Quantitative Cannabidiol Oil Analysis with the Pyroprobe

Application Note
Pharmaceutical -Cannabis

Author:

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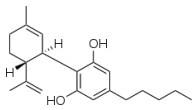
Abstract
This application note demonstrates quantitative analysis of CBD oil including RSDs and a calibration curve.

A previously published application note[1] describes Cannabidiol (CBD) oil qualitatively examined using a multi-step temperature sequence[2,3]. This article revealed specific active compounds like the sesquiterpenes α-Bisabolol, α-Caryophyllene, and cannabinoids in addition to CBD, including δ9-Tetrahydrocannabinol (THC), whose amount must be small enough to pass regulations. Performing a multi-step thermal analysis technique separates compounds based on their boiling points which simplifies chromatograms, therefore providing valuable information for the analyst.

In this application, a CBD calibration curve was further developed and RSDs were measured for a quantitative study. To decrease the sample's viscosity for easy syringe additions, 1mL CBD oil was diluted to 2mLs with hexane. Three 0.5µL aliquots of this sample were each added to 3 DISC tubes. Each DISC tube was analyzed twice at one of the 3 preset temperatures: 200°C, 300°C, and 400°C. The first run of each temperature was a sample run, and the second run of the same sample was to characterize residual CBD. A setpoint of 300°C for 30 minutes extracted nearly all the CBD, leaving 0.22% for the following 300°C step (Figure 1). So 300°C was chosen to create calibration curve and perform RSD measurements. Five consecutive sample runs yield an Area Count RSD for cannabidiol in CBD oil at 2.18%. Replicate extracted ion chromatograms of m/z 231.2, the mass spectrum's base peak, are shown in Figure 2, and the RSD measurements are found in Table 1.



Cannabidiol

A CBD calibration curve was created by adding 4, 6, 8, 10, 12, and 14 µL of a 1000µg/mL CBD standard (Restek P/N 34011) to DISC tubes, resulting in 4, 6, 8, 10, 12, and 14 µg of CBD, respectively, in each of the DISC tubes. These masses are equivalent to concentrations of 8, 12, 16, 20, 24, and 28mg/mL from 0.5µL of the tested CBD sample. These tubes were run at 300°C for 30 minutes, and the resulting six-point calibration produced a linear regression >0.99 r2 (Figure 3). By comparing the average area of the CBD in the replicates in relation to the calibration curve (using the generated calibration line equation and solving for x, concentration), it was determined that the sample has 24mg/mL of CBD. As the sample was diluted in half, the original CBD oil has 48mg/mL of CBD, close to what was claimed by the manufacturer (50mg/mL). As shown here, typical reproducibility and quantitative results experienced with the Pyroprobe can proceed into the analysis of complex samples such as CBD oil.

Performing multi-step thermal analysis on both pharmaceuticals and natural products like CBD oil, can help an analyst find distinct components, providing valuable information for competitive analysis and product development. Additionally, the linearity and RSDs demonstrate that the CDS Pyroprobe is adept at analyzing active substances like CBD in complex sample matricies, making it suitable to use for qualification and regulation purposes.

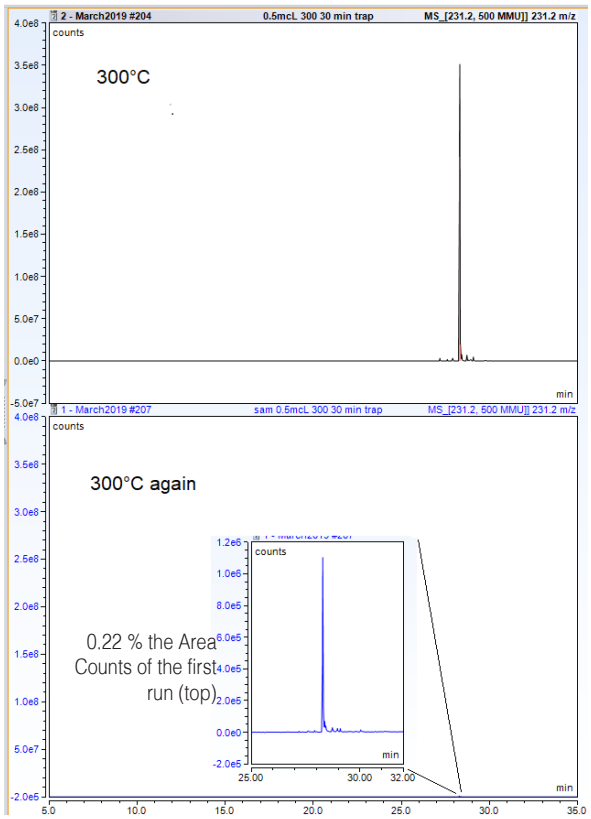


Figure 1. CBD 300°C (top) then 300°C again (bottom), m/z 231.2.

Figure 2. Five analyses of CBD oil, 300°C, m/z 231.2.

	Area Counts	
Rep 1	17579813	
Rep 2	16921013	
Rep 3	17600523	
Rep 4	16980330	RSD
Rep 5	16836796	2.18%

Table 1. Area counts of m/z 231.2 for CBD in CBD oil

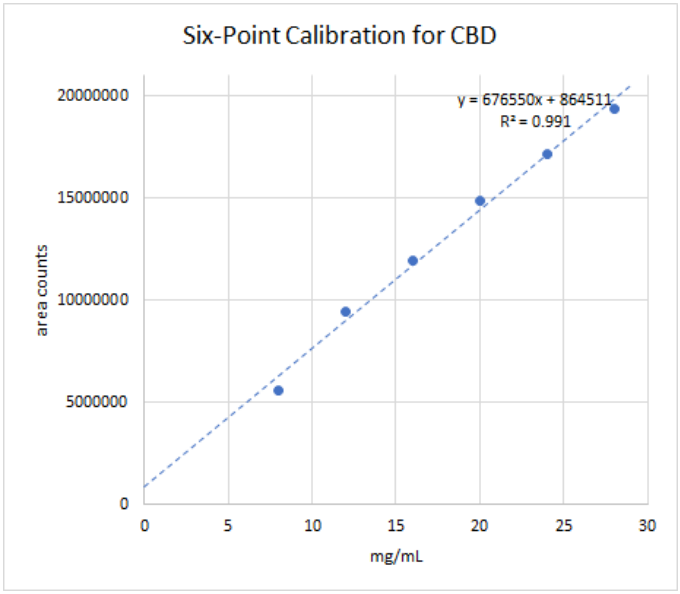


Figure 3. CBD Calibration Plot

Experimental Parameters
Samples were pyrolyzed in a DISC tube, using a CDS Model 6200 Pyroprobe.

Quantitative Analyses
Trapping Mode
Pyroprobe:
DISC Chamber: 300°C 30min

Trap Material:	Tenax
Trap Rest:	50°C
Trap Final:	325°C 10min

Interface:	300°C
Transfer Line:	300°C
Valve Oven:	300°C

GC/MS

Column:	5% phenyl (30m x 0.25mm)
Carrier:	Helium 1.25mL/min, 75:1 split
Injector:	300°C
Oven:	80°C for 10 minutes 10°C/min to 300°C
Ion Source:	230°C
Mass Range:	35-600amu

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READINGS:

[1]CDS Application Note #195 Cannabidiol Oil Analysis with the Pyroprobe

[2]CDS Application Note #184a Pyrolysis GC-MS of Pharmaceutical Packaging

[3]Sam, K. American Lab Multistep Thermal Characterization of Liquid-Filled Capsules and Medication Packaging Using GC/MS. Am. Lab, April 2018.