Analysis of Terpenes Using Gas Chromatography with Vacuum Ultraviolet Detection (GC-VUV)

Changling Qiu[a], Jonathan Smuts[b], Phillip Walsh[b], Kevin A. Schug[a,*]

a The University of Texas at Arlington, Arlington, TX, 76019, Untied States

b VUV Analytics, Inc., Cedar Park, Texas, 78613, United States

VUV ANALYTICS

INTRODUCTION

- Terpenes are natural products that are structurally related to isoprene (2-methyl-1,3-butadiene). Terpene mixtures contain many different compounds and these compounds are usually geometric, positional, and optical isomers. The separation and identification of these compounds in natural mixtures of terpenes is challenging and laborious.

- A new gas chromatography detector based on vacuum ultraviolet (VUV) spectroscopy, which collects full scan absorption in the range of 125-240 nm, was developed and applied to analyze terpenes. VUV features deconvolution of coeluting signals from different analytes, making it a great tool for analysis from complex matrices.

- In this study, the VUV absorption spectra for different terpenes (42) were evaluated and compared. GC-VUV was applied to the analysis of natural turpentines for qualitative and quantitative determination of terpenes.
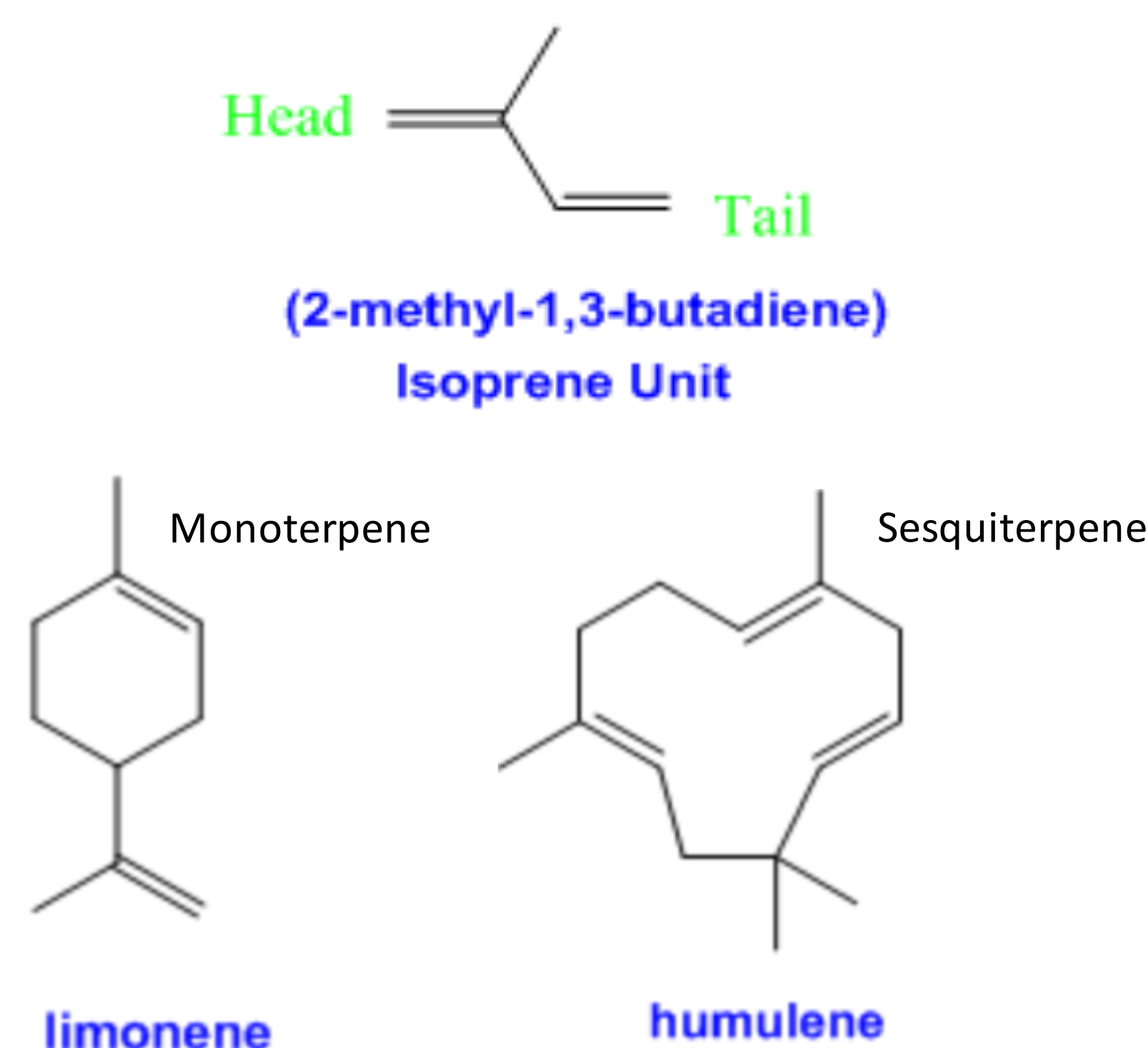

Fig. 1 Structures of Terpenes.

INSTRUMENTATION

- A VGA-100 VUV detector coupled to a Shimadzu GC-2010 gas chromatograph
- Rtx-5 column (30 m, 0.25 mm I.D., 0.25 μm d_f)
- Injection: 250 °C, 0.2 μL splitless
- Oven: 35 °C (1.5 min), 20 °C/min to 76 °C, 0.5 °C/min to 80 °C, 6 °C/min to 200 °C (4 min)
- Carrier gas: 26 cm/s He
- Interface and flow cell@ 275 °C
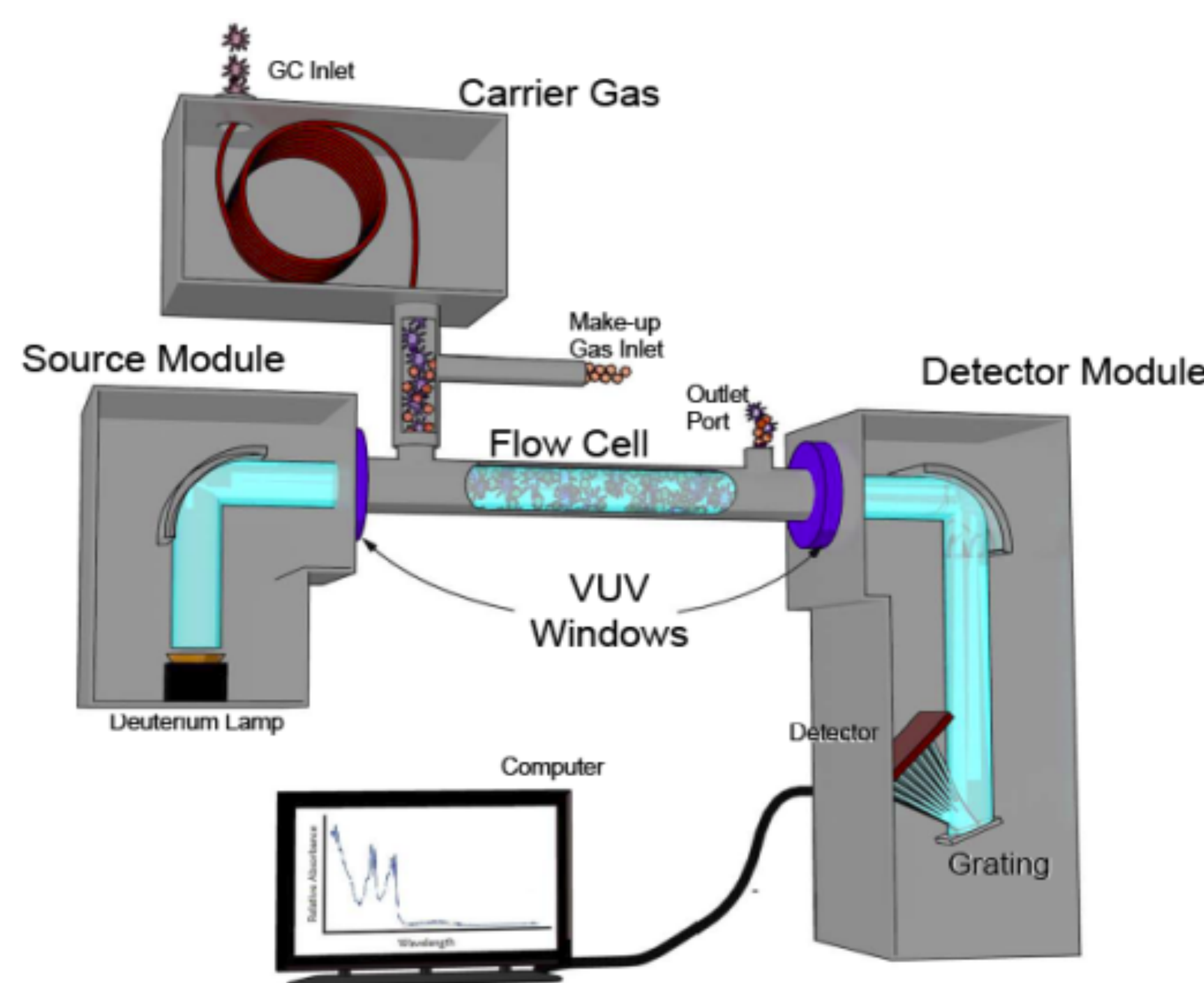- Make-up gas@ 0.15 psi
- Wavelength 125-240 nm

Fig. 2 Schematic of the GC-VUV instrument.

RESULTS

- The VUV absorption spectra for different terpenes (21) were distinctive and highly featured.
- Good LODs and LOQs were observed for some selected terpenes by GC-VUV.
- GC-VUV was applied to analyze turpentine samples and demonstrated excellent specificity for qualitative identification analysis of terpenes.
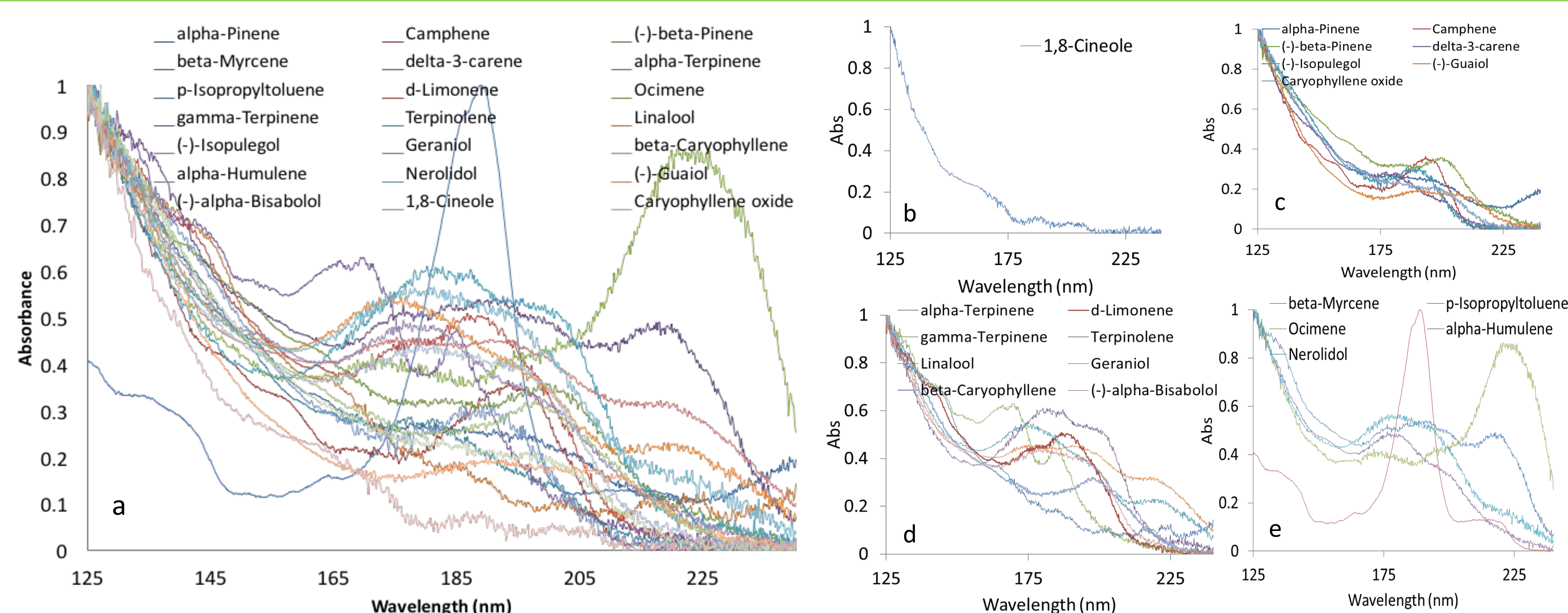- Deconvolution of the co-eluting signals of terpenes was achieved utilizing the VUV data analysis software.

Fig. 3 Spectra of terpenes (a) and grouped based on the No. of double bonds , 0 (b), 1 (c) , 2 (d), and 3 (e).
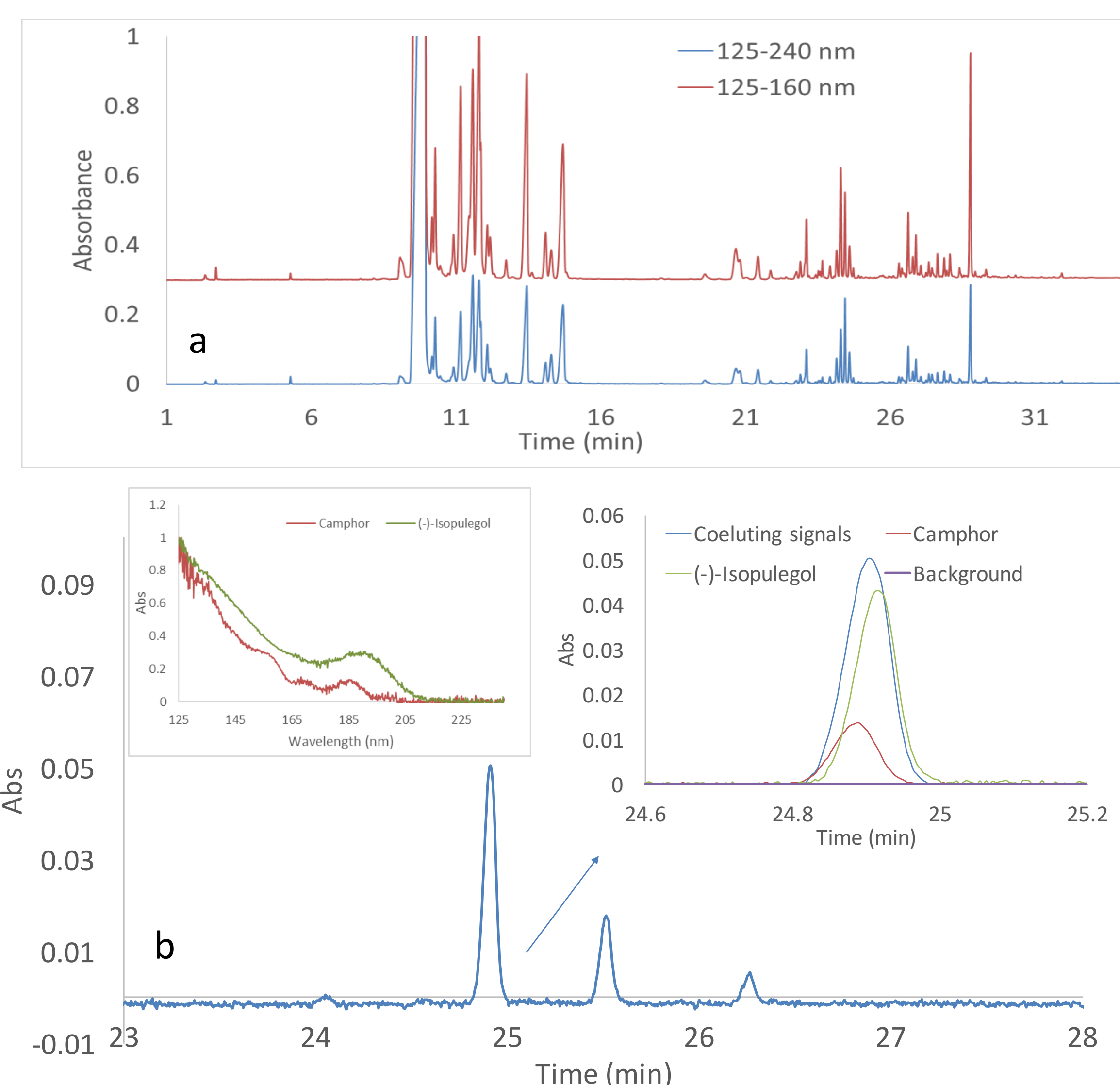

Fig. 4 Chromatogram of turpertine (a) and deconvolution of the coeluting signals in terpene mixture (b).

Table 1 LOD and LOQ data.

Compound	Class	No. of double bonds	LOD (μg/mL)	LOQ (μg/mL)
Alpha-Pinene	Mono	1	0.40	1.25
Camphene	Mono	1	1.25	5.00
(-)-beta-Pinene	Mono	1	0.40	1.25
Beta-myrcene	Mono	3	0.50	1.50
Delta-3-carene	Mono	1	0.50	1.50
Alpha-Terpinene	Mono	2	0.50	1.50
p-Isopropyltoluene	Mono	3	0.50	1.50
d-Limonene	Mono	2	0.50	1.50
Ocimene	Mono	3	0.63	2.50
Gamma-terpinene	Mono	2	0.50	1.50
Terpinolene	Mono	2	0.50	1.50
Linalool	Mono	2	0.63	2.50
(-)-Isopulegol	Mono	1	0.40	1.25
Geraniol	Mono	2	0.40	1.25
Beta-caryophyllene	Sesqui	2	0.50	1.50
Alpha-humulene	Sesqui	3	0.50	1.50
Nerolidol	Sesqui	3	0.63	2.50
(-)-Guaiol	Sesqui	1	0.50	1.50
(-)-alpha-Bisabolol	Sesqui	2	0.50	1.50

CONCLUSIONS

The VUV absorption spectra for different terpenes were distinctive and differentiable. GC-VUV demonstrated the capabilities for qualitative and quantitative analysis of terpenes in turpertine mixtures. Chromatographic coeluting signals can be deconvolved by the VUV data analysis software.

REFERENCES

1. Schug, K. A.; Sawicki, I.; Carlton Jr, D. D.; Fan, H.; McNair, H. M.; Nimmo, J. P.; Kroll, P.; Smuts, J.; Walsh, P.; Harrison, D. Analytical chemistry **2014**, 86, 8329-8335.
2. ICTWiki.http://ictwiki.iitk.ernet.in/wiki/index.php/Terpenes#Sesquiterpenes accessed Feb. 23, 2016.

ACKNOWLEDGEMENTS