Development of a Quantitative Method of Eight Cannabinoids Including Total THC and GCMS

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1. Introduction

Due to the legalization of marijuana both recreationally and medicinally, use of the drug has increased drastically. It's reported in 2018, more than 11.8 million young adults reported marijuana use in the past year[1]. With this rapid expansion of marijuana use, testing has become even more critical. The prevalence of marijuana use and the passage of legislation regulating its use has mandated the development of analytical procedures for detecting Δ^9-tetrahydrocannabinol (THC), the major psychoactive component of marijuana, and its metabolic products in biological matrices. Along with THC several other cannabinoids have become of interest such as: CBDV, CBC, CBD, Δ^8 THC, and CBG. With CBD products on the rise, it is important to be able to detect total CBD content, while also ensuring the product contains little to no THC. Having the ability to analyze the previously mentioned cannabinoids on GC-MS is extremely advantageous due to the fact terpenes, residual solvents and some pesticides can also be analyzed via GC-MS.

2. Experimental Methods

A cannabinoid mix from Cayman Chemical was procured for GC-MS analysis. The mix contained 11 cannabinoids; however only 9 are resolvable without derivatization. CBDV, THCV, CBC, CBD, Δ^8 and Δ^9 THC, CBG and CBN were are resolved in five minutes. The standards were prepped at concentration levels of 0.5, 1.0, 5.0, 10.0, 50.0, 100.0ppm. These standards were run via GC-MS with a liquid autosampler (Figure 1) and were run in the with goal of full chromatographic separation and detection, while also proving to be linear from lowest to highest concentration. To validify this method with a real world scenario, CBD lotion was prepped to determine CBD content. The four samples were weighed, and then underwent a methanol extraction performed twice to ensure all CBD was collected.

Figure 1. GCMS-QP2020NX

3. Analytical Conditions

System Configuration

GC-MS:	GCMS-QP2020NX (Shimadzu)
Autosampler:	AOC-20i/s

GC Parameters

Column:	RTX-35
Injector:	275 ˚C
Oven Temp.:	230 ˚C (1min), 260 ˚C @15/ min, 320 ˚C @ 25/min (.7min)
Carrier Gas Control:	Helium, Constant Linear Velocity, 46.0 cm/sec
Injection Mode:	Split 1:10
Total Program time:	6.07min

MS Parameters

Interface Temp.:	250 ˚C
Ion Source Temp.:	200 ˚C
Ionization Mode:	EI
Acquisition Mode:	SIM

Table 1. SIM m/z

Start	End		m/z		
3.34	4.55	SIM	314	299	286
			271	258	246
			232	231	218
			204	203	174
4.55	5.39	SIM	314	299	296
			295	258	246
			238	232	231
			193	174	123

Table 1 refers to the SIM masses used during the run for identifying specific cannabinoids at specific retention time ranges

4. Results

The cannabinoids standards were run from low to high and were qualitatively processed using the Wiley Mass Spectra of Designer Drugs 2019 providing high accuracy and exact match. The chromatograms below represent the high, middle and low concentrations (Figures 3, 4 and 5 respectively). Each peak is labeled with the respective cannabinoid.

Figure 3. Chromatogram of 100ppm

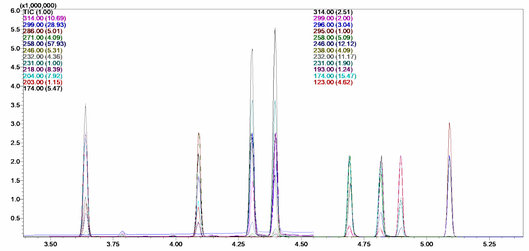


Figure 4. Chromatogram of 10ppm

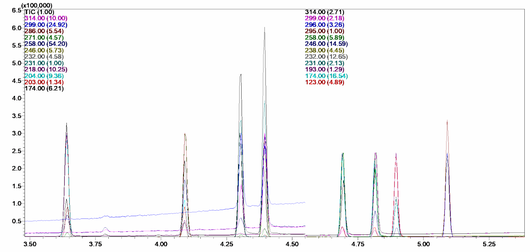
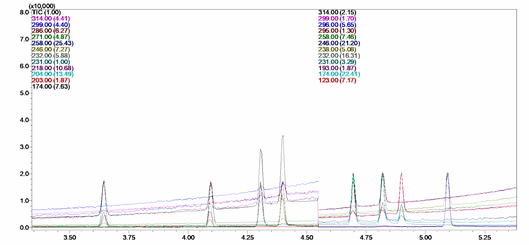


Figure 5. Chromatogram of 0.1ppm



The calibration curves shown in figure 6 and 7 represent Δ^9 THC and CBD. The linearity for the calibration curves are 0.9996 and 0.9991 respectively.

Figure 6. Calibration curve for Δ^9 THC

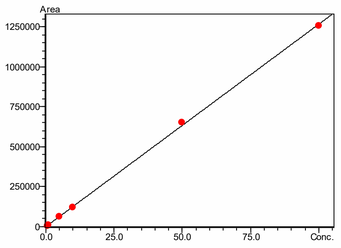
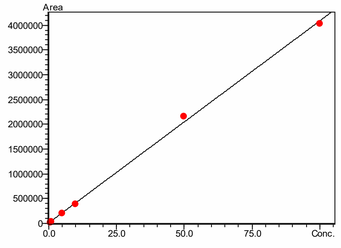


Figure 7. Calibration curve for CBD



The CBD lotion samples detected CBD concentrations while also detecting no THC. The chromatogram for a lotion sample can be seen in figure 8. Table 2 also shows the concentrations of CBD recovered

Figure 8. Chromatogram of CBD oil lotion

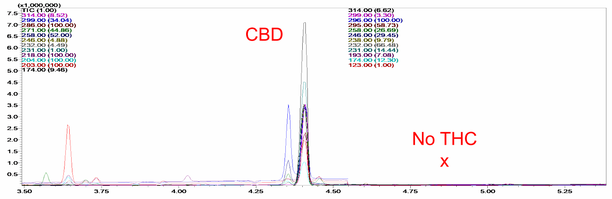


Table 2. Concentrations recovered of CBD

Sample	calculated result	weight	concentration in lotion (ppm)	combined result of both extracts (ppm)
1	99.847	0.1943	256.94	302.23
2	145.378	0.2946	246.74	324.01
3	146.359	0.3262	224.34	307.06
4	147.288	0.2968	248.13	313.26
1_2	17.599	0.1943	45.29	
2_2	45.529	0.2946	77.27	
3_2	53.968	0.3262	82.72	
4_2	38.661	0.2968	65.13	

5. Conclusion

The GC-MS method has proven to be extremely efficient and accurate in separating 9 major cannabinoids while also detecting them at low levels. Due to the fact this is run with pure standards, the next step in developing this method is to match matrices and continue with an LOD and LOQ study to validify the method. The GC-MS method has also proven to be a useful technique for customers currently using GC-MS for other cannabis applications like terpenes and residual solvents.

6. References

1) Substance Abuse Center for Behavioral Health Statistics and Quality. Results from the 2018 National Survey on Drug Use and Health: Detailed Tables, SAMHSA. https://www.samhsa.gov/data/report/2018-nsduh-detailed-tables. Accessed December 2019.