SHIMADZU Analysis of 21 Terpenes in 3 Cannabis Cultivars by HS-GCMS Vikki Johnson, Jordan M. Frost, Bob Clifford Shimadzu Scientific Instruments; Columbia, MD

Introduction

Terpene and terpenoid compounds are naturally occurring aromatic compounds that give cannabis its unique flavor and fragrance. Aside from their aromatic properties, terpenes have advantageous health benefits. They also have a synergistic relationship with cannabinoids, which further enhance the therapeutic effect of THC. Cannabis has over 140 terpene components, many of which are of medicinal interest.^{1, 2, 3} The concentration of individual terpenes varies by strain, can be anywhere from 0.1 to 1.5% of its total dry weight, and can vary depending on harvest time, drying and storage conditions. ^{1, 4}

Recent proliferation of new terpene profiling methods can be attributed to the ever-increasing state legalization of cannabis use. Due to the uniqueness of terpene profiles, they can be used by cultivators as a "fingerprint" to partially ID the specific strain in question. This poster describes the analysis of several strains of cannabis for 21 terpenes using Gas Chromatography Mass Spectrometry (GCMS) with headspace injection.

Experimental

Terpene analyses were conducted using a Shimadzu GCMS-QP2010SE single quadrupole mass spectrometer with the HS-20 headspace autosampler for sample introduction. The MS was run using the FAAST (Fast Automated SCAN/SIM Type) where the SCAN mode was used for identification and the SIM mode was used for greater quantitation. The instrument and operating conditions are shown in Table 1.



Table 1. Instrument Operating Conditions and Method Parameters

Headspace	: 1 mL sample loop 30 minute equilibrium at 150°C	Flow Mode	: Constant linear velocity (47.2 cm/sec)
Inj. Mode GC Column	: Split 50:1 :Rxi-624 Sil MS (30 m x 0.25 mm ID,	Interface Temp Ion Source Temp	: 300°C : 230°C
GC Oven	df = 1.4um) : 80 °C (1 min), 12 °C/min to 150 (1	Sample	: 10uL in 10mL vial
Temp	min), 9 °C/min to 250 (1 min)		

Sample Preparation

As plant material does not dissolve in solvent, the full evaporation headspace technique (FET) was used for quantitation. Using FET, a small amount of standard and sample was used to create a single phase gas system, as compared to a two phase liquid-gas system as in traditional headspace techniques. The terpene standard was purchased from Restek (Bellefonte, PA) and included 21 terpenes in a 2500 μ g/mL stock solution. A seven-point calibration curve was created with the concentration range from 78.125 µg/mL to 2500µg/mL. A part of the flower weighing 1.0 gram was frozen, followed by grinding to ensure a representative sample. Ten to 30mg of the flower were then weighed into a headspace vial and capped.

Results Chromatography Figure 1 shows the TIC for 21 terpenes

Figure 1. TIC chromatogram from Restek standard

10.0

11.0

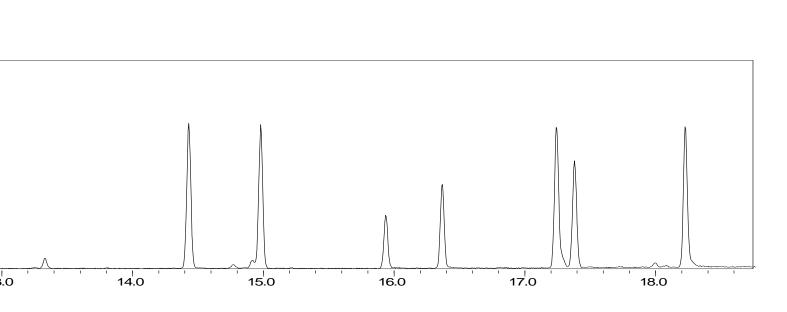
2.5

0.5

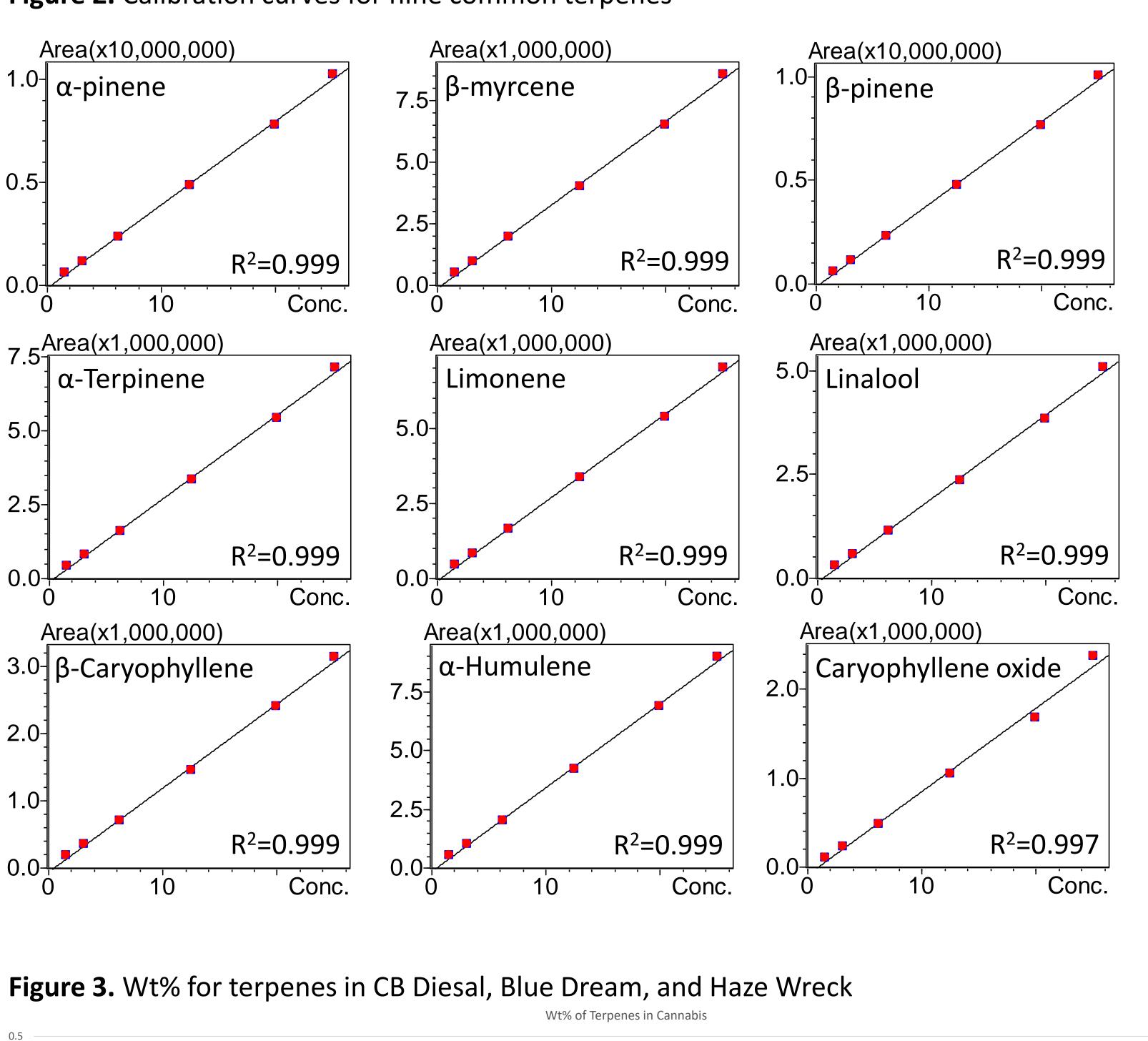
6.0

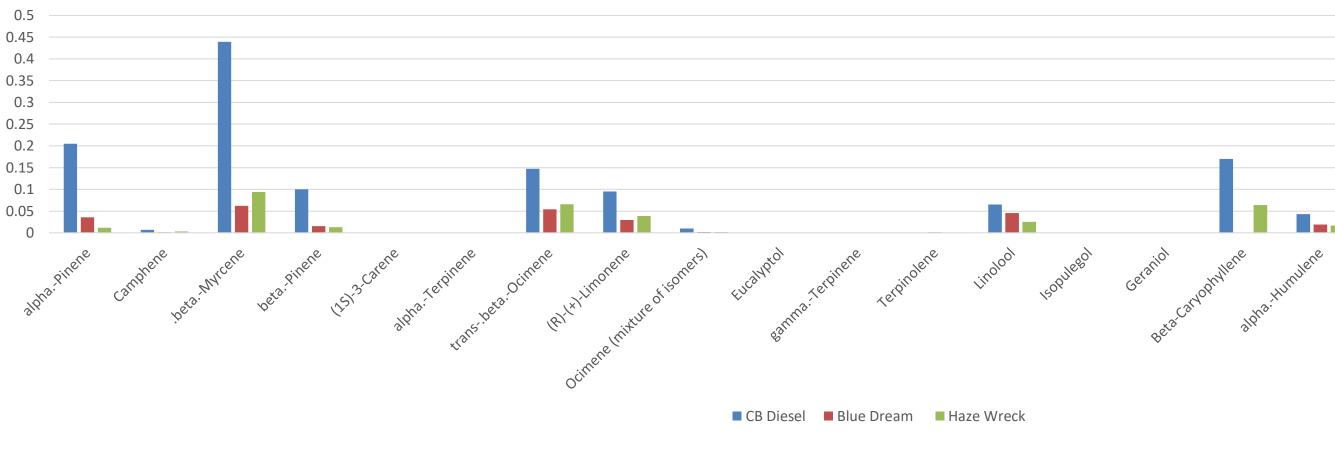
7.0

8.0



Calibration standards were prepared and analyzed using the optimized HS-20 and GC-MS parameters with concentrations ranging from 0.78µg to 25µg on column concentration. Figure 2 shows common terpene calibration curves with corresponding correlation coefficients illustrating linearity. Three strains of cannabis were tested and the results are shown in Figure 3.





Conclusion

The first sample of cannabis analyzed, CB Diesel, was analyzed shortly after harvest. The resulting wt% of terpenes is similar to that in current literature.¹ The other two samples, Blue Dream and Haze Wreck, were stored at ambient temperature and exposed to light for one month prior to analyzing. It has been demonstrated that different storage conditions can change terpene results over time,⁴ and this should be taken into consideration when analyzing cannabis samples as the results show less than expected results. Varying storage conditions and degradation experiments should be the next study in the ever changing world of regulatory cannabis testing.

References

- California, 2015.

Calibration

Figure 2. Calibration curves for nine common terpenes

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