APPLICATIONS

Scott Krepich
Scott enjoys surfing and eating. He is crazy about chromatography, because his mom is really into CSI and thinks that is what he does.

Analysis of Sensory-Active Volatile Phenols in Smoke-Exposed Grapes by Gas Chromatography

Matthew Noestheden[1,2], Katelyn Thiessen[1], Wesley Zandberg[1] ,Scott Krepich[3] and Allen Misa[3]
[1]University of British Columbia Okanagan, Chemistry Deparment, 1177 Research Rd, Kelowna, BC Canada V1V 1V7
[2]Supra Research & Development, 4532 Sallows Rd, Kelowna, BC Canada V1W 4C2
[3]Phenomenex, Inc., 411 Madrid Ave., Torrance, CA 90501 USA

Background

Volatile phenolic compounds (VPs; **Figure 1**) are expressed endogenously in wine grapes and have been shown to increase following on-vine smoke exposure due to forest fire. Depending on their relative concentrations (among other factors) VPs can possess negative organoleptic properties, with sensory descriptors such as 'ashtray' and 'Band-aid' often associated with wines made using smoke-exposed grapes. The evaluation of smoke exposure is a significant quality control issue for vineyards, but also for wineries, as the concentration of VPs may increase during fermentation or aging. With climate models predicting an increase in the frequency of forest/brush fires and the localization of many key wine producing regions near or in areas prone to such fires (e.g., California, British Columbia, Australia, South Africa), it is critical to expand our current understanding of this economically important phenomenon.

Introduction

The compounds outlined in **Figure 1** demonstrate that the VPs associated with smoke-exposure are structurally similar, with several existing as positional isomers (e.g., cresols). This structural similarity can make their chromatographic resolution challenging. The VPs discussed herein are suitably volatile, such that they are amenable to GC separation. It was hypothesized that the use of hydroxyl groups around a polar WAX-type stationary phase would provide an advantageous refinement in positional isomer resolution when compared to more traditional bonded silica GC phases.

Experimental Conditions

Column:	Zebron™ ZB-WAX
Dimensions:	30 m x 0.32 mm x 0.50 µm
Part No.:	7HM-G007-17
Injection:	Splitless @ 220, 5 µL, 1 min
Carrier Gas	Helium @ 1.5 mL/min (constant flow)
Oven Program:	10 °C/min for 9.5 min to 170 °C, 2.5 °C/min for 4 min to 180 °C, 75 °C/min for 1 min to 250 °C, 1 min hold @ 250 °C, Total gradient run time = 14min
Detector:	Triple Quadrupole
Transfer Line:	230 °C
Ion Source:	250 °C
Ionization Sample:	**EI**

1. Guaiacol
2. 4-Methlyguaiacol
3. o-Cresol
4. 4-Ethylguaiacol
5. p-Cresol
6. m-Cresol
7. Eugenol
8. 4-Ethylphenol
9. Syringol

Figure 1.
Volatile phenolic compounds (VPs) that may be present in smoke-exposed wine grapes and in wine made using smoke-exposed grapes. Guaiacol has an aesthetic objective of 20 – 80 µg/L in wine, and may be present at over 500 µg/L. The relatively low aesthetic objectives for VPs makes their quantitation vital for smoke-exposed wine grapes.

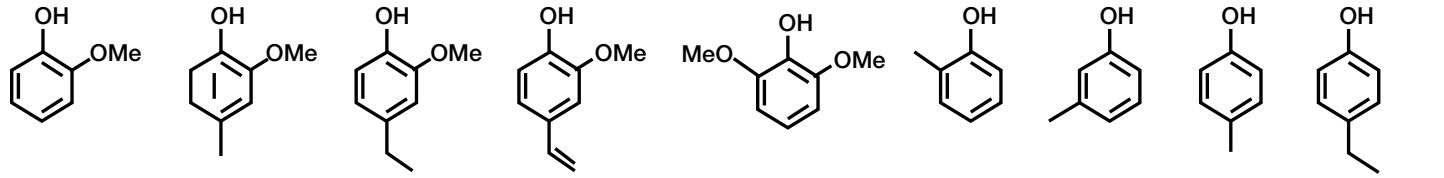
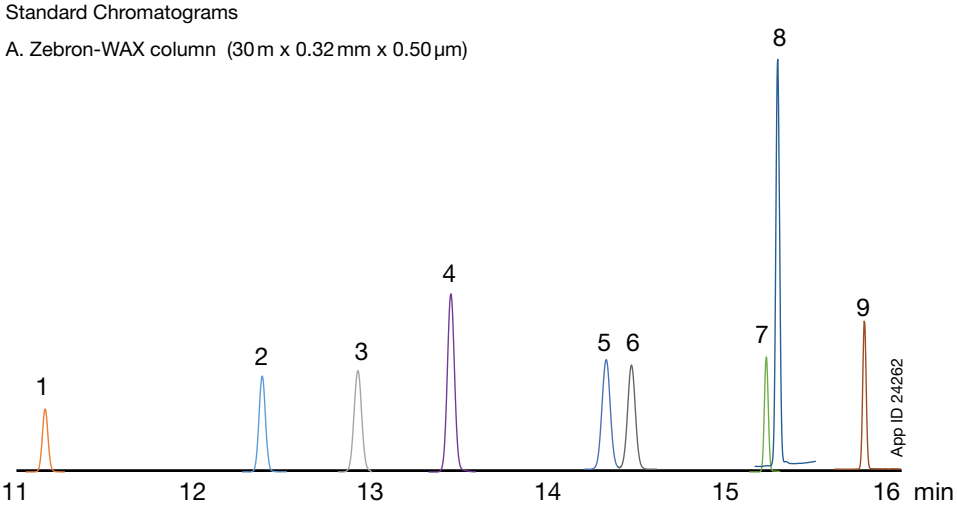


Figure 2.
Standard Chromatograms

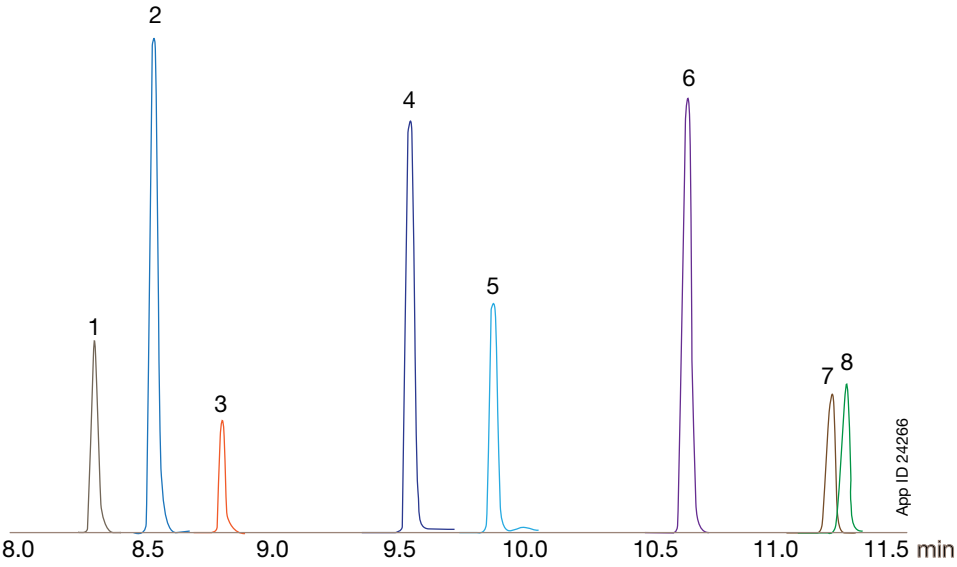
A. Zebron-WAX column (30 m x 0.32 mm x 0.50 µm)



1. Guaiacol
2. 4-Methylguaiacol
3. o-Cresol
4. 4-Ethylguaiacol
5. p-Cresol
6. m-Cresol
7. Eugenol
8. 4-Ethylphenol
9. Syringol

Broad selectivity differences and the co-elution of m/p-cresol on the 5 %-phenyl column highlight the advantages of the Zebron-WAX column for the analysis of VPs in smoke-exposed wine grapes.

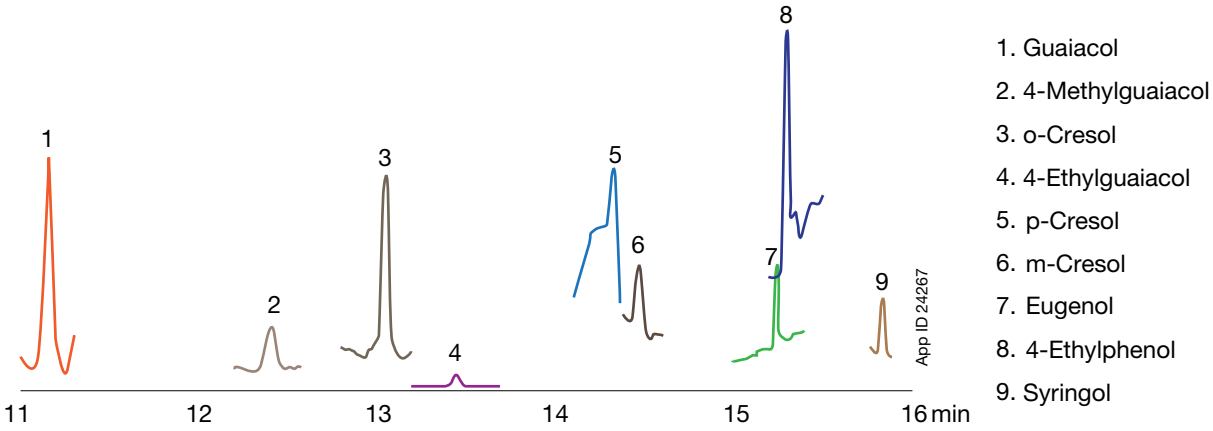
B. 5 %-phenyl-bonded phase GC column (60 m x 0.25 mm x 0.25 µm)



1. o-Cresol
2. m/p-Cresol
3. Guaiacol
4. 4-Ethylphenol
5. 4-Methylguaiacol
6. 4-Ethylguaiacol
7. Syringol
8. Eugenol

Comparative separations may not be representative of all applications.

Figure 3.
Sample chromatogram from smoke-exposed Pinot Noir grape extract, analyzed on the Zebron-WAX column.



Results & Discussion

The polyethylene glycol Zebron-WAX GC phase resolved all critical pairs of VPs, including the very challenging cresol positional isomers (**Figure 2**, **A.**). Chromatographic resolution is required for accurate quantitation of all three cresol isobars, as their structural similarity results in identical product ions when analysed by GC-MS/MS. Similar chromatographic resolution of the cresols was not feasible in a reasonable time scale using a 5 %-phenyl GC phase, despite using a 60 m column (**Figure 2**, **B.**). In fact, resolution of m/p-cresol was never observed on the 5 %-phenyl column, despite running extended gradient and isothermal methods (data not shown). The obtained resolution of m/p-cresol on the Zebron-WAX column will facilitate the accurate characterization of VPs in smoke-exposed grapes (**Figure 3**). In turn, this will aid the development of more accurate models for predicting wine quality issues when using smoke-exposed grapes and may also inform remedial and preventative strategies.

Acknowledgement

We would like to provide special thanks to Supra Research & Development contributing this application.

APPLICATIONS

Ordering Information

Zebron™ ZB-WAX GC Columns

ID(mm)	df(µm)	Temp. Limits °C	Part No.
10-Meter			
0.10	0.10	40 to 250/260	7CB-G007-02
15-Meter			
0.25	0.25	40 to 250/260	7EG-G007-11
0.32	0.25	40 to 250/260	7EM-G007-11
0.32	0.50	40 to 250/260	7EM-G007-17
0.53	1.00	40 to 250/260	7EK-G007-22
20-Meter			
0.18	0.18	40 to 250/260	7FD-G007-08
30-Meter			
0.25	0.15	40 to 250/260	7HG-G007-05
0.25	0.25	40 to 250/260	7HG-G007-11
0.25	0.50	40 to 250/260	7HG-G007-17
0.25	1.00	40 to 250/260	7HG-G007-22
0.32	0.15	40 to 250/260	7HM-G007-05
0.32	0.25	40 to 250/260	7HM-G007-11
0.32	0.50	40 to 250/260	7HM-G007-17
0.53	0.50	40 to 250/260	7HK-G007-17
0.53	1.00	40 to 250/260	7HK-G007-22
60-Meter			
0.25	0.15	40 to 250/260	7KG-G007-05
0.25	0.25	40 to 250/260	7KG-G007-11
0.25	0.50	40 to 250/260	7KG-G007-17
0.32	0.25	40 to 250/260	7KM-G007-11
0.32	0.50	40 to 250/260	7KM-G007-17
0.53	1.00	40 to 250/260	7KK-G007-22

Note: If you need a 5 in. cage, simply add a (-B) after the part number, e.g., 7HG-G007-11-B. Some exceptions may apply. Agilent 6850 and some SRI and process GC systems use only 5 in. cages.

Australia
t: +61 (0)2-9428-6444
f: +61 (0)2-9428-6445
auinfo@phenomenex.com

Austria
t: +43 (0)1-319-1301
f: +43 (0)1-319-1300
anfrage@phenomenex.com

Belgium
t: +32 (0)2 503 4015 (French)
t: +32 (0)2 511 8666 (Dutch)
f: +31 (0)30-2383749
beinfo@phenomenex.com

Canada
t: +1 (800) 543-3681
f: +1 (310) 328-7768
info@phenomenex.com

China
t: +86 400-606-8099
f: +86 (0)22 2532-1033
phen@agela.com

Denmark
t: +45 4824 8048
f: +45 4810 6265
nordicinfo@phenomenex.com

Finland
t: +358 (0)9 4789 0063
f: +45 4810 6265
nordicinfo@phenomenex.com

France
t: +33 (0)1 30 09 21 10
f: +33 (0)1 30 09 21 11
franceinfo@phenomenex.com

Germany
t: +49 (0)6021-58830-0
f: +49 (0)6021-58830-11
anfrage@phenomenex.com

India
t: +91 (0)40-3012 2400
f: +91 (0)40-3012 2411
indiainfo@phenomenex.com

Ireland
t: +353 (0)1 247 5405
f: +44 1625-501796
eireinfo@phenomenex.com

Italy
t: +39 051 6327511
f: +39 051 6327555
italiainfo@phenomenex.com

Luxembourg
t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

Mexico
t: 01-800-844-5226
f: 001-310-328-7768
tecnicomx@phenomenex.com

The Netherlands
t: +31 (0)30-2418700
f: +31 (0)30-2383749
nlinfo@phenomenex.com

New Zealand
t: +64 (0)9-4780951
f: +64 (0)9-4780952
nzinfo@phenomenex.com

Norway
t: +47 810 02 005
f: +45 4810 6265
nordicinfo@phenomenex.com

Puerto Rico
t: +1 (800) 541-HPLC
f: +1 (310) 328-7768
info@phenomenex.com

Spain
t: +34 91-413-8613
f: +34 91-413-2290
espinfo@phenomenex.com

Sweden
t: +46 (0)8 611 6950
f: +45 4810 6265
nordicinfo@phenomenex.com

United Kingdom
t: +44 (0)1625-501367
f: +44 (0)1625-501796
ukinfo@phenomenex.com

USA
t: +1 (310) 212-0555
f: +1 (310) 328-7768
info@phenomenex.com

All other countries
Corporate Office USA
t: +1 (310) 212-0555
f: +1 (310) 328-7768
info@phenomenex.com

www.phenomenex.com

Phenomenex products are available worldwide. For the distributor in your country, contact Phenomenex USA, International Department at international@phenomenex.com

guarantee

If Phenomenex products in this technical note do not provide at least an equivalent separation as compared to other products of the same phase and dimensions, return the product with comparative data within 45 days for a FULL REFUND.

TN51110317_W