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Reliably Confirm Cannabinoids by GC-MS

Using a 12m x 0.20mm ID 0.33µm Rxi®-5ms Column

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- Baseline resolution for all major metabolites.
- Ultra-low bleed at 300°C, for accurate data.
- Bake column at 340°C, to remove derivatization by-products and prolong column life.

Marijuana is one of the most abused substances in the United States. Its common abuse stems from its widespread availability and because it is inexpensive, compared to other abused substances such as cocaine and heroin. Marijuana use typically is determined by screening for its major metabolite in urine, 11-nor-9-carboxy-Δ^9-tetrahydrocannabinol (Δ^9-carboxy-THC), using an immunoassay. When screening results are positive, gas chromatography/mass spectrometry (GC/MS) is employed for confirmation. Marijuana use also can be determined by analyzing other sample matrices, such as blood, hair, oral fluid, or body tissues but, again, positive results require GC/MS confirmation.[1]

GC/MS confirmation methods require sample clean-up and derivatization of target analytes, and call for a capillary GC column that can produce reliable identification and quantification results. Δ^9-carboxy-THC is the primary target in GC/MS confirmation analysis, but other marijuana metabolites present in samples include cannabinol, cannabidiol, 11-hydroxy-Δ^9-tetrahydrocannabinol (Δ^9-hydroxy-THC), Δ^9-tetrahydrocannabinol (Δ^9-THC), and Δ^8-tetrahydrocannabinol (Δ^8-THC). Further, a guard column typically is recommended for this analysis, to prevent non-volatile residue in the sample matrix from contaminating the analytical column. The guard column should have an internal diameter approximately equal to that of the analytical column, to minimize changes in flow rate.

For the analysis we show in this article, we used MTBSTFA (N-methyl-N-(tert-butyldimethylsilyl)-trifluoroacetamide) to derivatize the target compounds.[2] The analytical column we chose is our new 12m x 0.20mm ID x 0.33µm Rxi™-5ms column (5% diphenyl / 95% dimethylpolysiloxane stationary phase). The small internal diameter makes this column compatible for use with mass spectrometers, because the column can be operated using a 1.0mL/min. flow rate. The short length produces analysis times of less than 15 minutes for the major metabolite, Δ^9-carboxy-THC, which elutes last. Because the target compounds have relatively high molecular weights (310-358 amu, underivatized — see Figure 1), the GC oven must be programmed to a relatively high temperature, 300°C, to keep analysis time short.

The column and conditions we used ensure baseline resolution for all of the metabolites in Figure 2. Figure 2 also shows that the ultra-low bleed exhibited by the Rxi™-5ms column does not interfere with the analysis. The GC oven must heated to an even higher temperature between samples, 340°C, to bake sample matrix interferences and derivatization by-products from the system. Derivatization by-products can be seen in the baseline in Figure 2.

The results of this analysis demonstrate that a 12m x 0.20mm ID x0.33µm Rxi™-5ms column has the selectivity and inertness needed to provide baseline resolution, suitably short analysis times, and no interference from bleed at high temperature. We highly recommend it for this analysis.

Figure 1 Cannabinoids have relatively high molecular weights, so high temperatures must be used in their analysis.

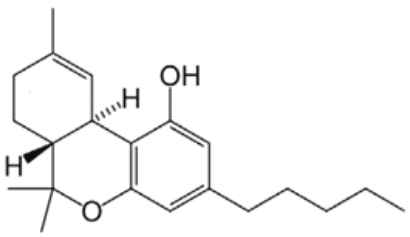
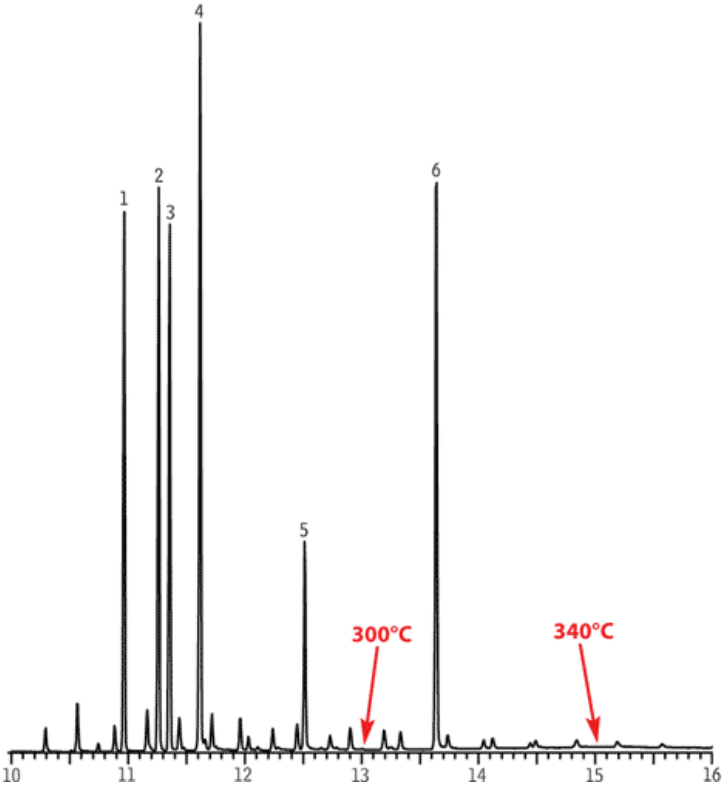


Figure 2 A 12m x 0.20mm ID x 0.33µm Rxi™-5ms column provides baseline resolution and short analysis time for cannabinoids.

1. cannabidiol
2. Δ^8-tetrahydrocannabinol
3. Δ^9-tetrahydrocannabinol
4. cannabinol
5. 11-hydroxy-Δ^9-tetrahydrocannabinol
6. 11-nor-Δ^9-tetrahydrocannabinol carboxylic acid



GC_PH00891

Column:	Rxi™-5ms 12m, 0.20mm ID, 0.33µm (cat.# 13497)
Sample:	1000µg/mL each component in methanol
	1.0µL, split, split ratio 25:1, 4mm ID base-deactivated single gooseneck inlet liner w/wool
Inj.:	(cat.# 20798-211.1)
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1mL/min.
Oven temp.:	40°C to 340°C @ 20°C/min. (hold 5 min.)
Det:	MS

Transfer line	
temp.:	280°C
Scan range:	100-550 amu
Ionization:	EI
Mode:	scan

References

1. Smith, F. and J. Siegel *Handbook of Forensic Drug Analysis* Elsevier Academic Press, 2005, pp. 98-151.
2. Clouette, R., M. Jacob, P. Koteel, and M. Spain *Journal of Analytical Toxicology* 17 (1): 1-4 (Jan./Feb. 1993).

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marijuana, *cannabinoid metabolites*, *Rxi-5ms*, *THC*