# Development of an analytical sampling technique to study the aroma profile of Pinot noir wine

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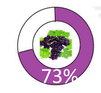
## 1 BACKGROUND

Pinot noir is the most-common red wine variety produced in New Zealand[1].




- Aroma is one of the most important factors in determining wine character and quality.
- Typical Pinot noir wine exhibits a complex mixture of odours including plum, cherry, raspberry and mushroom.




*It is quite challenging to differentiate and quantify aroma compounds due to the complexity of the matrix and low concentrations of analytes.*

## 2 AIM

To develop and validate a comprehensive volatile sampling methodology, which can profile the wide variety of aroma compounds present in Pinot noir wine.

- Esters
- Aldehydes
- Norisoprenoids
- Phenols
- Alcohols
- Terpenes
- Fatty acids

## 3 APPROACH

A Dispersive Liquid-Liquid Microextraction (DLLME) sampling method was developed to analyse aroma compounds in Pinot noir wine.

- Low-cost
- Simple method
- Environmentally friendly

## 4 EXPERIMENTAL DESIGN

D-optimal experimental design was used to investigate the optimal parameters for analysis[2].

Four independent variables were explored.

| X1 | X2 | X3 | X4 |
|---|---|---|---|
| Chloroform | 500 | Acetone | 500 |
| Dichloromethane | 750 | Acetonitrile | 1000 |
| Hexane | 1000 | Methanol | 1500 |
| Pentane | 1500 | | |
| Chloroform:Pentane (2:1) | 2000 | | |

*X1 - Extraction solvent type*

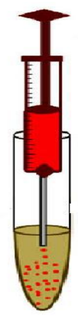
*X2 - Extraction solvent volume (µL)*

*X3 - Disperser solvent type*

*X4 - Disperser solvent volume (µL)*

## 5 METHOD

ii) Rapidly inject extraction solvent + disperser solvent mixture



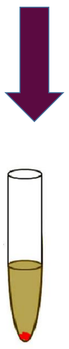
iii) Gently shake for 5 min



iv) Centrifuge for 5 min at 5000 rpm



v) Sedimented phase

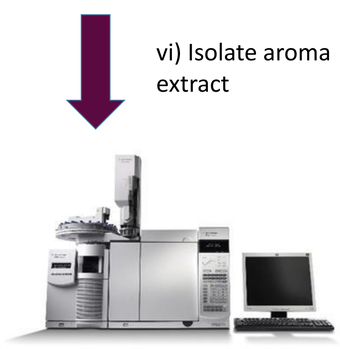


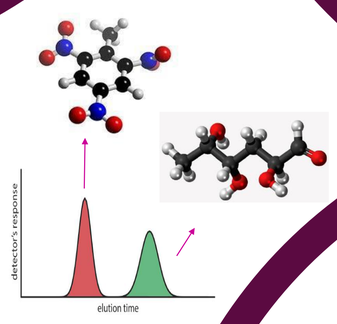
i) Wine sample with internal standards



## 6

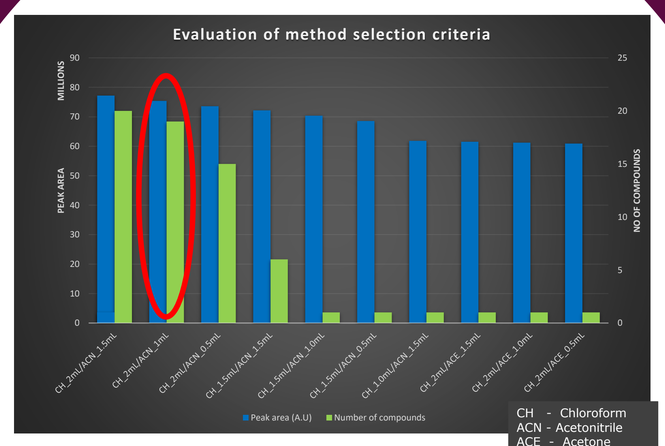
vi) Isolate aroma extract






vii) Analyse aroma compounds using GCMS

## 7 RESULTS




***Selected conditions***

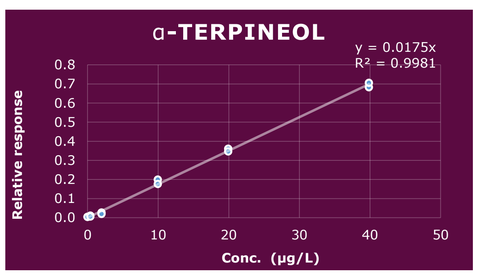
*Extraction solvent: chloroform (2 mL)*

*Disperser solvent: acetonitrile (1 mL)*

## 8 METHOD VALIDATION

Pinot noir wine was spiked with 33 target compounds at low, medium and high concentrations.

- All samples were prepared in triplicate.
- Repeatability ranged from 4.3 to 10.6% (RSD%).
- Recoveries ranged from 80-95.6% .
- All calibration curves had an $R^2 > 0.992$.
- LOD and LOQ for this method were calculated.




## 9 METHOD APPLICATION

- 33 volatile compounds were analysed.
  - *in 30 PN wines*
  - *from 3 different regions* – Marlborough, Central Otago, Martinborough
- Some of the results:

| Compound | Olfactory description | Olfactory perception threshold (µg/L)[3] | Concentration of aroma compound (µg/L) |
|---|---|---|---|
| **β-damascenone** | Apple, honey, floral | 0.05 | 1.7 – 11.5 |
| **β-ionone** | Violets, raspberry | 0.09 | nd – 1.68 |
| **α-ionone** | Woody, floral, violet | 2.6 | 0.8 – 1.3 |
| **Geraniol** | Rose, geranium | 20 | 0.7 – 12.8 |
| **Nerol** | Rose | 400 | 0.8 – 20 |
| **Linalool** | Floral, rose, lavender | 25 | 1.3 – 35.7 |
| **α-terpineol** | Floral, lily of the valley | 250 | 0.8 – 8.4 |

nd – not detected

β-damascenone and β-ionone are known to play significant roles in the aroma profile in wines.

## 10 CONCLUSION

**A novel DLLME approach combined with GC-MS was developed for the determination of volatile compounds in Pinot noir wines.**

**A D-optimal design, varying four different factors, was used to develop an experimental plan. 225 different extraction conditions were analysed. The combination of extraction solvent: chloroform 2.0 mL, and disperser solvent: acetonitrile 1.0 mL was selected as the best conditions.**

**Linearity, precision and detection limits of the developed DLLME method confirmed its suitability for the determination of volatile compounds.**

## REFERENCES

1) Winegrowers, N.Z., New Zealand Winegrowers Annual Report 2020.

2) Fedrizzi, B., Carlin, S., Franceschi, P., Vrhovsek, U., Wehrens, R., Viola, R., & Mattivi, F. (2012). D-optimal design of an untargeted HS-SPME-GC-TOF metabolite profiling method. Analyst, 137(16), 3725-3731.

3) Escudero, A., E. Campo, L. Farina, J. Cacho and V. Ferreira, Analytical characterization of the aroma of five premium red wines. Insights into the role of odour families and the concept of fruitiness of wines. J. Agric. Food Chem.,