

Analysis of Smoke Derived Volatile Phenols in Grapes and Wine

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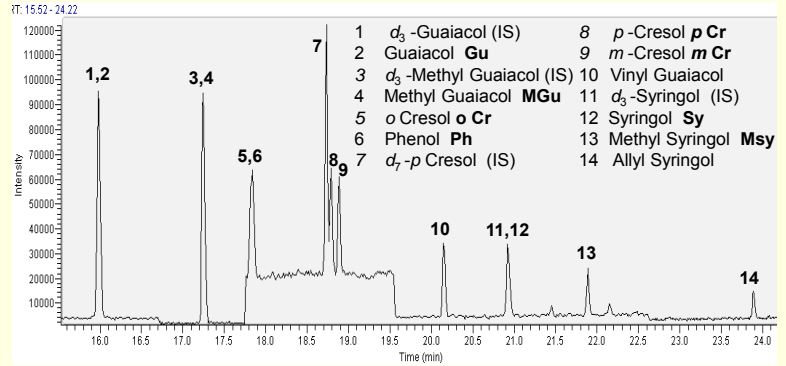
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The Aim of this Study

To develop a method for the quantification of the smoke-derived volatile phenols in grapes and wine and apply it to investigations of susceptibility of their glycosidic forms to release free volatiles as a result of acid hydrolysis.

Method Development

GC-MS SIM analysis of a 50 µg/L volatile phenol standard mix



- Calibration functions for each volatile phenol of interest were made over a concentration range of 0 to 200 µg/L (0 to 1000 µg/L for syringol and methyl syringol).
- Linearity of all compounds was >0.99 over the range of concentrations.
- A method for the analysis of free volatile phenols for grapes and wine was successfully developed and validated.

Sample preparation

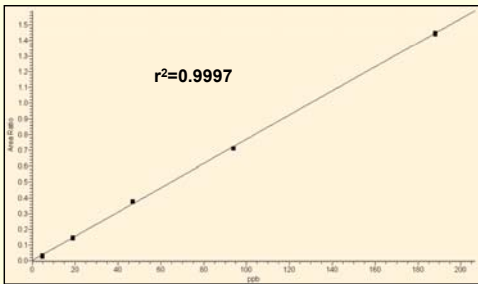
5 mL of juice, wine or hydrolysate

Added to a 10 mL screw capped vial with 100 µg of internal standard (*d*₃ guaiacol, *d*₃ methyl guaiacol, *d*₇ *p* cresol and *d*₃ syringol)

Mixed then extracted with 2 mL of 1:1 pentane: ethyl acetate.

The solvent extract is analysed by GCMS in SIM mode.

Example calibration function (*m* cresol)



Acid Hydrolysis of Smoke-Affected Grapes and Wine

Experiment protocol

- Bushfire smoke-affected Chardonnay and Cabernet Sauvignon grapes and their resulting wines were used.
- A 10 mL aliquot of wine or supernatant obtained from the grape homogenate was acidified to pH 1.0 with concentrated sulfuric acid and heated at 100 °C for 1 hr.
- Samples were subjected to GC-MS for the analysis of volatile phenols and HPLC-MS/MS for the analysis of their glycosides.
- The analyses were carried out before and after hydrolysis.

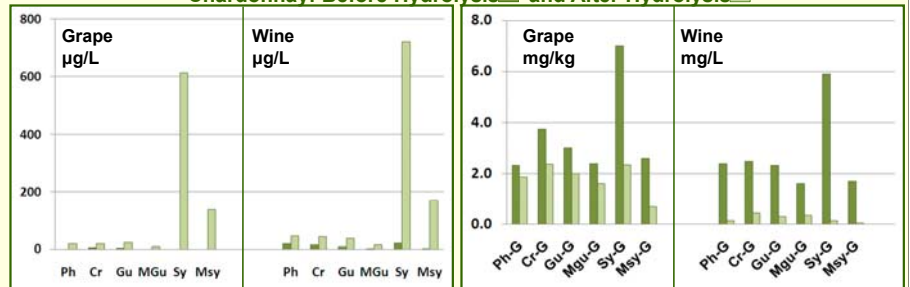
Results

- Free volatile phenols were found in both smoke-affected grapes and wine in elevated concentrations after hydrolysis.
- The concentrations of the glycosides in grape and wine decreased after hydrolysis.
- The loss of glycosides and gain in free volatile phenols was greater in the wine than the grapes after hydrolysis.
- The release of free syringol and methyl syringol and the decrease of their glycosides was greater in both grapes and wine, than the other phenols.

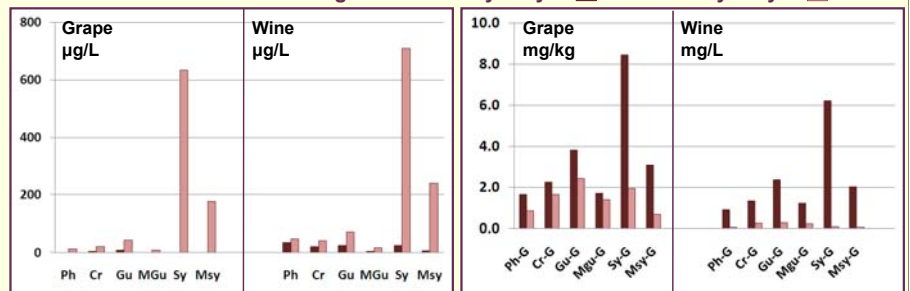
Free volatile phenols

Glycosides of volatile phenols

Chardonnay: Before Hydrolysis and After Hydrolysis



Cabernet Sauvignon: Before Hydrolysis and After Hydrolysis



Conclusions

- Significant amounts of volatile phenols were released after hydrolysis while concomitant reductions in their glycosides were noticed indicating the existence of the precursors (glycosides) resulting from grape vine exposure to smoke.
- Control (no smoke exposure) grape and wine released only negligible free volatiles following hydrolysis which is probably due to the trace amounts of glycosides present as naturally occurring components of grape vines. (data not shown).
- A study of non-smoke affected grapes and wines of different varieties from across Australia is needed and currently underway to determine the expected baseline values of these compounds.
- Bound and acid-sensitive forms of free volatile phenols represent a substantial pool of smoky, medicinal compounds in smoke affected juice and wine.