FRONTIER LAB

Rapid Screening of Cannabinoids in Edibles by Thermal Desorption (TD)-GC/MS & TD-GC/FID

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Why Thermal Desorption-GC/MS?

Most of the traditional methodologies for the determination of cannabinoids are based on solvent extraction, filtration, and concentration. These techniques are cumbersome, time-consuming, and suffer from analyst-to-analyst variability while producing data of limited value.

As the demand for the analysis of cannabinoids increases, it is imperative that the day-to-day analytical protocols be reproducible, accurate and efficient. Many laboratories routinely “screen” each sample in order to quickly determine the potential for matrix interference and instrument contamination while providing an estimate of the target compound’s concentration. A good “screening” method is simple (i.e., minimal or no sample preparation), fast and semi-quantitative.

One of the most widely used analytical techniques for “screening” is Thermal Desorption-GC/MS (TD-GC/MS). This technique does not require any of the traditional solvent extraction or sample pretreatment. Milligram quantities are put in an inert sample cup which is then ‘ready to analyze’. This technique is performed by the multi-mode Pyrolyzer with a vertical micro-furnace design that allows programmable and multiple thermal desorption analysis on a single sample.

Thermal desorption analysis eliminates conventional sample prep regimes; the sample is heated to the point that the cannabinoids desorb from the edible matrix (thermal extraction). It is fast, uses minimal or no solvent, and eliminates the need for expensive glassware. TD-GC/MS is a “volatiles only” analysis; high boiling sample constituents remain in the sample cup which eliminates system contamination, increases system stability and reduces run-to-run analysis time.

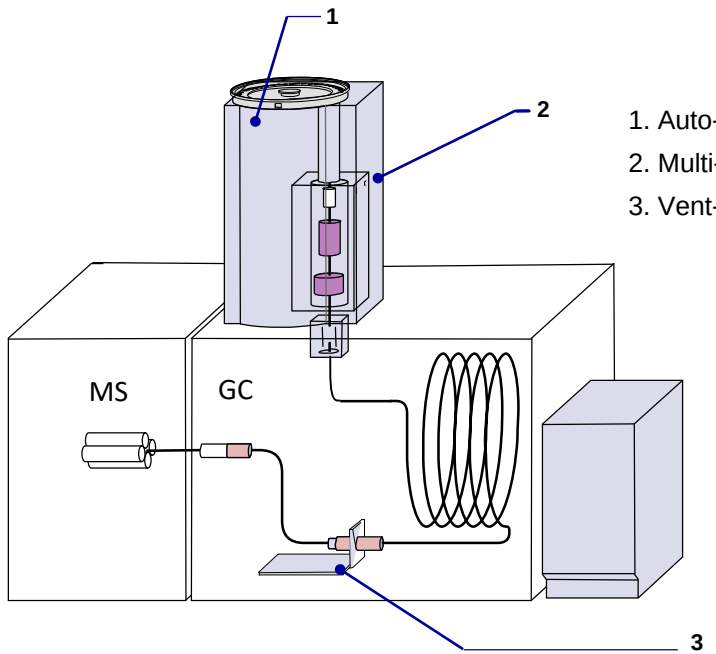
The Multi-Mode Pyrolyzer provides the users with multiple modes of operations and techniques. Evolved Gas Analysis (EGA) is another technique that can be performed as the rapid screening of evolved gases and identifying the optimal thermal desorption temperature zone(s).

What is TD-GC/MS Technique?

TD-GCMS is a powerful and straightforward technique that utilizes a Frontier Pyrolyzer as a programmable temperature inlet to a Gas Chromatography-Mass Spectrometer (GCMS) system. The material of interest (liquid or solid) is uniformly heated in an inert atmosphere. Volatile organics evolve at temperatures below 300ºC. At higher temperatures, covalent bonds break and the complex structure is degraded into smaller (stable and volatile) molecules which are referred to as pyrolyzates. The pyrolyzates formed and their relative intensities provide insight into the structure of the original material.

The technical data in this monograph were obtained using one or more of the listed accessories. Each accessory is described in more detail in the system configuration section.

The Frontier Pyrolyzer is interfaced directly to the GC inlet. The sample is placed in a small deactivated cup which is, in turn, positioned in a micro-furnace. The temperature of the sample is carefully controlled (±0.1ºC) to ensure that the sample-to-sample thermal profile is identical. Frontier's well-engineered technology ensures that the sample is maintained at ambient temperature, in an inert atmosphere, prior to pyrolysis; thus eliminating evaporation, thermal degradation, and thermosetting before analysis.



1. Auto-Shot Sampler (AS-1020E)
2. Multi-Shot Pyrolyzer (EGA/PY-3030D)
3. Vent-Free GC/MS adapter

Analysis of Cannabis Infused Chocolate Brownie by TD-GC/MS

A commercial package of cannabis-infused chocolate brownie containing 10 brownie bites with the total of 100 mg THC (Tetrahydrocannabinol) is used. According to the product label, each brownie bite contains 10 mg of THC. In this experiment, one of the brownie bites from the package was placed on an analytical balance, and the weight was recorded as 10.17 grams. So based on the label, there is 10mg/10g=1mg/g of THC present in that brownie bite. The same brownie bite with the recorded weight was used to perform the TD-GC/MS analysis to confirm the theoretical THC value according to the label.

To calculate and confirm the amount of THC, EGA was first performed on THC standard-Absolute Standard. The pyrolyzer furnace was programmed from 100 to 800ºC (20ºC/min). The EGA thermogram obtained is shown in Figure 1. For more information on the “Method Map” for material characterization, refer to page 19.

From the EGA thermogram, the optimal thermal desorption zone of THC was identified as 100 to 300ºC. In fact, using the MS interpretation library, the peak with the apex of 185ºC (100 to 300ºC temperature zone) was identified as THC.

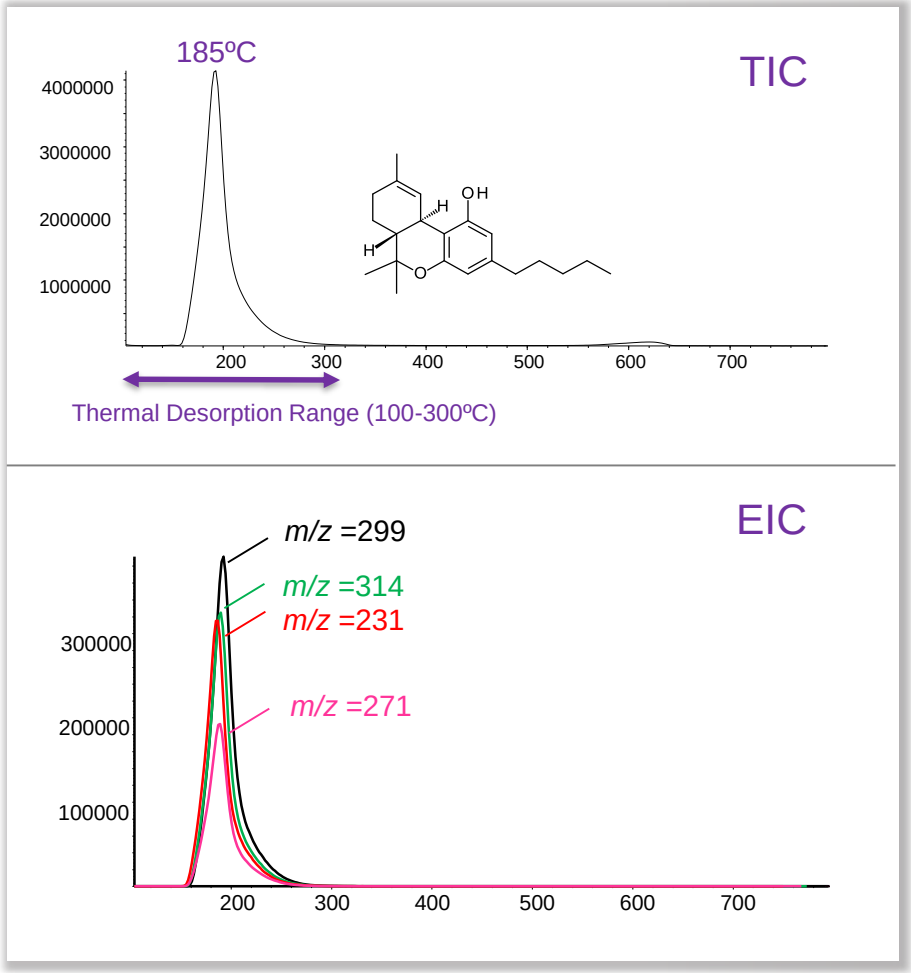


Fig. 1 EGA Thermogram of THC Standard & Extracted Ion Chromatograms (EICs)

Analysis of Cannabis Infused Chocolate Brownie

TD-GC/MS analysis was then performed on the brownie bite in triplicate as shown in Figure 2. To perform TD-GC/MS analysis, the pyrolyzer furnace was programmed from 100 to 300ºC (100ºC/min) after the EGA tube was replaced by a separation column (easily facilitated by using the vent-free GC/MS adaptor). The amount of sample used to obtain the TD chromatograms was 0.25-0.26 mg.

The peaks shown in Figure 2 (*) at 12.4 minutes were identified as THC using the MS interpretation library. The RSD% of 4.96% was calculated based on the area counts of the THC peak. The most intense peak around 6 minute was identified as 5-Hydroxymethylfurfural (dehydration of sugar).

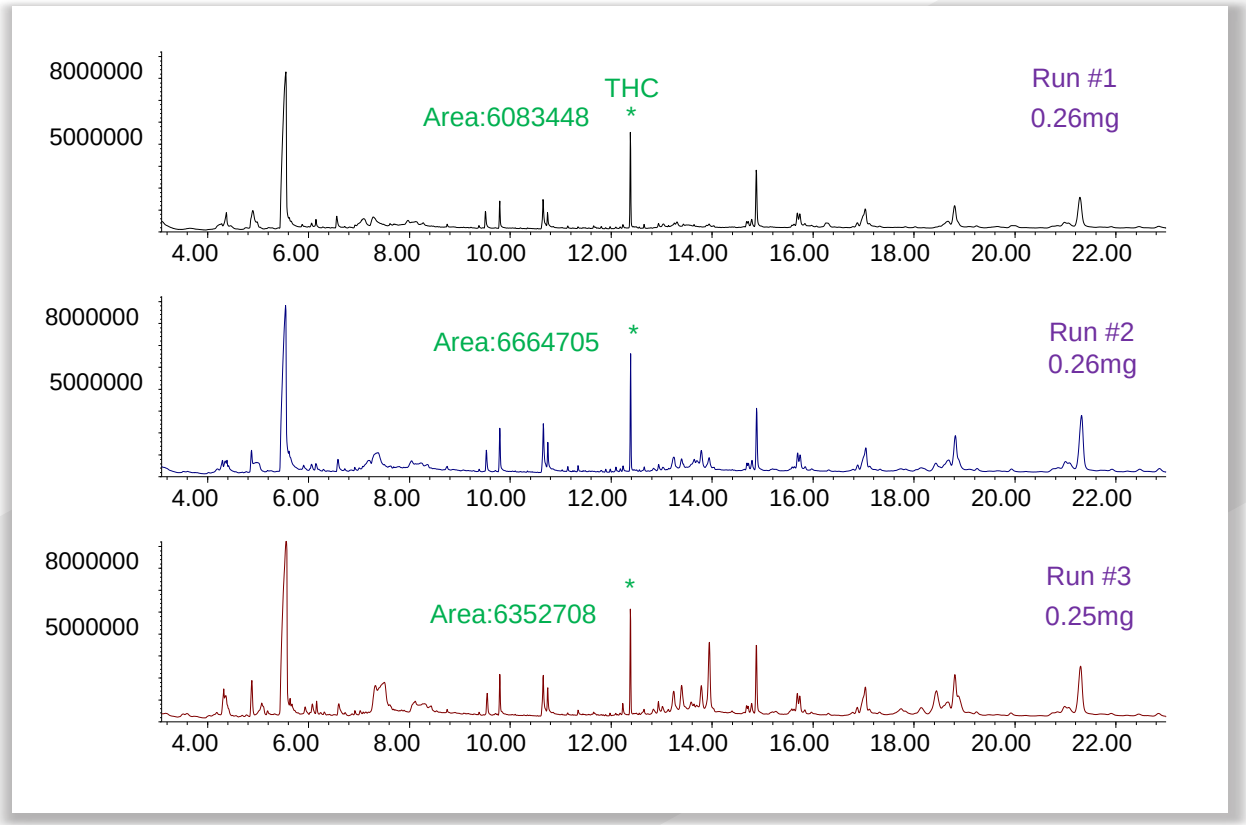
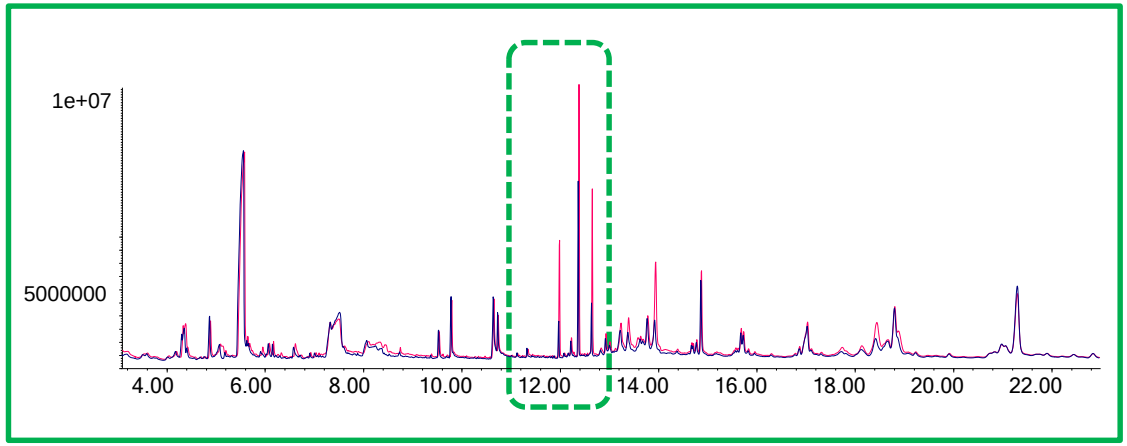
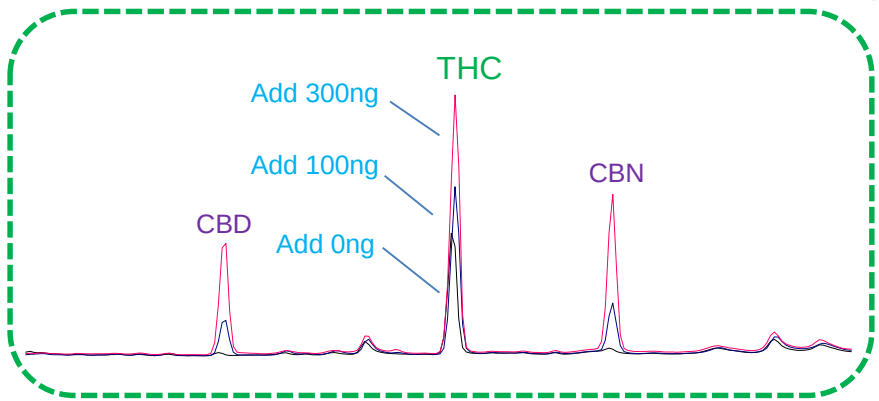


Fig. 2 TD-GC/MS Chromatograms of the Brownie Bite in Triplicate RSD 4.96%

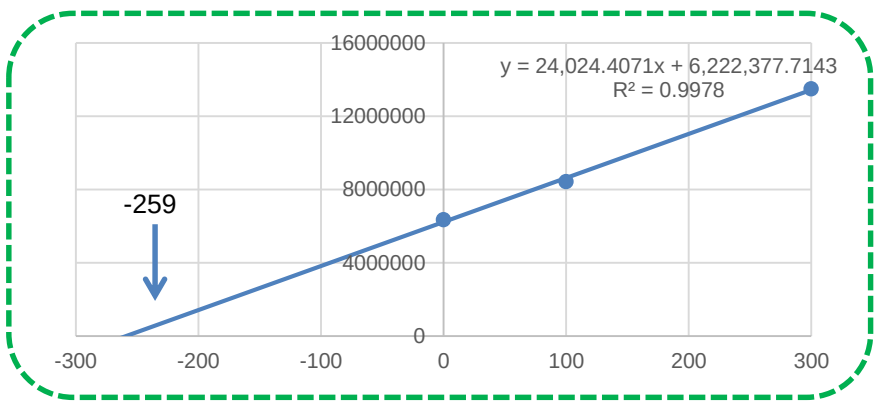
Analysis of Cannabis Infused Chocolate Brownie

To confirm the THC concentration in the brownie bite, standard addition calibration curve was created. The standard solution was a mixture of CBD (Cannabidiol), THC, and CBN (Cannabinol). Figure 3 shows the calibration curve and calculated amount of THC in the brownie bite. Using the calibration curve, the THC concentration is calculated as 0.996mg/g which is in an excellent agreement with the product label indicating 1mg/g of THC.





*STD solution is the mixture of CBD,THC and CBN
Label THC
10mg/10g= 1mg/g



Brownie Standard addition calibration curve
X intercept : -259.0 (ng)
Sample weight 0.26mg
THC concentration: 996.2 ng/mg=0.996mg/g

Fig. 3 Brownie Sample Standard Addition Calibration Curve

Analysis of Cannabis Infused Chocolate Bar by TD-GC/MS

A commercial cannabis-infused dark chocolate bar with the total of 100 mg THC and net weight of 50g (1.7oz) is used. According to the product label, there are 20 pieces of chocolates and each piece of chocolate contains 5 mg of THC, so there is 100mg/50g=2mg/g of THC present in each piece. To demonstrate the accuracy and precision of the methodology, the analysis was performed in triplicate. The weights of each piece were recorded using an analytical balance as 0.099 mg, 0.097mg, and 0.105mg.

The same methodology (the “Method Map” described in page 19) as the chocolate brownie sample was used for analyzing the chocolate bar. First, the EGA was performed as the rapid screening technique. Then the optimal thermal desorption zone of THC was identified. The pyrolyzer furnace was programmed from 100 to 300ºC (100ºC/min) to obtain the thermal desorption chromatograms for all three pieces of the chocolate. Figure 1 shows the TD chromatograms in triplicate.

The peaks indicated by (*) between the retention time of 12 to 14 minutes were identified as THC using the MS interpretation library. The RSD% of 7.18% was calculated based on the area counts of the THC peak.

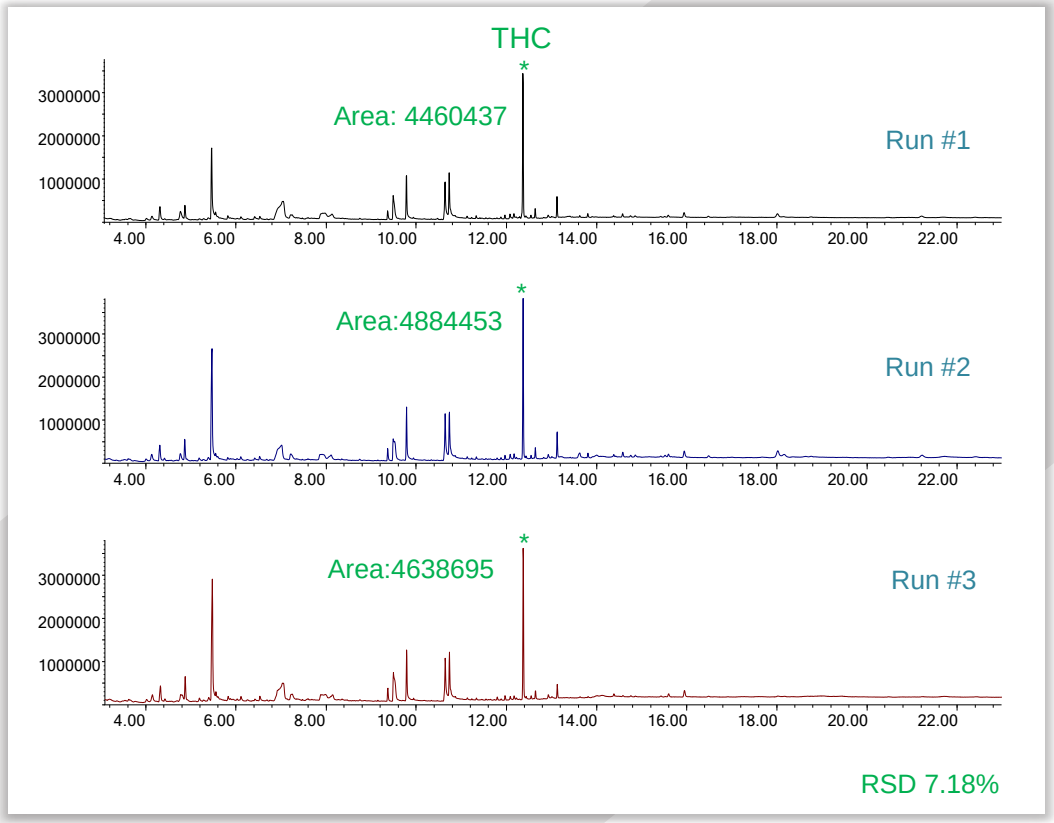


Fig. 1 TD-GC/MS Chromatograms of the Chocolate Pieces in Triplicate

Analysis of Cannabis Infused Chocolate Bar

To confirm the THC concentration in the chocolate pieces, Standard Addition calibration curve was created. The standard solution was a mixture of CBD (Cannabidiol), THC, and CBN (Cannabinol). Figure 2 shows the calibration curve and calculated amount of THC in the pieces. Based on the calibration curve, the THC concentration is calculated as 2.1mg/g per piece which is in an excellent agreement with the product label (2mg/g of THC).

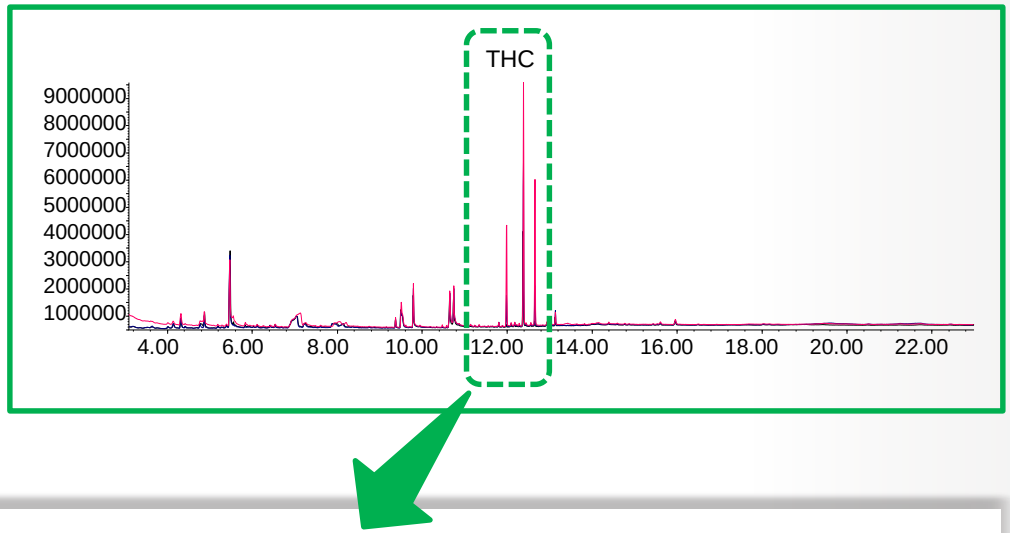


Fig. 2 Chocolate Pieces Sample Standard Addition Calibration Curve

Analysis of Cannabis Infused Tincture (Hemp Extract Pain Relief) by TD-GC/MS

A commercial hemp extract pain relief tincture sample with the net volume of 30mL (1oz) is used. According to the label, this product contains 100mg Hemp Extract (CBD) in the bottle. There is no specific label of each required cannabinoid concentration.
To perform the TD-GC/MS analysis, viscous liquid sample (milky lotion) was placed in the eco-cup and weighed on the analytical balance. The weight was recorded as 0.045mg.

The “Method Map” methodology is used in this experiment. Figure 1 shows the TD-GC/MS chromatograms of the tincture sample. There are many matrix peaks such as triglyceride or menthol. However, those matrix peaks did not cover CBD peak.

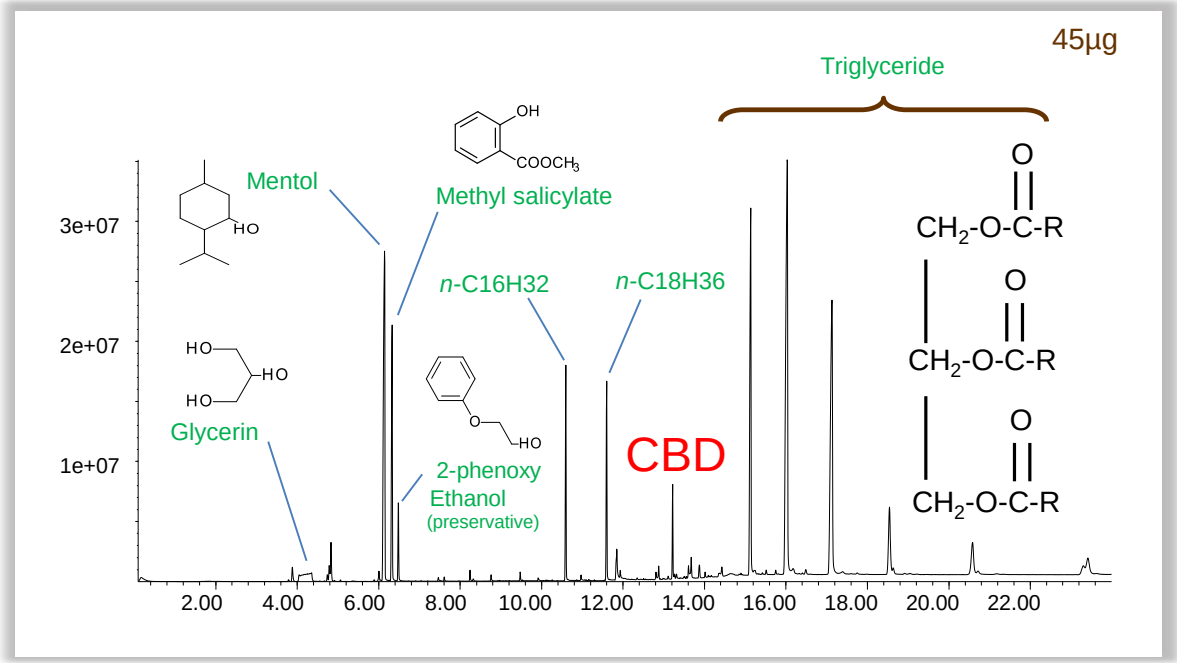


Fig. 1 TD-GC/MS Chromatograms of the Tincture Sample

The ingredients indicate that this product contains Hemp Extract. It is easy to confirm what cannabinoid(s) is (are) in the sample. There is no sample preparation. The sample was placed in the cup and analyzed with TD-GC/MS. When MS is used as the detector of GC, other ingredients can be identified simultaneously. CBD peak intensity is quite high, and it is close to be “saturated peak”.

Analysis of Cannabis Infused Tincture (Hemp Extract Pain Relief)

There are two choices for TD. One is “isothermal temperature 300ºC. The other one is “ramping temperature program” which is 100ºC-300ºC (100ºC/min). Both methods were performed and demonstrated the same results.

Depending on the sample shape, sample concentration and target compounds, “isothermal temperature method” and “ramping program method” can be chosen by analyst.

Figure 2 shows the comparison of “isothermal” and “ramping temperature program” for the tincture sample.

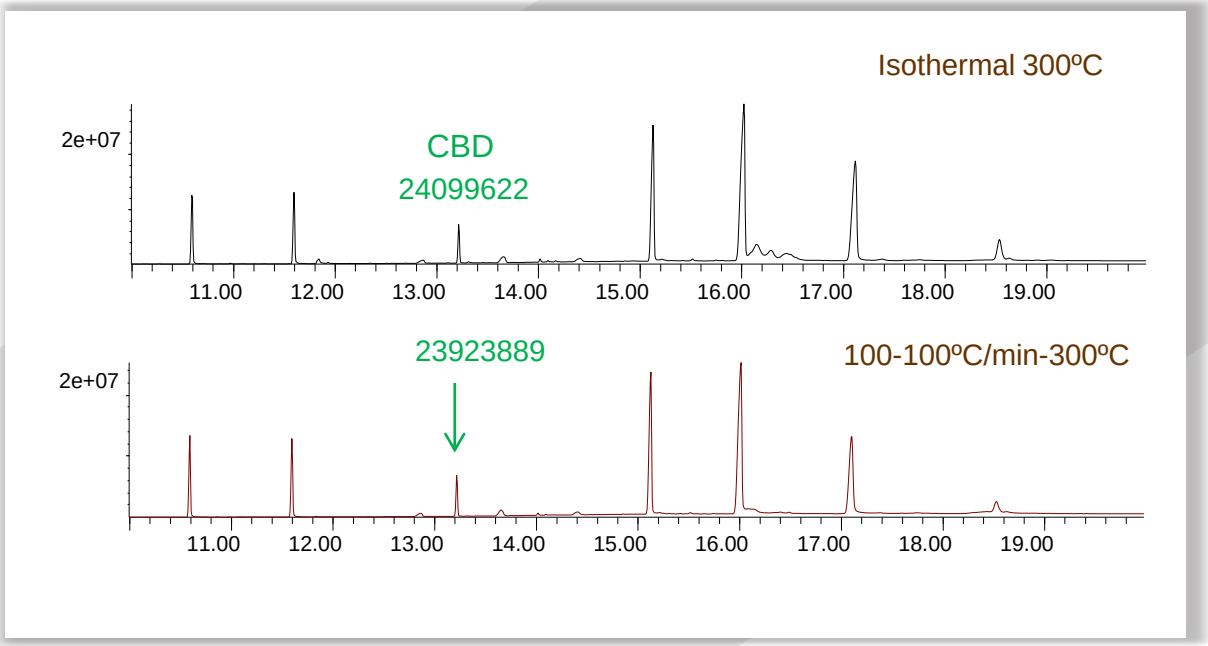


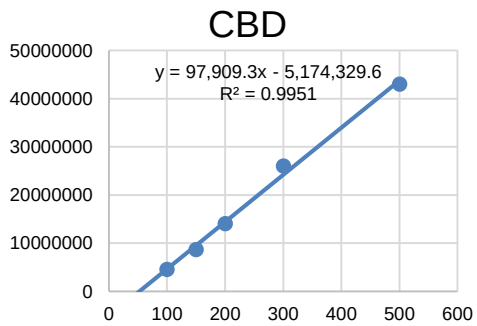
Fig. 2 Comparison of Two TD Methods (Isothermal & Temperature Ramping) for the Tincture Sample

Analysis of Cannabis Infused Tincture (Hemp Extract Pain Relief)

Next approach was solvent extraction to obtain a homogeneous sample. IPA was used to extract CBD from the tincture sample. 1mL of sample (milky lotion) was extracted by 2mL of IPA shaken for 10 minutes or by ultrasonic, then the extracted solution was diluted by IPA again until getting appropriate sample peak size.

Figure 3 shows the TD chromatogram of the extracted sample (5 runs) using 300ºC isothermal TD. The quantitation result using External Standard Calibration technique is also shown. The label indicates 100mg Hemp extra in the bottle and 89.1mg of CBD in the bottle (average in 5 times) is the quantitation result by TD-GC/MS.

Peak area (m/z=299)	Quant result (ng)	Quant result (mg)*
23587577	293.8	88.1
22050239	278.1	83.4
24651147	304.6	91.4
24099622	299.0	89.7
25095794	309.2	92.7
Avg.		89.1
RSD (%)		4.1



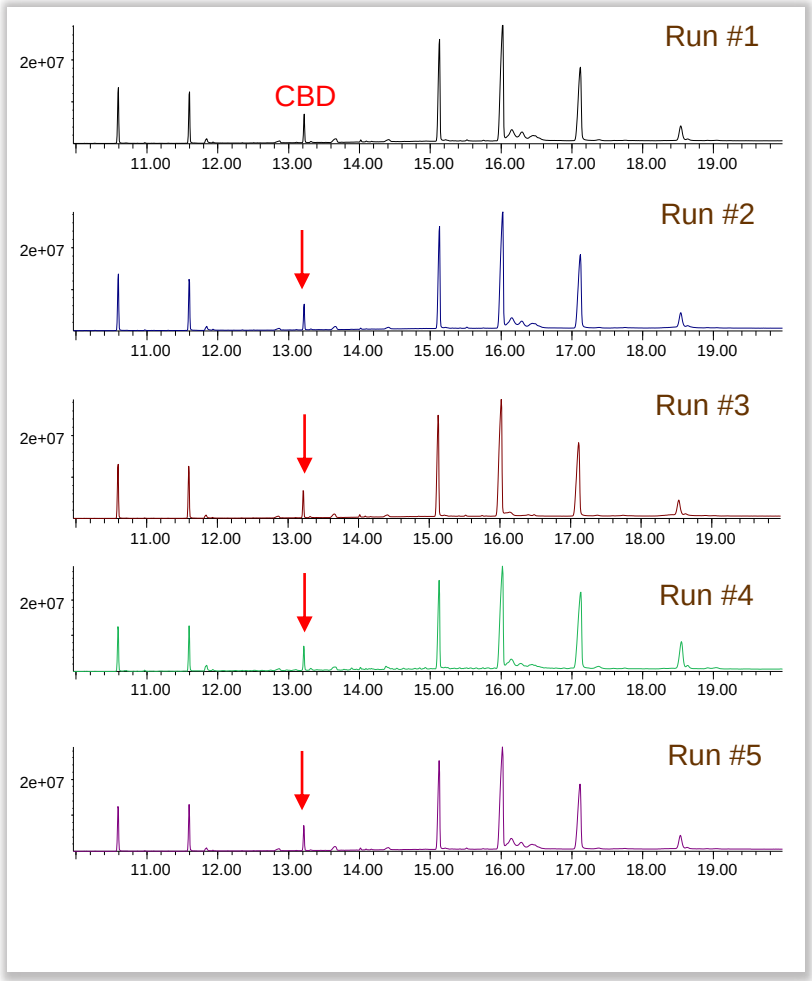


Fig. 3 Repeatability and Quantitation Result

*Tincture CDB concentration was calculated using sample size, dilution, and TD volume

Analysis of Cannabis Infused Candy by TD-GC/MS

A commercial individually wrapped candy containing 5mg of THC (according to the product label) is used for this experiment. To confirm the amount of THC from the label, the candy sample is analyzed using direct TD-GC/MS analysis as well as thin film technique.
Analysis of the solid is often referred to as a “direct” method. Analysis of a microliter aliquot of a quantitative solution of the sample is referred to as a “thin film” method.

From the EGA thermogram, the optimal thermal desorption zone of THC was identified as 100 to 300ºC.

For the direct TD-GC/MS analysis, the multi-mode Pyrolyzer's furnace was programmed at 300ºC isothermal. The candy sample was crushed and powered. Small amount of powdered candy (0.54mg) was placed in an Eco-cup to perform TD-GC/MS analysis (in this approach the candy was analyzed “as is” for direct method).

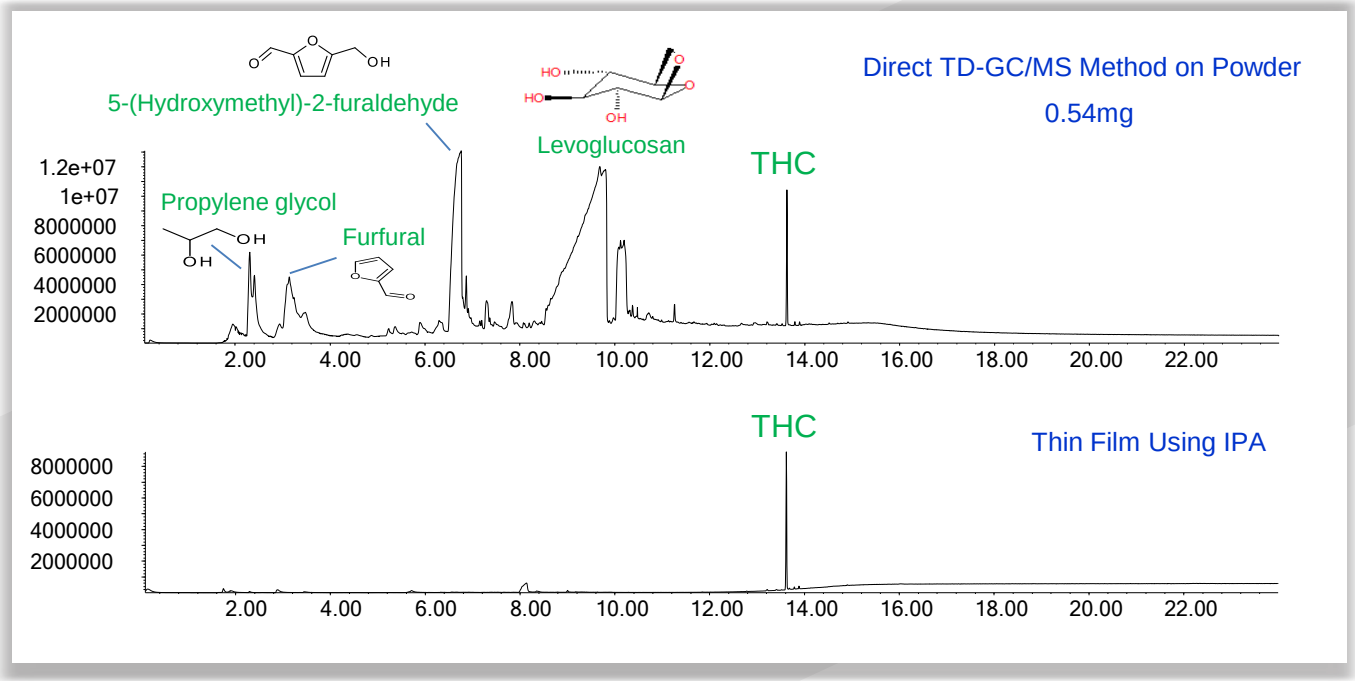


Fig. 1 Comparison of Direct TD and Thin Film Using IPA TD Chromatograms

Analysis of Cannabis Infused Candy

In the thin film method, 1.0 g of the powdered candy was mixed with 2mL of IPA. The mixture was then shaken and sonicated for about 10 minutes to extract cannabinoids.
2µL of extracted solution was spiked into the cup to perform TD-GC/MS analysis (TD temperature was 300ºC isothermal).

Figure 1 shows the obtained TD-GC/MS chromatograms of both analysis on the candy sample. THC is identified in both TD chromatograms.

Direct TD Quantitation		IPA Extraction Quantitation	
Run	Quantitation result (µg/g)	Run	Quantitation result (µg/g)
#1	876	#1	997
#2	807	#2	989
#3	1128	#3	1007
#4	1085	#4	971

Table. 1 Comparison of Quantitation Result Between Direct TD and Thin Film Methods

To calculate the THC concentration, External Standard (ESTD) was used. Table 1 shows the obtained results for n=4 of direct TD and thin film using IPA. From the calculated values, it is concluded that both techniques are in very close agreement with the product labeling (1mg/g) of THC.

It is important to note that the thin film using IPA approach in this experiment has less variation for quantitative analysis due to the nature of the sample. In fact, this experiment illustrates the importance of sample homogeneity when performing quantitative analysis. In this experiment, mixing the candy's powders with IPA and creating a solution provided a more homogeneous sampling in compare to the direct method.

Analysis of Cannabis Infused Soda by TD-GC/MS

A commercial cannabis-infused soda with a label total of 45 mg THC and net weight of 12FL OZ is used. According to an additional sticker label, the THC and CBN concentrations are 46.9mg/mL and 1.0mg/mL, respectively. However, the concentration of THC from the original label (total 45mg THC) does not match with the sticker label value (46.9mg/mL THC). To analyze the accurate value of THC concentration, TD-GC/MS technique is performed.

Using this technique the sample preparation step from traditional methodologies is eliminated. Some aliquot of the sample was spiked into the Eco-Cup to perform Thermal Desorption analysis. Due to the nature of the sample as a soda, the sample was not shaken prior the spike. Using a glass syringe two samples were collected from different sections of the beverage container; the top and the bottom sections.

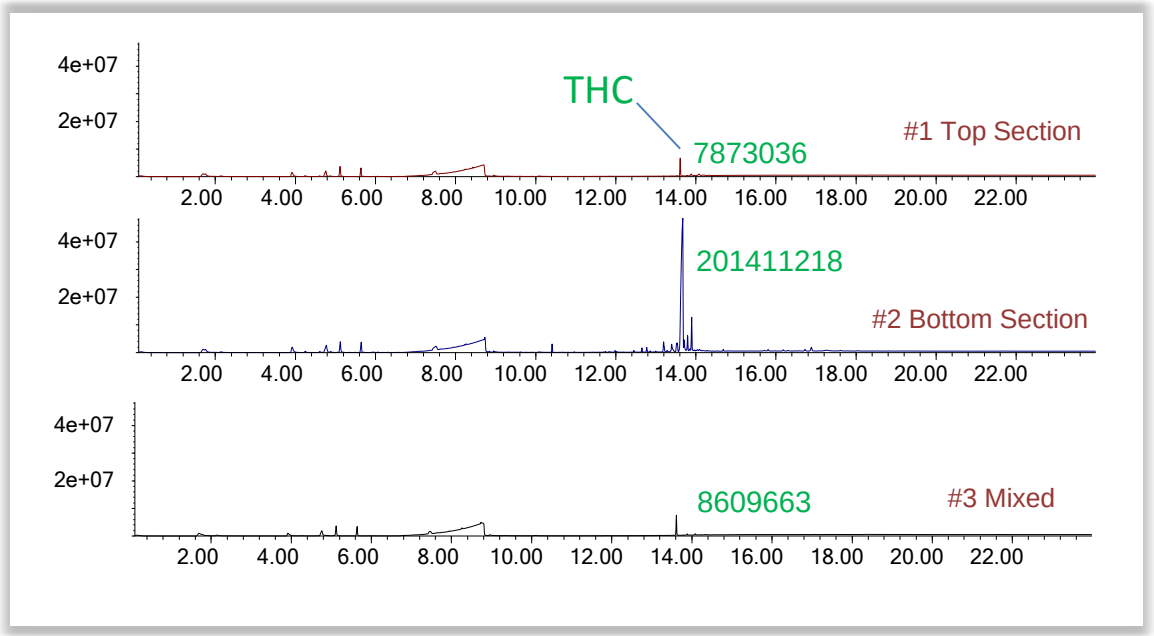


Fig. 1 TD-GC/MS Chromatograms of the Samples from Top and Bottom Sections of the Beverage Container as well as the Mixed Solution

Figure 1 shows the TD-GC/MS chromatograms (Isothermal TD at 300ºC) of the sample collected from the top section of the can as well as the sample collected from the bottom. As the obtained chromatograms indicate the solution inside the can is not homogenous. To make homogeneous sample solution, the entire solution was sonicated for 10 minutes to remove CO_2 gas from soda, then stirred gently on Magnetic stirrer with stirrer rod. Using the syringe, 3µL of the mixed and homogenous solution was collected to perform TD-GC/MS analysis (Isothermal at 300ºC). The obtained TD-GC/MS of the mixed solution is also shown in Figure 1.

Analysis of Cannabis Infused Soda

After mixing the solution, the repeatability of chromatogram was improved. However, the quantitation result by ESTD were lower than the product label which is “total 45mg THC” or “46.9mg/mL THC”. To further investigate the amount of THC, all the liquid from the can was transferred to a glass beaker and the inside of the can was rinsed with IPA. TD-GC/MS was performed on the aliquot of the IPA rinsed.

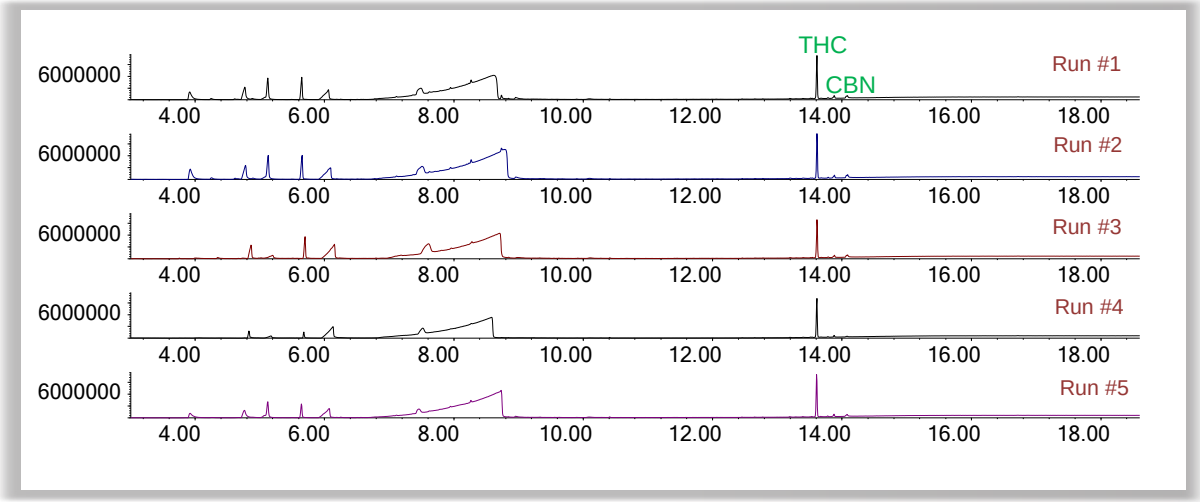


Fig. 2 The repeatability of TD-GC/MS Chromatograms and Quantitation Result of Soda

It turned out that 7.7mg of THC was left on the surface of the can. This finding revealed the reason why the original quantitation result was lower than expected. The total amount of the original quantitation result (34.6mg/can) and the leftover on the surface of the can (7.7mg/can) is 42.3mg/can. The value is in close agreement to the label with 45mg THC.

It is important to note that in the manufacturing process of cannabis-infused beverages, different techniques are used to create emulsion of cannabinoids as THC does not dissolve in water.
In some instances the emulsion is not stable. As the result, the cannabinoids concentration of beverage differs depending on the sampling place in the beverage container. [1]
This experiment demonstrates the applicability of TD-GC/MS for analyzing an accurate concentration of the cannabinoids present in different types of cannabis-infused drinks and beverages. This technique thermally extract the cannabinoids from the mixture while eliminates the sample preparation or solvent extraction steps.

Analysis#	mg / can	
	THC	CBN
#1	35.4	2.7
#2	37.5	2.6
#3	31.5	2.2
#4	32.5	2.1
#5	36.3	2.4
Avg.	34.6	2.4

[1] White paper: The Art and Science of Cannabis Beverages. Le Herbe: a 420 Venture Company (Page 19)

Analysis of Cannabis Infused Gummy by TD-GC/FID

A commercial package of THC-infused gummy is used for this experiment. The package contains one piece of gummy with the net weight of 15g. The product label states 60mg THC and 30mg CBD. To confirm the product label and perform TD-GC/MS based on the “Method Map” methodology, 0.16g of the gummy sample was dissolved in 5mL IPA: water=1:1 solution. Then 5 µL of the solution was spiked in an Eco-Cup to perform TD-GC/FID.
This experiment illustrates the applicability of FID as the detector instead of MS. FID has a wide dynamic range and provides stable data. External Standard (ESTD) and Standard Addition calibration techniques were used for the quantitation. To confirm the repeatability of the chromatograms and calculated results for CBD, THC, and CBN. TD-GC/FID analysis was performed 15 times. The RSD% for n=15 were calculated as CBD=1.6%, THC=1.6%, and CBN=1.2%. The obtained low RSD values indicate the high repeatability of the analysis. Figure 1 shows the overlaid chromatograms of 15 runs.

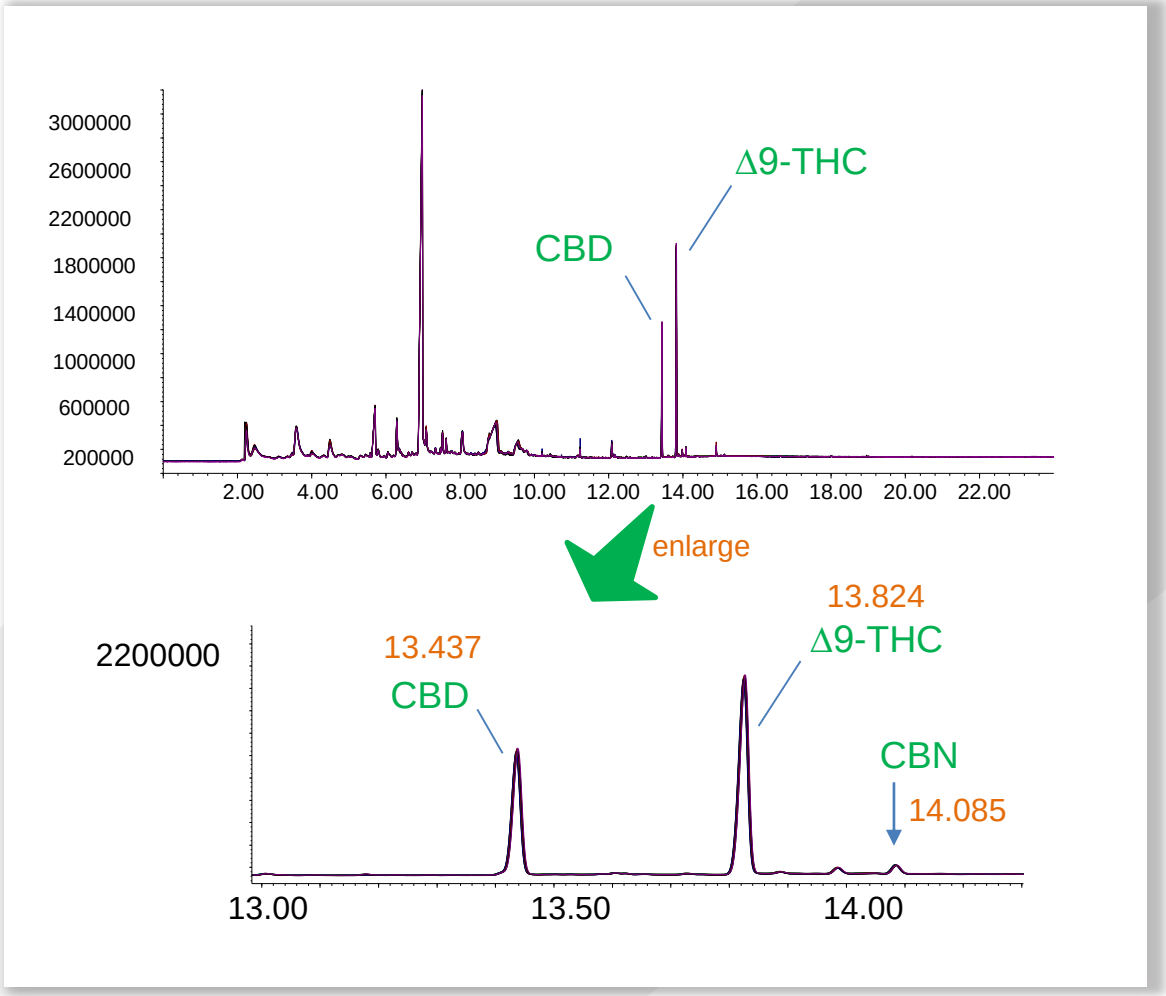


Fig. 1 The Overlaid Chromatograms of the15 Runs for the Gummy Sample

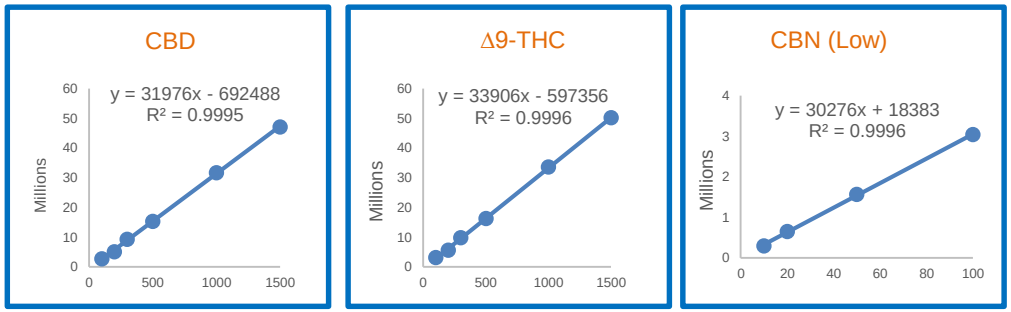
Analysis of Cannabis Infused Gummy

The Standard mix (CBD, THC, and CBN) solution was added to the sample cup and calibration curve was established for ESTD technique. The Thermal Desorption (TD) temperature was set as 300ºC isothermal. CBN peak was much smaller than CBD and THC in the sample. So a CBN low concentration calibration curve was also established as shown in Figure 2.

The calibration curves for CBD, THC, and CBN were also obtained using the Standard Addition technique. The gummy solution was spiked in several cups and then the standard mix (CBD, THC, and CBN) solution was spiked 0, 100, 300, and 600ng. Due to the low concentration of CBN presence in the sample, a low calibration curve was created for CBN.

Figure 3 (next page) shows the obtained calibration curves using Standard Addition as well as the quantitative results.

The calculated values from Figure 3 indicates that the CBD concentration is slightly higher than the product label. One benefit of FID is that ECN (Effective Carbon Number) can be used to estimate the relative responses for any compounds. As shown in Figure 4, CBD and THC has similar ECN. [1][2]



mg/package (15g)	CBD(mg/pack)	THC (mg/pack)	CBN (mg/pack)
Trial#1	38	57	2.5
Trial #2	37	56	2.5
Trial #3	38	58	2.5

Fig. 2 ESTD Calibration Curves and the Quantitation Result

Compound	ECN(1)	ECN(2)
THC	19.15	19.48
CBD	19.30	19.52
CBN	19.25	19.58

Fig. 4 Effective Carbon Numbers of Cannabinoids According to 2 Different Methods

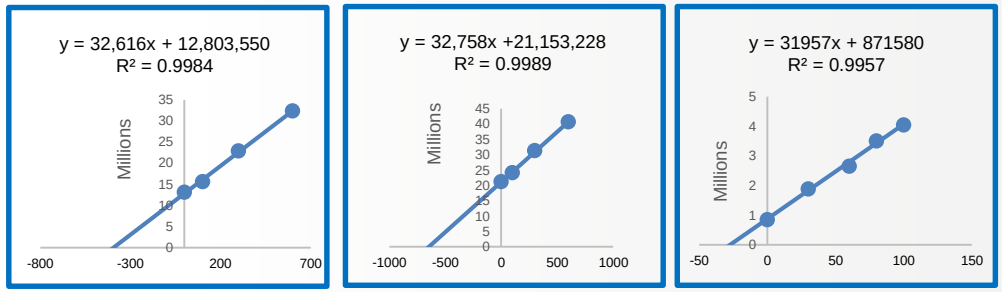
Analysis of Cannabis Infused Gummy

Figure 5 demonstrates one example of a STD (each 400ng) chromatogram.

The peak area of CBD and THC is similar. The obtained values confirm the concept of similar ECN values for CBD and THC.

According to the product label, there are 30mg CBD and 60mg THC in the gummy package. Based on the label, THC peak area should be approximately twice as the CBD peak area. The average peak areas of CBD and THC were calculated as 11792954 and 19506798, respectively from the 15 chromatograms ran for repeatability.

The calculated values of the peak areas indicate the ratio of 0.6 to 1. Also as the quantitation results of External Standard and Standard Addition indicate (shown in Figure 6), the CBD and THC concentration ratio is similar to the peak areas ratio (0.6:1).



CBD amount (ng) Δ9-THC amount (ng) CBN amount (ng)

mg/package (15g)	CBD(mg/pack)	THC (mg/pack)	CBN (mg/pack)
	38	62	2.3

Fig. 3 Standard Addition Calibration Curves and Quantitation Result

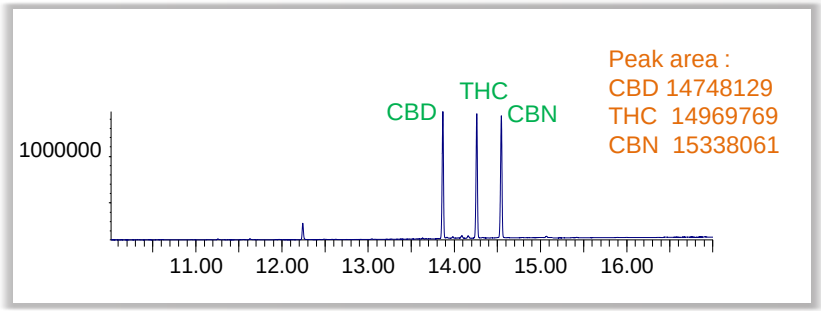


Fig. 5 Standard (CBN, THC, and CBN) Each 400ng FID Chromatogram

Analysis of Cannabis Infused Gummy

From the experimental results, it is concluded that the product label does not report an accurate concentration of the cannabinoids. In fact, CBD and THC ratio is higher in the sample.

This experiment illustrates the capability of FID to evaluate cannabinoids ratios in a sample due to the similarity of ECN values for CBD, THC, and CBN. It is also determined that as the product or standard solution get older, THC peaks becomes smaller and CBN peaks larger.

FID also provides insight about the product and its shelf life.

mg/package (15g)	CBD(mg/pack)	THC (mg/pack)	Ratio (CBD/THC)
ESTD result			
Trial#1	38	57	0.67
Trial #2	37	56	0.66
Trial #3	38	58	0.66

mg/package (15g)	CBD(mg/pack)	THC (mg/pack)	Ratio (CBD/THC)
	Standard Addition result		
	38	62	0.61

Fig. 6 Quantitation Result and the Ratio (CBD/THC)

[1] Suurkuush, G. (2010), Validation of the Gas Chromatographic Method for THC, CBD, and CBN Differentiation. University of Tartu: Faculty of Science and Technology.
[2] Poortman-van deer Meer, A.J. & Huizer, H., A Contribution to the Improvement of Accuracy in the Quantitation of THC. Forensic Science Laboratory of the Ministry of Justice, Volmerlaan 17, NL-2288 GD Rijswijk, Netherlands. Forensic Science International 101 (1999) 1-8, Elsevier Science Ireland Ltd (1999).

Analysis of Cannabis Infused Cookie by TD-GC/FID

A commercial package of cookies is used in this experiment. The package label indicates 100 mg of each THC and CBD. There are a total of 10 cookies in the package with the net weight of 100g. So the concentration of THC and CBD is 1mg/g (100mg/10g) each. Using a commercial coffee grinder, one of the cookies was grounded. 0.5g of the cookie powder was then mixed with 5mL of IPA. The mixture was then shaken (ultrasonic can also be used). 10µL of the solution was then placed in the sample cup to perform thermal desorption analysis.

This experiment is performed by FID as well as MS as the detector. Following the "Method Map" methodology and based on the identified optimal TD zone for THC from the EGA, the TD-GC/FID was performed at 300ºC isothermal. For quantitative analysis, External Standard and Standard Addition were used. To demonstrate the high repeatability, the analysis was performed 15 times and the RSD% for CBD, THC, and CBN were calculated as 1.4%, 1.7%, and 5.8%, respectively (CBN peak is very small). Figure 1 shows the overlaid chromatograms and the cannabinoids identification.

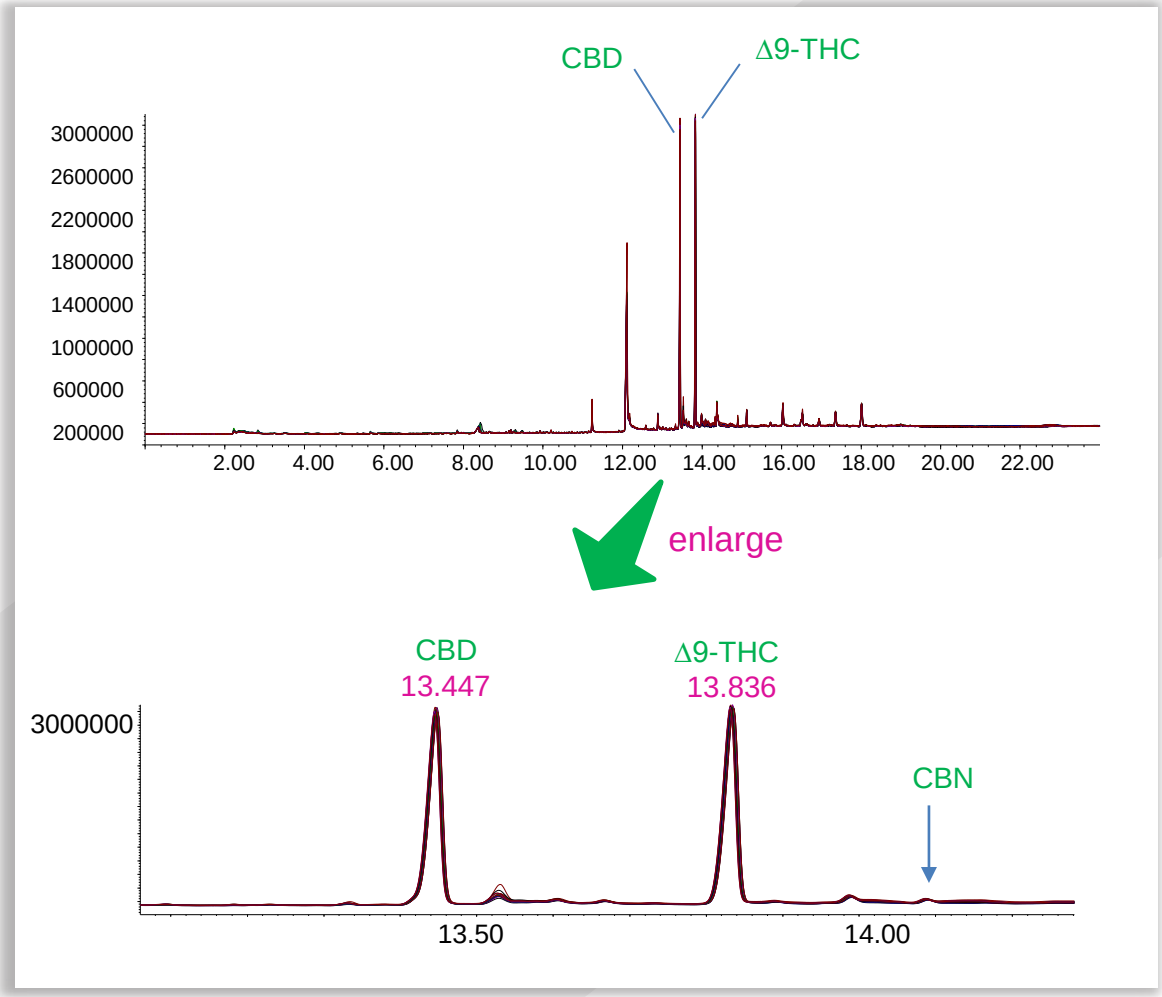
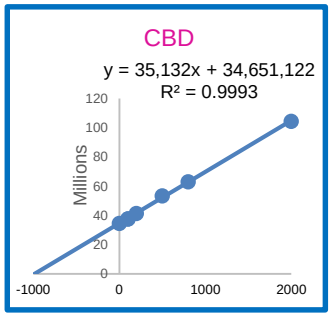


Fig. 1 The Overlaid Chromatograms of the15 Runs for the Cookie Sample

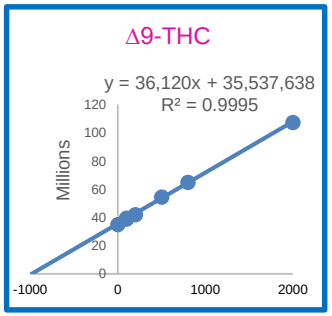
Analysis of Cannabis Infused Cookie

Using the ECN concept to the obtained FID chromatograms, it can be concluded that the response factor for CBD and THC are almost the same. The FID allows the analyst to utilize the External Standard calibration curve for multiple different samples. The gummy's External Standard calibration curve was used to perform the quantitation analysis on the cookie sample as shown in Figure 2.

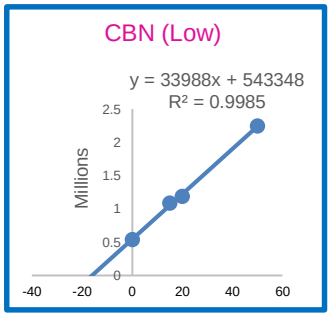
The Standard Addition curve was created for the cookie sample by spiking the cookie and IPA solution in several cups and then spiking standard mix (CBD, THC, and CBN) solution at 0, 100, 300, and 600 ng. A low concentration calibration curve was also created for CBN as the identified peak was small. Figure 3 shows the obtained calibration curves using Standard Addition as well as the quantitative results.



CBD amount (ng)



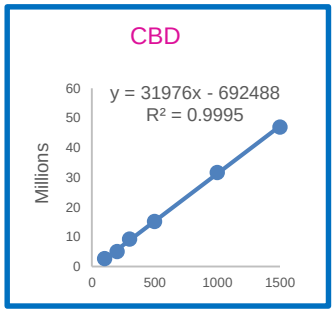
Δ9-THC amount (ng)



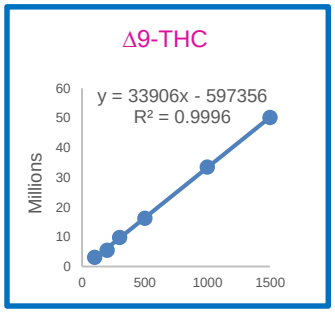
CBN amount (ng)

mg/package (100g)	CBD(mg/pack)	THC (mg/pack)	CBN (mg/pack)
	98.6	98.4	1.6

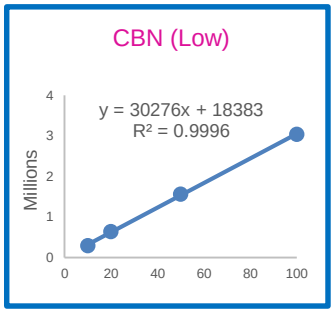
Fig. 3 Standard Addition Calibration Curve and the Quantitation Result



CBD amount (ng)



Δ9-THC amount (ng)



CBN amount (ng)

mg/package (100g)	CBD(mg/pack)	THC (mg/pack)	CBN (mg/pack)
Trial#1	110	105	1.5
Trial #2	106	102	1.5
Trial #3	108	102	1.4

Fig. 2 ESTD Calibration Curve (same as gummy ESTD curve) and the Quantitation Result

Analysis of Cannabis Infused Cookie

TD-GC/MS (using MS as the detector) was also performed on the cookie solution (mixed with IPA) at 300ºC isothermal. Using the MS interpretation library, CBG (* labeling) was identified from the TD-GC/MS chromatogram.

The quantitation result summary is shown in Figure 5. The product label indicated 100mg CBD and 100mg THC. The quantitation result of both techniques are in an excellent agreement with the label.

mg/package (100g)	CBD (mg/pack)	THC (mg/pack)	CBN (mg/pack)
ESTD result			
Trial#1	110	105	1.5
Trial #2	106	102	1.5
Trial #3	108	102	1.4
Standard Addition result			
mg/package (100g)	CBD (mg/pack)	THC (mg/pack)	CBN (mg/pack)
	98.6	98.4	1.6

Fig. 5 Quantitation Result Summary

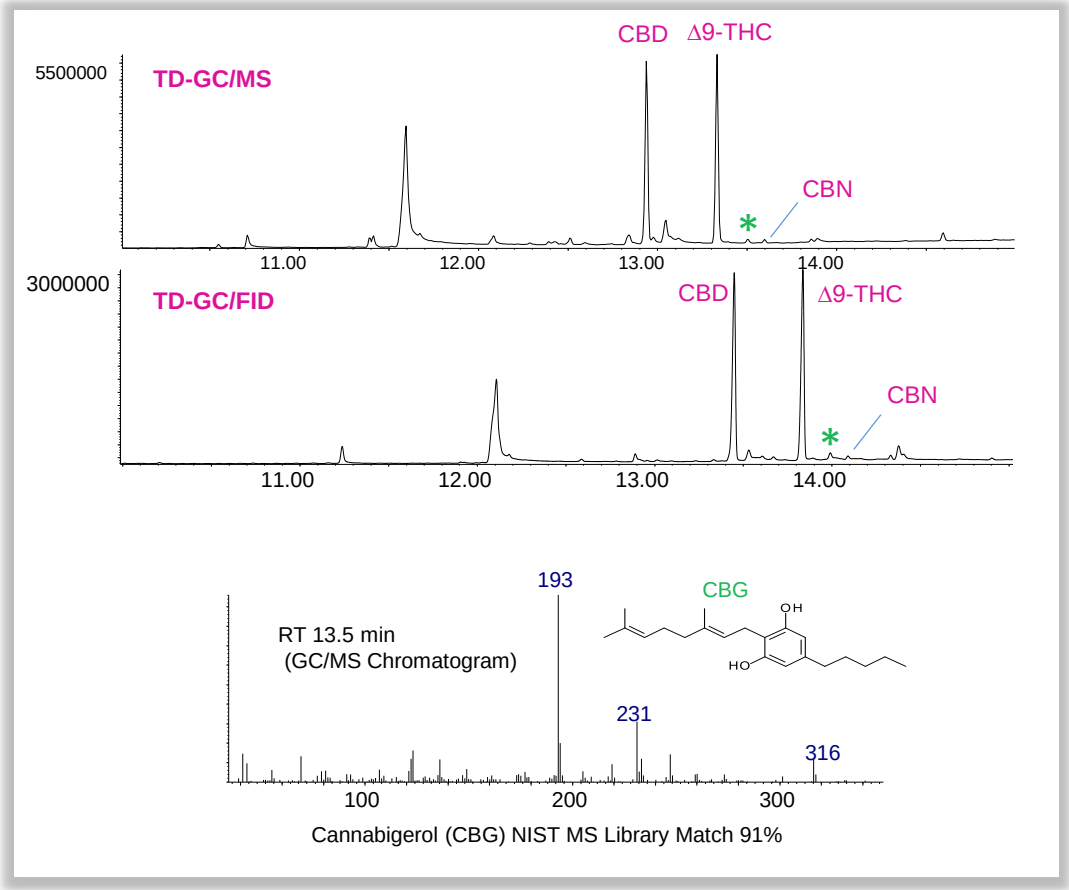


Fig. 4 TD-GC/MS and TD-GC/FID Chromatograms of IPA Extracted Solution and MS Spectrum of RT 13.5min peak

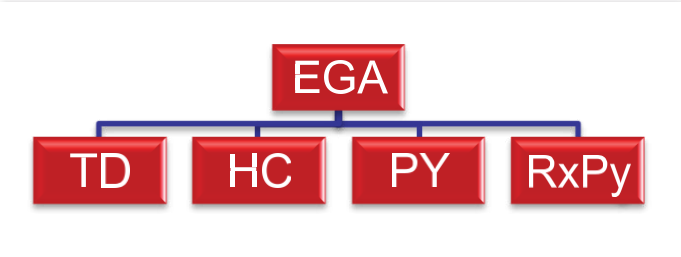
“Method Map” for Material Characterization

Frontier Lab has developed a sequence of tests referred to as the “method map” to chemically characterize samples using the EGA/PY-3030D Multi-Functional Pyrolyzer System in conjunction with a benchtop GC/MS. This sequence is applicable when characterizing virtually any organic material from volatiles to high molecular weight polymers.

The **“method map”** provides scientists with two simple steps for determining the organic composition of any unknown material:

i.

The first step is to perform an Evolved Gas Analysis (EGA). In this technique, the sample is dropped into the furnace which is at a relatively low temperature (ca. 40-100 ˚C). The furnace is then programmed to a much higher temperature (ca. 600-800 ˚C). Compounds “evolve” continuously from the sample as the temperature increases. A plot of detector response versus furnace temperature is obtained.



ii.

The second step is to use the EGA thermogram and selected ion chromatograms (EIC) to define the thermal zones of interest and then perform one or combination of the following techniques:

Use the links below for more information.

Thermal Desorption (TD)

Flash Pyrolysis (Py)

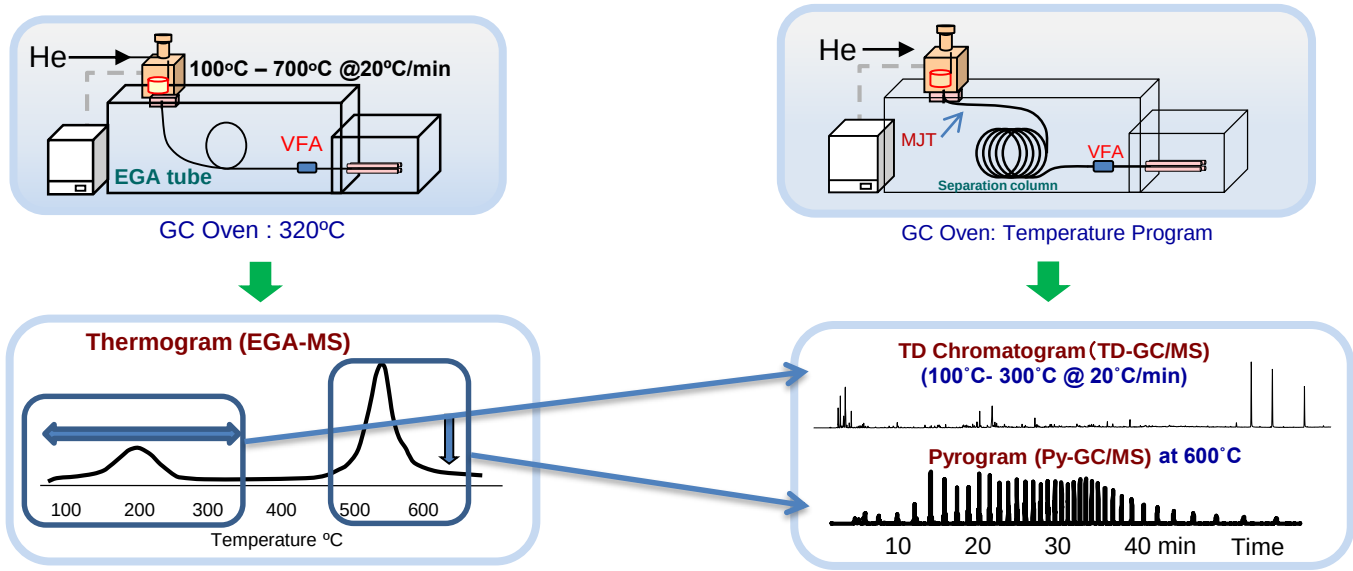
Heart Cutting (HC)

Reactive Pyrolysis (RxPy)

EGA & "Method Map"

EGA CONFIGURATION: No column is used; a short, small diameter (1.5m X 0.15mm id) deactivated tube connects the injection port to the detector. All thermal zones (interface temperature, GC injection port, column oven and detector cross-over) are held at elevated temperatures to prevent condensation. The figure below shows the EGA-MS configuration and a typical EGA thermogram

Following EGA, the instrument is re-configured. The EGA tube is replaced by an analytical column. The Frontier Vent-Free Adaptor enables this to be done easily and quickly; there is no need to vent the MS. MS vacuum equilibrium is re-established within a few minutes, and the exposure of the ion source to oxygen is minimized.



This process which refers to Evolved Gas Analysis (EGA) starts with the acquisition of a thermal profile (i.e., detector response as a function of sample temperature) of each sample type.
The sample is dropped into the furnace at a relatively low temperature (40-100ºC). The furnace is then programmed to a much higher temperature (600-800ºC). Compounds "evolve" continuously from the sample as the temperature increases. A plot of detector response versus furnace temperature is obtained. Extracted ion chromatograms (EIC) are used to identify the thermal zone over which specific compounds of interest evolve from the sample.

EGA & "Method Map"

Now, these optimum TD temperatures can be used in subsequent TD-GC/MS experiments to introduce the key components of interest while minimizing introduction of the matrix.

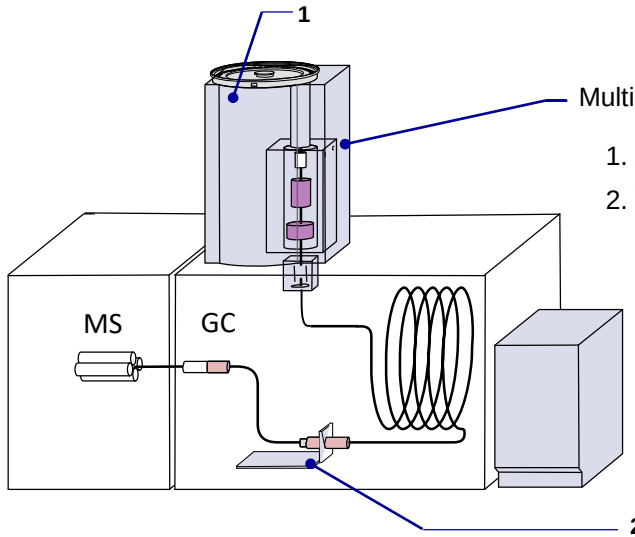
Only this portion of the sample is actually transferred (i.e., injected on) to the analytical column. Injecting only a small portion of the sample provides immediate benefits to the laboratory:

- The high boiling fraction of the sample remains in the sample cup. This eliminates the need for a high-temperature bake out. Thus, column lifetime increases, there is little to no system contamination and run-to-run cycle time decreases.
- More sample can be put in the sample cup which has the effect of lowering detection limits – without affecting instrument performance or cycle time.

With respect to the analysis of cannabinoids, it is important to keep in mind that TD-GC/MS is based on the volatilization of target compounds from the matrix. Those compounds that are thermally labile or easily converted to a alternative compound need to be identified. In these instances, it is the reformed compounds that are identified and monitored. Decarboxylation is forced to completion which could give the "screening determinations" higher values: the concentration range increases and dilution factors more accurately determined.

Using Thermal Desorption GC/MS, solid samples can be analyzed. There is no solvent required as opposed to traditional GC/MS techniques. In other words, the solid and liquid samples can be injected (using an inert sample cup) into the Pyrolyzer without any solvent and sample pretreatment. This advantage, as well as rapid screening capability, is one of the primary reasons many laboratories integrate the multi-mode Frontier Pyrolyzer into both their day-to-day QC and analytical research protocols.

TD-GC/MS
System Configuration



Use these links for more information.

Multi-Mode Pyrolyzer (EGA/PY 3030D)

1. Auto-Shot Sampler (AS-1020E)
2. Vent-Free GC/MS adapter

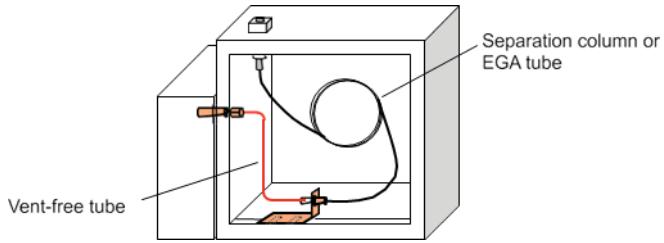
1. Auto-Shot Sampler (AS-1020E)

Up to 48 samples can be automatically analyzed using any of the analytical modes (e.g., TD, Py, Double-Shot, Heart-Cutting. Etc) with enhanced reliability.

Pyrolyzer is located in the housing of Auto-Shot Sampler.

2. Vent-Free GC/MS Adapter

Without venting MS, separation column and/or EGA tube can be switched.



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- **India**
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