



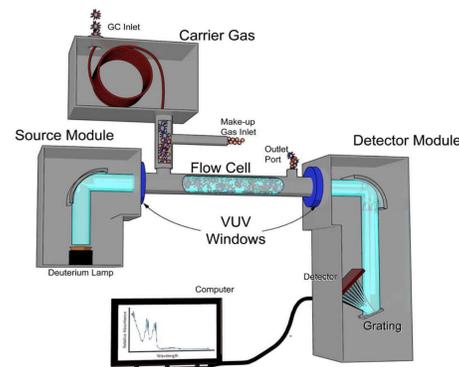
Overview of Vacuum UltraViolet Absorption Detection for Gas Chromatography

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OVERVIEW/INTRODUCTION

- Gas chromatography coupled with vacuum ultraviolet spectroscopy (GC-VUV) is a powerful tool for detecting and analyzing cannabinoids and their metabolites.
- GC-VUV offers enhanced differentiation of structurally similar compounds.
- Provides advantages over traditional methods like GC-MS and LC-MS.
- Highlights research from Leghissa et al. (2018), Skultety et al. (2017), and others.
- Focuses on GC-VUV's applications in forensic and pharmaceutical sciences.

METHODS



Sample Preparation:

- Cannabinoids & Synthetic Drugs: Derivatization with BSTFA + 1% TMCS for enhanced volatility
- Biological Samples: Liquid-liquid extraction with ethyl acetate, followed by centrifugation, filtration, and derivatization

Instrumentation:

- Agilent 7890 GC system with VGA-100 VUV detector
- DB-5MS column (30 m x 0.25 mm x 0.25 μ m)
- Injector: 280°C, split ratio 10:1, 1 μ L injection

Analytical Conditions:

- GC Oven: 100°C (initial), ramped to 280°C (final)
- Carrier gas: Helium (1.2 mL/min)
- VUV Detector: 120-240 nm, 0.5-second spectral data collection

Previous Studies: Skultety et al. (2017) used:

- GC Column: RTX-1, 30 m, 0.25 mm i.d., 0.25 μ m df
- Autoinjector: Shimadzu AOC-20i, 1.0 mL sample, 100 mg/mL standard concentrations
- Injector: 250°C, splitless mode
- VUV Transfer Line/Flow Cell: 300°C, 10 cm path, 80 mL volume
- VUV Lamp: Deuterium, Data Rate: 1.3 Hz
- Carrier Gas: Helium, constant velocity 28 cm/s
- Oven Program: 50°C to 300°C, various ramp rates
- LOD: Determined by S/N ratio (3:1)

RESULTS

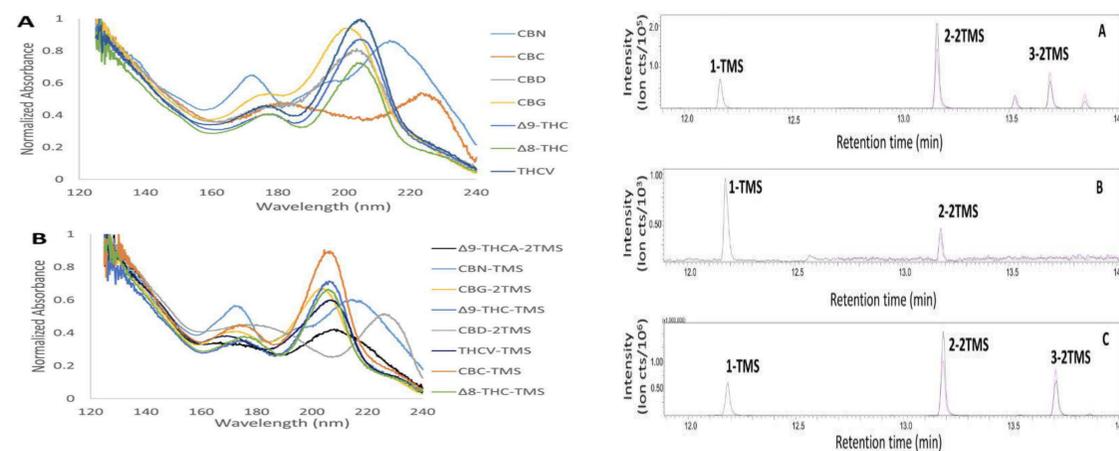


Figure 1: Normalized gas phase VUV/UV absorbance spectra for (A) underivatized and (B) silylated cannabinoids.

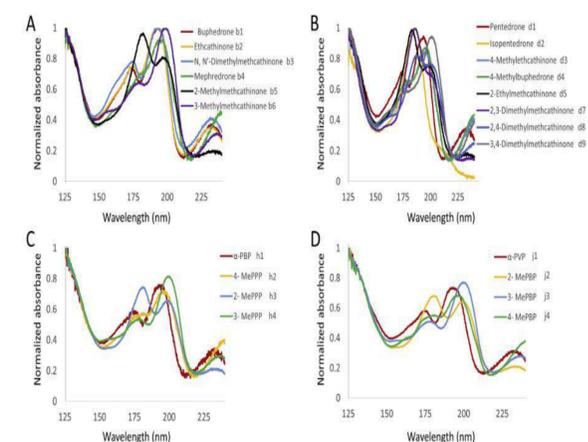


Figure 3: VUV reference spectra of selected isomeric NDS sets: (A) b1-b6, 177.1154 g/mol; (B) d1-d9, except of d6, 191.1310 g/mol; (C) h1-h4, 217.1467 g/mol; (D) j1-j4, 231.1623 g/mol.

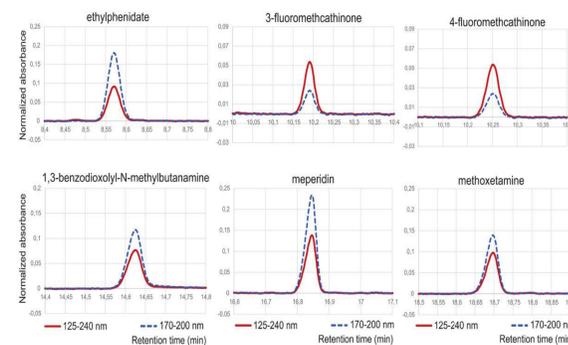


Figure 4: Normalized absorbance plotted against retention time for six NDS and two wavelength windows [125-240 nm (full range) and 170-200 nm (preferential for aromatics)] in GC-VUV analysis of standards.

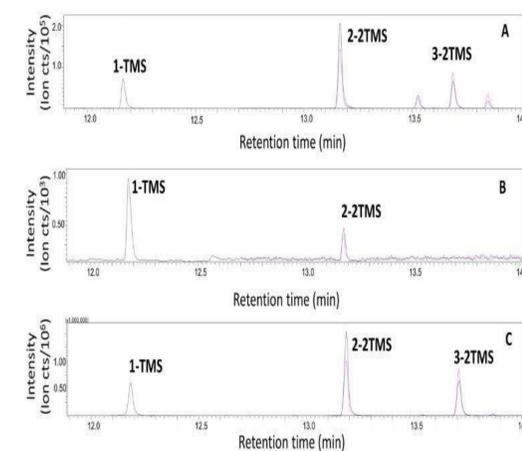


Figure 2: Representative MRM chromatograms for: (A) a 4 ppm mixture extracted from urine matrix; (B) 0.6 ppm mixture extracted from a plasma matrix; and (C) a mixture of standards at 15 ppm. Analytes correspond to the indicated silylated forms of: 1 Δ 9-THC; 2 Δ 9-THC-OH; and 3 11-nor-9-carboxy- Δ 9-THC

- Legend:
- 1 - THCV (Tetrahydrocannabivarin)
 - 2 - CBD (Cannabidiol)
 - 3 - CBC (Cannabichromene)
 - 4 - Δ 8-THC (Delta-8-Tetrahydrocannabinol)
 - 5 - Δ 9-THC (Delta-9-Tetrahydrocannabinol)
 - 6 - CBG (Cannabigerol)
 - 7 - CBN (Cannabinol)

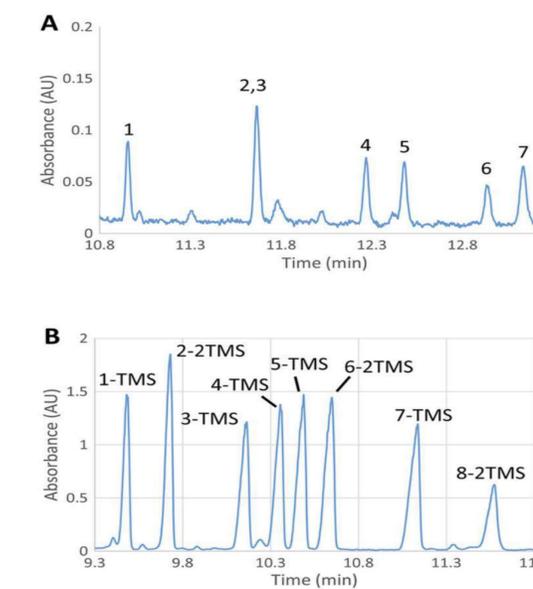


Figure 5: Representative chromatograms of (A) underivatized and (B) silylated cannabinoids. Shown are signals filtered for response in the 125-160 nm wavelength range.

CONCLUSIONS

GC-VUV Analysis:

- Provides a powerful approach for analyzing cannabinoids and synthetic drugs
- Allows clear differentiation of structurally similar compounds without full chromatographic separation

Comparison to GC-QQ-MS:

- GC-VUV simplifies data analysis
- May require sensitivity improvements for biological matrices

Future Research Directions:

- Focus on optimizing sensitivity for better analysis
- Expand applications to forensic toxicology, petroleum analysis, and pharmaceutical impurity detection

REFERENCES

Leghissa A, Smuts J, Qiu C, Hildenbrand ZL, Schug KA. Detection of cannabinoids and cannabinoid metabolites using gas chromatography with vacuum ultraviolet spectroscopy. *Sep Sci plus* 2018;1:37-42.

Leghissa A, Hildenbrand ZL, Foss FW, Schug KA. Determination of the metabolites of Δ 9-tetrahydrocannabinol in urine and plasma using multiple reaction monitoring gas chromatography with triple quadrupole mass spectrometry. *Sep Sci plus* 2018;1:43-47.

L. Skultety, P. Frycak, C. Qiu, J. Smuts, L. ShearLaude, K. Lemr, J.X. Mao, P. Kroll, K.A. Schug, A. Szewczak, C. Vaught, I. Lurie, V. Havlicek. Resolution of isomeric new designer stimulants using gas chromatography - vacuum ultraviolet spectroscopy and theoretical computations. *Analytica Chimica Acta* 971 2017; 55-67

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