

**AGRICULTURAL RESEARCH FOUNDATION
FINAL REPORT
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Title - Investigating “reductive aromas” in Oregon Pinot noir wine; Comparison and Quantitation of aromatic compounds in non-reductive and reductive wines throughout the winemaking process.

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Hypothesis – Pinot noir wines with reductive off odors have different aromatic composition beyond those compounds associated with reduction.

Introduction - Maintaining wine quality is of utmost importance to wine regions to ensure prices and wine sales remain high. This wine from Oregon, most notably Pinot noir, has established itself as a premium product retailing at \$40 a bottle and above. To ensure that this wine remains a premium product, quality must be good every year. One issue that has proven to be problematic for the local wine industry is the occurrence of reductive aromas towards the end of the wine aging process. Reductive aromas are considered negative factors of wine quality around the world and the direct cause is not clearly understood.

Reductive aromas are described as stinky, rotten eggs and cabbage, and have traditionally been linked to aroma compounds containing sulfur, specifically H₂S, dimethyl sulfide, diethyl disulfide, dimethyl sulfide and mercaptan containing compounds. Traditionally many believe the occurrence of these reductive aromas is caused by an insufficient amount of nitrogen in the grapes and must prior to fermentation (Ugliano et al. 2009). However much of the research has proven to be inconsistent. Additionally, grapes in Oregon do not typically have nitrogen deficiencies, suggesting that a lack of nitrogen is not the cause of this quality issue if it is indeed linked to sulfide aromatic compounds.

Other aromatic compounds may play just as an important of a role if not greater to “reductive aromas” in wine. This project will identify other compounds that may be influencing the reductive aromas, providing a starting point for future research into the sensory impacts of these compounds on wine quality. For instance there is a possibility that esters, responsible for fruity aromas, may be reduced in reductive wines, which would provide insight on how biosynthesis may vary in reductive versus nonreductive wines.

Method – Two wineries from the Willamette Valley collected Pinot noir wine samples from wines that were reductive and wines that were not reductive.

Wine samples were stored in 40mL amber vials with PTFE lined melamine caps at -4°C until analyzed by head-space-solid-phase-microextraction-gas chromatogram-mass spectrometry (HS-SPME-GCMS). HS-SPME-GCMS based method used for analysis was taken from (Tomasino, Harrison, Breitmeyer, Sherlock, & Frost, 2012). However unlike in Tomasino et al. (2012), only qualitative information was obtained, not quantitative (Table 1).

Peak areas for each identified aromatic component was compared for the final wines, reductive wines (R) versus nonreductive wines (C, M, N and W). Discriminant analysis was run on the chemical data to determine how the wines differed (Fig 1). As can be seen the wines do differ with 99% of all variance due to the first factor (F1). 95% confidence intervals around wine centroids do not overlap, therefore all wines are statistically different from each other.

Interestingly, total S (the 4 chemical compounds associated with reduction; hydrogen sulfide, dimethyl disulfide, diethyl disulfide and ethyl mercaptan) were associated with N wines. These wines were not considered to have any reductive aromas by their respective winemakers. R wines were actually located close to the 0, 0 point which means that all aroma compounds were fairly balanced.

Pearson correlations were run to determine if any of the measured aroma compounds showed correlations with the total sulfur composition (Table 2). Significant correlations were found between total sulfur compounds and isoamyl acetate, gammanonalactone, B-ionone and ethyl heptanoate. B-ionone is a compound known to originate in grapes, gammanonalactone comes from oak and isoamyl acetate and ethyl heptanoate are fermentation derived aromas. None of these compounds contain sulfur in their molecular structure and as far as the authors know production of these compounds are in no way related to sulfur metabolic pathways in yeast fermentation.

Our conclusions show that there appears to be no relationship to reductive odors in wine and lower levels of non-sulfur related compounds. We additionally did not see high concentrations of sulfur related compounds in reductive smelling wines, although this may be due to the difficulty in producing accurate measurements of these compounds. We suggest that future work use labeled sulfur to be able to trace the metabolic pathway in reductive wines.

Table 1 – Percent area of total chromatogram peaks for each compound and wine. R – wines that smell reductive. C, M, N and W – are nonreductive wines.

wine code	isobutyraldehyde	2-methyl-1-butanal	hexanal	hexyl acetate	furfural	linalool	B-damascenone
C1	2.2	1.2	3.5	2.0	2.0	3.9	3.8
C2	2.9	1.2	2.7	1.4	1.6	4.2	2.0
C3	3.2	5.3	4.0	2.8	3.4	1.2	3.4
C4	3.0	4.7	3.1	2.1	2.1	1.4	2.8
M1	3.3	1.5	3.3	4.1	2.7	3.2	2.1
M2	2.2	0.9	4.4	7.5	2.3	3.4	3.8
M3	3.2	5.1	0.0	5.5	0.9	3.2	4.2
M4	4.5	6.6	5.5	3.2	4.3	2.4	2.7
M5	4.5	7.4	0.0	2.7	3.5	2.8	4.2
N1	1.7	0.7	2.3	2.6	1.8	3.7	1.5
N2	1.6	0.6	2.7	1.0	2.0	3.1	2.6
N3	3.9	3.6	2.6	2.7	2.5	2.9	4.5
N4	3.7	3.1	0.0	1.9	2.9	2.4	4.7
N5	3.1	3.9	4.8	3.3	2.2	1.9	4.7
R1	3.0	1.5	2.5	1.6	1.5	2.6	1.3
R2	2.4	0.9	3.1	4.0	4.0	2.9	2.1
R3	2.7	4.8	3.4	1.9	1.3	1.5	2.6
R4	2.9	3.5	0.0	2.4	4.9	3.5	5.6
R5	2.9	1.2	4.2	6.7	3.4	3.9	2.8
R6	3.4	1.4	4.1	7.3	4.1	4.7	2.8
R7	5.4	4.8	0.0	4.0	4.6	2.8	4.3
R8	1.6	0.6	1.8	1.7	3.5	3.9	1.4
R9	2.4	1.2	2.5	1.5	1.0	4.1	4.6
R10	2.6	4.8	5.0	3.8	6.4	1.8	3.2
R11	2.4	1.5	2.0	2.9	2.1	4.8	1.7
R12	3.4	5.8	0.0	2.6	6.5	2.2	3.3
R13	4.1	7.3	4.5	3.2	4.3	2.9	3.4
W1	2.0	0.9	2.3	3.2	2.1	4.7	2.5
W2	2.2	1.2	3.8	3.0	1.7	4.9	1.9
W3	3.5	1.5	3.7	3.5	2.5	3.6	2.0
W4	4.1	5.5	14.6	1.7	4.0	2.5	3.4
W5	5.8	5.9	3.5	2.2	7.8	3.1	4.2

Table 1- continued

wine code	geraniol	B-ionone	4-ethyl guiaucol	Gamma-nonalactone	eugenol	ethyl anthranilate	Ethyl Isobutyrate
C1	9.5	3.8	1.3	5.8	3.3	1.8	2.9
C2	2.3	2.8	1.7	3.8	2.2	2.7	3.3
C3	0.0	4.9	1.6	2.5	3.0	8.3	4.8
C4	0.6	3.7	1.1	2.6	1.8	3.2	3.2
M1	1.8	2.8	2.6	1.8	3.4	1.9	2.8
M2	3.6	3.2	2.1	2.3	3.0	1.2	2.3
M3	2.2	5.7	0.4	1.7	1.2	1.0	5.0
M4	2.4	2.1	1.5	2.0	1.5	2.2	2.5
M5	0.0	4.1	2.5	2.3	2.2	1.4	3.3
N1	4.5	2.1	1.7	5.2	2.6	1.1	1.7
N2	5.8	2.2	4.7	4.5	2.7	1.0	2.4
N3	0.0	3.9	2.9	3.0	2.9	3.7	5.1
N4	0.0	2.3	5.3	3.0	1.4	1.2	3.0
N5	0.0	4.9	1.1	3.1	3.1	2.3	2.7
R1	3.8	2.4	0.9	3.8	3.4	8.0	3.3
R2	7.2	3.4	1.4	4.4	2.4	1.5	2.3
R3	1.5	4.1	0.7	1.9	1.2	3.2	4.4
R4	1.4	3.0	0.7	5.8	1.9	0.7	3.8
R5	6.8	2.4	2.3	2.5	4.2	1.9	2.5
R6	3.3	3.4	1.5	2.6	3.0	1.8	3.0
R7	1.2	3.0	1.5	1.8	2.4	1.1	4.2
R8	2.6	1.9	1.3	3.5	8.4	1.5	2.5
R9	5.1	3.4	1.1	5.5	3.4	1.8	1.7
R10	1.1	2.7	1.5	2.3	2.9	1.7	1.4
R11	8.6	2.0	1.5	3.2	4.8	1.4	1.5
R12	0.9	2.4	17.5	2.2	2.9	9.7	4.1
R13	1.0	1.8	1.2	2.3	1.6	1.8	2.6
W1	13.6	2.6	1.9	4.2	4.0	2.0	2.0
W2	4.2	1.8	1.2	4.3	6.1	0.8	1.8
W3	3.5	3.3	10.9	2.3	4.6	12.3	2.9
W4	0.8	3.5	1.8	2.0	3.6	2.8	3.5
W5	0.6	4.1	20.9	1.7	5.1	13.0	7.3

Table 1 – continued

wine code	Ethyl butanoate	ethyl isovalerate	isoamyl acetate	3-methyl-1-butanol	Ethyl hexanoate	ethyl heptanoate
C1	2.5	2.8	2.9	3.2	2.9	4.5
C2	2.8	3.2	2.6	2.7	2.8	3.2
C3	3.3	4.7	3.2	4.4	2.9	2.8
C4	4.4	3.8	5.2	3.9	4.1	2.8
M1	2.5	2.7	3.2	2.4	2.6	2.0
M2	2.6	2.9	3.7	2.3	2.8	2.0
M3	3.3	2.9	3.0	3.0	3.1	1.8
M4	2.7	2.7	5.1	3.1	2.7	2.4
M5	3.3	4.5	4.1	3.0	3.0	2.7
N1	3.0	1.9	2.3	2.4	2.6	3.3
N2	2.5	2.9	2.2	3.8	4.5	2.2
N3	4.2	4.8	3.2	3.4	3.5	3.4
N4	3.0	3.2	2.9	3.2	2.6	2.4
N5	5.2	3.3	3.1	3.8	4.1	2.9
R1	2.2	2.3	2.2	2.4	2.4	4.0
R2	1.8	2.3	2.4	2.7	2.6	2.4
R3	3.4	4.2	4.3	4.9	3.1	3.0
R4	4.2	4.0	2.9	4.2	4.3	5.5
R5	2.5	3.1	3.7	2.5	2.8	2.6
R6	2.6	3.8	3.1	2.1	2.7	2.7
R7	4.4	4.1	3.8	3.5	3.1	2.6
R8	2.4	2.5	2.8	2.6	2.9	4.2
R9	3.6	2.3	2.1	3.1	3.2	3.9
R10	4.2	2.3	4.1	3.4	3.3	2.4
R11	2.7	1.1	2.2	2.2	2.5	4.2
R12	2.1	2.5	2.9	2.3	2.5	2.1
R13	3.9	2.9	3.0	3.5	4.3	3.0
W1	2.5	2.2	2.1	2.7	2.3	4.4
W2	2.4	2.0	2.6	2.8	3.4	6.2
W3	2.7	3.4	2.5	2.7	3.0	2.9
W4	3.2	4.2	3.1	3.5	3.5	2.7
W5	4.1	4.7	3.4	4.5	4.0	2.7

Table 1 – continued

wine code	Hexanol	trans-3-Hexen-1-ol	Ethyl octanoate	benzaldehyde	ethyl decanoate	phenethyl alcohol
C1	3.6	4.5	2.8	1.9	1.5	1.8
C2	2.6	3.0	2.3	1.4	1.8	1.7
C3	3.0	3.5	3.0	1.7	3.4	3.3
C4	2.7	2.9	4.3	1.0	7.9	8.0
M1	2.9	3.0	2.6	1.9	2.4	1.6
M2	2.8	3.9	3.0	1.9	2.1	1.4
M3	3.5	4.4	2.9	6.2	4.2	4.0
M4	3.0	2.9	3.1	7.9	3.5	3.7
M5	2.9	2.6	2.5	3.0	3.6	3.5
N1	3.3	3.0	2.9	2.1	1.2	1.4
N2	2.7	3.0	2.7	2.3	1.7	1.7
N3	3.4	2.4	3.5	2.2	3.1	2.8
N4	2.1	2.2	2.3	6.5	1.7	1.6
N5	3.2	2.7	3.6	3.0	4.8	4.9
R1	2.6	2.4	2.9	2.2	1.9	2.6
R2	3.2	3.1	3.1	2.0	1.7	1.6
R3	3.2	3.4	3.2	1.7	3.5	3.4
R4	4.0	4.5	3.1	1.2	2.7	2.7
R5	2.8	3.0	3.4	3.4	2.1	1.8
R6	2.5	2.6	3.1	1.5	2.1	1.5
R7	3.2	3.9	2.8	1.9	2.9	2.9
R8	3.3	3.1	2.9	3.1	1.8	1.9
R9	3.8	2.9	3.1	2.4	2.0	1.7
R10	2.4	2.3	3.4	4.4	5.1	5.1
R11	3.4	3.4	3.1	2.7	1.7	1.7
R12	2.2	2.0	3.0	13.2	4.0	4.1
R13	5.1	3.7	4.3	2.5	6.9	7.7
W1	3.2	3.4	2.9	2.7	1.8	2.0
W2	4.6	3.9	2.5	3.7	1.4	1.4
W3	2.6	2.8	3.8	2.6	1.9	3.3
W4	3.8	3.1	3.3	3.7	7.6	8.0
W5	2.2	2.2	4.9	2.2	5.7	5.2

Table 1 – continued

wine code	ethyl pentanoate	butyric acid	isovaleric acid	hexanoic acid	octanoic acid	acetic acid	total S
C1	3.0	3.3	3.3	3.6	3.1	3.5	13.6
C2	2.9	2.6	2.4	3.5	3.5	2.9	10.1
C3	2.7	3.0	4.6	2.6	3.0	3.5	9.5
C4	4.0	5.3	4.1	4.0	3.7	3.6	9.6
M1	1.3	2.2	2.6	3.3	3.5	3.0	10.8
M2	1.9	2.6	2.4	3.5	3.5	2.9	10.2
M3	2.7	3.9	2.9	2.7	2.7	3.5	9.4
M4	2.5	3.1	4.9	2.8	3.2	4.6	8.7
M5	3.9	3.3	4.5	2.6	2.3	4.0	11.5
N1	3.5	3.0	0.2	0.3	0.3	0.3	14.0
N2	2.0	2.3	2.7	3.5	3.0	2.7	19.5
N3	4.8	3.7	3.9	3.2	3.5	3.7	13.3
N4	2.5	3.4	3.1	2.8	2.4	2.7	12.8
N5	4.9	5.0	2.8	3.6	3.4	3.2	11.3
R1	3.1	1.5	2.9	3.0	3.3	3.2	10.1
R2	1.6	2.3	2.9	3.2	3.3	3.4	12.2
R3	3.4	4.0	4.2	3.7	3.7	3.4	10.8
R4	6.2	3.2	2.3	3.1	2.5	2.2	17.7
R5	2.2	2.2	3.2	3.4	3.7	3.3	13.3
R6	3.1	2.3	3.2	3.1	3.2	3.1	11.2
R7	4.5	3.4	3.3	2.6	2.8	2.9	10.0
R8	3.3	2.6	2.2	2.9	2.9	3.1	14.0
R9	2.3	3.6	2.3	3.9	3.4	2.9	11.1
R10	2.7	3.9	2.9	2.7	2.7	3.5	12.2
R11	4.1	2.7	1.3	2.6	2.2	3.3	15.0
R12	2.3	2.6	4.1	3.0	3.9	3.4	16.8
R13	3.0	2.7	2.1	2.7	2.4	1.8	14.2
W1	2.5	2.4	2.9	2.8	2.7	3.4	14.5
W2	4.6	3.0	2.4	4.2	2.2	3.3	15.2
W3	2.7	2.0	2.8	3.3	4.0	3.2	13.1
W4	2.6	3.8	5.9	3.4	3.3	2.8	11.6
W5	3.2	5.0	4.5	4.5	6.5	3.7	12.7

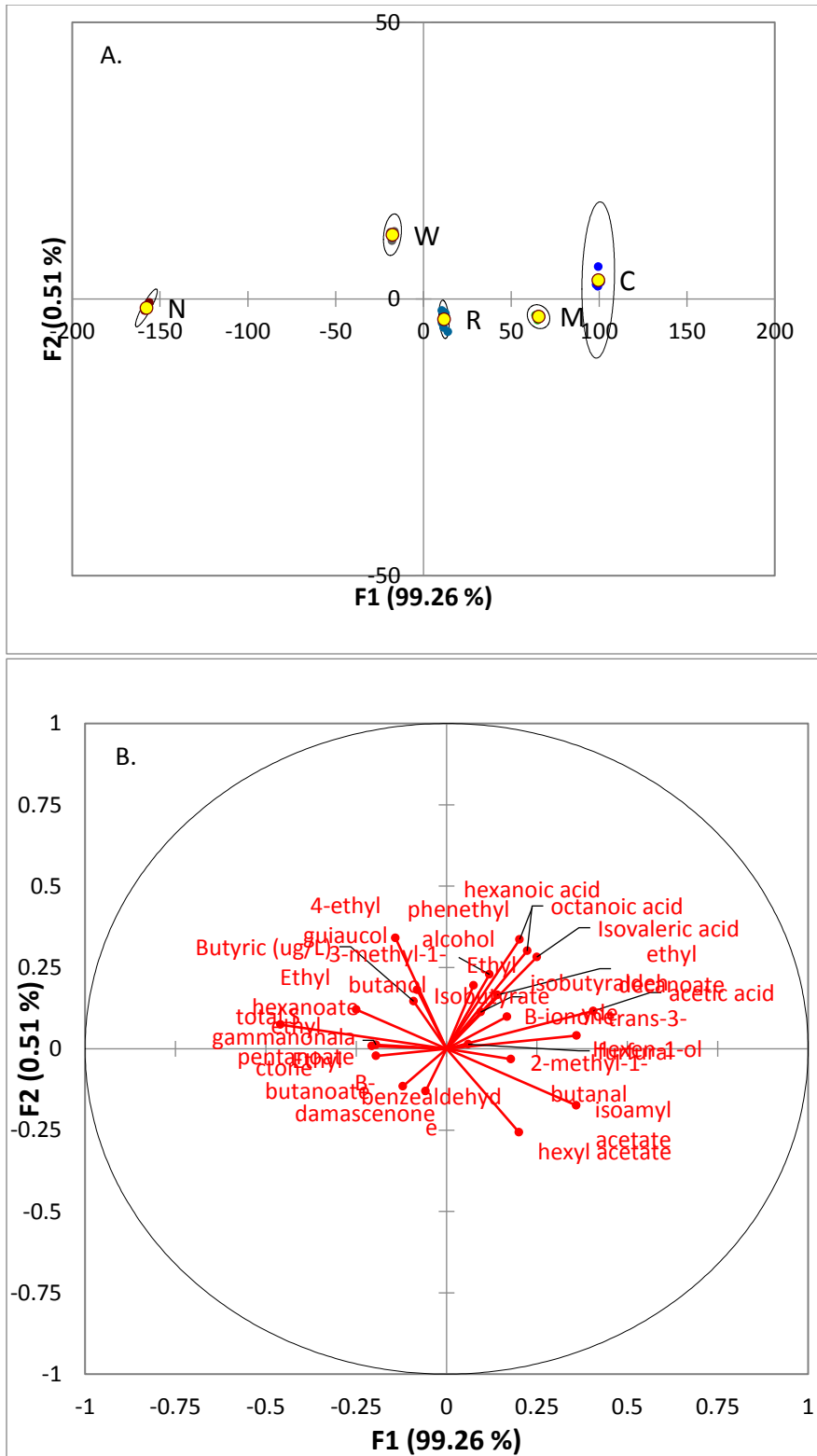


Figure 1 – Pinot noir wine means and 95% confidence intervals (A) with chemical vector loadings for F1 and F2 that are important for wine differentiation (B). R - wines with reduction aromas, C, W, N and M - wines that have no reduction off aromas.

Table 2- Pearson correlations with total sulfur and aroma composition.

Variables	total S
isobutyraldehyde	-0.36
2-methyl-1-butanal	-0.25
hexanal	-0.20
hexyl acetate	-0.26
furfural	0.17
linalool	0.34
B-damascenone	-0.01
geraniol	0.31
B-ionone	-0.47
4-ethyl guiaucol	0.28
gammanonalactone	0.48
eugenol	0.28
ethyl anthranilate	-0.05
Ethyl Isobutyrate	-0.19
Ethyl butanoate	-0.19
ethyl isovalerate	-0.26
isoamyl acetate	-0.50
3-methyl-1-butanol	-0.03
Ethyl hexanoate	0.29
ethyl heptanoate	0.38
Hexanol	0.19
trans-3-Hexen-1-ol	0.06
Ethyl octanoate	-0.04
benzaldehyde	0.14
ethyl decanoate	-0.24
phenethyl alcohol	-0.19
ethyl pentanoate	0.17
butyric acid	-0.25
isovaleric acid	-0.34
hexanoic acid	-0.04
octanoic acid	-0.21
acetic acid	-0.34