Application Note

Cannabis and Hemp Testing

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Spectral Fidelity of Terpenes in Cannabis with Hydrogen Carrier Gas

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Abstract

This application note assesses terpene spectral fidelity in cannabis using a single quadrupole GC/MS in selective ion monitoring (SIM) mode with hydrogen as the carrier gas. The study complements Agilent application notes 5994-6216EN[1] and 5994-6511EN[2]. Samples in matrix were analyzed in SCAN mode with both helium and hydrogen carrier gases. MassHunter Unknowns Analysis (v 10.2) software was used for spectral analysis, resulting in match scores between 79.7% and 98.8% for spectra generated in hydrogen carrier gas against the NIST 23 Library. Notably, menthol exhibited similar match scores in hydrogen (79.7%) and helium (80.2%). Excluding menthol, hydrogen-generated match scores ranged from 90.7% to 98.8%. This application underscores the viability of hydrogen carrier gas, paired with the Agilent HydroInert source, for terpene analysis in cannabis flower. The HydroInert source minimizes redox chemistry in the MS source, ensuring conserved spectral fidelity and allowing confident identification of target analytes from library spectra generated in helium.

Introduction

Terpenes are a class of compounds that are responsible for the unique flavor and aroma in each strain of *Cannabis spp*. Characterizing the flavor and aroma profiles of commercial cannabis strains is often done using GC/MS, with helium as the preferred carrier gas. However, recurring helium shortages and mounting costs have increased demand for applications using hydrogen as the carrier gas. This application brief focuses on the spectral fidelity of terpenes in cannabis on a single quadrupole GC/MS in selective ion monitoring (SIM) mode with hydrogen as the GC carrier gas. The following study and results are meant to supplement the work in Agilent application note 5994-6216EN and 5994-6511EN.[1,2]

When adopting hydrogen for GC/MS analysis, there are several factors to consider. First, hydrogen is a reactive gas, and it may potentially cause chemical reactions in the inlet, column, and sometimes the MS EI source, which can change analysis results. To address potential issues in the MS source, the Agilent HydroInert source was used. Additional information can be found in the Agilent technical overview of the HydroInert source.[3] Second, for GC/MS applications, hardware changes in the gas chromatograph and mass spectrometer may be required when switching to hydrogen carrier gas. The Agilent Helium to Hydrogen Carrier Gas Conversion Guide[4] describes in detail the steps for conversion from helium to hydrogen carrier gas. Lastly, it is recommended that when working with flammable or explosive gases, laboratories maintain proper safety in gas handling and use. Further information on the safe use of hydrogen can be found in the Agilent Hydrogen Safety Manual[5] and Hydrogen Safety for the Agilent GC System Guide.[6,7]

Experimental

Instrumentation and sample preparation

Terpenes standard mixes CAN-TERP-MIX1H and CAN-TERP-MIX2H, each containing 21 terpenes, were purchased from SPEX CertiPrep (Metuchen, NJ, US). The GC/MS instrument configuration and method parameters for analysis with helium carrier gas followed the methodology described in Agilent application note 5994-2032EN.[8] The GC/MS instrument configuration and method parameters for analysis with hydrogen carrier gas followed the methodology described in Agilent application note 5994-6162EN.[1] Preparation of samples followed the methodology described in Agilent application note 5994-2032EN.[8] Optionally, preparation of all standards, calibrators, and samples can be automated using the Agilent PAL3 Series II RTC instrument, as described in Agilent application note 5994-6007EN.[9]

Spectral analysis and library match

Samples in matrix were run in SCAN mode on the GC/MS in helium and hydrogen carrier gas to collect spectra for all target analytes. Spectra were analyzed using MassHunter Unknowns Analysis (v 10.2) software. Unknowns Analysis was set up to deconvolute the spectra then match the deconvoluted results against the NIST 23 library. Default setting in Unknowns Analysis were used for all deconvolution and library matching.

Results and discussion

Table 1 shows the library match scores for spectra of target analytes generated in both helium and hydrogen against the NIST 23 library. Observed results show high-quality match scores, ranging from 79.7% to 98.8%, for spectra generated in hydrogen carrier gas against the NIST 23 Library. It should be noted that menthol had a match score of 79.7% in hydrogen and 80.2% in helium, demonstrating very similar match results. If the match score for menthol is removed from the hydrogen generated match scores, the range would fall between 90.7% to 98.8%. An example comparison of hydrogen-generated spectra and the spectra from NIST 23 are shown in Figure 1.

Table 1. Comparison of library match scores for spectra generated in helium and hydrogen carrier gas against the NIST 23 Library.

Target analyte	CAS number	Helium match score (%)	Hydrogen match score (%)
alpha-Pinene	80-56-8	98.9	92.5
Camphene	79-92-5	98.8	96.2
Sabinene	3387-41-5	98.4	96.6
beta-Myrcene	123-35-3	98.2	96.7
beta-Pinene	127-91-3	97.6	97.4
alpha-Phellandrene	99-83-2	97.0	95.1
delta-3-Carene	13466-78-9	97.9	97.0
alpha-Terpinene	99-86-5	96.7	96.1
E-beta-Ocimene	3779-61-1	98.8	95.2
D-Limonene	5989-27-5	99.3	98.1
p-Cymene	99-87-6	98.4	96.1
Z-beta-Ocimene	3338-55-4	98.9	97.5
Eucalyptol	470-82-6	99.3	98.4
gamma-Terpinene	99-85-4	99.3	98.4
Terpinolene	586-62-9	98.6	97.7
Sabinene hydrate	546-79-2	98.7	97.4
Linalool	78-70-6	99.2	97.2
[+/-]-Fenchone	7787-20-4	98.6	98.8
Endo-fenchyl alcohol	2217-02-9	95.3	97.8
Isopulegol	89-79-2	97.6	96.5
[+/N/-]-Camphor	464-49-3	99.4	98.5
Isoborneol	124-76-5	81.6	91.1
Menthol	89-78-1	80.2	79.7
[+/-]-Borneol	464-45-9	98.6	97.7
alpha-Terpineol	98-55-5	97.7	96.9
gamma-Terpineol	586-81-2	94.5	93.1
Nerol	106-25-2	98.2	94.7
Geraniol	106-24-1	98.1	95.6
Pulegone	89-82-7	98.4	97.0
Geranyl acetate	105-87-3	98.4	94.3
alpha-Cedrene	469-61-4	98.0	97.0
E-Caryophyllene	87-44-5	99.2	96.6
alpha-Humulene	6753-98-6	99.3	96.4
Valencene	4630-07-3	98.1	96.5
Z-Nerolidol	142-50-7	96.6	96.9
E-Nerolidol	40716-66-3	98.0	97.0
Guaiol	489-86-1	98.3	98.1
Caryophyllene oxide	1139-30-6	97.0	93.3
Cedrol	77-53-2	97.9	90.7
alpha-Bisabolol	23089-26-1	95.9	94.9

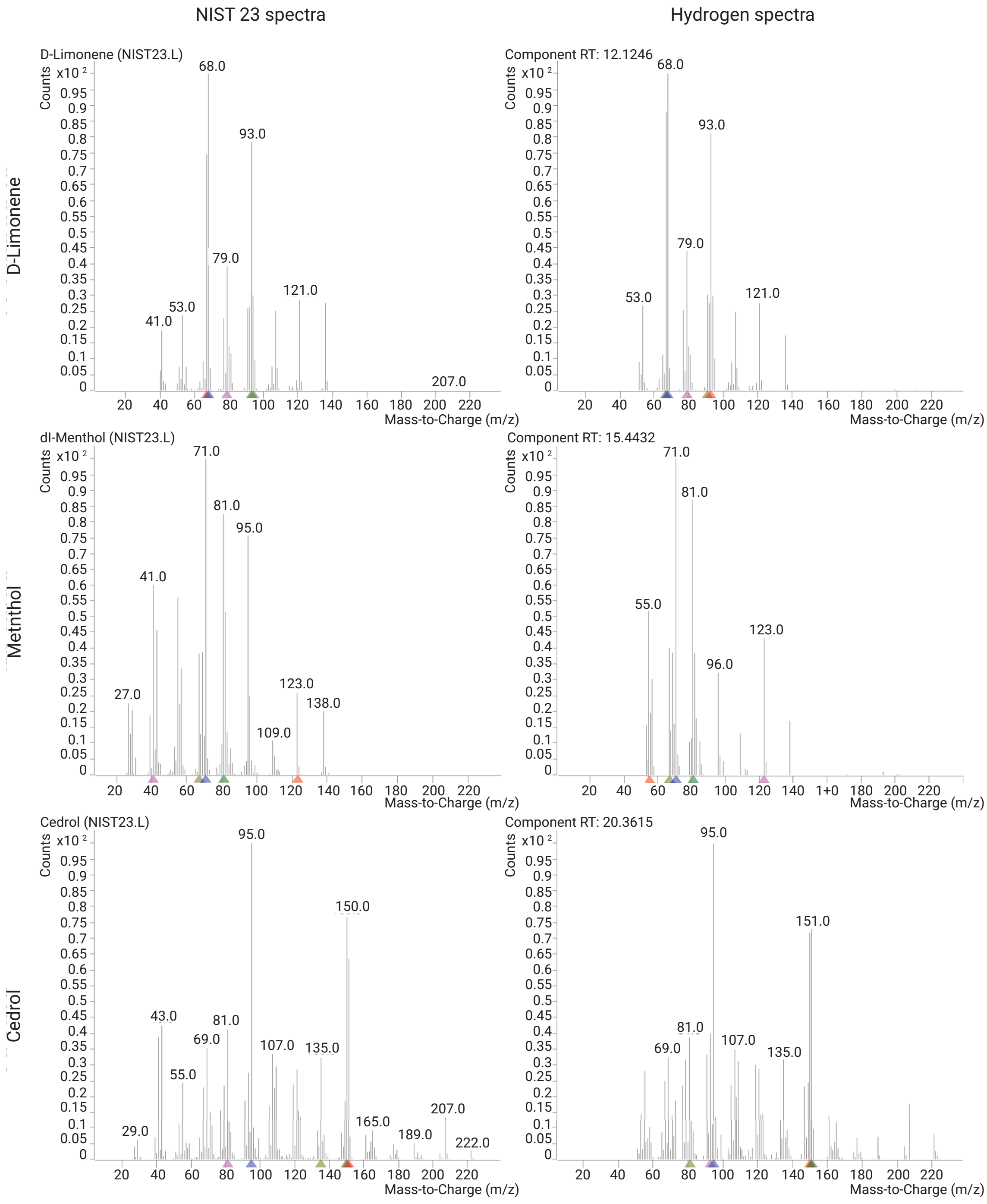

Figure 1. Comparison of NIST 23 spectra (left) to spectra observed in matrix (right) using hydrogen carrier gas for D-limonene, menthol, and cedrol. Spectral scale is relative and out of 100%.

Conclusion

This application demonstrates that hydrogen carrier gas, when paired with the Agilent HydroInert source, is a viable option for the analysis of terpenes in cannabis flower. Use of the HydroInert source greatly reduces the occurrence of redox chemistry in the MS source when using hydrogen carrier.[3] The reduction of chemical reactions in the source leads to conserved spectral fidelity, where spectra generated in hydrogen can be used with a high degree of confidence to identify target analytes from library spectra generated in helium. Further, the same quantification and qualifier ions used in helium methods can be used in SIM modes with hydrogen because of the conserved spectral fidelity.[1,2,8]

References

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