

Introduction

Climate change has increased the incidence of forest and bushfires in the west of the United States. When the grapevine is exposed to wildfire smoke, the grapes and grapevine can absorb the smoke compounds and further convert them into the corresponding glycosides or other bound forms. The amounts of smoke compounds absorbed by the grapes depend on smoke intensity, smoke duration, grape variety, and many other factors. Guaiacol, 4-methylguaiacol, *o*-, *m*-, and *p*-cresols have been suggested as indicators for smoke exposure. These compounds (free and total) were investigated in over 400 wine samples during the 2020 wild fire season in Oregon using a fast (30min) SPME-GC-MS method.

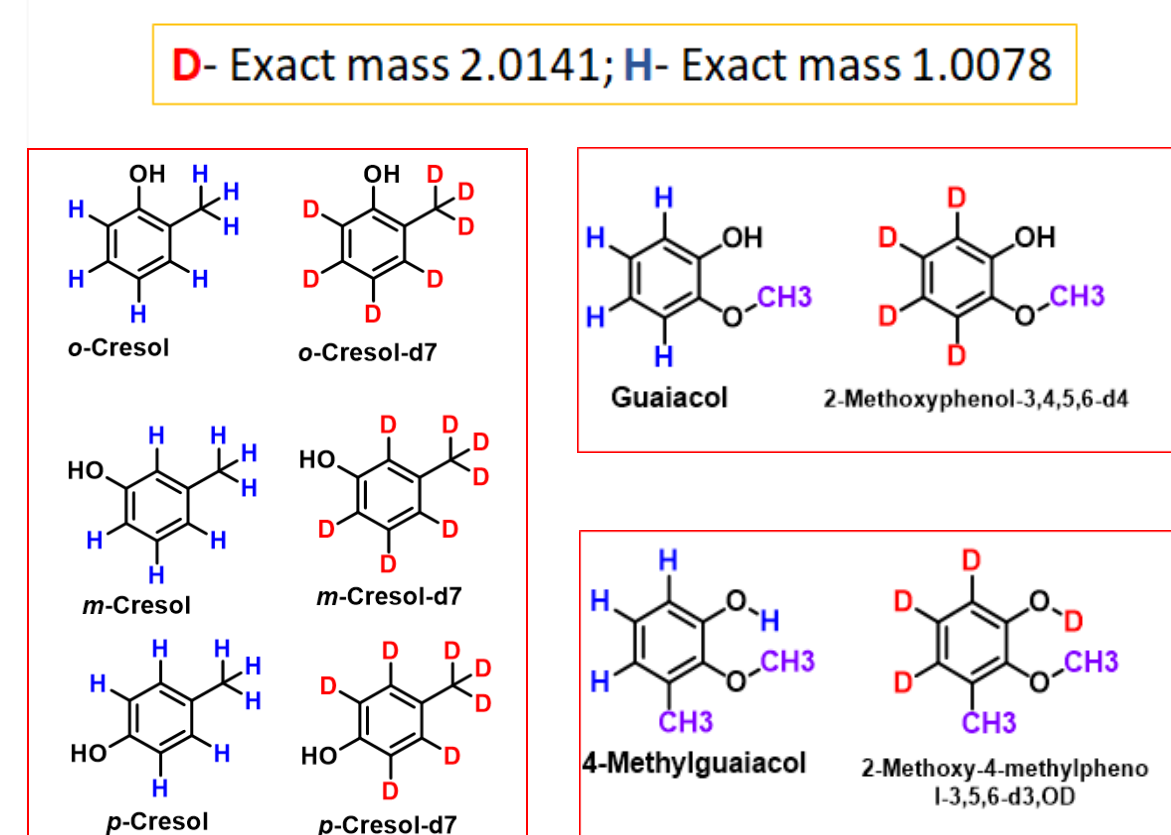
Materials

Samples:

Over 370 smoke-exposed red wines and 90 smoke-exposed white wines (including rose wine) were analyzed during the 2020 wildfire season.

Internal standard (IS):

Stable isotope-labeled compounds were used as IS to quantitate volatile phenol compounds.



Methods

Extraction for free-form volatile phenols:

-10 mL of wine with a magnetic stir bar was placed to a 20 mL headspace glass vial, and 10 µL of IS was added.

-The free-form VPs in wines were extracted using 50/30 µm three phase fiber at 50 °C for 25 min after 2 min equilibration with 250 rpm stirring

-Samples were desorbed at 250 °C for 7 min

Extraction for total volatile phenols:

-2 mL of wine was added into a 20 mL headspace glass vial, and then 20 µL of 10 M HCl was added to get the pH value 1.2.

-The vials were kept in a 100 °C water bath for 4 h.

-After the vials cool down, 8 mL citric acid buffer with saturated salt (0.2 M, pH 3.5) and 10 µL of IS were added in.

-Samples were extracted with SPME at 50 °C for 25 min

GC-MS analysis:

For free-form VPs: Agilent 6890 GC-5973 MSD system and SUPELCOWAX-10 column (59m × 0.25mm × 0.25µm) was adopted to separate VPs with the flow rate of He 2 mL/min.

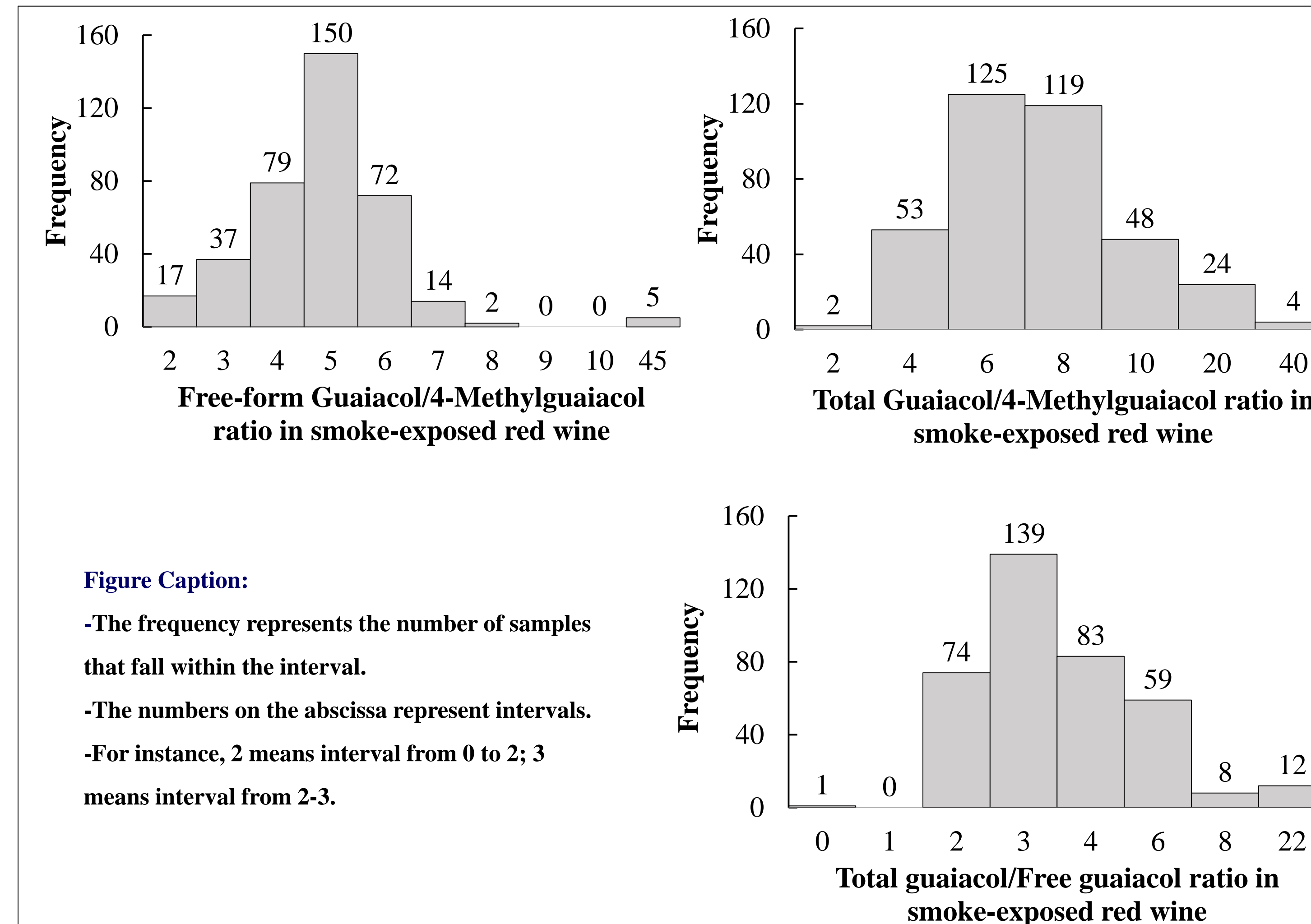
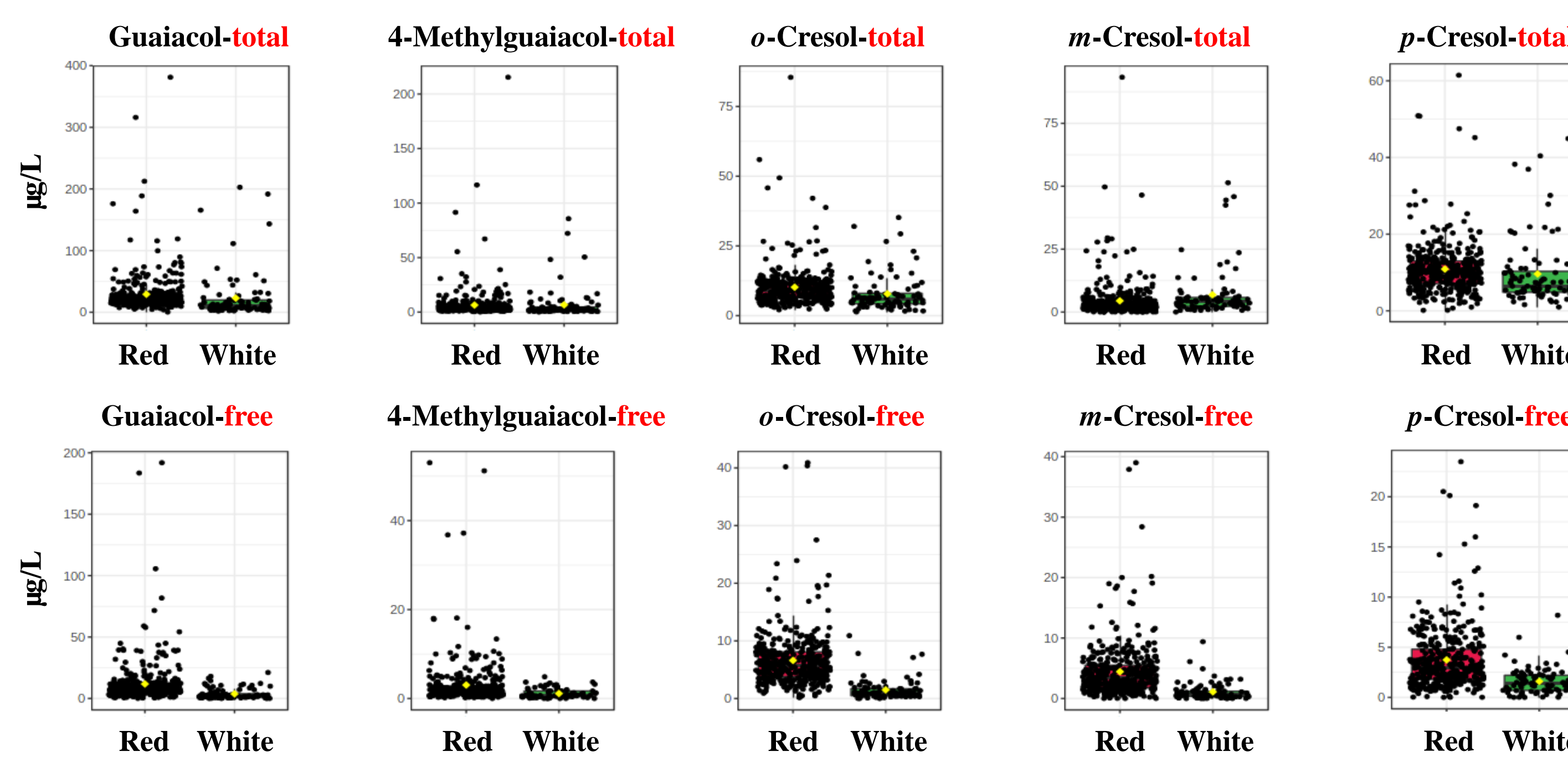
For total VPs: Agilent 7890-5975MSD (Optima FFAP plus, 60m × 0.25 mm × 0.5µm) was used for VPs analysis with the flow of He 1.5 mL/min.

The oven program: 60 °C hold for 3 min, then ramped to 160 °C at a rate of 20 °C/min, then to 200 °C at 5 °C/min and held for 10 min, finally raised to 220°C at 20 °C/min and held for 5 min.

Quantitative ions for SIM:

(1)109, 113, 124, 128 (2)123, 126, 138,141 (3)107,108, 113, 115

Results



Summary

1. Out of the 370 smoke-exposed red wine, 84 samples had free guaiacol 0-5 µg/L, 159 samples had free guaiacol 5-10 µg/L. 78% of the samples had 4-methylguaiacol < 3 µg/L.
2. In red wine, the highest frequency of guaiacol/4-methylguaiacol ratio in free-form and total was 4-6
3. The average ratio of free-form guaiacol/4-methylguaiacol was 4.5:1; total guaiacol/4-methylguaiacol was 6.6:1
4. Except for *m*-cresol, levels of other total VPs appeared approximately two-five times higher than the corresponding free VPs.
5. The highest frequency of total guaiacol/free-form guaiacol ratio was 2-4, and the average ratio was 3.3:1.

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