Quantitative Analysis of δ9-Tetrahydrocannabinol in Cannabidiol Oil Using Pyroprobe

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Application Note

Cannabis

Abstract

This application note demonstrates a quantitative analysis of δ9-Tetrahydrocannabinol (THC) from a cannabidiol oil sample. The calibration curve yields a correlation coefficient R^2 of 0.9997. Combining the low RSDs at 3.4% from 7 runs, the Pyroprobe is qualified for quantitative thermal extraction of THC.

Introduction

Cannabidiol (CBD), derived from the cannabis plant, has a growing interest in nutraceutical and pharmaceutical industries. Similar in structure to psychoactive δ9-Tetrahydrocannabinol (THC) found in marijuana, non-intoxicating CBD is being credited with helping treat many medical issues. Diluted in hemp seed oil, CBD oil is a complex natural product, containing many volatile and non-volatile constituents. As demonstrated in an earlier application note[1], the Pyroprobe could be used to clarify constituents in CBD oil by first separating ingredients based on their volatility from thermal extraction, then pyrolyzing the non-volatile portion at higher temperature.

Due to the concern from public on distinguishing CBD products from marijuana, the Agriculture Improvement Act of 2018, Pub. L. 115-334, (the 2018 Farm Bill) was signed into law on Dec. 20, 2018, which states that cannabis products containing less than 0.3% (3 μg/μL) THC are exempted from controlled substances under federal law. To assist in law enforcement activities, this application note describes a novel approach to use a Pyroprobe and GC/MS to quantify the THC concentration from commercially available CBD products.

Experimental Setup

The internal standard solution of methyl stearate was prepared at a concentration of 1 μg/μL. The calibration standards were prepared from a Restek THC standard in methanol (Restek P/N 34067) to four concentrations of 0.5, 0.62, 0.76 and 1 μg/μL. The internal standard solution was added to each of the THC calibration standard at a 50:50 ratio. The resulting concentrations of the THC in the final dilutions were 0.25, 0.31, 0.38, and 0.5 μg/μL respectively, and the concentration of the internal standard in these dilutions were the same as 0.5 μg/μL.

In the reproducibility study, seven aliquots of 1 μL volume of 0.5 μg/μL calibration standard were added to DISC tubes by a 1.0 μL syringe. Samples were then thermally extracted at 300°C using a CDS Model 6200 Pyroprobe coupled to a mainstream GC/MS. Base peaks of m/z 74 and 299.2 were chosen for methyl stearate and THC respectively to calculate area ratios.

Experimental Parameters

Pyroprobe	
DISC Chamber:	300°C 30min
Trap Rest:	50°C
Trap Final:	325°C 10min
Interface:	300°C
Transfer Line:	350°C
Valve Oven:	350°C

GC/MS	
Column:	5% phenyl (30m x 0.25mm)
Carrier:	Helium 1.25mL/min 75:1
Injector:	360°C
Oven:	80°C for 10 minutes
	15°C/min to 320°C
Ion Source:	230°C
Mass Range:	35-600amu

In the linearity study, a four point calibration curve of THC to ISTD area ratio vs. concentration was completed by running all four calibration standards at 0.25, 0.31, 0.38, and 0.5 $\mu g/\mu L$.

Finally, a commercially purchased CBD oil was diluted in methanol by 6 fold, and quantified on THC against the calibration curve to obtain the THC concentration.

Results

Figure 1 showed the Total Ion Chromatograms from 7 runs. The RSDs were calculated based the ion ratio between the internal standard and the THC. An RSD of 3.4% was observed.

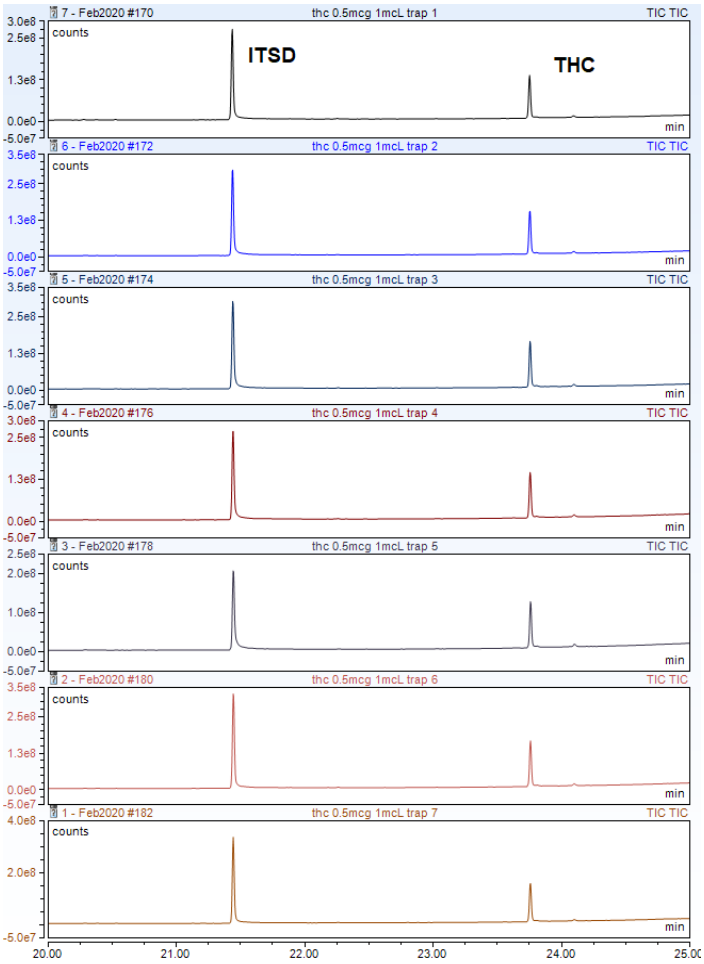

Figure 1. Seven analyses of THC at 300°C.

Table 1. Area Ratio of 299.2 (THC): 74 (ITSD) from seven runs of a THC standard

	Area Ratio	
Rep 1	0.18	
Rep 2	0.19	
Rep 3	0.18	
Rep 4	0.19	
Rep 5	0.20	
Rep 6	0.18	RSD
Rep 7	0.19	3.4%

Figure 2 showed the four point calibration curve. From the linear regression, the correlation coefficient R^2 was obtained as 0.9997.

Figure 2 also had the data point collected from the diluted CBD sample. From the data fitting, it was determined that the diluted CBD oil had 0.44 $\mu g/\mu L$ of THC. Multiplying by the dilution factor of 6, the original CBD oil sample had 2.64 $\mu g/\mu L$ of THC, which is equivalent to 0.26% THC. This concentration meets the less than 0.3% THC federal guideline.

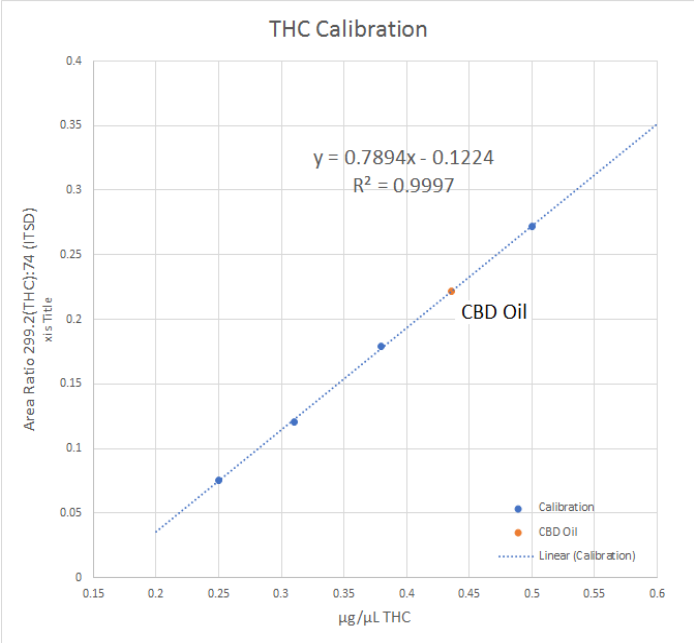

Figure 2. THC Calibration curve and diluted CBD oil sample.

Conclusion

This application note demonstrated a qualitative thermal extraction technique of THC using a Pyroprobe. This method has sufficient reproducibility and linearity to test the THC concentration from unknown CBD samples based on federal regulations.

References

1. Sam, K., App Note #201: Investigative Multi-Step and Quantitative Analysis of Cannabidiol Oil using the Pyroprobe, CDS Analytical