incubated initially for 10 min at 60 °C during which time the vial was agitated at 500 rpm. After 10 min the SPME fiber (2 cm long Stableflex DVB/CAR/PDMS) was exposed to the headspace of the vial for a period of 30 min at 60 °C. During this exposure period the headspace volatiles were adsorbed onto the fiber. Desorption of these volatiles occurred for 5 min at 270 °C.

2.2.4. Fermentation derived volatile aroma compounds

Sixteen fermentation-derived aroma compounds were quantified using a GC-MS procedure similar to that described above but with some minor differences including differing carrier flow rates and temperature ramps as described by Green et al. (2011). The quantification parameters for the two compounds not reported in Green et al. (2011), namely isobutanol and diethyl succinate, are shown in Table 5.

2.2.5. HS-SPME MD-GC-MS of 3-isobutyl-2-methoxypyrazine (IBMP)

3-isobutyl-2-methoxypyrazine concentrations in the wines were determined using an adaption of the automated HS-SPME (headspace solid-phase micro-extraction) method described elsewhere (Parr et al., 2007). Automated multi-dimensional gas chromatography mass spectrometry (MD-GC-MS) was used to enhance the single dimension GC separation method described by Parr et al. (2007) into a two-dimensional procedure. The two dimensional procedure provided more sensitivity through improved chromatographic separation and was a more prudent approach given the work of Schmarr et al. (2010) who reported co-elution anomalies with standard GC-MS methods for methoxypyrazine analysis in wines that included HS-SPME techniques.

The automated HS-SPME MD-GC-MS analysis of IBMP was carried out using a Shimadzu GC-2010 oven containing a special pressure switch (Dean's switching valve) coupled to a Shimadzu GC-MS (Model QP2010-Ultra) equipped with a CTC Combi-Pal auto-sampler. Chromatographic separation was performed using a Rtx-Wax (polyethylene glycol) 60 m × 0.25 mm ID × 0.25 μm film thickness (Restek, Bellefonte, PA, USA) connected to the "Dean's switching valve" (DSV) in the first GC oven (first dimension). The DSV (Deans, 1968) is pressure-controlled and was used to transfer a timed segment of the column outflow (heart cut) of the first dimension to a GC column in the second

Table 4
Quantification parameters for the 5 volatile organic acid analytes.

Analyte	ISTD[a]	RT (min)	Target ion m/z	Confirming ions m/z (% to target ion)	Calibration range μg/L (1/10 dilution)	R^2 #	Purity of standards (%)	CAS No.	Supplier
d_4-Acetic acid	(1)	12.37	46	63 (72)	–	–	99.9A%	1186-52-3	CDN isotopes
d_{11}-Hexanoic acid	(2)	17.25	63	77 (43), 93 (12)	–	–	98A%	95348-44-0	Sigma
d_2-Octanoic acid	(3)	19.69	62	74 (33) 102 (12)	–	–	98 A%	64118-36-1	CDN isotopes
Acetic acid	1	12.43	43	60 (82), 45 (84)	0–204,878	0.9999	99	64-19-7	Merck
Isobutyric acid	1	14.06	88	42 (170)	0–395.12	0.9994	99	79-31-2	Sigma
Butanoic acid	1	14.81	60	73 (27)	0–2195	0.9994	99	107-92-6	AppliChem
Hexanoic acid	2	17.41	60	73 (41), 87 (12)	0–1170.7	0.9999	99	142-62-1	Sigma
Octanoic acid	3	19.74	60	73 (56), 101 (20)	0–878	0.9999	97.5	124-07-2	BDH (Merck)

All fitted standard (calibration) curves were quadratic functions.

[a] Internal standards used are in brackets.

Table 5
Quantification parameters for additional fermentation-derived aroma compounds, method in Green et al. (2011).

Analyte	ISTD[a]	RT (min)	Target ion m/z	Confirming ions m/z (% to target ion)	Calibration range μg/L (1/10 dilution)	R^2 #	Purity of standards (%)	CAS No.	Supplier
Isobutanol	4	13.42	43	41 (72), 74 (10)	0–1272	0.9999	99.5	78-83-1	Sigma
Diethyl succinate	–	33.50	129	128 (27), 102 (23)	0–320.3	0.9981	99	123-25-1	Sigma

#All fitted standard (calibration) curves were quadratic functions.
[a] Internal standard 4 is d_{13}-hexanol (Green et al., 2011).

oven (second dimension). The DSV utilises an extra pressure controller to ensure no loss of carrier flow in the second dimension. In this case, a Rtx-5 ms (5% diphenyl 95% dimethyl polysiloxane) 30 m × 0.25 mm ID × 0.25 μm film thickness (Restek, Bellefonte, PA, USA) column was connected to the outflow of the DSV and was installed in the GC-MS oven (second dimension). A 0.75 mm ID SPME inlet liner (SGE Analytical Science Pty. Ltd., Ringwood, Victoria, Australia) was used in the injection port. The helium carrier gas was programmed to a linear velocity of 20 cm s^{-1} in the first dimension column with a resultant initial column flow of 1.69 mL min^{-1} in the second dimension column. A separate switching pressure (92 kPa programmed to 163 kPa) was applied to the DSV to ensure 100% of the material switched from the first dimension column was loaded onto the second dimension column. A constant flow of 1.3 mL min^{-1} was maintained in the second dimension column from the 45th to the 62nd minute to ensure adequate separation of IBMP as described in Parr et al. (2007). The injector was set to a temperature of 270 °C and operated in split mode at a ratio of 20:1 to reduce column overloading. The first dimension column was held at 35 °C for 3 min then increased to 100 °C at 8 °C min^{-1}, held for 5 min then increased to 120 °C at 1 °C min^{-1} and held for 5 min before a final increase to 250 °C at 40 °C min^{-1} where this was held for 17 min. The second dimension column was held at 35 °C for 15 min then increased to 90 °C at 3 °C min^{-1} and held for 19.67 min then increased to 250 °C at 40 °C min^{-1} and held for 5 min. The total runtime was 62 min.

The temperature of the flame ionisation detector (FID) (first dimension GC oven) was set at 250 °C. The mass spectrometer (MS) was operated in electron impact mode (EI) at an ionisation voltage of 70 eV with the interface and source temperatures set at 250 °C and 200 °C, respectively. IBMP was quantified using selected ion monitoring (SIM) mode with the selected ions m/z 124 and 151 with the internal standard d_3-IBMP using selected ions m/z 127 and 154. The m/z ratio 124/127 was used to quantify the IBMP concentrations in the wine samples. The retention time of IBMP and d_3-IBMP in the first dimension was 34.82 min with the second dimension retention times being 46.24 min for IBMP and 45.97 min for d_3-IBMP. The cut window used to switch IBMP and d_3-IBMP from the first to the second dimension column was 34.11–35.82 min.

A 2 cm Stableflex DVB/CAR/PDMS SPME fiber was used to increase adsorption capacity and hence sensitivity. As a result, the dilution of the wine was changed from a 5- to a 3-fold dilution, namely 3.0 mL of wine and 4.85 mL of deionised water were added to a 20 mL SPME sample vial followed by 150 μL of (d_3)-isobutyl-2-methoxypyrazine (d_3-IBMP) deuterated internal standard solution. To this, 1 mL of 4 M NaOH was added followed immediately by 3.0 g of crystalline sodium chloride. The sample vial was then quickly capped.

2.2.6. Non-volatile organic acids—HPLC

The five non-volatile organic acids (Table 6) were quantified using an adapted HPLC procedure described elsewhere (Shi et al., 2011). The more important procedural details are given below.

The HPLC analysis was conducted using a Shimadzu LC system (Shimadzu Corporation, Kyoto. Japan) consisting of a system controller CMM-20A, pump LC-20CE, degassing unit DGU-20A5, autosampler SIL-10AF with sample cooler, UV detector SPD-20A, and column oven CTO-10 ASvp. Chromatographic separation was achieved using a Prevail™ organic acid column (250 mm × 4.6 mm, 5 μm particle size; Grace Davison Discovery Sciences) fitted with a guard column (7.5 mm × 4.6 mm, 5 μm particle size; Grace Davison Discovery Sciences). The mobile phase used was 25 mM KH_2PO_4 (pH 2.5, adjusted by H_3PO_4) filtered through a 47 mm × 0.45 μm cellulose acetate membrane (Advantec supplied by MicroAnalytix, New Zealand). The column flow rate was set to 0.6 mL min^{-1}, at a temperature of 50 °C. The UV detector wavelength was set a 210 nm with a sample injection volume of 20 μL.

Standard L-tartaric acid (99.5%), shikimic acid (99%), and malonic acid (99%) were purchased from Sigma-Aldrich (Auckland, New Zealand), L-malic acid (>99%) and acetic acid (100%) were obtained from Merck (Auckland, New Zealand) with DL-lactic acid (85%) obtained from Acros Chemicals (ThermoFisher Scientific, Auckland, New Zealand). A total of 11 working standards were prepared in 13.3% ethanol (Table 6) to cover a range of concentrations required to calibrate the HPLC system.

Adapted from the work of Kordis-Krapez, Abram, Kac, and Ferjancic (2001) wine samples were diluted 4 times with 13.3% ethanol in deionised water, vortex-mixed, and filtered through a 0.2 μm Phenex glass fiber/cellulose acetate membrane (Phenomenex, Auckland, New Zealand). Samples were thawed and diluted just prior to analysis. The identification of the organic acids was obtained by comparing the retention time of the samples to the standards. Quantification was determined by the comparison of peak heights of organic acids, obtained from the chromatograms, using an external calibration standard curve. All data was processed using Shimadzu's LC Solution software version 1.24 SP.

2.2.7. Wine elements—ICP-OES

One milliliter of wine was taken from 30 mL specimen vials and digested with 3 mL of Aristar™ nitric acid (69%) in sealed 100 mL Teflon tubes in a microwave digester (CEM MARS Xpress, CEM Corporation, USA). The temperature was ramped to 150 °C over ten minutes and samples were held for an additional ten minutes at that temperature. Cooled samples were made up to 10 mL using MilliQ (Barnstead Easy Pure RF) water. Concentrations of Al, As, B, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, S, and Zn were determined using inductively coupled plasma optical emission spectroscopy (ICP-OES Varian 720 ES, USA).

2.2.8. Analysis of volatile thiols

2.2.8.1. Specific extraction of volatile thiols. The five volatile thiols were specifically extracted by reversible combination of the thiols with sodium-*p*-hydroxymercuribenzoate (*p*-HMB), as described by Tominaga and Dubourdieu (2006). A total of 7.5 mL p-HMB solution (2 mM in 0.1 M Tris) was added to 50 mL wine samples previously supplemented with 1.2 nmol 4-methoxy-2-methyl-2-mercaptobutane (Oxford Chemicals, Hartlepool, UK) and 6-sulfanylhexan-1-ol as internal standards with the pH raised to 7.0 using a NaOH solution (10 N) under magnetic stirring for 10 min. Wine samples were then percolated for 10 min through a strongly basic anion-exchange column (Dowex 1–1 × 2–100). The column was rinsed with 50 mL sodium acetate buffer (0.1 M, pH 7). The volatile thiols were released from the thiol-p-HMB complex fixed on the column by percolating with 60 mL cysteine solution (10 gL^{-1}) adjusted to pH 7 with NaOH (10 N). The eluate containing the volatile thiols was collected in a 100 mL flask and 0.5 mL ethyl

Table 6
Quantification parameters for 6 non-volatile organic acids.

Analyte	RT (min)	Calibration range mg/L (1/4 dilution)	R^2 #	Purity of standards (%)	CAS No.	Supplier
Tartaric acid	5.53	0–800	0.9998	99.5	87-69-4	Sigma
Malic acid	6.94	0–800	0.9998	99	6915-15-7	Merck
Malonic acid	7.27	0–800	0.9999	99	141-82-2	Sigma
Shikimic acid	8.00	0–40	0.9998	99	138-89-0	Sigma
Lactic acid	8.52	0–800	0.9999	85	50-21-5	Acros
Acetic acid	9.30	0–800	0.9999	99	64-19-7	Merck

All fitted standard (calibration) curves were quadratic functions.

acetate was added. The eluate was extracted twice with dichloromethane (4 and 3 mL, respectively) for 10 min each time, under magnetic stirring. The two organic phases were combined, dried over anhydrous sodium sulfate, and then concentrated under nitrogen flow in a 10 mL graduated tube to approximately 200 μL. The concentrate was then transferred to a 1 mL vial, concentrated to 25 μL, and injected into a gas chromatograph/mass spectrometer (GC-MS) within 24 h.

4-Methyl-4-sulfanylpentan-2-one (4MMP) (1% in polyethylene glycol) and 3-sulfanylhexyl acetate (3MHA) (98% purity) were supplied by Interchim (Montluçon, France). 3-Sulfanylhexan-1-ol (3MH) (>95% purity) was obtained from Acros Organics (Illkirch, France). Ethyl-2 sulfanylacetate (E2MA) (>98% purity) was from Alfa Aesar (Schiltigheim, France), 6-Sulfanylhexan-1-ol (6SH) and benzemethanethiol (BMT) (both 99% purity) were from Sigma-Aldrich (L'Isle d'Abeau Chesnes, France).

2.2.8.2. Quantification of volatile thiols by GC-MS. GC-MS analysis was carried out on a 6890N gas chromatograph (Agilent Technologies) coupled with an MS 5973 Agilent Technologies series mass selective detector (MSD). A 3 μL sample of each concentrated extract was injected in splitless mode (250 °C; purge time = 1 min, purge flow = 50 mL min^{-1}) at an initial oven temperature of 45 °C into a BP20 capillary column (SGE, 50 m × 0.22 mm ID × 0.25 μm film thickness). The GC oven temperature program was as follows: initially held at 45 °C for 10 min then increased to 230 °C at 3 °C min^{-1} and held for 20 min. Helium 55 (Air Liquide, France) was the carrier gas used with a column-head pressure of 22 psi and a flow rate of 1 mL min^{-1}. The mass spectrometer, functioning in electron impact mode (electron energy = 70 eV), was connected to the GC with a transfer line heated to 250 °C. 3MH, 3MHA, 4MMP, BMT, E2MA and the internal standards were detected in SIM mode by selecting the following ions: $m/z = 134$ and 100 for 3MH and the internal standard, 4-methoxy-2-methylbutane-2-thiol; $m/z = 132$, 99, and 75 for 4MMP, $m/z = 124$ and 91 for BMT, $m/z = 116$, 101, 98 for 3MHA, $m/z = 74$ for E2MA, m/z 116, 101 for 6MH. The quantification ions were $m/z = 134$ for both 3MH and 4-methoxy-2-methylbutane-2-thiol (internal standard), $m/z = 75$ for 4MMP, $m/z = 74$ for E2MA, m/z 124 for BMT, m/z 116 for 3MHA, m/z 101 for 6MH (internal standard).

2.2.9. Analysis of volatile sulphur compounds

The analytical method used for the analysis of six volatile sulphur compounds was adapted from Fedrizzi, Magno, Moser, Nicolini, and Versini (2007). Briefly, volatile compounds were extracted by HS-SPME, and then desorbed in a gas chromatograph coupled to a mass spectrometer operating in SIM mode. Sulphur compounds analysed were hydrogen sulphide (H_2S), methylmercaptan (MeSH), ethylmercaptan (EtSH), dimethyl sulphide (DMS), diethyl sulphide (DES), and dimethyl disulphide (DMDS) from Sigma-Aldrich (L'Isle d'Abeau Chesnes, France) with level of purity higher than 98%. Thiophene (99% pure, Sigma-Aldrich) was used as the internal standard.

Volatile compounds were directly extracted using a HS-SPME fiber (75 μm CAR/PDMS fused silica 24 gauge, p/n 57,319, Supelco Bellefonte, PA, USA, through Sigma-Aldrich) after adding the internal standard to the wine sample. Briefly, 10 mL of wine was transferred into a 20 mL SPME vial under nitrogen, and then supplemented with thiophene (10 μL at 100 μg/L) and 5 g of crystalline sodium chloride was added. The sealed sample was placed on the MPS2 GERSTEL autosampler tray and extracted for 30 min with agitation at 35 °C.

The SPME fiber was desorbed in the injection port of an Agilent 6890 gas chromatograph coupled to a 5975 Agilent mass detector. The analysis was performed on a BP 20 column (30 m × 0.32 mm i.d.; 1 μm film thickness fused-silica capillary). Gas chromatography conditions were as follows: GC injector temperature, 250 °C; injection in splitless mode for 1 min; GC oven temperature program was as follows: initially held at 35 °C for 5 min then increased to 40 °C at 1 °C min^{-1} then further increased to 250 °C at 10 °C min^{-1}. The mass spectrometer, functioning in electron impact mode (electron energy = 70 eV), was connected to the GC with a transfer line heated to 250 °C. H_2S, MeSH, EtSH, DMS, DES, DMDS, and the internal standard thiophene were detected and quantified in SIM mode by selecting the following ions: $m/z = 34$, 33 for H_2S, $m/z = 47$, 48, 45 for MeSH, $m/z = 62$ and 47 for EtSH as for DMS, $m/z = 90$, 75, 61 for DES, $m/z = 94$, 79, 45 for DMDS, $m/z = 84$, 58, 45 for thiophene.

3. Data analysis

3.1. Sensory data

Data concerning judgments of perceived minerality from the global condition of the sensory experiment only were included in the analyses. A two-way mixed ANOVA with wine as a within-subject variable and subject origin as a between-subject variable was carried out on the perceived minerality ratings.

3.2. Chemical data

A one-way ANOVA with wine as the main effect was carried out on the concentration of each chemical compound.

3.3. Wine characterisation by multivariate analysis of sensory and physico-chemical data

We employed partial least squares regression (PLSR) with the aim of predicting perceived minerality for each culture from wine chemical composition. PLSR combines features of PCA and multiple regressions and is particularly suitable when there is a need to predict a set of dependent variables from a large set of independent variables. Further, PLSR is appropriate when multicolinearity between independent variables is expected. Since some of the chemicals estimated are assumed metabolically related, multicolinearity was expected. Prior to undertaking the PLSR, independent variables were pre-selected by computing their correlation with French and/or NZ perceived minerality average scores. In order to check possible quadratic relationships, squared values of concentrations were also included in the variables' pre-selection. A significant quadratic predictor with a positive sign can be interpreted as a “minimum effect”, the curve having an apex at the bottom, whereas a quadratic predictor with a negative sign can be interpreted as an “optimum effect” in which perceived minerality reaches a maximum, the apex of the curve being at the top. Only independent variables with a correlation coefficient above 0.30 were considered as PLSR predictors. PLSR analyses were performed with XLstat 2014 1.08 (Addinsoft, New York, USA).

4. Results

4.1. Sensory analyses

ANOVA results on the minerality scores showed significant effects for wine ($F_{15,885} = 2.53$; $p = 0.001$), subject origin ($F_{1,59} = 87.26$; $p < 0.0001$) and the interaction subject origin × wine ($F_{15,885} = 2.22$; $p = 0.0048$). These effects can be observed in Fig. 1, which shows the average minerality scores for each wine and for each culture. For clarity, the wines were ranged from the most mineral to the least mineral according to the perception of French participants.

Fig. 1 shows that the NZ tasters gave higher minerality ratings overall than did the French tasters. This is in keeping with results reported in Parr et al. (2015), where a three-way, mixed design ANOVA conducted on an extended version of the dataset (with culture as the between-subject variable and perception mode and wine as within-subject variables) showed a significant effect of tasters' culture, with NZ scores higher than French scores (Parr et al., 2015; Fig. 1, global).

Fig. 1 also shows the interaction between subject origin and wine. The NZ tasters gave higher minerality scores only to some wines (NZRS, NZAVFB, FSBAGA, NZLWWH, and FSFC), while for other wines, the average scores were similar for French and NZ tasters. This interaction cannot be explained in terms of wine origin as half of the wines that received higher scores from NZ participants were from NZ and the other from France, suggesting that prior exposure was not a major factor in the differences in minerality ratings to the wines. Due to the differences in minerality judgment between cultures for many of the wines, a decision was made to separate the data as a function of culture in subsequent data analyses.

4.2. Chemical analyses

The average concentrations and SD of each measured compound is given in Table 7 (see Supplementary Material). One-way ANOVA showed significant differences between wines for all but four of the compounds estimated: Ba, Cu, Ni, and H_2S (data not given).

4.3. Association of physico-chemical and sensory data by PLSR

The PLSR gave an optimal solution for 2 latent vectors (Stone–Geisser $Q^2 = 0.401$), explaining 40.1% of the X variance and 72.4% of Y. Fig. 2 shows the correlation plot with the projections of the Y variables (NZ and French perceived minerality average scores), the X variables (chemical compounds) and the observations (wines).

4.3.1. Cultural differences

Fig. 2 shows that NZ participants' minerality perceived scores project onto the positive side of the first dimension and the main positive predictors were bound SO_2, hexanoic acid, Na, Ca, and malic acid for both linear and quadratic relationships apart from Ca, which was not significant in its quadratic form. The negative predictors of perceived minerality by NZ participants were isoamyl alcohol, diethyl succinate, and isobutanol for both linear and quadratic relationships (the normalized regression coefficients and their confidence intervals can be seen in Fig. 3a).

Consistent with our hypothesis predicting cultural differences, the French participants' perceived minerality scores project onto the positive side of the second dimension. The significant positive predictors were isoamyl acetate and free SO_2 for both the linear and quadratic relationships, and the negative predictors were tartaric acid and titratable acidity (the normalized regression coefficients and their confidence intervals can be seen in Fig. 3b).

4.3.2. Absence of flavour in wine

With respect to our hypothesis concerning the relation between perceived flavour and minerality, and hence influence of the major impact compounds of sauvignon blanc, namely IBMP, 3MHA, 3MH, 4MMP, BMT, E2MA, and the C6 compounds, PLSR results show that 3MH only, a compound implicated in citrus-type aromas, had a significant and positive effect on perceived minerality for NZ participants. This result is at odds with our hypothesis since 3MH is considered among the most important varietal, sauvignon blanc impact compounds. However, if we consider that 3MH is involved in citrus/grapefruit aromas this result is consistent with Parr et al. (2015) who reported that the citrus aroma was a positive predictor of minerality for wine professionals from both France and NZ.

4.3.3. Wine acidity

Results related to aspects of wine acidity are mixed in terms of support for our hypotheses. More specifically, the results demonstrate that

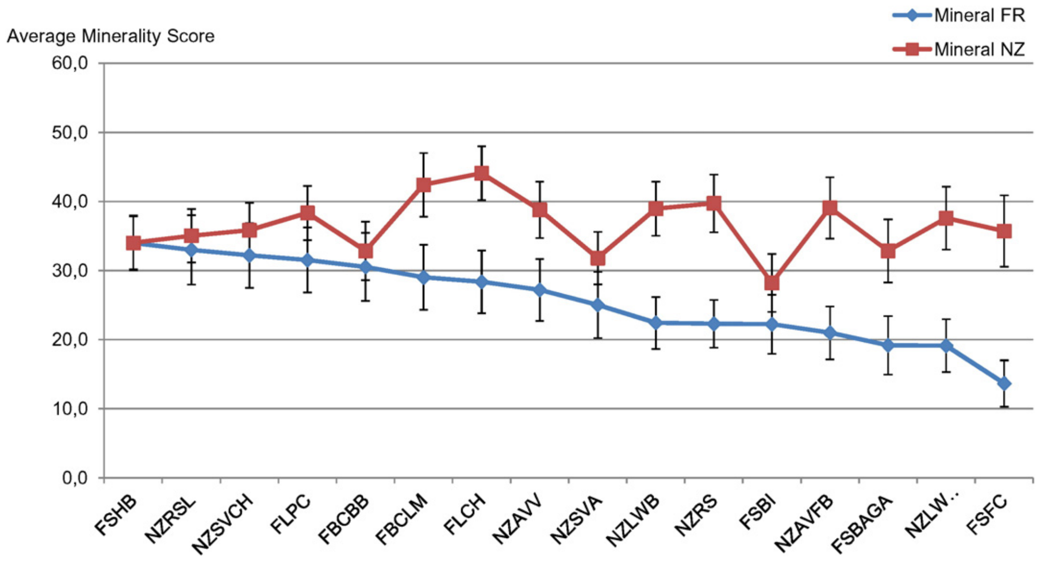

Fig. 1. Average perceived minerality scores for each wine and each culture. Wines with the same letter are not significantly different according to a SNK test ($\alpha = 0.05$).

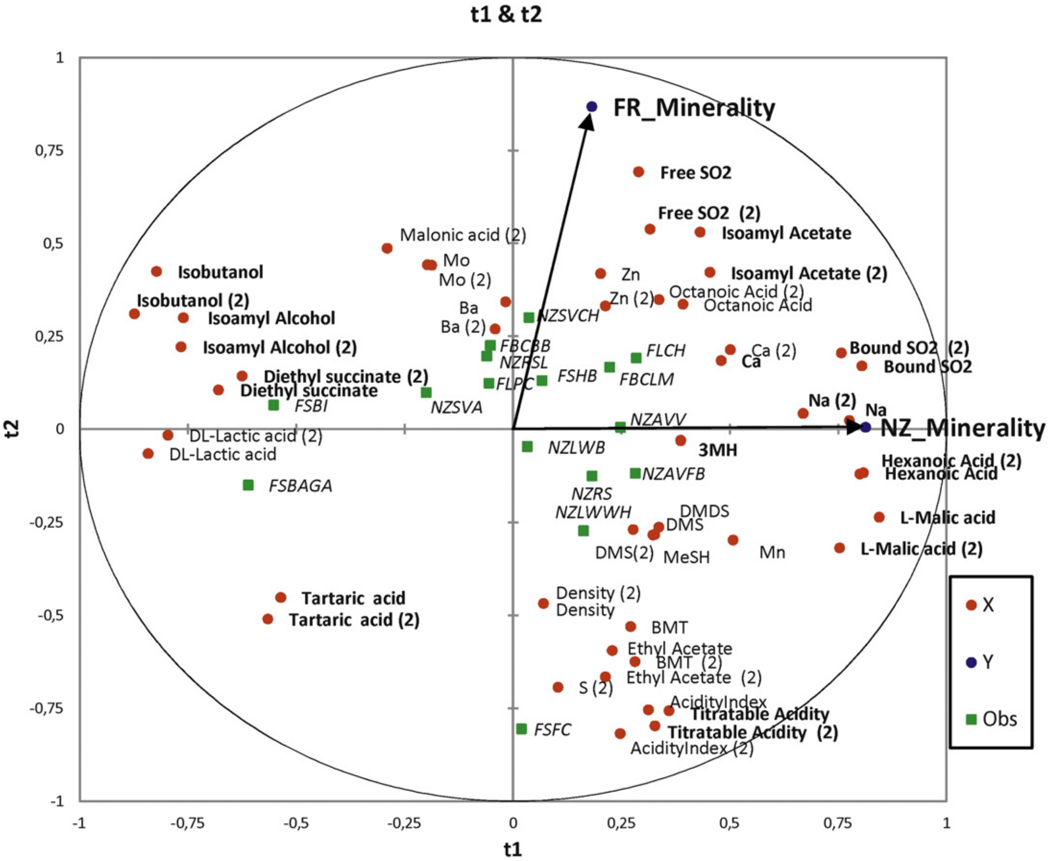

Fig. 2. Correlation plot with projections of the *Y* variables (NZ and French perceived minerality mean scores), the *X* variables (chemical compounds) and the observations (wines). The variables in bold have normalized regression coefficients different from zero for French minerality and/or for NZ minerality; (2) = quadratic variable.

for NZ participants, malic acid was a positive predictor of perceived minerality, while lactic acid tended toward a negative association with minerality. For French participants, tartaric acid and titratable acidity were both significant negative predictors of minerality. Neither wine pH nor the derived acidity index were significant variables in predicting perceived minerality for either culture (see Fig. 3).

4.3.4. Wine elements

In terms of the elements, Ca^{2+} and Na^{+} were positive predictors of minerality for NZ participants. On the other hand, none of the measured elements had a significant role in the prediction of perceived minerality by French participants.

4.3.5. Association of minerality with sulphur compounds

Contrary to our hypothesis, the sulphur compound BMT, which has been reported to be a source of perceived flinty characteristics, was not found to be a significant predictor of perceived minerality for either culture.

The final results important to report in light of our a priori hypotheses concern volatile sulphur compounds commonly associated with perceived reductive phenomena. Our results show that none of the reductive sulphur compounds estimated were significant predictors of perceived minerality for participants of either culture. Interestingly, free SO_2 concentration was the most important predictor of minerality for French participants, and bound SO_2 the most important for NZ participants, both associations being positive. This result is congruent with the sensory data demonstrating a greater reliance on olfactory information by French than NZ tasters in their global wine judgments since free SO_2 would be detectable via olfaction whereas bound SO_2 would be assumed to be experienced primarily as a palate sensation (Bueno, Carrascon, & Ferreira, 2016; Coetzee, Brand, Jacobson, & Du Toit, 2016; Jackowetz & Mira de Orduna, 2013; Liu & Pilone, 2000).

5. Discussion

The current study aimed to investigate the nature of perceived minerality in sauvignon wines from a wine composition perspective. The major result is demonstration that several aspects of chemical composition associated significantly with perceived minerality in sauvignon blanc wines. Further, the specific compounds that served as significant predictors of perceived minerality varied as a function of participant culture. That is, not only did French and NZ participants draw on different sensory information to make their minerality judgements, with French relying more on olfaction while New Zealanders relied equally on olfaction and palate phenomena (Parr et al., 2015), but also the aspects of wine composition predicting each culture's judgments of perceived minerality differed. For French participants, the significant positive predictors of their minerality judgments were isoamyl acetate and free SO_2 while the negative predictors were tartaric acid and total acidity. For NZ participants, positive predictors were bound SO_2, hexanoic acid, Na, Ca, and malic acid while negative predictors were isoamyl alcohol, diethyl succinate and isobutanol. Several of these results are more interpretable than others and these are discussed below in relation to our a priori hypotheses.

Contrary to our hypothesis, the major impact compounds giving sauvignon blanc wines their varietal character, namely the volatile thiols (Dubourdieu et al., 2006) and IBMP (Allen et al., 1991), were not found to be negative predictors of perceived minerality for either culture. This negative result includes not only the thiol compounds considered the source of fruity characters in sauvignon wines, but also the volatile sulphur compound BMT which was identified by Tominaga et

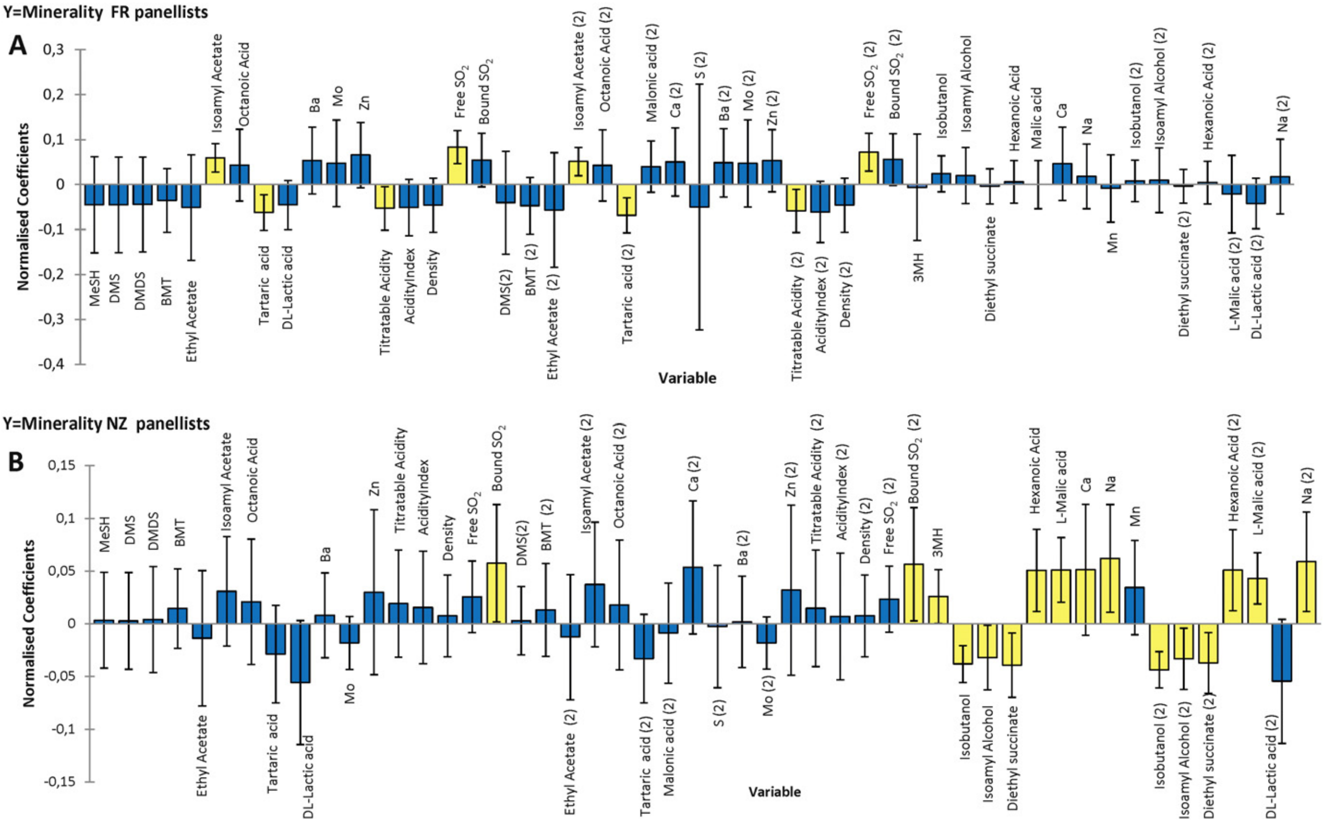

Fig. 3. Normalized regression coefficients and their confidence intervals for the prediction of perceived minerality by French participants (A) and NZ participants (B). Coefficients whose confidence intervals do not cross zero are considered significant.

al. (2003) as associated with empyreumatic and spicy aroma of sauvignon blanc. The lack of a significant association between BMT and perceived minerality was an unexpected finding in that Parr et al. (2015) reported their sensory data as showing stony/flinty notes to be good predictors of global minerality for both cultures. These results do not mean necessarily that the negative relationship between perceived minerality and intensity of varietal character reported by Parr et al. (2015) does not have some of its basis in wine concentration of varietal thiols and IBMP, but that the wine composition effects are potentially more complex (e.g., masking effects as perceptual interaction phenomena may be at play) than the present study has been able to consider. Interestingly, the one compound assumed to contribute a fruity character that was a significant predictor, isoamyl acetate, contributed positively to perceived minerality, rather than negatively, further eroding any support for our first hypothesis.

Our second hypothesis concerning an influence of qualitative and quantitative aspects of wine acidity on perception of mineral character received some support. However, overall the data demonstrate that the relationship between perceived minerality and acidity is complex. First, our derived acidity index relating wine pH and titratable acidity failed to serve as either a positive or negative predictor of perceived mineral character in the wines, contrary to Heymann et al. (2014) results and much anecdotal evidence from wine producers and wine writers. Second, the relation between perceived minerality and the organic acids seems particularly complex. In support of the hypothesis linking mineral character to qualitative aspects of acidity is demonstration that for NZ participants, malic acid, an acid imparting a sharp, green-apple character to musts and wine, was a positive predictor of minerality, whereas lactic acid correlated negatively with minerality. Lactic acid in wine results from the oenological process known as malolactic fermentation (MLF), with the conversion of malic acid to lactic acid resulting in not only a decrease in total acidity but as well a modification in the aromatic profile of a wine with the emergence of lactic notes which tend toward buttery characteristics. Such buttery, milky characters were found negatively correlated to minerality by Heymann et al. (2014) and conceivably could have a negative effect on perception of wine freshness, freshness being a character that Parr et al. (2015) reported to positively associate with perceived minerality for both French and NZ participants. Finally, the result demonstrating that for French participants perceived minerality associated negatively with tartaric acid and total acidity was not entirely unexpected in that Parr et al. (2015) failed to show a positive relationship between perceived sourness and perceived minerality for either culture in the global tasting of these same sauvignon wines.

In terms of the relation between wine volatile sulphur composition and perceived minerality, the present data lend little support to the notion that reported mineral notes in wines bottled under inert bottle closures (the NZ wines) have their source in reductive phenomena. This however does not imply that reported mineral notes do not at times have their source, at least in part, in reductive phenomena, and further research is needed to clarify the complex situation. Indeed, the sensory data published to date are equivocal with respect to this issue, Ballester et al. (2013) and Heymann et al. (2014) reporting a positive relationship, while Parr et al. (2015) did not. What we can say is that in the present results, the concentrations of reductive sulphur compounds did not serve as significant positive predictors of perceived minerality for either culture. However, it is interesting to note that concentration of free SO_2, a compound considered to give a distinctive pungent note when in high concentration, was the most important predictor of perceived minerality for French participants, who were shown in the sensory data to rely to a large degree on olfactory information in their global wine judgments. This result is in agreement with Heymann et al. (2014). Also consistent with the sensory data is demonstration that bound SO_2, the non-volatile component expected to produce a palate sensation rather than a significant odour (Coetzee et al., 2016), was the most important predictor for NZ participants who were shown to rely equally on olfaction and palate information when making their global judgments of perceived minerality. This result is particularly relevant to wine sensory research where in general researchers do not report concentrations of either free or bound SO_2 in their reports of wine sensory phenomena but rather focus on other standard measures such as ethanol and residual sugar as likely determinants of sensory outcomes. Clearly, further research is warranted in this area.

Our hypothesis concerning possible impact of wine elemental composition on perceived minerality in wine received some support. The present data show Ca and Na, likely present in wine as ionic Ca^{2+} and Na^{+}, to be positive predictors of perceived minerality for NZ participants. In keeping with this result, Parr et al. (2015) reported the sensory descriptor chalky/calcareous to be a significant positive predictor of perceived minerality irrespective of participant culture and sensory evaluation mode. However, this descriptor is somewhat ambiguous with both chalky odour and chalky texture possible, and the link between these linguistic terms and underlying sensory-derived concepts with actual ionic calcium is far from obvious. To complicate the situation further, we point out that Maltman's (2013) analysis suggesting that it is unlikely that any particular ion, due to its concentration in wine, plays a role in wine taste did not consider synergistic effects involving various ions at sub-threshold level (Dalton, Doolittle, Nagata, & Breslin, 2000).

Vignon et al. (2012) reported an exploratory investigation concerning sodium, magnesium, potassium and minerality, but there appear to be few published data on the effect of ions on wine taste and/or texture that are based on sound scholarship. Clearly further research in this area is warranted.

Having addressed each of our a priori hypotheses, it now behoves us to comment on the predictors of perceived minerality in sauvignon wines that have been identified in the current study. The negative contributions of fusel alcohols isoamyl alcohol and isobutanol, as well as diethyl succinate do not, at face value, fit any of our hypotheses. While the roles of isoamyl alcohol and isobutanol are far from clear, diethyl succinate could be linked to succinic acid. Diethyl succinate is one of two ethyl esters found in wine formed by esterification of ethanol with succinic acid, a diprotic acid (Ribereau-Gayon, Glories, Maujean, & Dubourdieu, 2006). Along with ethyl succinate, both are regarded as marker compounds in aging white wines with notable increases found in bottled white wines <12 months old (Câmara, Alves, & Marques, 2006; Francioli, Torrens, Riu-Aumatell, Lopez-Tamames, & Buxaderas, 2003; Garde-Cerdán & Ancín-Azpilicueta, 2007; Recamales, Gallo, Hernanz, Gonzalez-Miret, & Heredia, 2011). While it is fair to assume diethyl succinate was generated from succinic acid it is not possible to ascertain from this the quantity of succinic acid present in these wines, which according to Baron and Fiala (2012) influences wine perceived minerality.

5.1. Limitations and qualifications

Despite the present study's relatively comprehensive nature in terms of the physico-chemical measures undertaken on the sauvignon wines, there are some limitations worthy of reporting. In retrospect, there were some perceived attributes (e.g., the sensory descriptor "salty") and some wine compositional aspects (e.g., the organic acid succinic acid) that were not investigated due to feasibility but that had potential to strengthen the present study. Succinic acid has been recently suggested as a potential source of wine perceived minerality (Baron & Fiala, 2012) although there is no clear evidence in support of this. Succinic acid is known to have an ambiguous taste involving sourness, bitterness and saltiness (Peynaud & Blouin, 2013; Ribereau-Gayon et al., 2006) but its sensory impact in wines is still unknown.

6. Conclusion

Our sensory and chemical data demonstrate both cultural similarities and cultural differences in terms of what drives perception of mineral character in sauvignon wine. In extending our earlier work, these data show the complexity of the concept of minerality in wine from both a chemical and sensory perspective, with most of our specific hypotheses partially supported only. Clearly, further research is required, including with wine varietals other than sauvignon blanc, to delineate the wine attributes and compositional factors associated significantly with perception of mineral character in white wine. Of particular interest for both researchers and practitioners is the finding that concentrations of both free and bound sulphur dioxide were positive predictors of perceived mineral character in the wines, suggesting that these compounds require more serious consideration, maybe as chemical markers, in future wine sensory investigations.

Supplementary data to this article can be found online at http://dx.doi.org/10.1016/j.foodres.2016.06.026.

Acknowledgement

The research was funded by New Zealand Winegrowers, the New Zealand Institute of Plant & Food Research, Pernod Ricard NZ and Pernod Ricard Centre de Recherche, Paris, France, the Regional Council of Burgundy, the Bureau Interprofessionnel des Vins de Bourgogne and Conseil Interprofessionnel des Vins de Bordeaux. We thank Richard Hider and Joy Jiao, and the wine professionals in France and New Zealand who participated in the study.

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