APPLICATIONS

Update . . . >Jan 2016

re-published by Chromtech Jan 2016

Medical Cannabis / Marijuana

Page PIC Index TOC

Some prelim work . . . By Restek

- RESIDUAL SOLVENTS / PESTICIDES In Cannabis Extracts
- Detailed Quechers Extracts PLUS GCxGC TOF-MS
  - but Note . . . MS is a bit limited for cannabinoids, terpenes etc

Derivatisation of Cannabinoids - *proved "perfect" . . . but be wary of matrix effects!*

Cannabinoid Standards / Terpenes also available from Restek

some GC Configurations *(specialised)*

Some practical HINTS re GC set up / accessories

DISCLAIMER . . . In Australia use of Cannabinoids and even R&D on such STILL seems illegal (or at least highly restricted) on a State by State basis and potential customers require full ID and possible registration/certification for ANY purchase from Chromtech / work being done in this field

FULLY at customers responsibility - No authenticity . . . No Sale !

NEW 2015+ WebSITE / SHOPPE

www.chromalytic.net.au

Update 2016-6

SRI Gas Chromatographs

: Custom configurations › Application specific

Medical Cannabis 8610C GC
8610-0091 ~AUD18,000

8610C Gas Chromatograph for . . .

Medical Cannabis Analysis

- ***Medical Cannabis Gas Chromatograph Automated Hi-volume Configuration : 8610V***
- ***Medical Cannabis Gas Chromatograph (GC) Configuration choices February 2011.***
- ***Medical Cannabis Potency Testing using the SRI 8610C FID GC.***
- ***Medical Cannabis Pesticide Screening using the SRI 8610C GC.***
- ***Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC.***

Cannabis not Marijuana — drug policy australia

New 2016 *Model 420 Cannabis Potency GC*

- *THC/CBD . . . ALL inclusive accessories*
- *LOW COST; portable*
  8610-0420 ~AUD7000
- *built-in H2 Gen + Air*
- *BUT with limited column efficiency compromise*

Disclaimer / preamble . . .

The use of Cannabis in Australia is “arguably” deemed illegal . . . despite being legalised in many States and Countries for personal and/or medical use.

Governments seem more interested not necessarily in health issues but more-so of potential loss of revenue through difficult to control naturally grown and controlled substances like tobacco and alcohol. Deemed to be “protected” these substances are under more and more severe taxation tariffs . All this despite arguably both being proven to have far more damaging health and social problems.

Vested interest are the prime concerns and these include the medical/pharmaceutical legal and political “system” as well as the attracted criminal element all out to exploit the publics naivety an/or gullibility.

Chromalytic Tech does NOT condone the use of such illicit materials even though it may actually ameliorate the adverse social consequences otherwise resulting from social pressures arising as a consequence of poor government, management decisions etc.

Proven “hard” drugs where health, safety, addiction are a different issue and controls are obviously required.

There is in fact a valid argument for the legalisation of all drug use with proper quality control

Up to a point authorities seem to condone limited use of "reasonable" possession use of various drugs as they realise wide-spread cultivation is so easy
to produce but difficult if not impossible to enforce.

We’d argue that to reduce the huge criminal repercussions . . . better to control the production through sensible licensing and Quality Control testing of product to minimise adulteration/ dilution by producers through drug peddlers and unscrupulous re-sellers all out to exploit end-users.

At best all of these "middle men" are unaware of the risks involved in converting raw product in safe materials re pesticide contamination, solvent extraction impurities let alone the actual potency variances due to genetics and growth factors.

Trivial "saliva" and "potency" test kits are to varying degree legalised in this context but are for all intents and purposes of minimal usefulness except for
ill-defined law enforcement purposes.

Gas Chromatography (and perhaps to a lesser extents ***HPLC*** being less affordable and more complex) is recognised as a relatively simple low cost QC technique. HPLC by comparion is another promising complementary albeit more expensive technique.
Under suitable Laboratory control, licensing etc. with proper technical supervision. . . of course !
For "Cannabis" GC has been well researched and documented to the point where effective low cost Quality Control is now possible.

Chromalytic Technology now offers such GC equipment to qualified Labs and researchers (for R&D purposes).
By definition GC is such a Universal technique NO GC system can be defined as being applicable to marijuana ONLY!
(or for that matter any other drug testing purpose)

OUR DISCLAIMER :

"Catch-ALL legalities" Buyer BEWARE . . . To prevent diversion of such or other equipment that might be somehow related to potential drug manufacturethe authorities have deemed that in their "wisdom" anything can at any time and at their discretion be declared "for restricted use Only"

Analytical Test equipment in general ***including GCs*** and in principle are NOT classified.

Chromalytic Technology will NOT supply such equipment to unauthorised end-users.
- ***enquire !*** . . .Re setting up an account with **Chromalytic Technology** with full trace-ability; ID etc
- Declaration as to the intended use. . . *may still be required !*

We accept NO Responsibility whatsoever for the use or misuse of such equipment . . . either legal or illegal !
Any results and interpretation of such and ANY resulting consequences are entirely at the end-users risk

Medical Cannabis Gas Chromatograph (GC)
Configuration choices February 2011

SRI can configure a gas chromatograph (GC) in hundreds of ways to perform almost any analysis. Two chassis sizes are available. The smaller 310C chassis is very portable while the larger 8610C chassis allows for more complex hardware. All SRI GCs are portable and easily shipped by UPS, FedEx and even as airline baggage.

Medical Cannabis contains many active cannabinoid compounds, but three are considered important, cannabidiol (CBD), THC, and cannabinol (CBN). A GC is the perfect tool for measuring the amount of these three compounds in plant material, resin , tinctures and edibles. Other analytical techniques such as HPLC and GC/Mass Spec can also be used, but are much more expensive to buy, and vastly more complicated to operate yet they do **NOT** provide superior data. For this analysis, GC is the best solution. Unlike a HPLC, the GC naturally de-carboxylates the THCA (the original molecule produced by the plant) into Delta-9THC saving a processing and reporting step. Total cost to perform a GC analysis is less than one dollar, requires only .1 gram of sample and usually takes less than 5 minutes.

Four common configurations have become popular for measuring medical cannabis.

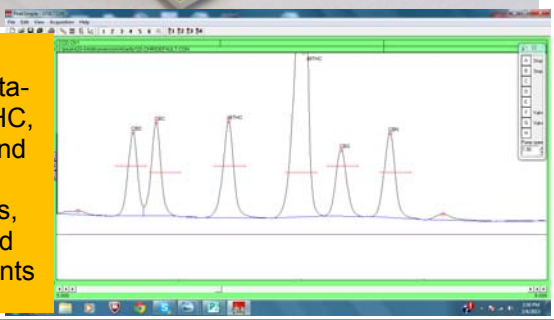
1) Gasless, ultra portable, simple
2) Industry standard FID
3) Automated, hi-volume
4) Pesticides and potency both

Comes with a heavy duty shipping case.

Weighs about 35 pounds (16 kilograms)

C Gas Chromatograph for edical Cannabis Analysis

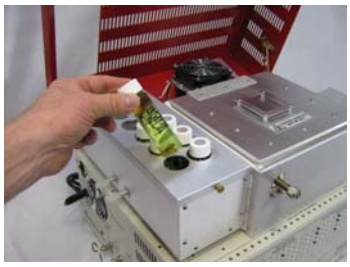
easure CBD,CBC, delta- and delta- THC, CBG, CBN and other cannabinoids, terpenes and residual solvents



Flame Ioni ation Detector (FID)
Heated Flash Injector
°C column oven
Built-in Incubator for heated extractions
meter Capillary column
PeakSimple Data System built-in
Hydrogen regulator/tubing kit
Field portable system
Heavy Duty shipping container
Low power consumption (watts)
Ships via Fed x/UPS or airline baggage
Small footprint for crowded lab benches
Friendly, easy to reach US tech support
Free training
Two Year warranty
ade in USA

Complete system
US , . plus shipping

The SRI C is the perfect si e GC (gas chromatograph) for measuring CBD, THC and CBN levels in medical cannabis. It can also be used to test for synthetic cannabinoids like SPIC , butane residuals, terpenes, aromas and most edibles. The SRI C is rugged enough for mobile applications and light enough to carry around. Simple operation makes training new operators easy. The built-in °C incubator speeds up the extraction process and is helpful in getting concentrates and/or butters to dissolve. A small cylinder of hydrogen (customer supplied) is used for carrier gas and lasts for months. The regulator and tubing for the cylinder is provided. Analysis time is about minutes so up to samples an hour can be analy ed. The included PeakSimple software (Windows P/Vista/Win /) controls the GC as well as acquiring and calibrating the data. Simple one click export of the data to xcel or Word makes your final report look professional. Get half a day of free training with your GC at our tech support center near LA (Los Angeles) airport.



System consists of two part numbers:

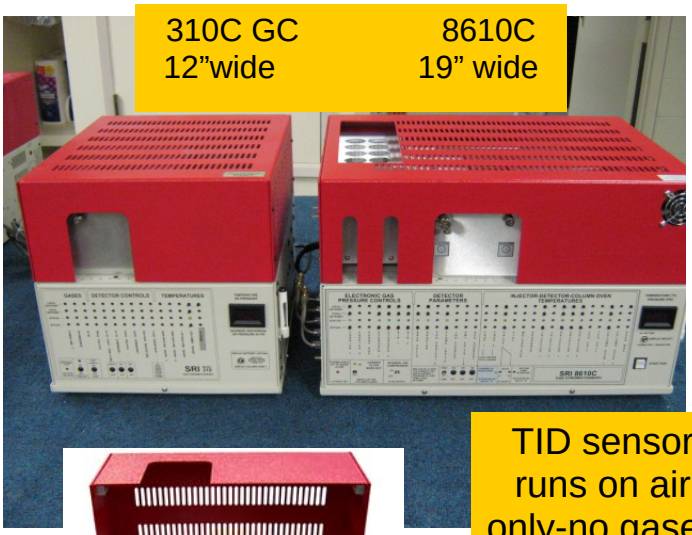
8610-0091	**Cannabis Potency Testing GC complete**	**$11,585.00**
8600-C350	**Hydrogen Gas line kit**	**430.00**
Total	**USD ADD Import Frt&GST In Australia**	**USD$12,015.00**

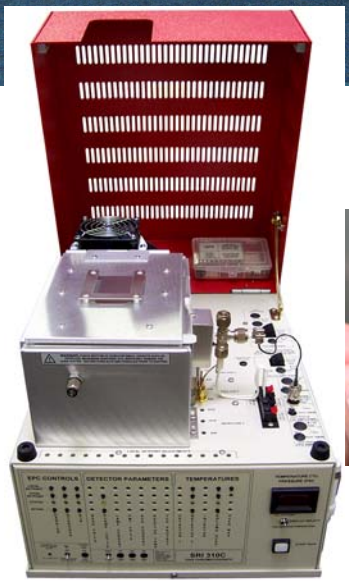
Medical Cannabis Gas Chromatograph (GC)
Gasless and Simple Configuration

Configuration #1
"Gasless" TID Detector based Potency Configuration
Part# 8610-0094 $9999.00

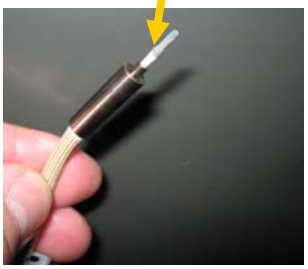
This GC is configured on the ultra-compact 310C chassis (only 12 inches wide) and includes an TID (thermionic ionization detector) which requires no gas cylinders to operate. All required gas is provided by the built-in "whisper quiet" air compressor and dryer. This GC configuration is appropriate for users with no prior GC experience, and/or for those who want maximum portability. You can literally carry the GC around under your arm, it's that portable.
Just add a Windows PC (XP, Vista, or Windows 7) desktop or laptop. SRI's easy to learn PeakSimple software is included.
The GC comes complete with syringes, and a starter pack of vials; everything you need except the standards and a balance.

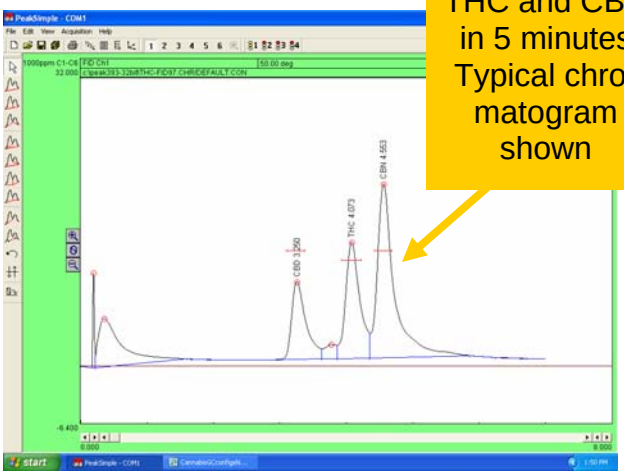
Run times can be as short as 3-4 minutes. A typical calibration chromatogram is shown at right.





TID sensor runs on air only-no gases required





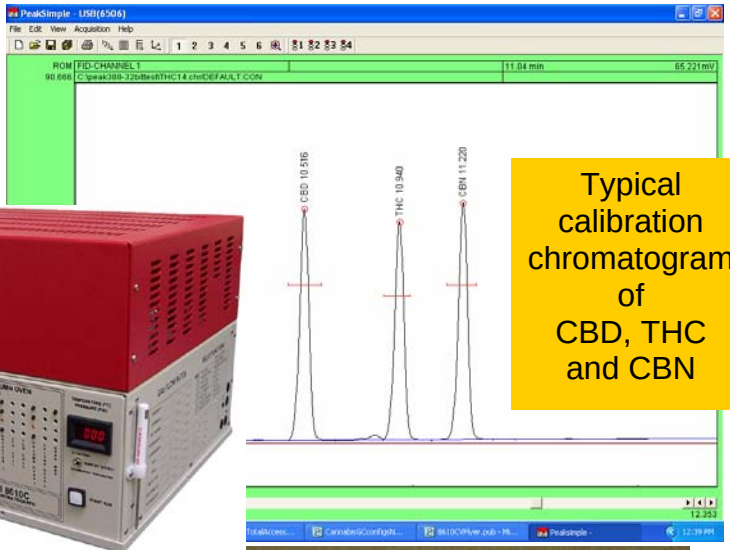
Measure CBD, THC and CBN in 5 minutes Typical chromatogram shown

Medical Cannabis Gas Chromatograph
Industry Standard FID Configuration

Configuration #2
FID Detector based
Potency Configuration
Part# 8610-0091 $10,210.00

This GC configuration includes an FID (flame ionization detector) which requires hydrogen gas to operate. Because hydrogen is used as a carrier gas, higher resolution is possible when measuring the CBD, THC and CBN molecules in cannabis. A photo of a typical hydrogen gas cylinder is shown at right. This GC configuration is appropriate for users with prior GC experience, for those who want to be equipped with industry standard hardware, or for those who may later wish to add the extra hardware required to measure the pesticide content of cannabis.
Run times can be as short as 3-4 minutes.

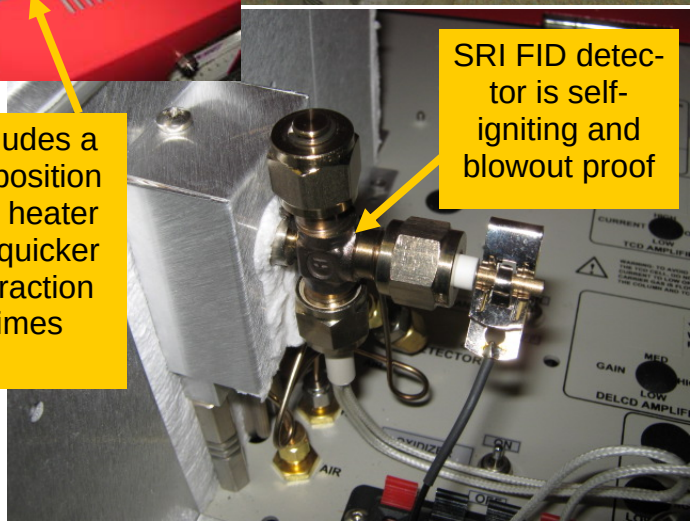
User's will need a hydrogen cylinder, Windows computer and AC power. Syringes and a starter pack of vials is included.



Typical calibration chromatogram of CBD, THC and CBN

Hydrogen Gas cylinder

Includes a 12 position vial heater for quicker extraction times



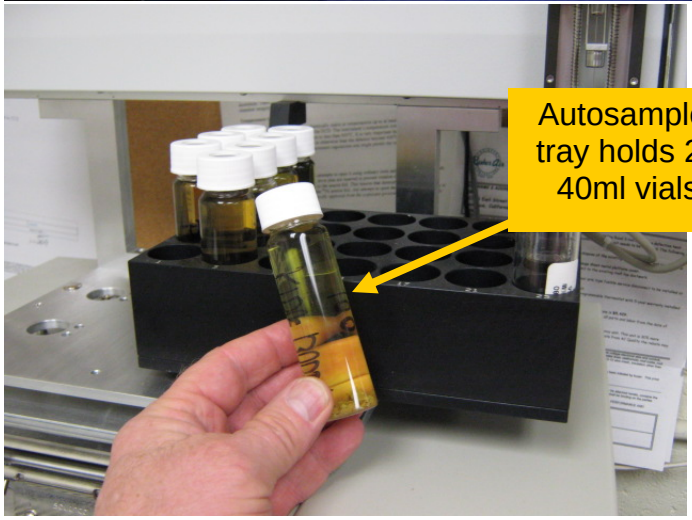
SRI FID detector is self-igniting and blowout proof

Medical Cannabis Gas Chromatograph
Automated Hi-volume Configuration

Configuration #3
Potency test with FID detector and Autosampler
Part#8610-0093 $25,530.00

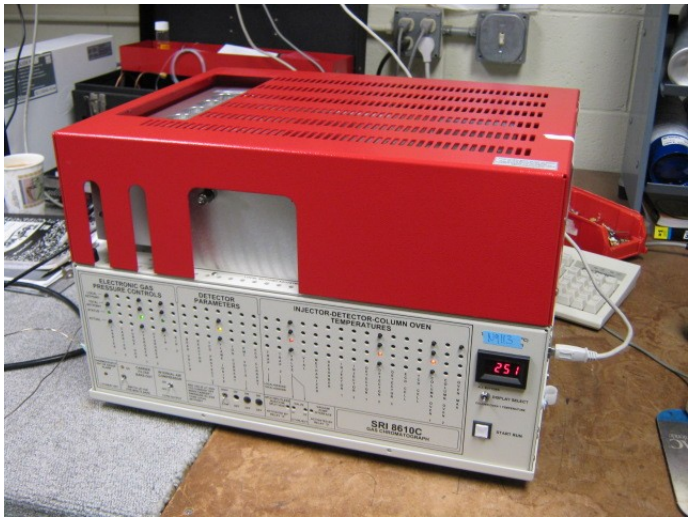
This GC configuration is appropriate for user's who have higher numbers of samples per day to analyze for CBD, THC and CBN. The autosampler accommodates 28 of the 40milliliter extraction vials so users do not have to transfer the THC extract from the extraction vial to a smaller autosampler vial thus saving an expensive and time consuming step. The autosampler makes it practical to take 2-3 samples from the same vial and average the results, leading to increased accuracy. The autosampler lets the user walk away or operate overnight. This configuration is appropriate for users with prior GC experience and who have or anticipate a high sample volume.

This configuration is not as portable as Configurations #1 or #2 since it is physically larger and the autosampler must be removed from the GC prior to transport.



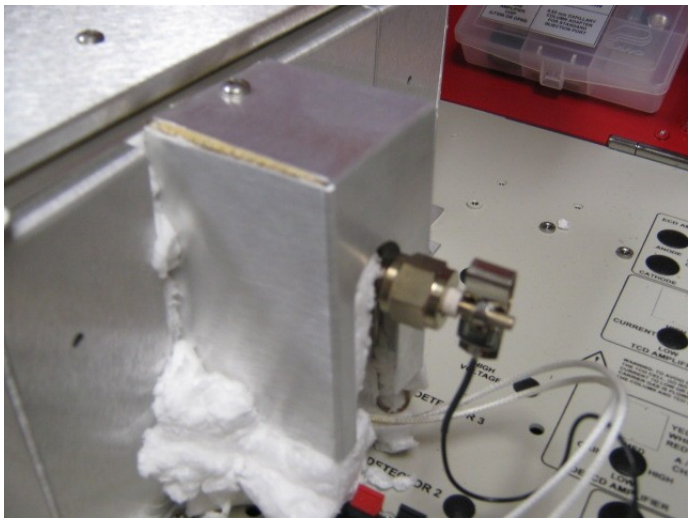
Medical Cannabis Potency Testing using the SRI 8610C FID GC

The SRI 8610C FID GC with a 12-vial sample heater is designed for testing the **potency of medical cannabis (cannabinoids)**. With minor configuration and procedural changes the GC can also test for **terpenes** and **residual solvents** in concentrates (see our documents on our website at www.srigc.com/documents.htm).



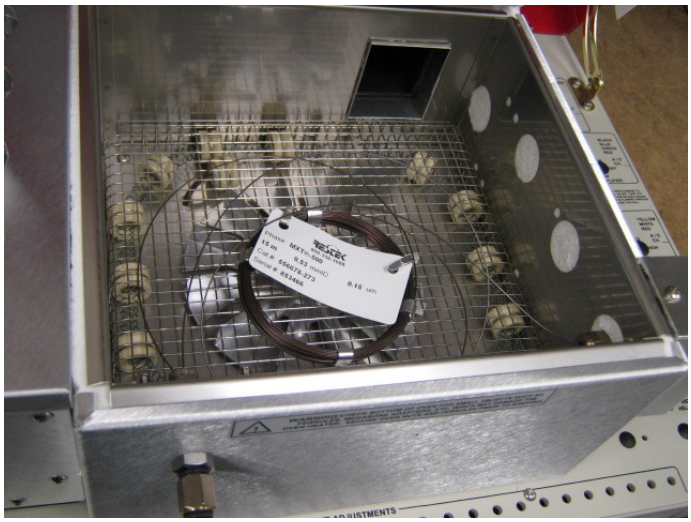
The 12-vial sample heater aids in a quicker extraction of the cannabinoids in solvent and maintains the extracted samples at 50° C for better reproducibility.

The GC includes SRI's Flame Ionization Detector (FID) which is able to measure the cannabinoid molecules based on its ability to detect the combustion of hydrocarbon molecules.

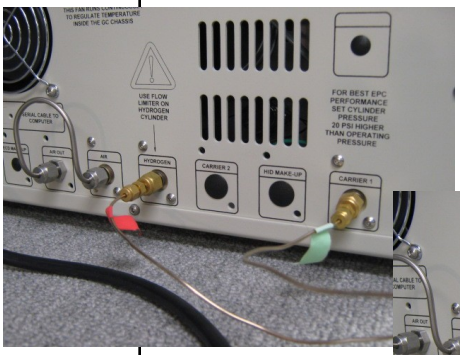


Medical Cannabis Potency Testing using the SRI 8610C FID GC

The cannabinoid molecules, Δ9-THC, CBD, and CBN (and for more advanced operators, CBC, Δ8-THC, and CBG) are separated by a 15-meter metal capillary column which is heated in the column oven.



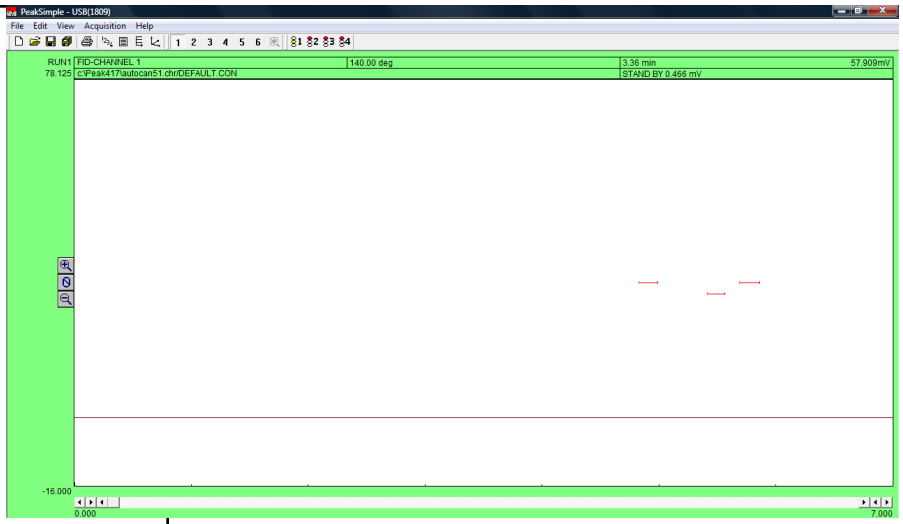
Hook up the gas lines to the left side of the GC. The GC can be operated with hydrogen or helium as a carrier gas. When using hydrogen as a carrier gas, cap off the hydrogen gas inlet and connect the hydrogen to the carrier 1 inlet.



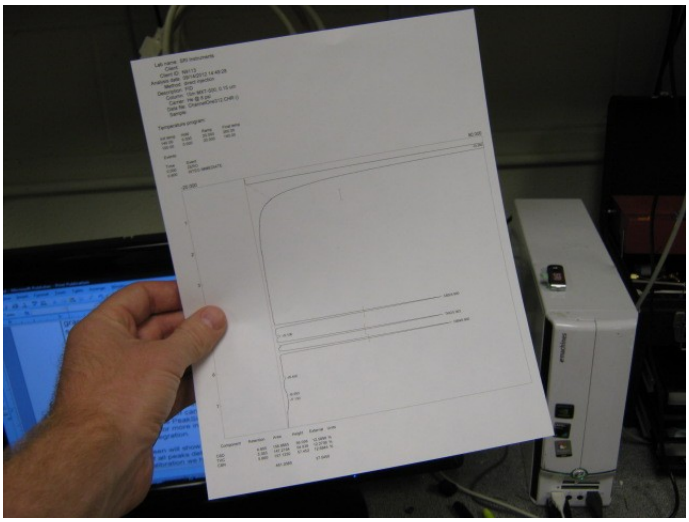
The entire GC plugs into any Windows computer using a USB cable.

Medical Cannabis Potency Testing using the SRI 8610C FID GC

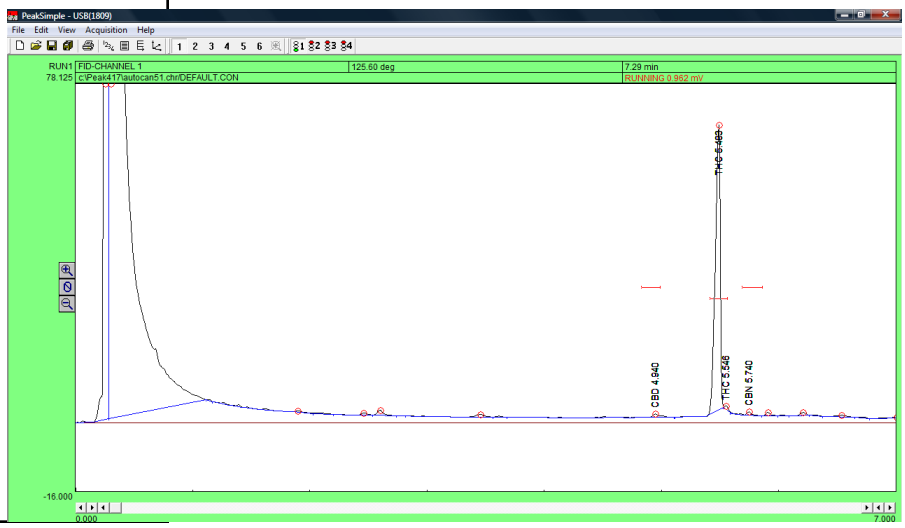
SRI's PeakSimple software is included with the GC. PeakSimple software collects the GC data and generates a calibrated result which can be printed or transferred to other programs such as Excel or Word.



The chromatogram hardcopy printout at right shows the three peaks CBD, THC and CBN which were injected to calibrate the GC.

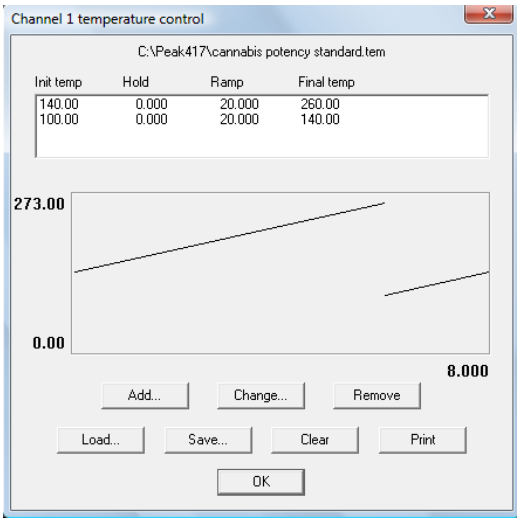


An actual cannabis sample is shown at right.

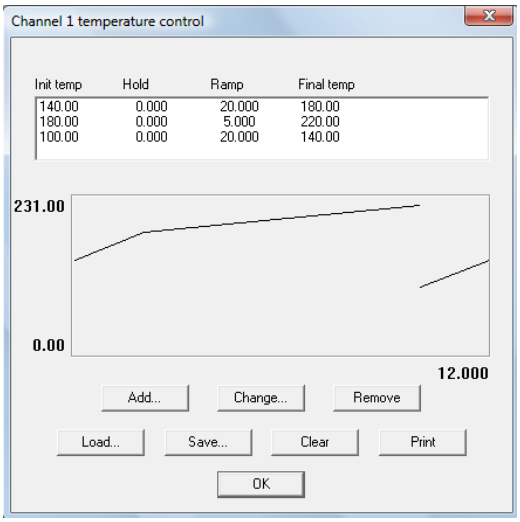


Medical Cannabis Potency Testing using the SRI 8610C FID GC

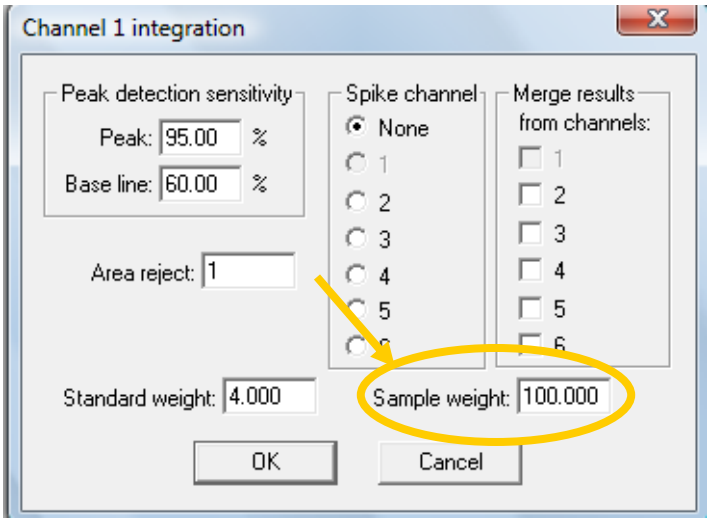
For a quick 8-minute analysis that optimizes speed *and* peak separation, set the column oven temperature as shown to the right.



Sometimes, a better separation is preferred (particularily with THC, CBG, and CBN) at the expense of speed. For a longer 12-minute analysis, set the column oven temperature as shown to the right.



Set the integration parameters as shown. Note the “Sample weight” box. When you calibrate the GC it will be set at 100. When you run actual cannabis samples, the weight of the sample will be entered. (ex. If the sample weighed 0.104 grams, then “104” should be entered).

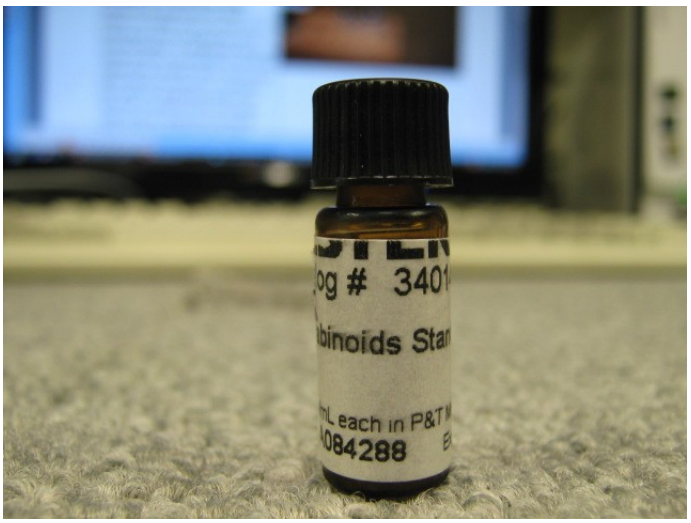


Medical Cannabis Potency Testing using the SRI 8610C FID GC

Obtain the cannabinoid calibration standard from a chromatography supplier like Restek (restek.com). The standards can be acquired individually, but SRI recommends a more convenient three-way (THC, CBD, CBN) cannabinoid standard. The standards are available at a concentration of 1000 ng/ul in Methanol. No license is required to purchase.

Break the glass ampoule and transfer the contents into a 2mL septum vial. Restek provides one free vial with each standard.

Whether you have three vials (individual standards of THC, CBD, or CBN) or one vial of 3-way standard, they will each be at a concentration of 1000ng/ul. We will refer to these standards as primary standards. Ideally, when not in use they should be kept in a refrigerator with an unpierced septum so that the methanol will not evaporate and increase the concentration of the cannabinoids in the standard. When calibrating with the primary standards the percent concentration of the cannabinoids will be approximately 40%.

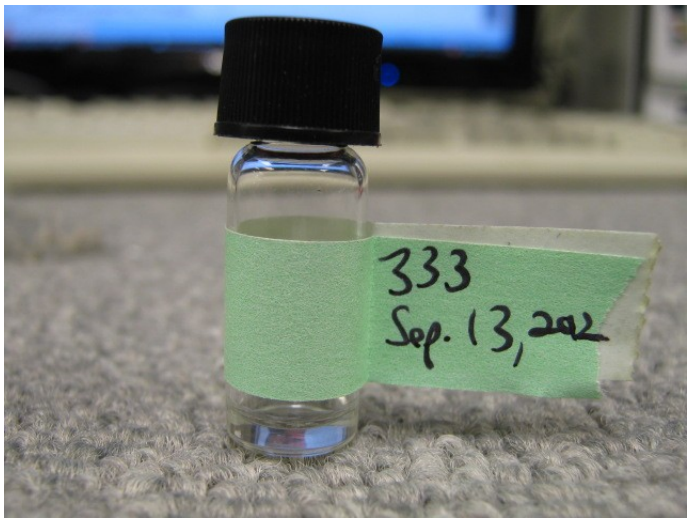


Medical Cannabis Potency Testing using the SRI 8610C FID GC

SRI recommends preparing a “333 working standard” rather than using a primary standard to calibrate. Not only will this help to preserve the purity of your primary standard and get more mileage out of it, but it will also calibrate the GC at percent concentrations that more closely resemble cannabis flowers (13.32% instead of 40%).

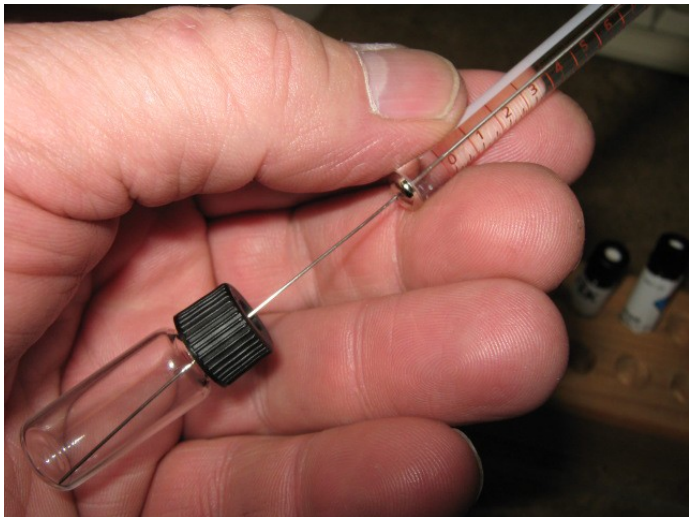
If you have separate cannabinoid standards, use the 100uL syringe, which is included with the SRI GC, (Restek#24863) to transfer 100uL of each 1000ng/uL (primary) standard into another 2mL vial. If you have the 3-way standard, use the 100uL syringe to transfer 100uL of the standard into another 2mL vial and then add 200uL of methanol.

After either method, you will end up with 300uL of working standard containing 333ng/uL each of the three compounds (CBD,THC and CBN). Label the primary and working standards with both a name and a date.

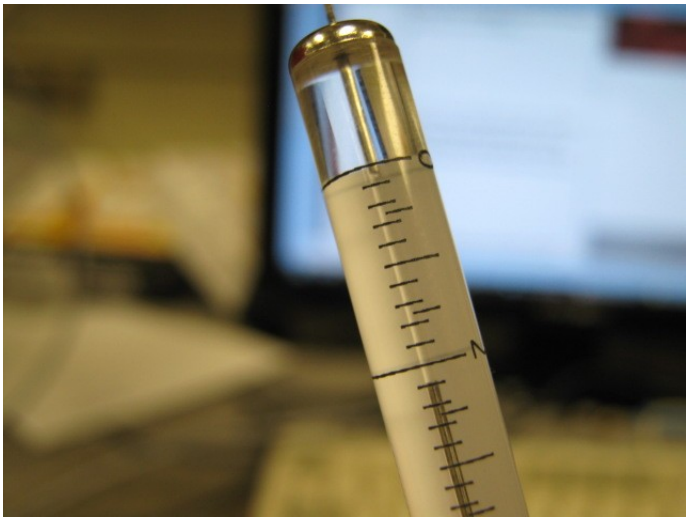


Medical Cannabis Potency Testing using the SRI 8610C FID GC

Rinse the syringe first then use the 10ul syringe delivered with the GC (SRI #8670-9550) to withdraw 2-3ul of the working standard. Puncture the septum rather than open the vial to avoid letting the methanol solvent evaporate each time the vial is opened. Pump the plunger several times to get rid of air bubbles.



With 2-3ul of liquid in the syringe, hold the needle vertically or at least slanted upwards so any air bubbles will rise towards the needle.

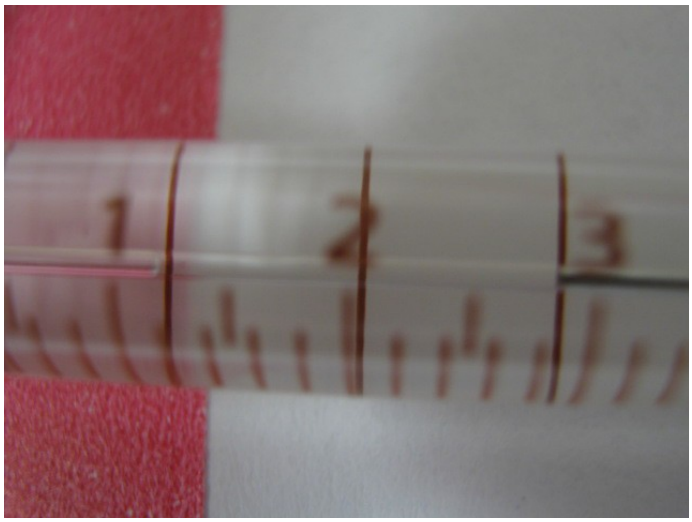


With air bubbles removed, push the plunger to the 1ul mark. It is important to be as precise as possible. Wipe the needle with your fingers or a tissue to remove any liquid from the outside of the needle.

Medical Cannabis Potency Testing using the SRI 8610C FID GC

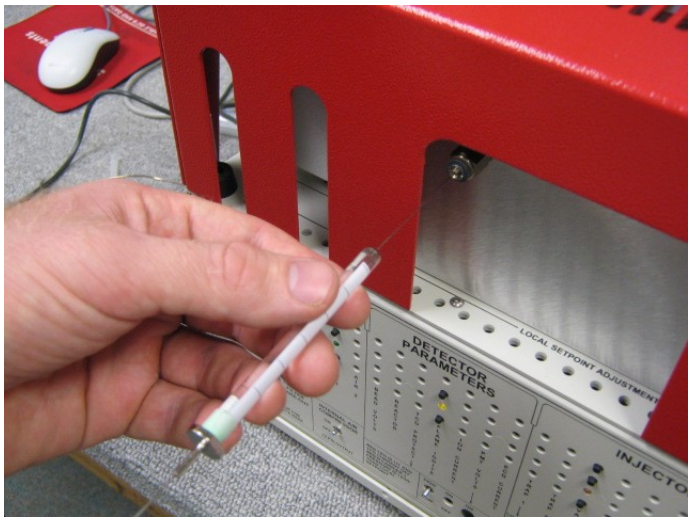
Pull the plunger back to the 3ul mark and note the amount of liquid. It should be 1.6-1.8ul because the needle also contains .6-8ul and this adds to the 1ul you measured with the plunger.

Leave the plunger at the 3ul mark.

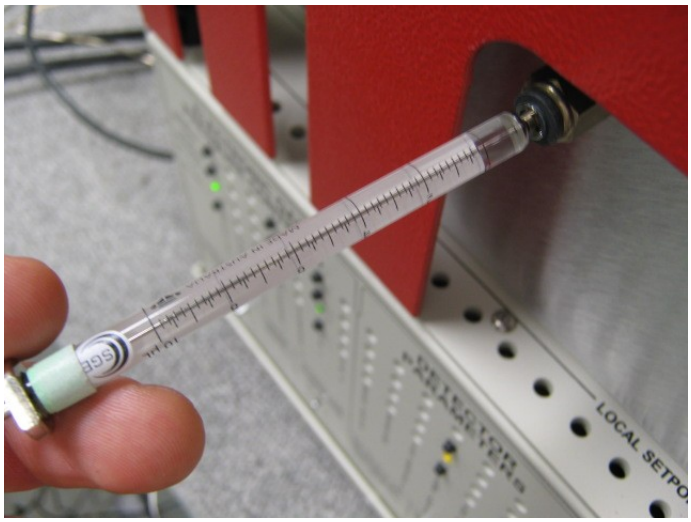


With the plunger still at the 3ul mark, place the needle up against the septum (but not poking through it yet).

Press the Start Run button or hit the Spacebar on the keyboard to start the run.

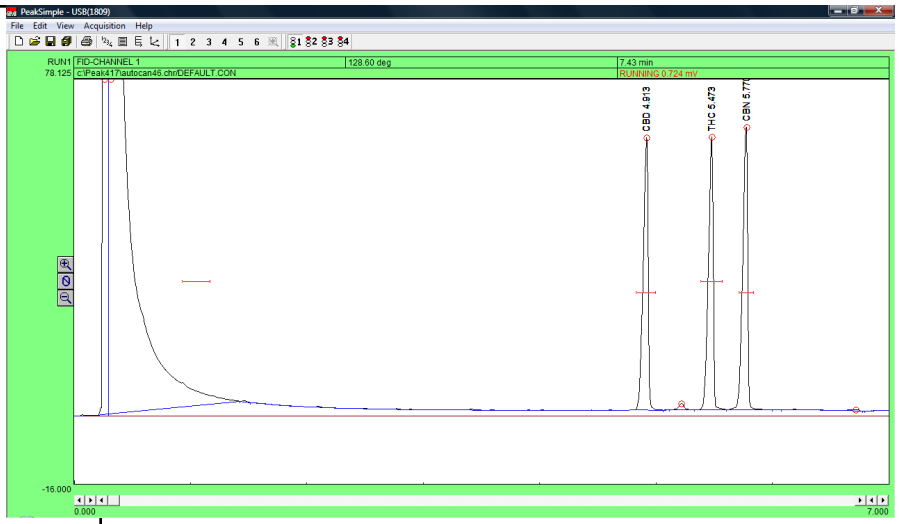


Insert the syringe all the way through the septum as far as it will go. Immediately depress the plunger. Twist the syringe one half turn (to wipe off any liquid on the tip of the needle) and then withdraw the syringe.

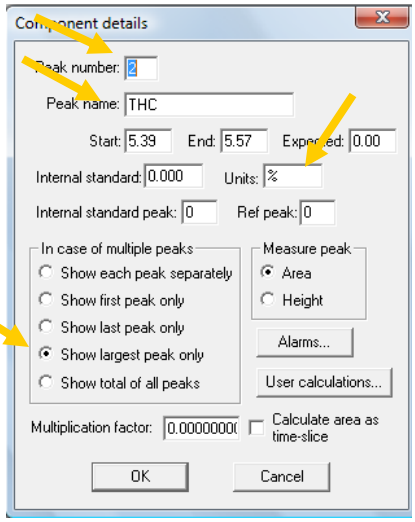


Medical Cannabis Potency Testing using the SRI 8610C FID GC

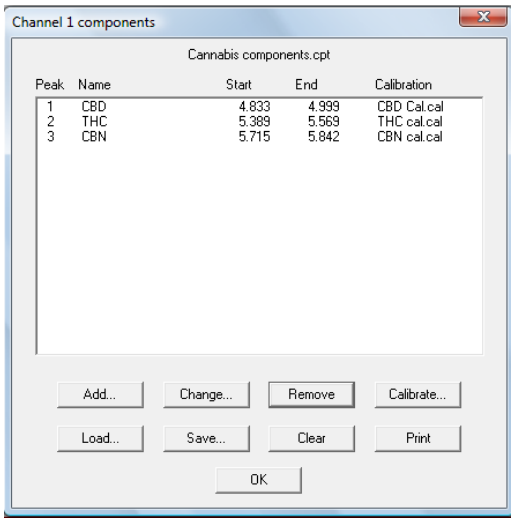
Once the run is completed you should see a large solvent peak near the beginning, then closer to the end, three peaks of roughly equal size (there will also probably be a small Delta-8 THC peak between the 1st and 2nd peak). Add retention windows to the three peaks by right clicking on the peak and selecting “Add component”. See the PeakSimple tutorial describing the process of creating retention windows.



Identify the three peaks (from left to right: CBD, THC, CBN) by right-clicking on each peak and selecting “Edit component”. Assign each peak a unique number and name (CBD, THC, or CBN), select “show largest peak only”, and add a “%” sign to the “Units” box. Press the “OK” button to exit back to the main chromatogram screen.



Right click on the chromatogram and select “Components” to open the “Channel 1 Components” Screen. Here will be displayed a list of all the components with named retention windows and unique peak numbers. Select “Save” and name the component file so that if you exit PeakSimple your component and calibration files will not be lost.

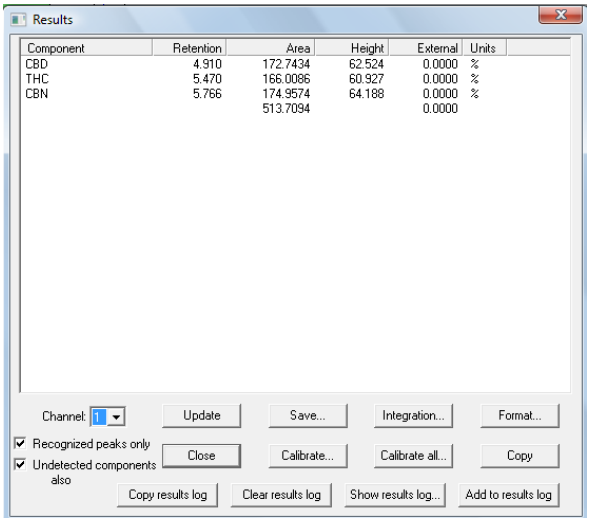


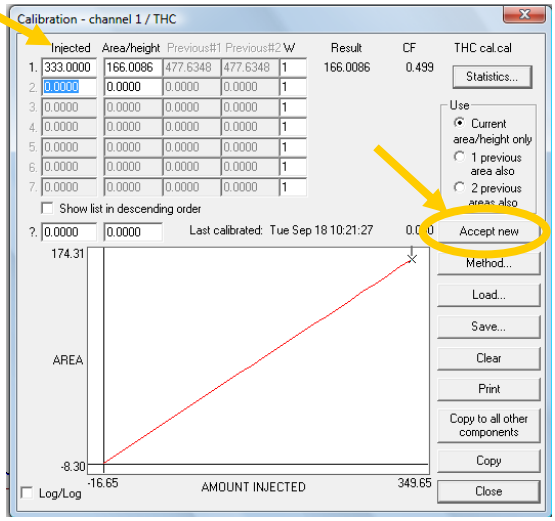
Medical Cannabis Potency Testing using the SRI 8610C FID GC

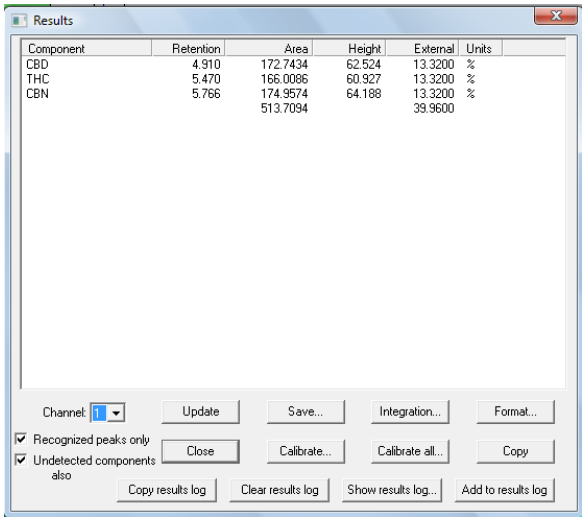
Check the Results screen. If you injected a primary standard the area counts for the cannabinoids should be between 420 and 540 and roughly equal to each other (+/- 30). If you injected the working standard the area counts should be between 140 and 180 and roughly equal (+/- 10).

Calibrate each peak by creating a calibration curve. See the Peak-Simple tutorial describing this process. In the calibration curve enter the amount of standard you just injected. This will be 333 (for 333ng/ul) or 1000 (for 1000ng/ul). Type this number in the top left cell of the spreadsheet in the calibration curve. Then click the Accept New button to transfer the peak's area into the top row 2nd column. Save the curve under some name. Do this for all the peaks.

Navigate to the View/Results screen to see the report. With the integration screen and components setup as discussed earlier in the document the percent concentrations of CBD, THC, and CBN will each be displayed as 13.32% (or 40% if primary standards were injected). You are now calibrated and ready to inject real cannabis samples.



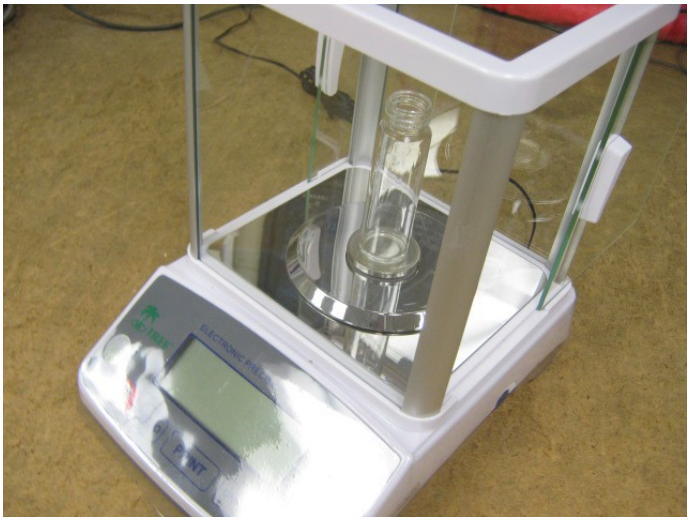




Medical Cannabis Potency Testing using the SRI 8610C FID GC

Remove the cap from a 40ml vial and place it on the balance. The balance should be capable of reading 1 milligram (.001 gram). A balance like this can be purchased brand new for less than $300 on E-bay.

With the 40 mL vial on the balance tare the reading (make the reading 0.000). Then carefully add 100 milligrams of manicured cannabis. Drop the bits of cannabis into the vial slowly until the reading is close to 100 milligrams. Make sure to write down the exact weight of the sample somewhere, preferably on the vial itself.

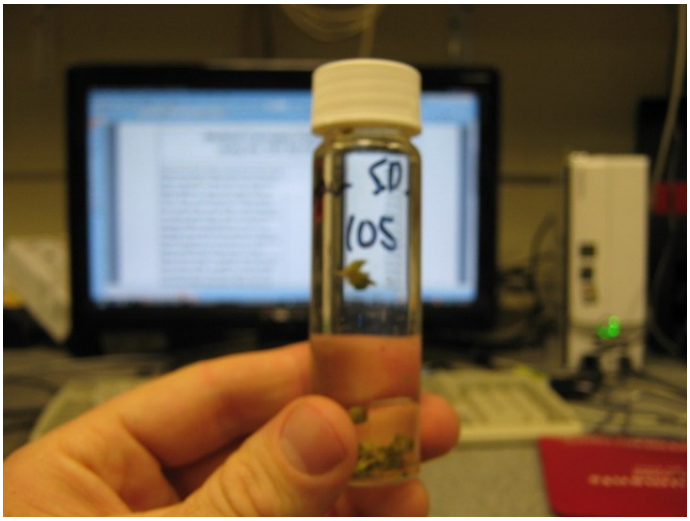


Don't worry if you are slightly under or above 100. In the photo at right, the reading is 98 milligrams which is close enough. You will enter the reading in the sample weight field in Peak-Simple software which will mathematically correct the calculated answer to compensate for weights slightly above or below 100.

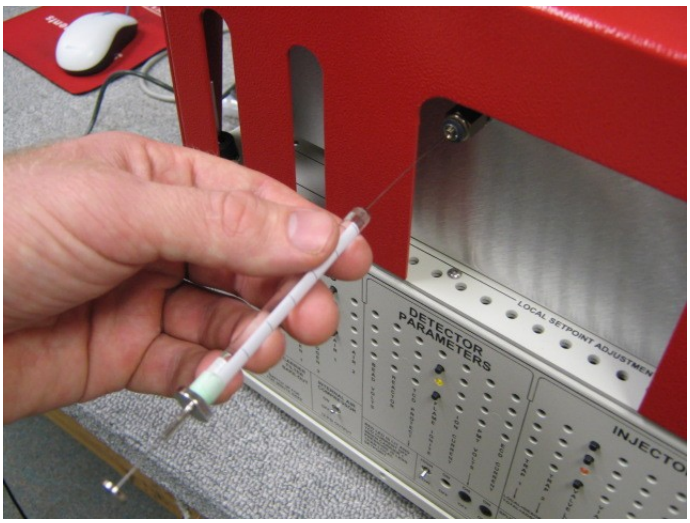
Medical Cannabis Potency Testing using the SRI 8610C FID GC

Remove the 40ml vial from the balance and fill it with 40ml of extraction solvent. You can use 70% or 91% IPA, methanol (methyl alcohol), ethanol, acetone, chloroform or other solvents. We recommend using either methanol, or for a cheap and efficient solvent, denatured alcohol (a mixture of ethanol and methanol) that can be obtained at most hardware stores for less than $20 a gallon. Non-polar solvents like hexane are not recommended because they do not extract the cannabinoids as well as polar solvents.

Shake the vial for a few seconds and then let it sit for about 20 minutes in the incubator (longer without heat).

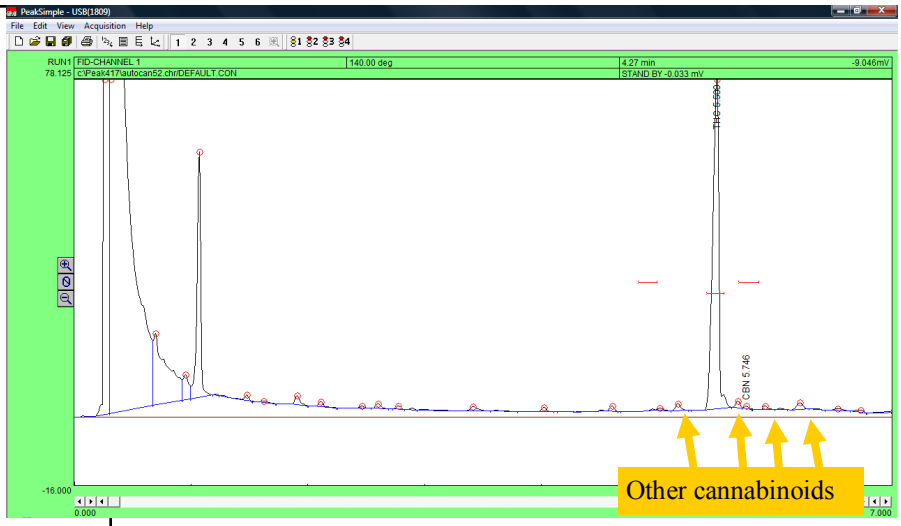


Use the 10ul syringe which comes with the GC to inject 1ul of the extract as shown previously with the calibration standard. It is important to be very precise with the syringe since the overall accuracy of the test depends on this. Don't forget to enter the exact Sample weight in the proper field on the integration screen.



Medical Cannabis Potency Testing using the SRI 8610C FID GC

A real cannabis sample will look something like the chromatogram at right. There will be one big peak (THC) and much smaller ones for CBD and CBN. In this case, CBD is so low that it is not detected.



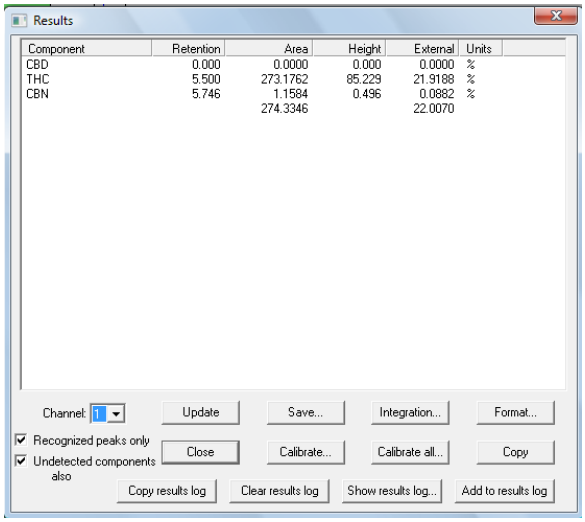
CBN may or may not be detected or it may blend into the much larger THC peak. When this happens you can use the slower temperature program and/or lower the carrier pressure to get better separation of the peaks.

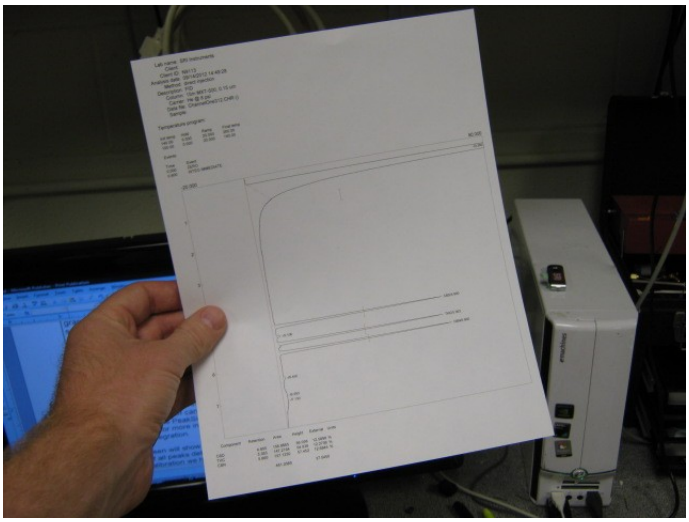
There may be other peaks which are not CBD, THC or CBN. These other peaks are cannabinoids (CBC, Delta-8 THC, CBG, and others) for which there may or may not be calibration standards available at this time.

It may be necessary to manually integrate some of the peaks for the most accurate quanitification of cannabinoid potency. See the PeakSimple Advanced Tutorial for more information on manual Integration.

The Results screen will show the concentration of all peaks detected based on the calibration we have previously done.

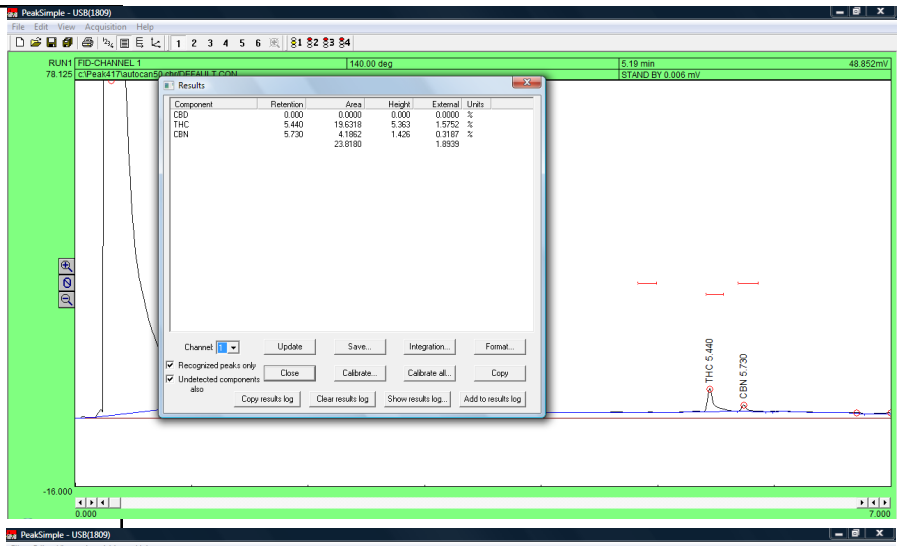
Print the chromatogram and results for a hardcopy record of the analysis.



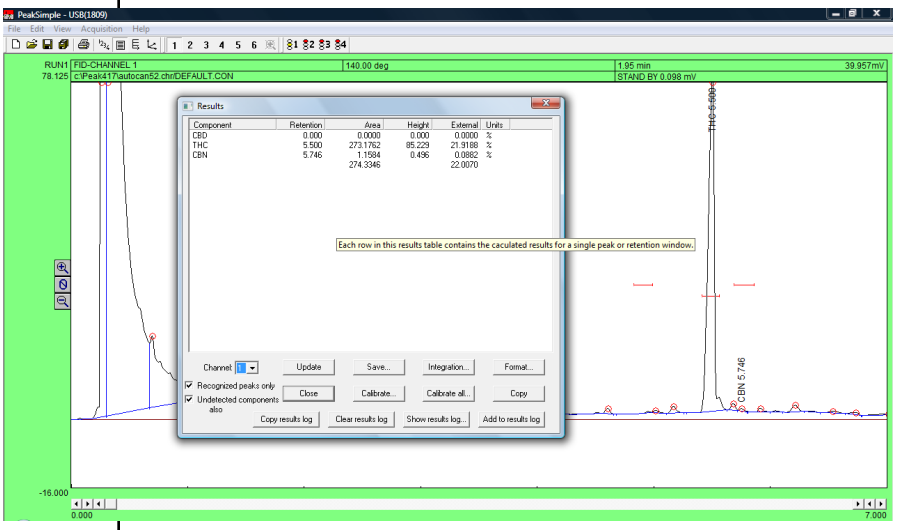


Medical Cannabis Potency Testing using the SRI 8610C FID GC

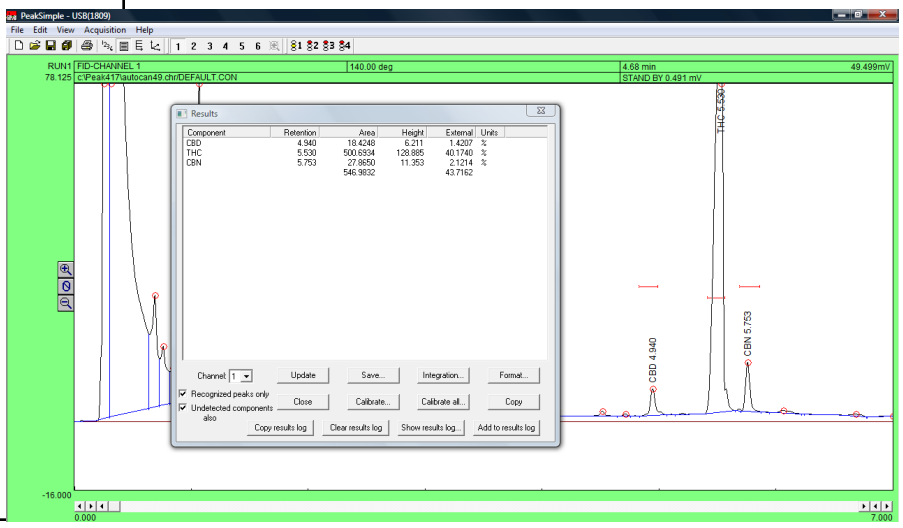
Chromatogram of a low-potency cannabis flower sample with 1.6 % THC.



Chromatogram of a high-potency cannabis flower sample with 21.9 % THC.



Chromatogram of a typical cannabis concentrate with 40.2% THC.



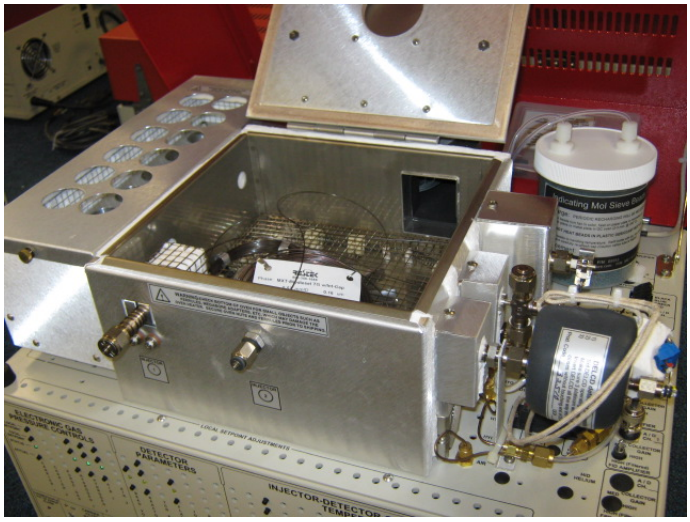
Medical Cannabis Gas Chromatograph Pesticides and Potency Configuration

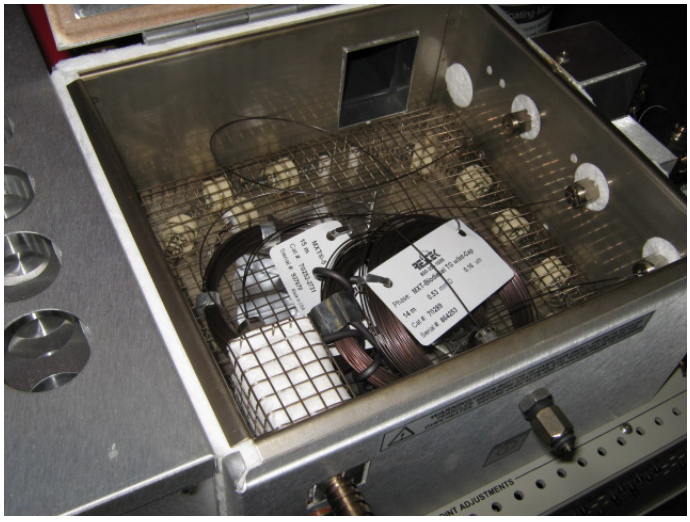
Configuration #4
Potency plus Pesticides
GC configuration
Part# 8610-0092 $21,889.00

This GC configuration permits two separate analyses which can be run simultaneously. The first analysis is for potency (CBD, THC and CBN) using a FID detector. The second analysis is for pesticides in cannabis using dual detectors. The NPD (nitrogen phosphorus detector) measures organo-phosphorus pesticides (Malathion) and many of the carbamate pesticides (Sevin).
The DELCD (dry electrolytic conductivity detector) measures organo-chlorine pesticides like Dursban, DDT, and Endrin.

The photos at right show the three columns, three detectors and dual injectors which make this possible.

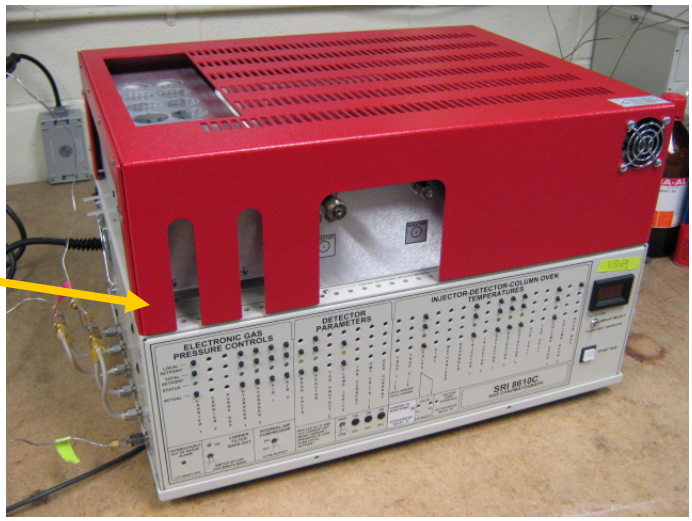
This GC configuration is appropriate for users with prior GC experience since the pesticide screen is more complex than the potency test. It should be understood that while 90% of all pesticides can be detected with this GC configuration, it is not possible to measure every possible pesticide since there are hundreds of pesticide molecules in a variety of chemical classes. It does allow the user to screen for most common pesticides in a very cost effective (less than 25 cents per analysis) manner using only .1 grams of sample.





Medical Cannabis Pesticide Screening using the SRI 8610C GC

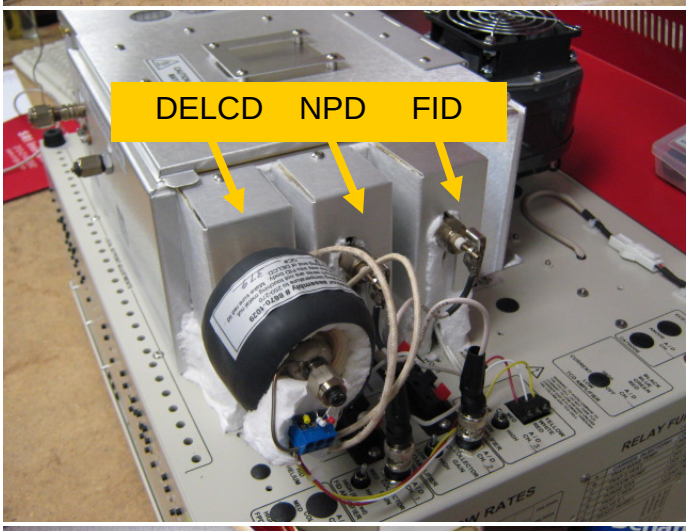
The SRI 8610C Gas Chromato-graph (GC) configured for Medical Cannabis Potency and Pesticide testing is shown at right.



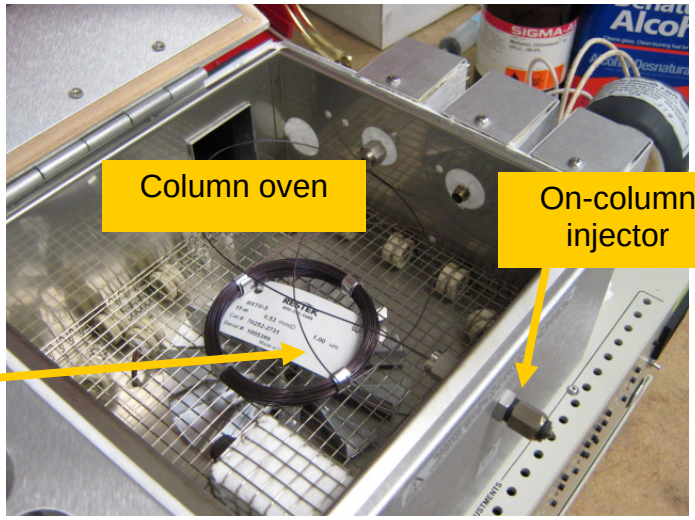
The GC is equipped with three detectors:

FID (flame ionization detector)
NPD (nitrogen/phosphorus)
DELCD (dry electrolytic conductivity)

Refer to the GC manual or pdf documents on the SRI website www.srigc.com for specific instructions on the detectors.



This GC can be used for potency testing only by using the on-column injector and the FID detector. In this case only a single column is re-quired in the column oven.

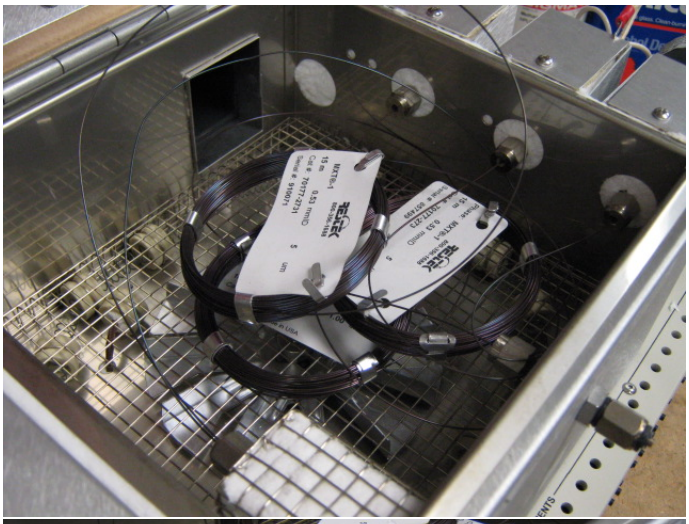


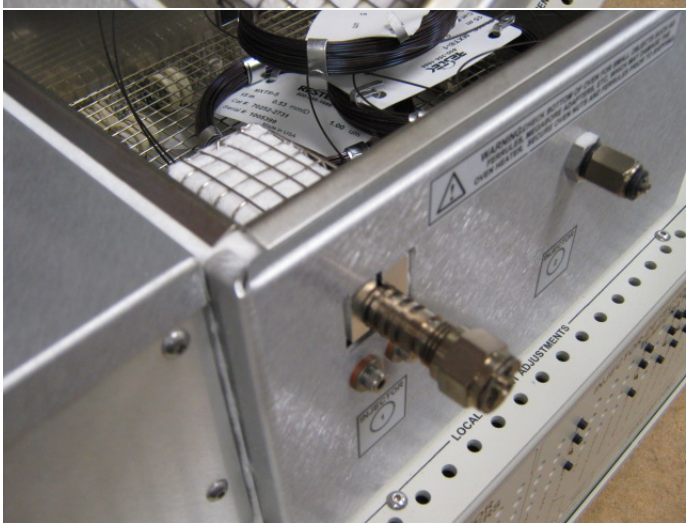
Medical Cannabis Pesticide Screening using the SRI 8610C GC

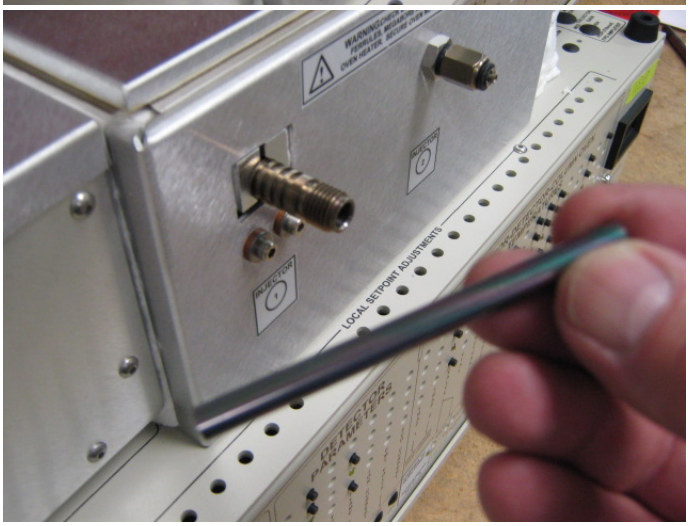
Two additional columns can be connected to the Heated Injector. One of these columns goes to the NPD detector and the other to the DELCD detector. The Heated Injector splits the sample onto the two columns using a two hole ferrule Restek part# 20246

The Heated Injector and on column injector are side by side on the front of the GC's column oven.

The Heated Injector includes a removeable quartz lined stainless steel tube. Cannabis samples (100 milligrams) are inserted into the tube and then into the Heated Injector which at 200C thermally desorbs pesticides off the cannabis and onto the two columns.



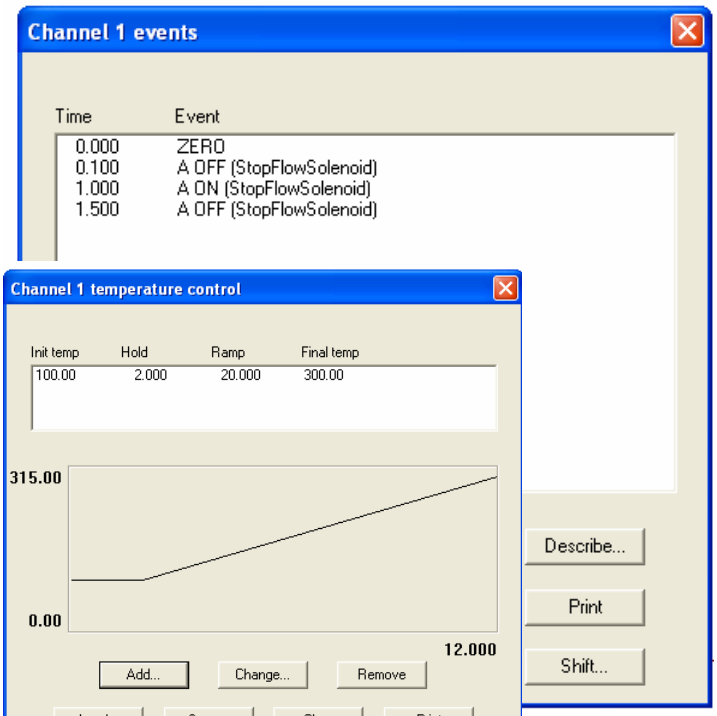


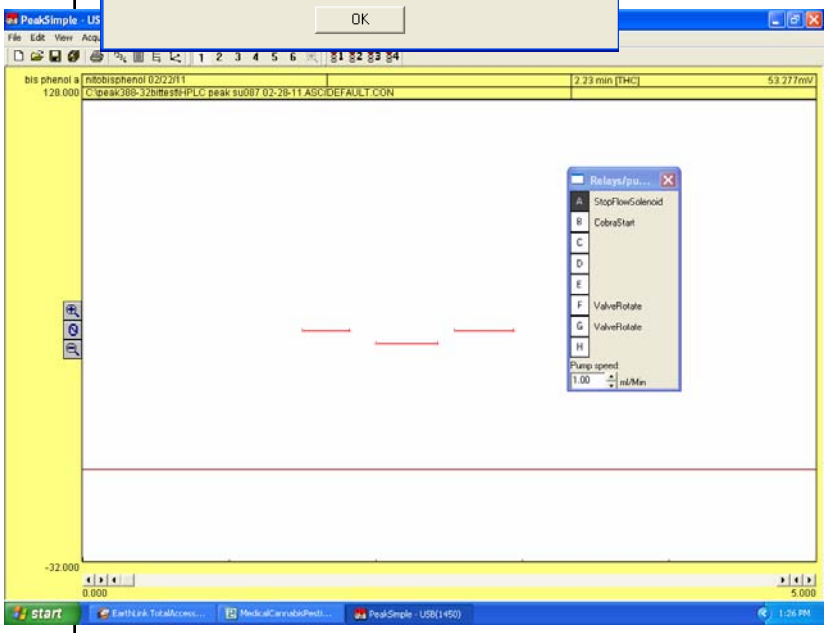


Medical Cannabis Pesticide Screening using the SRI 8610C GC

Edit the Event table in Channel 1 of the PeakSimple software to turn the carrier gas to the Heated Injector on and off at the times shown.

Enter the temperature program shown at right. The column oven starts at 100C for two minutes, then ramps at 20 degrees per minute to 300C.





Manually actuate Relay A prior to the start of the analysis. Display the Pump/Relay window and click the A button to actuate Relay A. When it is actuated, Relay A turns the carrier gas flow to the heated injector off.

Medical Cannabis Pesticide Screening using the SRI 8610C GC

Take a common cotton ball and make a small wad about the size shown.

Use a screwdriver or other tool to push the cotton wad about halfway down the tube.

Place the tube on the balance and then ‘tare” the balance to make it read 0.000 grams

Medical Cannabis Pesticide Screening using the SRI 8610C GC

Manicure the cannabis sample and scoop 100milligrams (.1gram) into the tube.

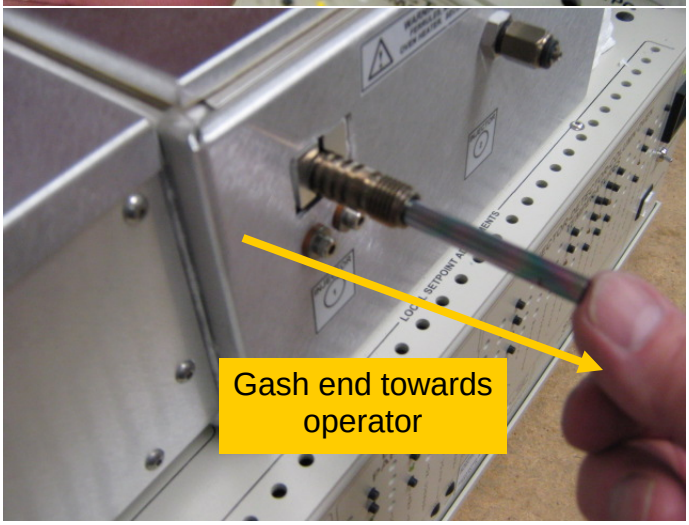
Weigh the tube until you get approximately 100 milligrams. You do not have to get exactly 100 so long as you are close (95-105 mg). The photo are right shows the weight at 99 milligrams. You can correct for the actual sample weight in the PeakSimple software after the analysis.

Stuff a little more cotton into the tube to hold the cannabis sample in place. Do not pack the cotton and cannabis tightly. The cotton should just be tight enough to prevent the cannabis from escaping the tube. The cannabis should be loose, NOT packed down.

Medical Cannabis Pesticide Screening using the SRI 8610C GC

Since the injector is HOT, use a tool like a 9/16” socket to remove the septum nut.

Insert the tube filled with cannabis into the injector. At this time the carrier gas is off so no gas will escape while you are inserting the tube.



The tube has a gash at one end.

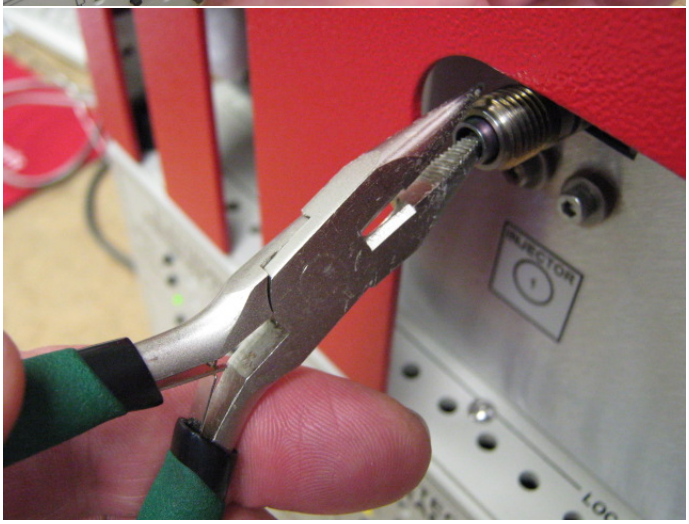
<u>The gash end MUST be towards the operator.</u>

Medical Cannabis Pesticide Screening using the SRI 8610C GC

Start the analysis by pushing the start button on the GC. You can also push the spacebar on the computer keyboard. The Event table in PeakSimple will de-actuate Relay A at .1 minutes into the analysis which will cause the carrier gas to strip the pesticides from the now HOT cannabis and deposit the pesticide molecules on the two columns.

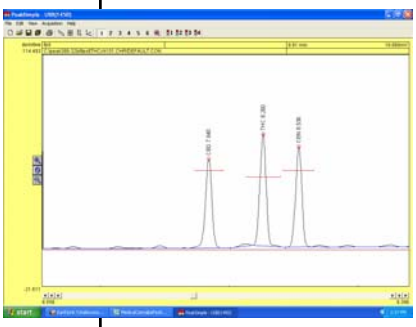
If your GC is equipped with a second injector and FID detector for potency measurement, you can inject the potency extract in the other injector anytime in the first 1 minute of the analysis.

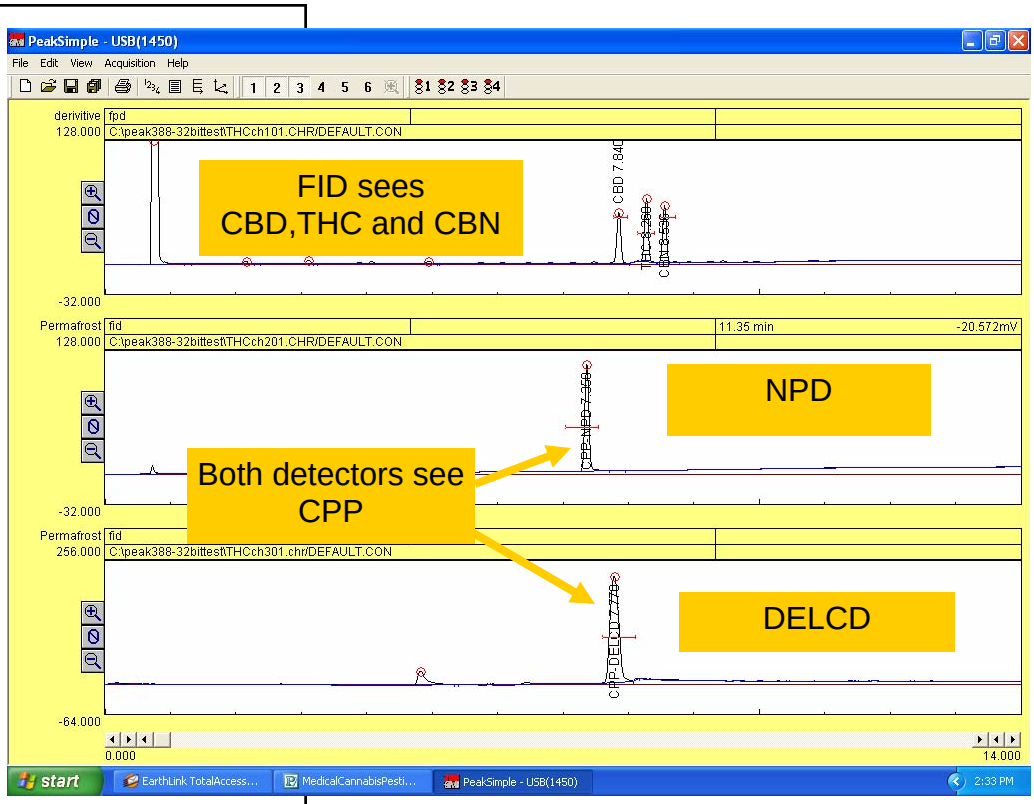
At 1 minute into the analysis, the carrier gas is turned off for 30 seconds. During that 30 second period remove the tube from the HOT injector using a tool to avoid burning your fingers. Place the HOT tube in a beaker to cool off. You must replace the septum nut within the 30 second window.



Medical Cannabis Pesticide Screening using the SRI 8610C GC

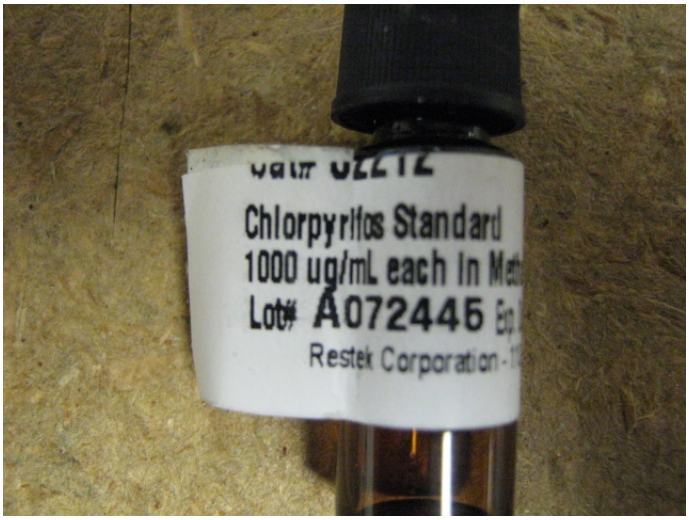
To calibrate the Potency channel (channel 1), inject 1ul of the 333ng/ul calibration mixture into the on-column injector. You should see three equal size peaks.





Preparation of the 333ng/ul working standard is described in another publication.

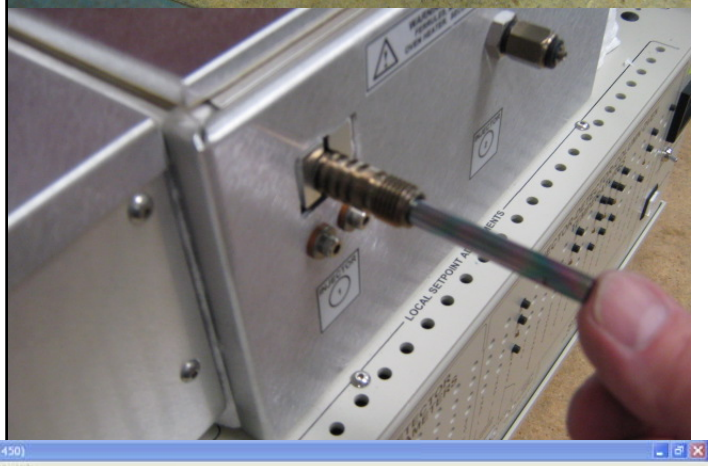
The two pesticide detectors (NPD and DELCD) are calibrated with a pesticide standard such as Chlorpyrifos. Restek part# 32212 is 1000ug/ml (1000ppm) of chlorpyrifos (CPP) in methanol. CPP was chosen as the calibration pesticide because it has both phosphorus (which the NPD detects) and chlorine (which the DELCD detects). So the one pesticide can be used to calibrate both detectors.



Medical Cannabis Pesticide Screening using the SRI 8610C GC

Deposit 1ul of the CPP standard on a clean cotton wad in the tube.

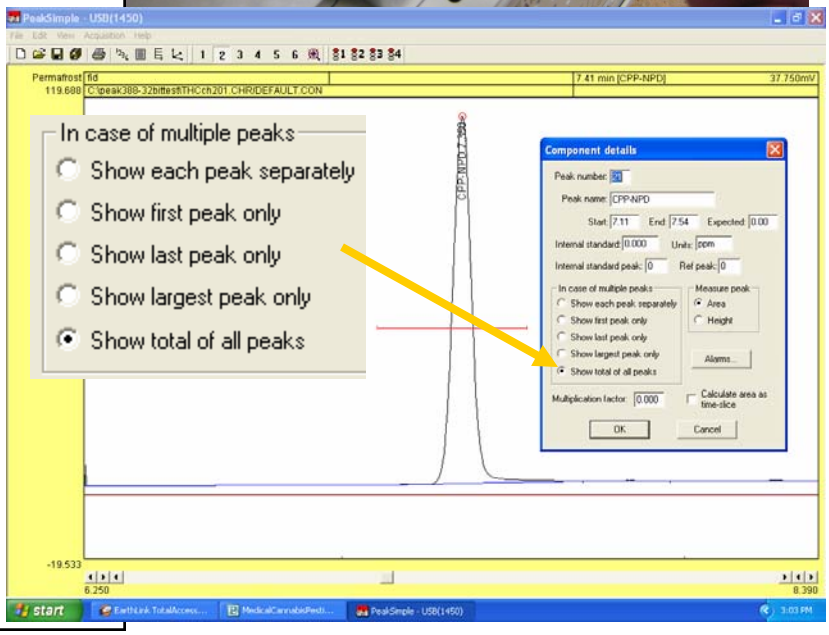
Then desorb using the standard program and events.



There should be a single peak on the NPD and DELCD channels.

Create a retention window for the CPP peak in the NPD channel and another similar retention window in the DELCD channel.

Notice that the retention window has "Show total of all peaks" selected



Medical Cannabis Pesticide Screening using the SRI 8610C GC

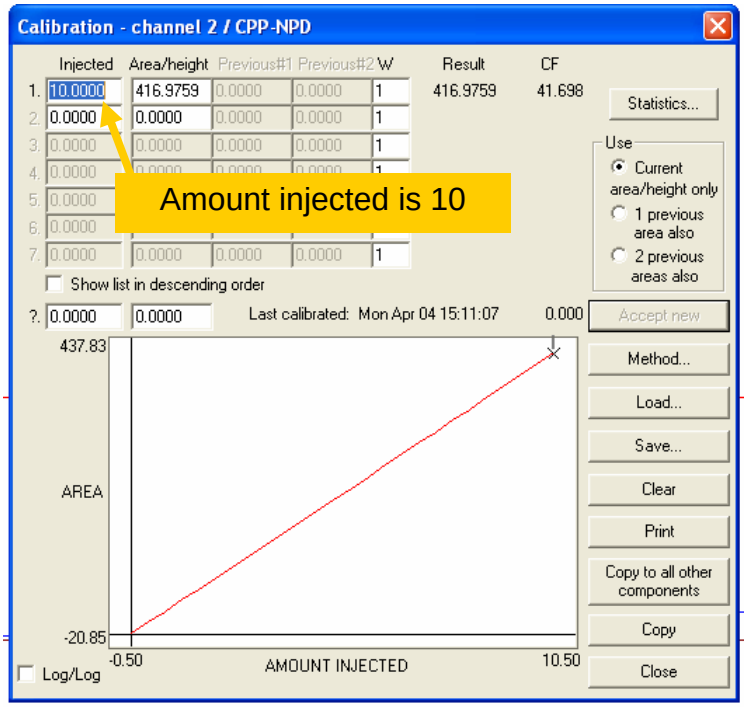
Create a calibration curve for the CPP in both NPD and DELCD channels. Note that the amount injected is set to 10.

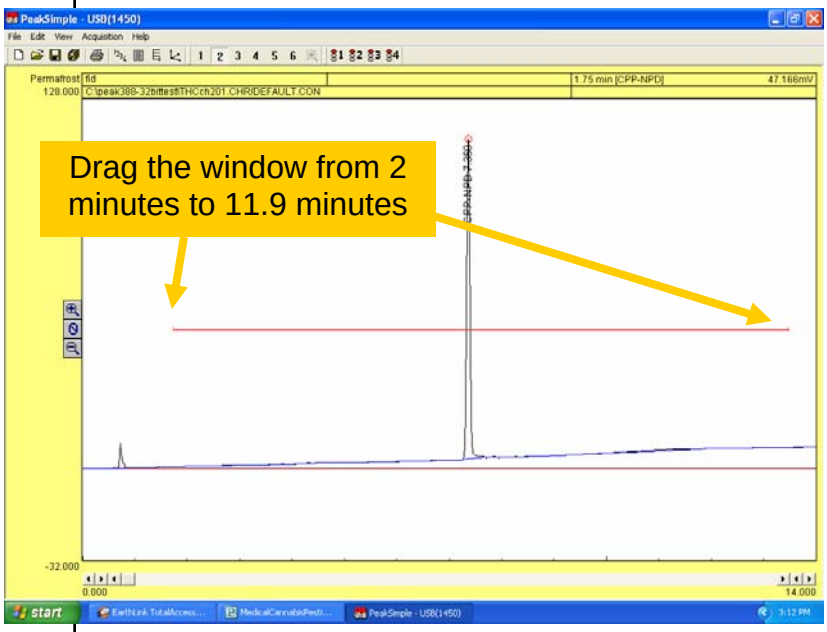
We injected 1ul of CPP standard which contains 1000 nanograms of CPP. Since we will be desorbing 100milligrams of cannabis, 1000 nanograms is 10ppm, hence the number 10 in the amount injected column.

Drag the retention window across the entire screen except for the first 2 minutes. This will have the effect of adding up all the peaks detected during the analysis and applying the CPP calibration to the total of the peaks, regardless of whether a particular peak is CPP or another pesticide.

Unlike the potency analysis where the results are reported in Percent, the pesticide results are reported in ppm (parts per million) because the concentration should be very low.

1,000,000 ppm =100%
100,000ppm=10%
10,000ppm=1%
1000ppm=.1%
100ppm=.01%
10ppm=.001%
1ppm=.0001%

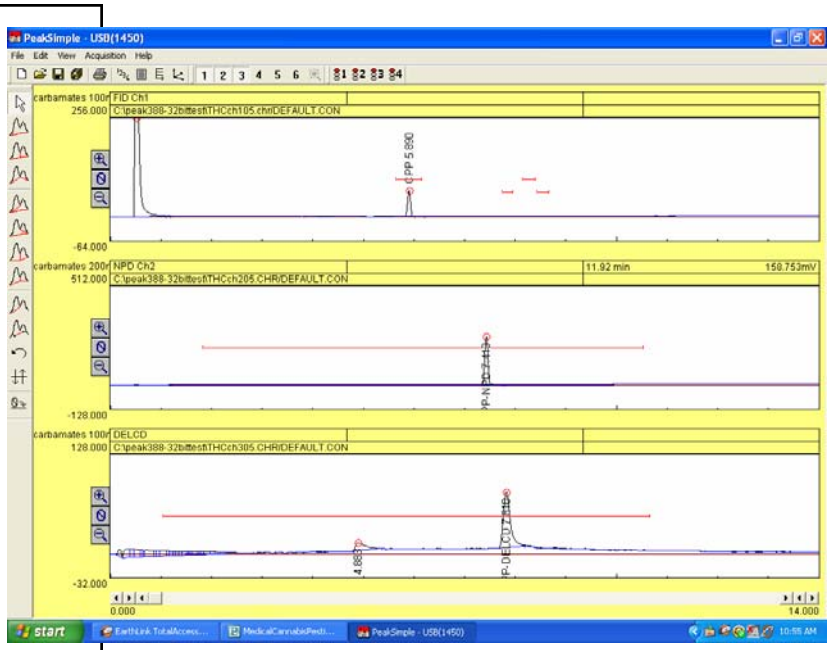




Medical Cannabis Pesticide Screening using the SRI 8610C GC

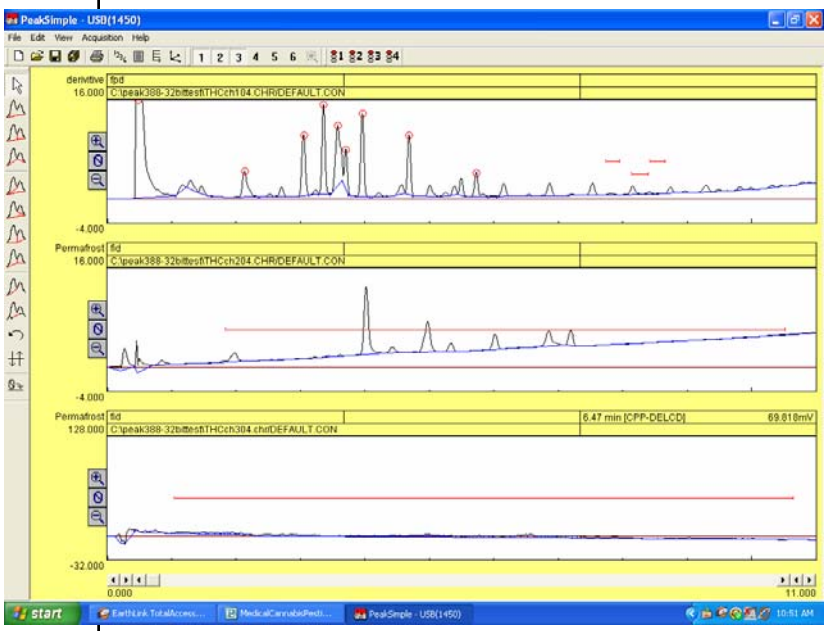
The chromatograms at right show the CPP peak on all three channels. The top channel (FID) was injected with the CPP standard just for comparison. Normally the FID channel is used for potency (CBD, THC, CBN).

The NPD (channel 2) and the DELCD (channel 3) show the CPP standard desorbed from the desorber tube.



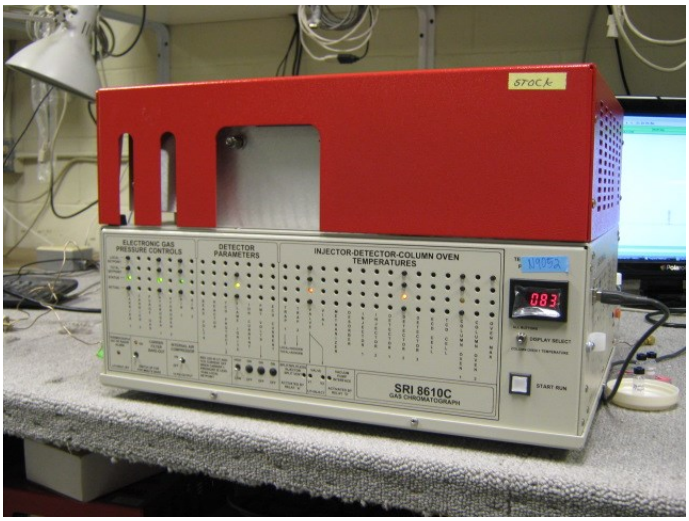
The chromatograms to the right show carbamate pesticides.

You can see the NPD responds but the DELCD does not. Since the carbamates do not have chlorine this makes sense.

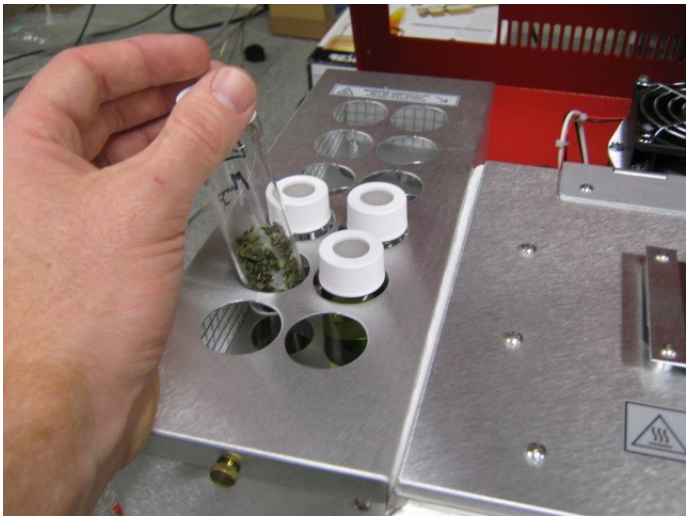


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

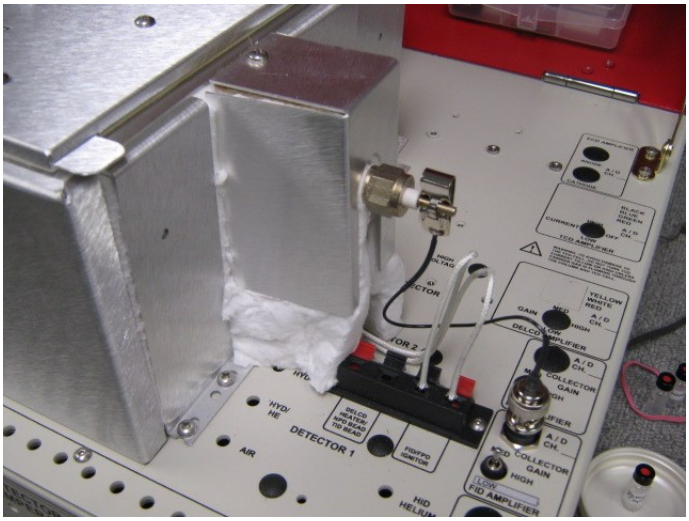
The SRI Medical Herb Potency 8610C GC is shown at right. This GC can also be used to test for residual solvents (i.e. butane, acetone, gasoline residue, etc.) in medical cannabis. These solvents are used in the extraction process to create medical cannabis hash oils and concentrates.



The 12 vial sample heater (incubator) aids in extraction of samples for potency testing, but can also be helpful in residual solvent analysis since the added heat makes any solvents more concentrated in the gas headspace in the vial.



The GC includes SRI's Flame-Ionization Detector (FID) which is sensitive to hydrocarbons (solvents, terpenes, and cannabinoid molecules).



Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

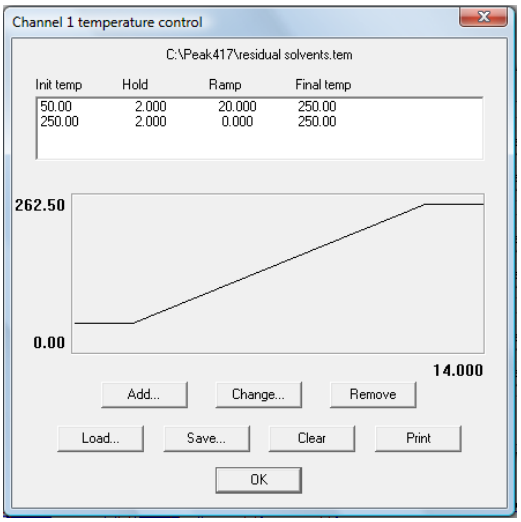
Solvents used to make cannabis extractions commonly include:
Butane
Isopropanal Alcohol
Acetone
Ethyl Alcohol (Ethanol)
Methyl Alcohol (Methanol)
Petroleum Ether
And in some cases Naphtha or even Gasoline (which contains hazardous chemicals like Benzene, Toluene, and Xylene, also known as BTEX).

Many types of columns could be used to separate these molecules, but SRI suggests a 15 meter MXT-1 with a 5 micron film thickness and .53mm id. This column can distinguish between solvents like pentane and hexane and does a good job of separating terpene molecules.

The residual solvent analysis can also be performed on the MXT-500 column that comes standard with the Potency GC, but the separation of volatile hydrocarbons will not be as good. For the best separation of terpene molecules, a 30 meter MXT-Wax is recommended but solvent separation will not be as good, and buying the column will be more expensive. As with all GC analysis, the operator must decide what compounds are most important to detect and select the proper column accordingly.

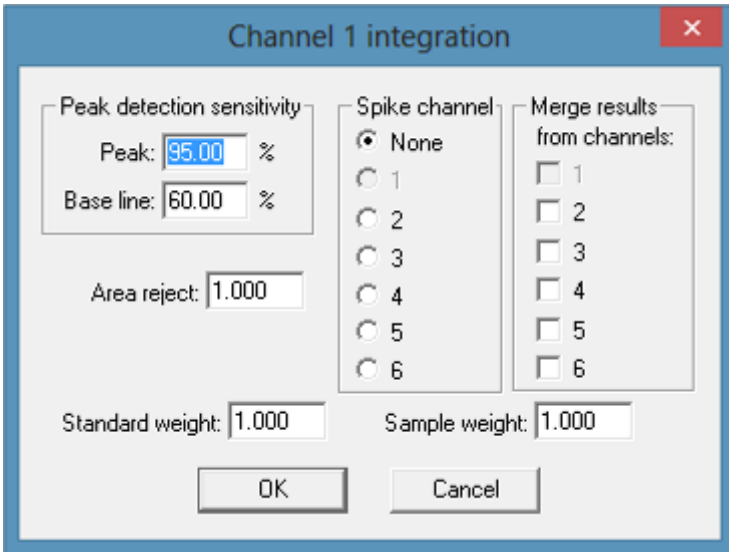
Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

Set the column oven temperature as shown at right. Although we are only interested in the early eluting solvents and adulterants, the "heavier" terpene molecules are also injected onto the column, and these must be allowed time to come out. The light hydrocarbons come out during the two minute hold, BTEX between 50 and 130 degrees, and the terpenes after that. The final temperature hold at 250 ensures that the heaviest molecules are "baked-out" of the column.



Thus, it can be convenient to perform butane and residual solvent ***and*** terpene analysis in one run. For more information on terpene testing, please see the tutorial describing medical cannabis terpene analysis.

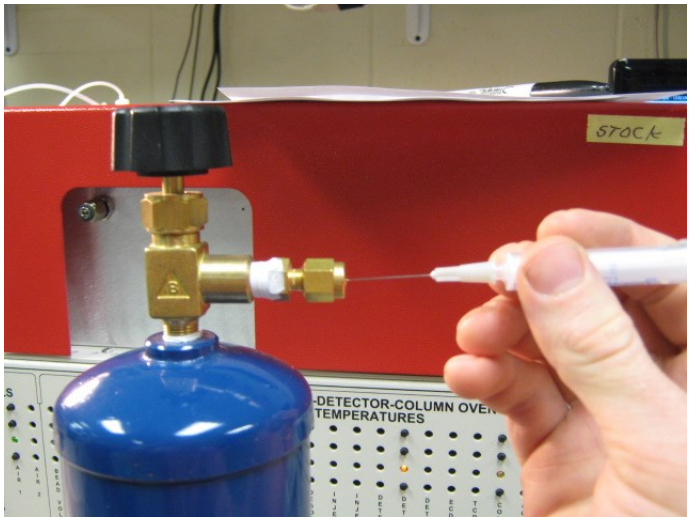
Set the Integration parameters as shown.



Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

In order to identify residual solvents in cannabis samples known standards must be injected. There are many ways to do this, but SRI recommends using a C1 to C6 gas standard at 0.1% concentration (1000 ppm for each gas). You can pick a gas standard from Grace Davison (part # M7017).

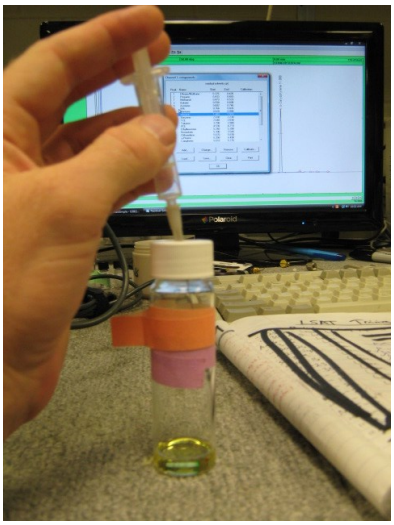
Pressurize the gas cylinder by turning the release valve slightly counterclockwise. Pierce the septum with a 3 mL gas syringe and withdraw 1 mL of gas. Remove the syringe from the gas sample bottle.



Or, alternatively, place the 3 mL syringe needle into a standard disposable lighter and suck out 1 mL of butane.

Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

To identify gasoline and its constituents that remain after evaporation (BTEX) obtain some gasoline and place it into an airtight vial. Using the 3 mL syringe, suck out 1 mL of headspace gas from the top of the vial.

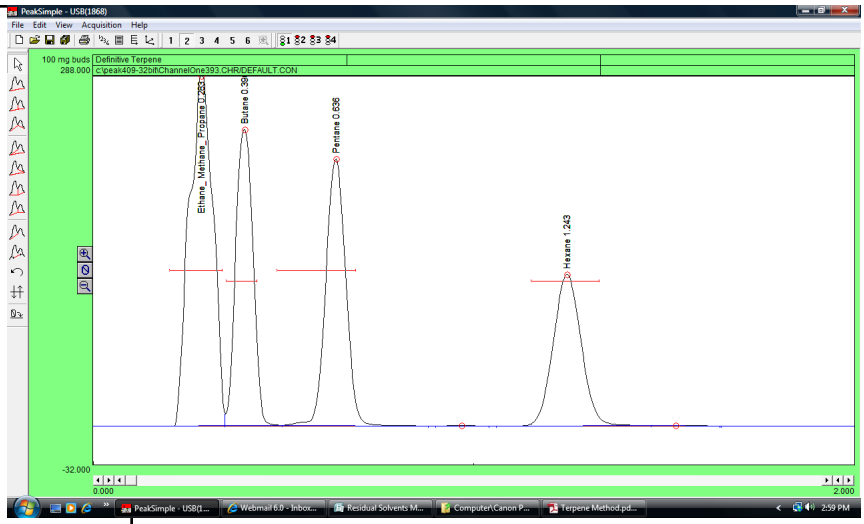


With the syringe plunger still at the 1mL mark, place the needle up against the septum of the injection port (but not poking through it yet). Press the Start Run button or press the spacebar on the keyboard.

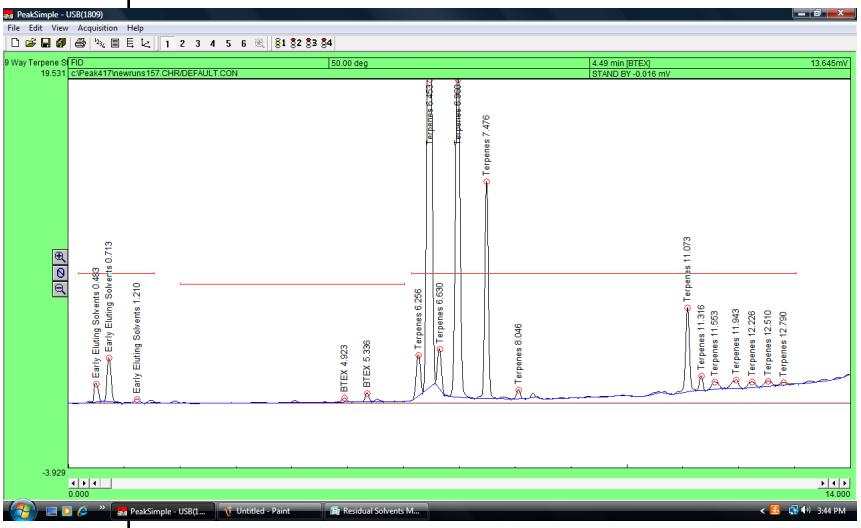
Insert the syringe all the way through the septum as far as it will go. Immediately depress the plunger and quickly withdraw the syringe.

Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

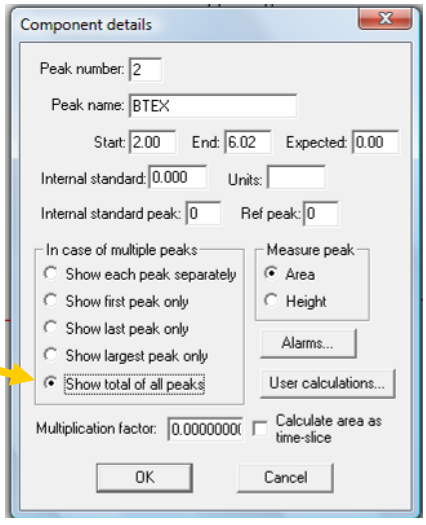
After injecting the C1—C6 standard we see four peaks: ethane, methane, and propane (which all elute together); butane; pentane; and hexane. Identify the peaks so that each peak is defined by a "retention window". See the PeakSimple tutorial describing the process of creating retention windows.



Since it may be difficult, if not impossible, to obtain reference standards for all the various residual solvents in cannabis it may be more practical to place blanket retention windows over categories of residual solvents. In the chromatogram to the right, one retention window covers the organic solvents, the second covers BTEX, and the third encompasses all the terpenes.



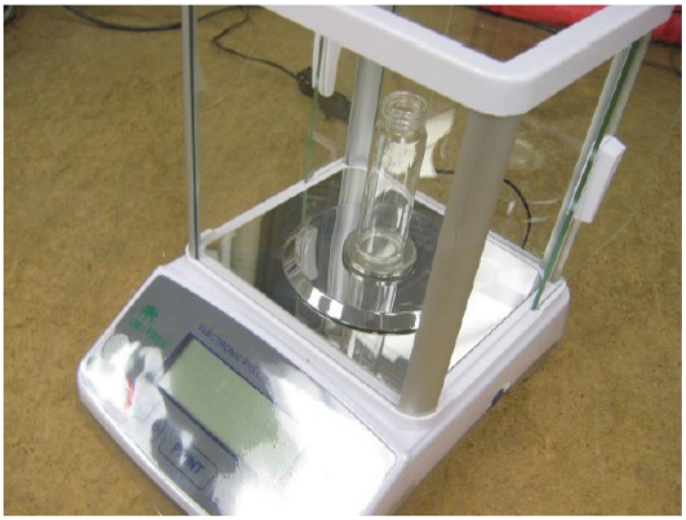
In this case, all the peaks under the retention window need to be quantified. In the Edit Component screen select "Show total of all peaks".



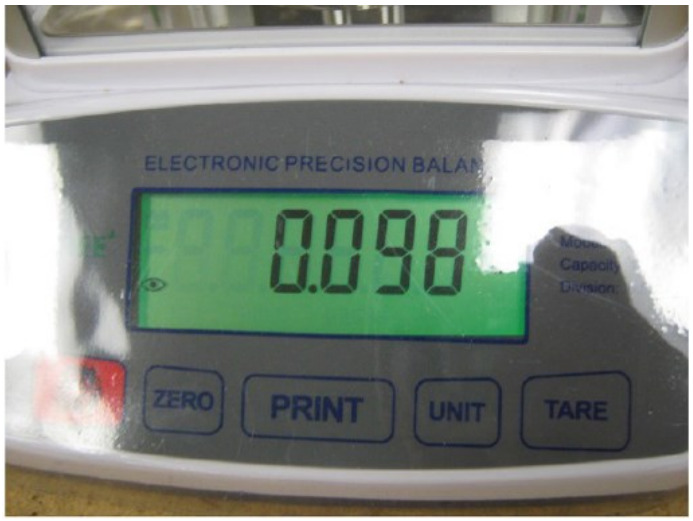
Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

Remove the cap from a 40mL vial and place it on a balance capable of reading 1 milligram (.001 gram). A balance like this can be purchased brand new for less than $300 on eBay.

With the 40mL vial on the balance, tare the reading (make the reading 0.000). Carefully add 100 milligrams of manicured cannabis to the vial. Drop the bits of cannabis into the vial slowly until the reading is close to 100 milligrams.

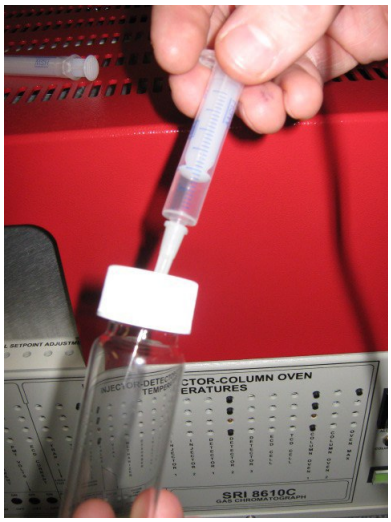


Don't worry if you are slightly under or above 100. In the photo at right, the reading is 98 milligrams which is close enough. Qualitative butane and residual solvent analysis does not depend on an exact measurement of sample, but the operator may find it advantageous to use the same sample for a subsequent potency analysis. In this case, the reading on the scale will be important in properly measuring the cannabis sample. See the PeakSimple tutorial describing Medical Cannabis Potency.



Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

Seal the cap of the 40mL vial and let it sit for at least 15 minutes in the incubator. Use a 3mL gas syringe to extract 1mL of gas from the “headspace” of the sample vial.

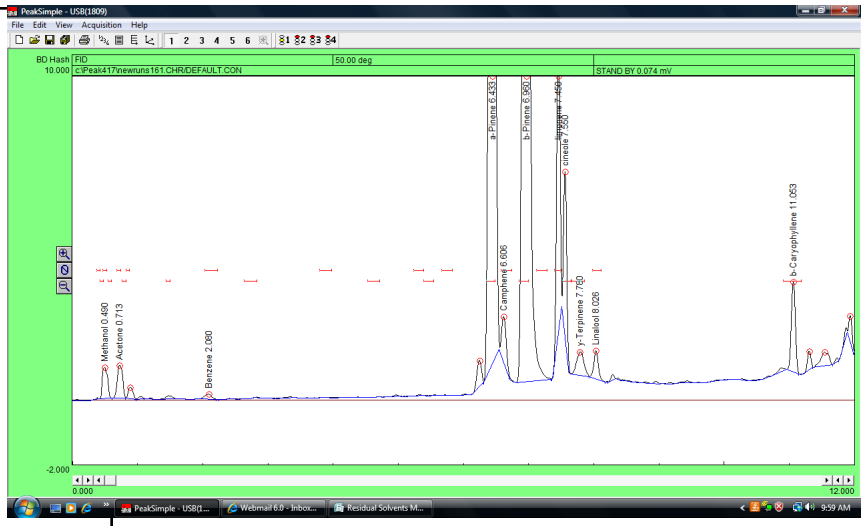


Inject the contents of the syringe into the injection port and start the run as shown previously.

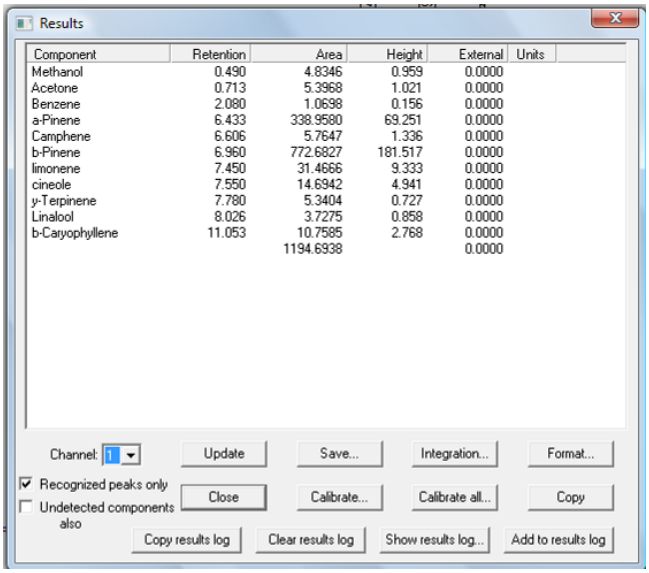
The picture at right shows a butane and residual solvent sample vial filled with 40 mL of extraction solvent and ready to be injected for cannabis potency analysis. See the PeakSimple tutorial describing the process for Medical Cannabis Potency testing.

Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

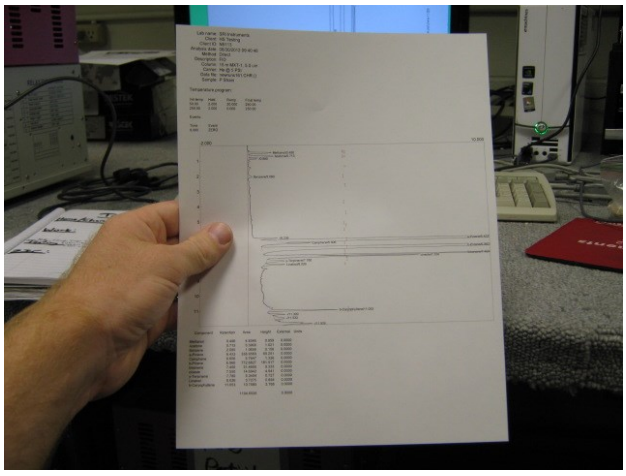
A real cannabis flower sample will look something like the chromatogram at right. This particular sample has standard levels of organic solvents (which are present in low levels naturally in plant matter) and multiple terpenes.



The Results screen will display the area counts of all peaks detected and identified with retention windows.

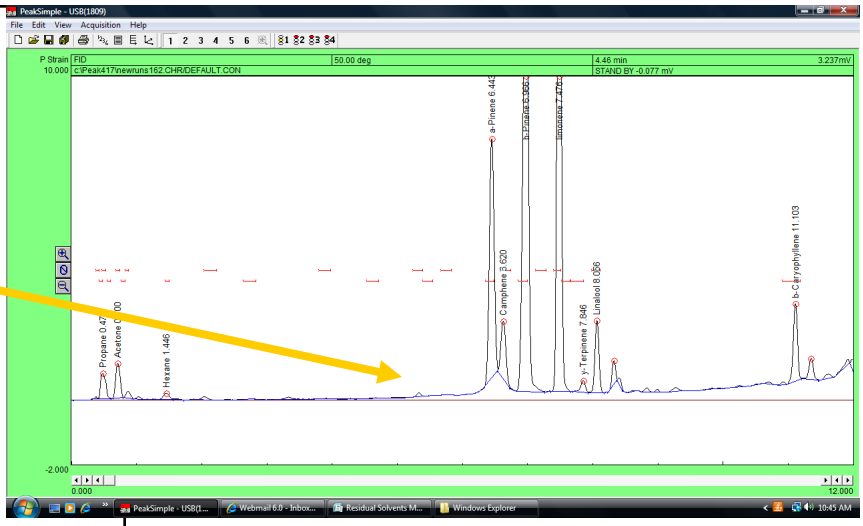


Print the chromatogram and results for a hardcopy record of the analysis.

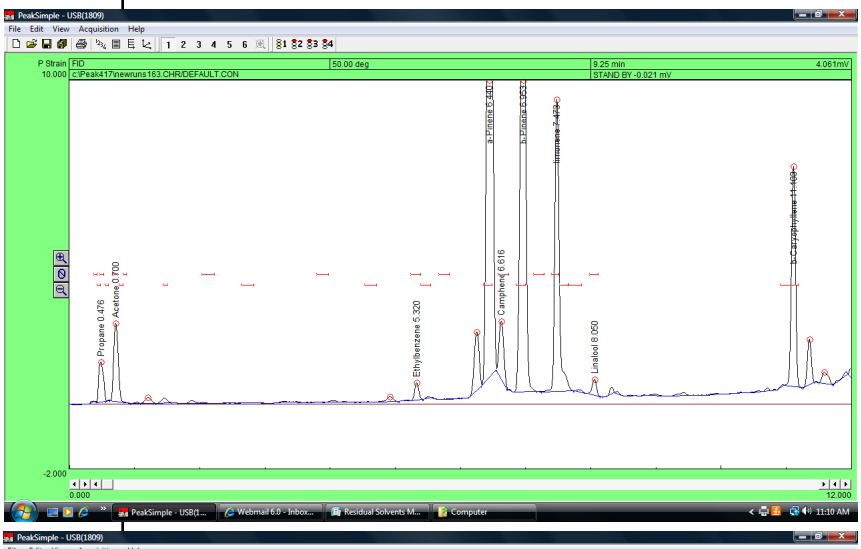


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

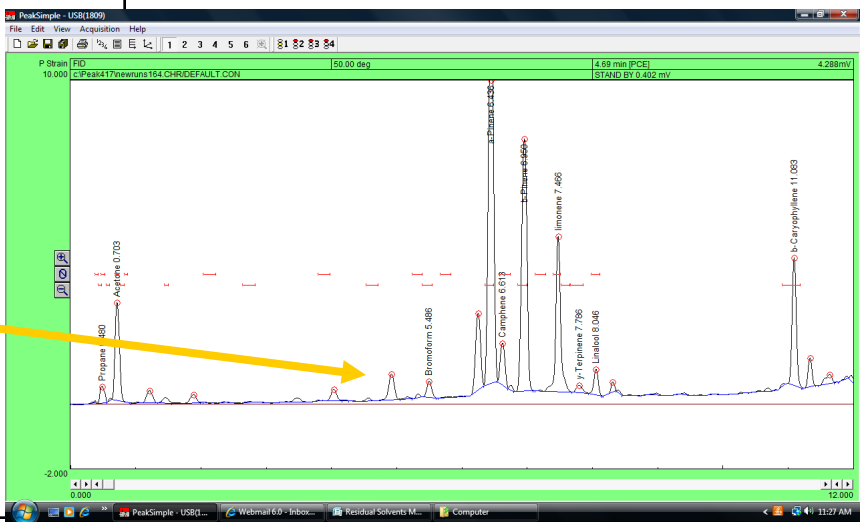
Super Sour Diesel flowers. Notice the lack of any BTEX-like adulterants.



Blue Dream concentrate.

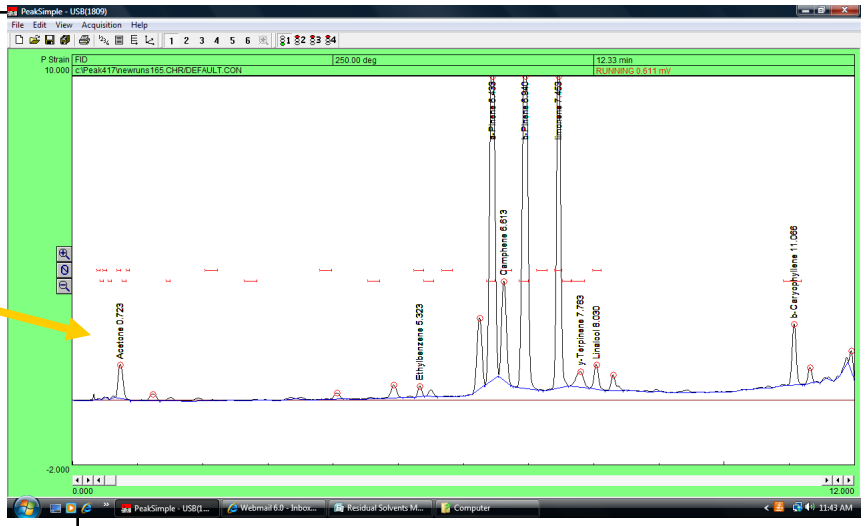


Mr. Nice concentrate. Notice the three small peaks in the BTEX area.

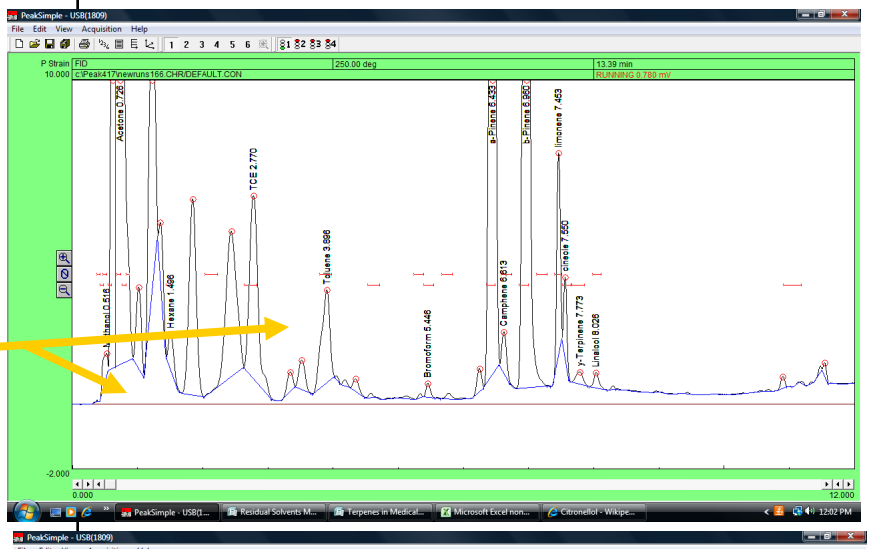


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

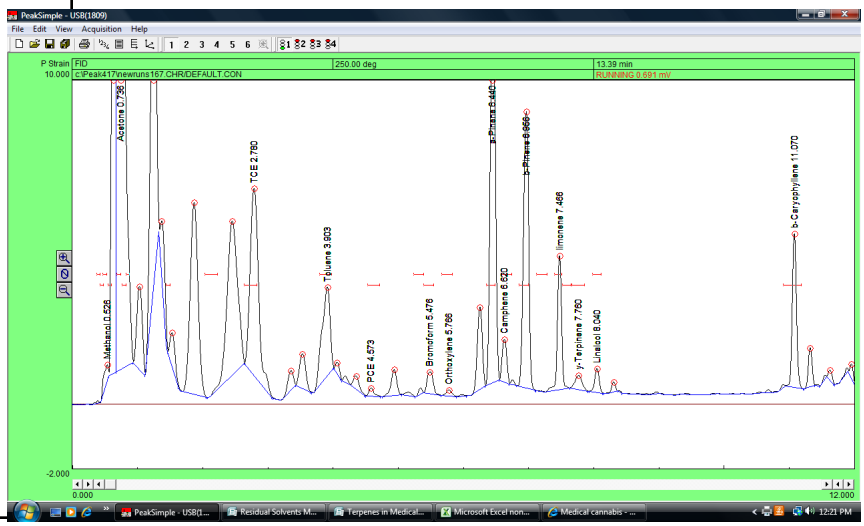
Sour OG Concentrate. Notice the low levels of organic solvents.



Outdoor-grown flower spiked with gasoline fumes. Notice the high concentrations of organic solvent and BTEX adulterants.

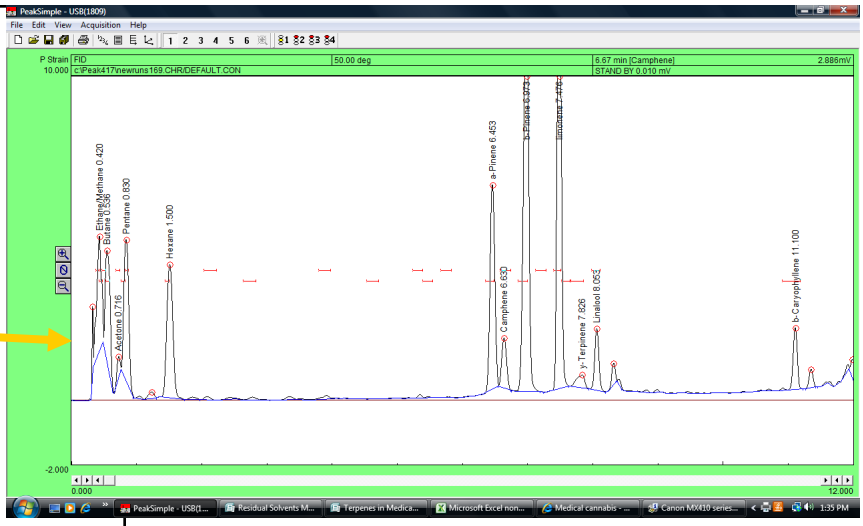


Mr. Nice Concentrate spiked with gasoline fumes.

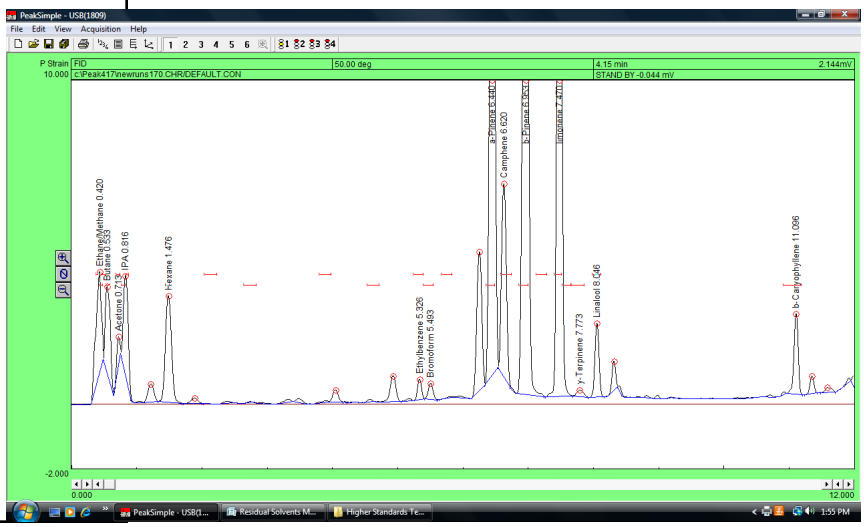


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

Super Sour Diesel Flowers spiked with organic solvents (C_1-C_6). Notice the elevated concentrations of organic solvents.



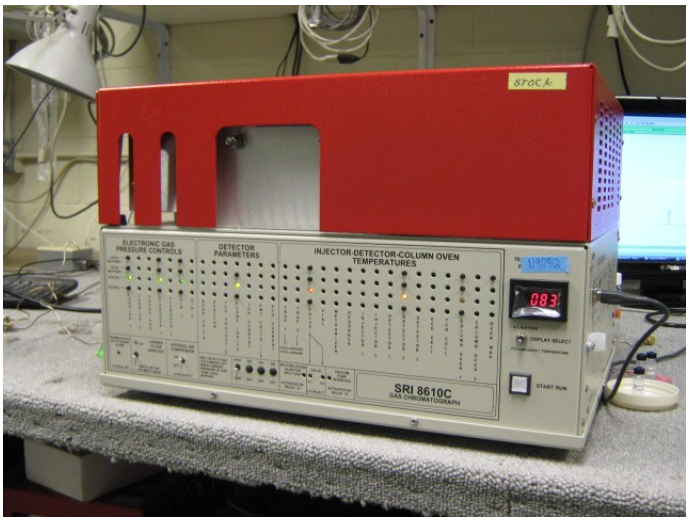
Sour OG concentrate spiked with organic solvents (C_1-C_6).



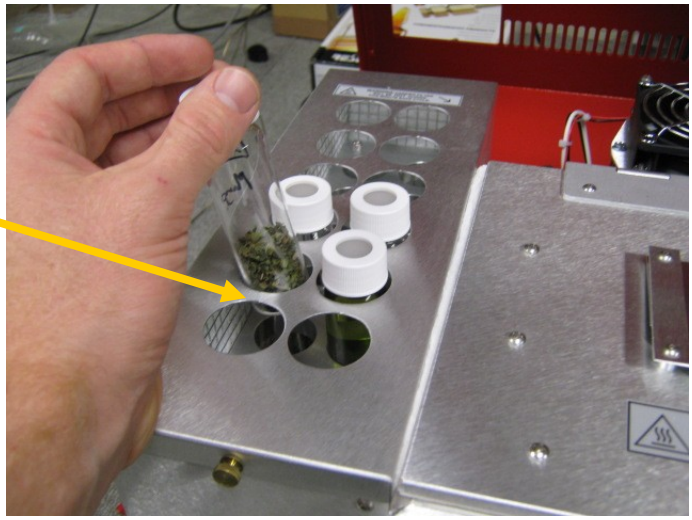
Real world medical cannabis samples will always contain some concentration of organic solvents (plant matter gives off trace amounts of ethane, methane and other gases as it slowly decays), so the presence of minute quantities of these gases should not be alarming. As the operator gains experience running samples they will be more qualified to determine what acceptable and unacceptable levels of these compounds are.

Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

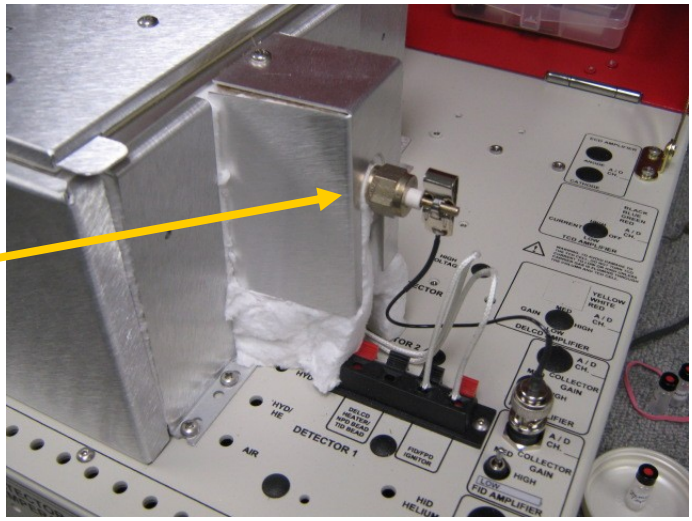
The SRI Medical Herb Potency 8610C GC is shown at right. This GC can also be used to test for the presence of terpenes in cannabis. The word terpene is usually taken to mean the non-psychoactive volatile molecules which make up the characteristic odor of cannabis even though delta-9-THC, CBD and other cannabinoids which are psychoactive, are also terpenes.



The 12 vial sample heater (incubator) aids in extraction for potency testing, but can also be helpful in terpene analysis since the added heat makes the terpenes more concentrated in the gas headspace in the vial.



The GC includes SRI's Flame-Ionization Detector (FID) which is sensitive to all the terpene and cannabinoid molecules.



Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

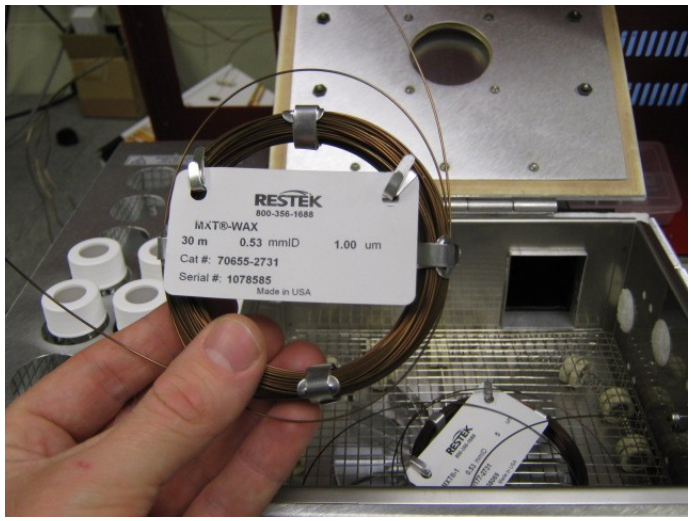
The terpene molecules commonly found in cannabis include:

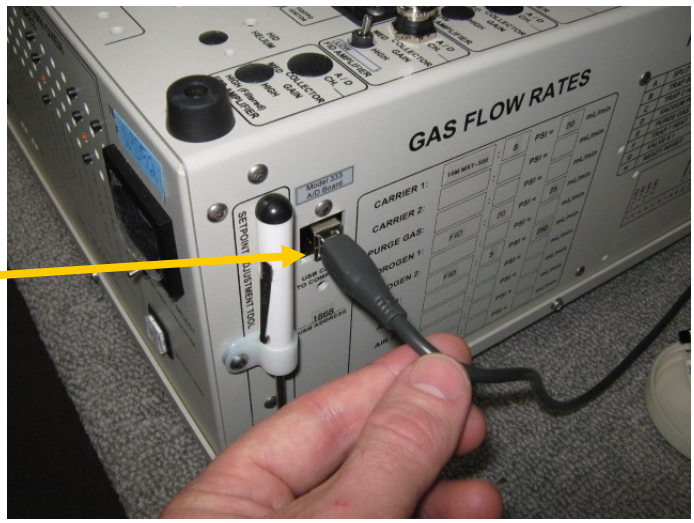
α-Pinene
β-Pinene
Camphene
Cineole (Eucalyptol)
γ-terpinene
β-Caryophyllene

But there are many more.

Many types of columns could be used to separate these molecules, but SRI currently suggests a 30meter MXT-WAX with 1 micron film thickness and .53mm id. The terpene analysis can be performed on other columns but the MXT-WAX provides the best separation.

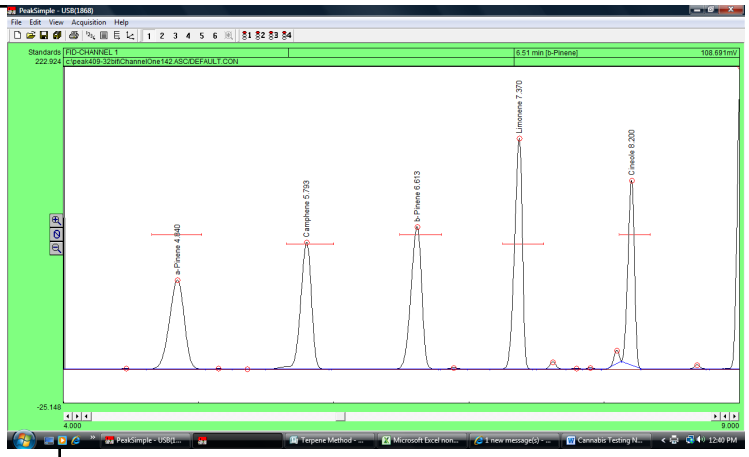
The entire GC plugs into any Windows computer using a USB cable.



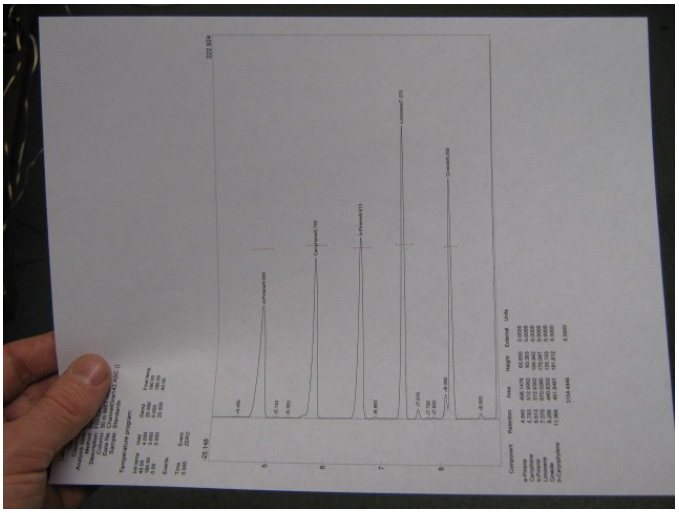


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

SRI's PeakSimple software is included with the GC. PeakSimple software collects the GC data and generates a qualitative result which can be printed or transferred to other programs such as Excel or Word.

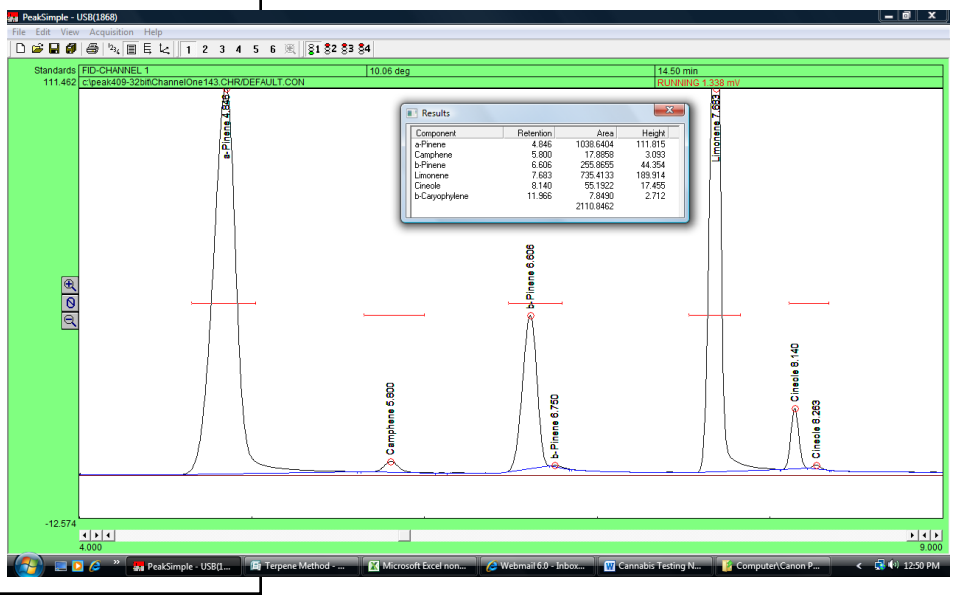


The chromatogram hardcopy printout at right shows a five terpene standard which was injected to identify these volatile odor compounds.



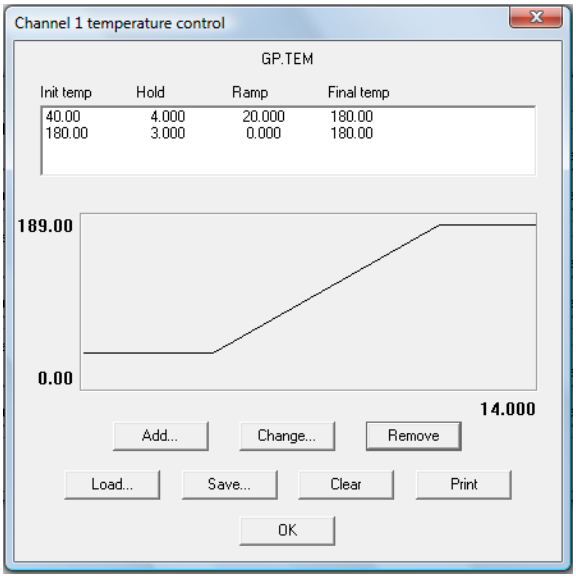
An actual cannabis sample run on the MXT-Wax column is shown at right.

The terpenes a-Pinene, Camphene, b-Pinene, Limonene, and Cineole are identified on the chromatogram.

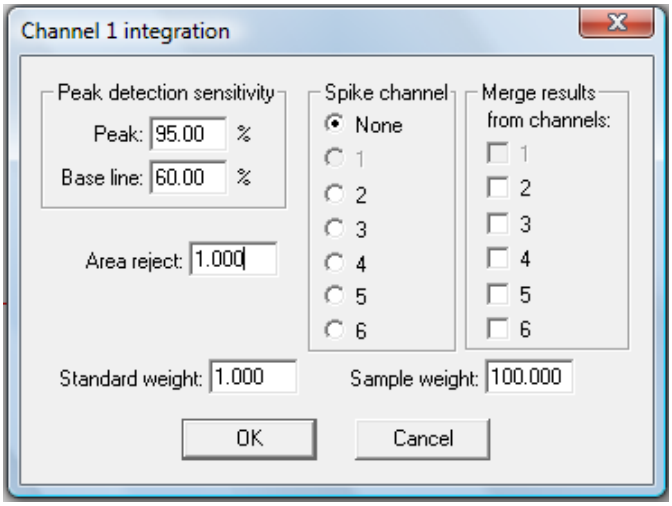


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

Set the column oven temperature as shown at right. It is best not to exceed 180C or the MXT-WAX column may be damaged.

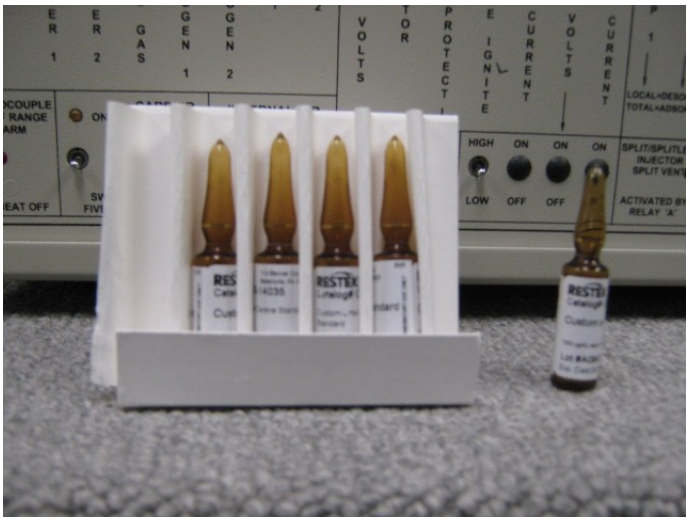


Set the Integration parameters as shown.

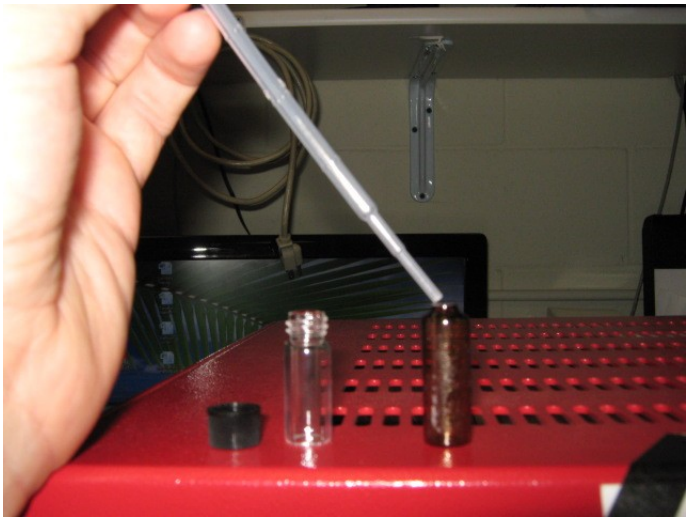


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

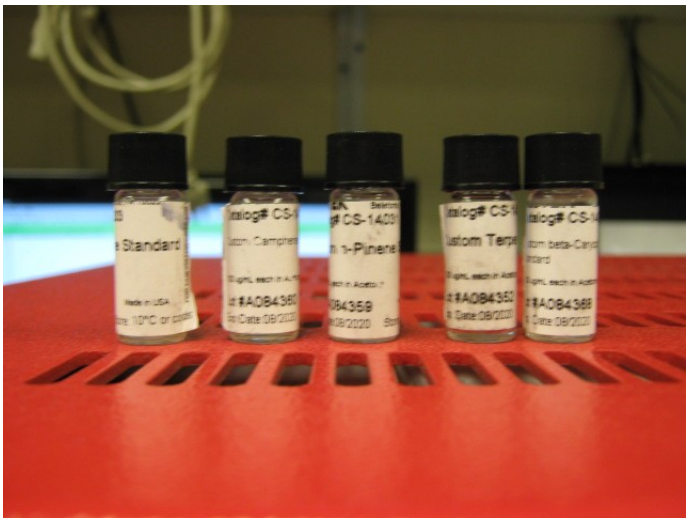
In order to identify terpenes in cannabis obtain the standards from a chromatography supplier like Restek (restek.com) (800) 356-1688.



Break the glass ampoule and transfer the contents into a 2ml septum vial (Restek #21154 and #24495). Restek provides one free vial with each standard.

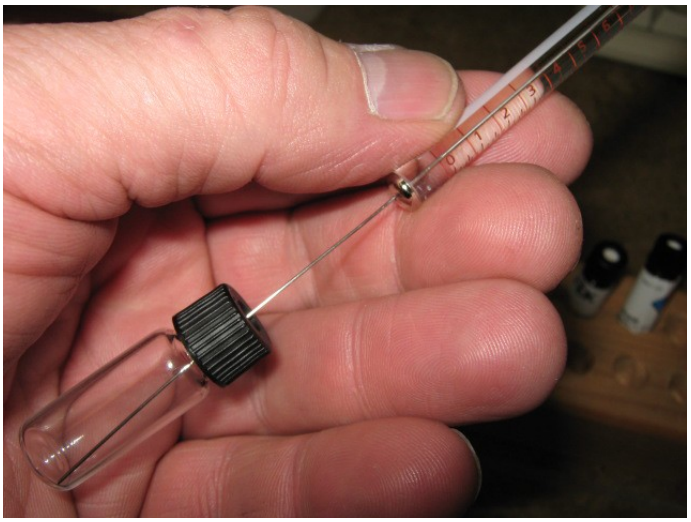


You will end up with one vial per terpene standard. There are 5-10 main terpenes in cannabis.

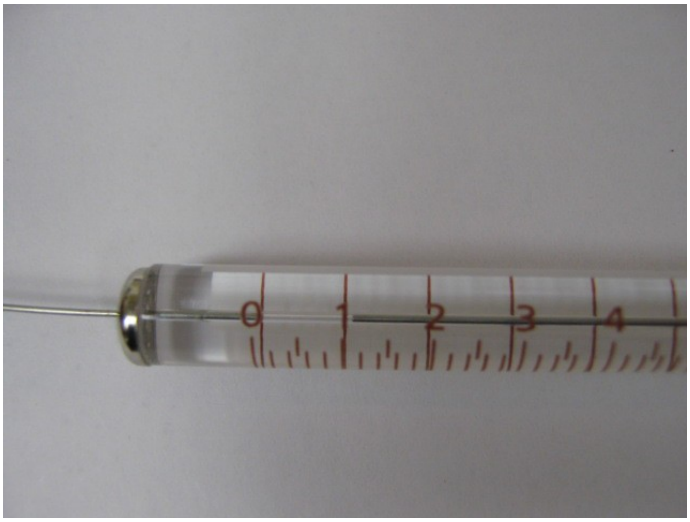


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

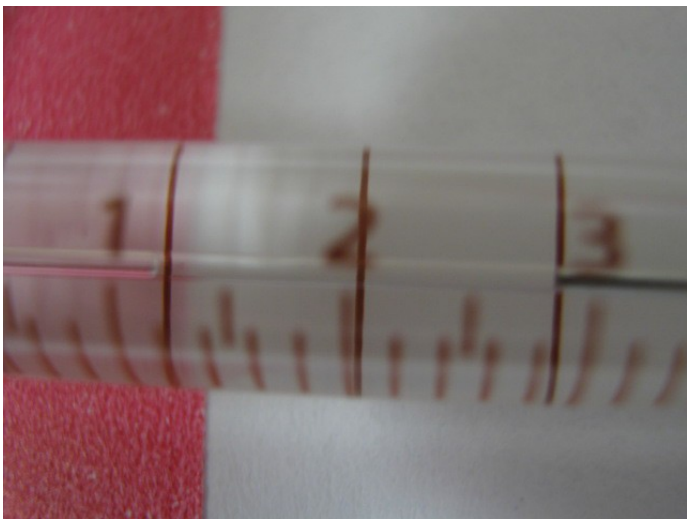
To qualitatively identify each terpene, the standard must be injected into the GC. Rinse the syringe first, then: use the 10uL syringe delivered with the GC (SRI #8670-9550) to withdraw 3-4uL of the standard. Puncture the septum rather than open the vial to avoid letting the methanol solvent evaporate each time the vial is opened. Pump the plunger several times to get rid of air bubbles.



With 3-4uL of liquid in the syringe, hold the needle vertically or at least slanted upwards so any air bubbles will rise toward the needle. With air bubbles removed, push the plunger to the 1uL mark. It is important to be as precise as possible. Wipe the needle with your fingers or a tissue to remove any liquid from the outside of the needle.



Pull the plunger back to the 3uL mark and note the amount of liquid. It should be 1.6-1.8 uL because the needle also contains .6-.8uL and this adds to the 1uL you measured with the plunger.



Leave the plunger at the 3uL mark.

Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

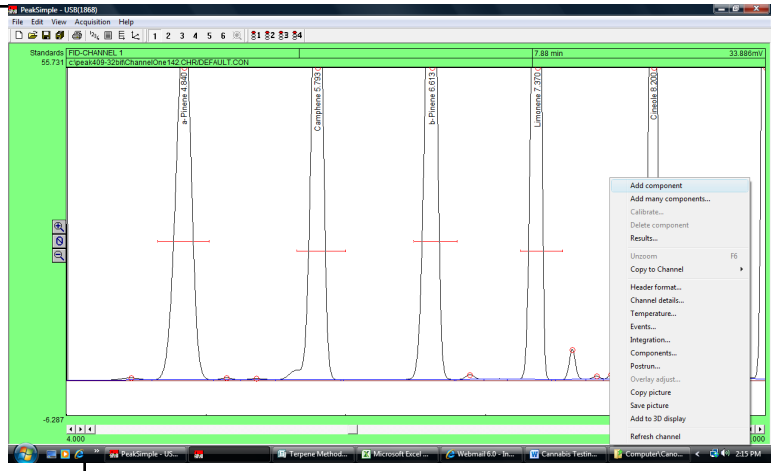
With the plunger still at the 3uL mark, place the needle up against the septum of the injection port (but not poking through it yet).

Press the Start Run button or press the spacebar on the key-board.

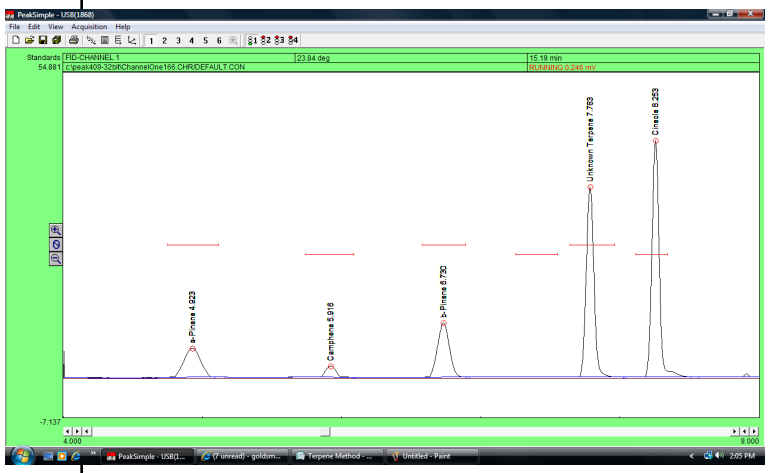
Insert the syringe all the way through the septum as far as it will go. Immediately depress the plunger. Twist the syringe one half turn (to wipe off any liquid on the tip of the needle) and then with-draw the syringe.

Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

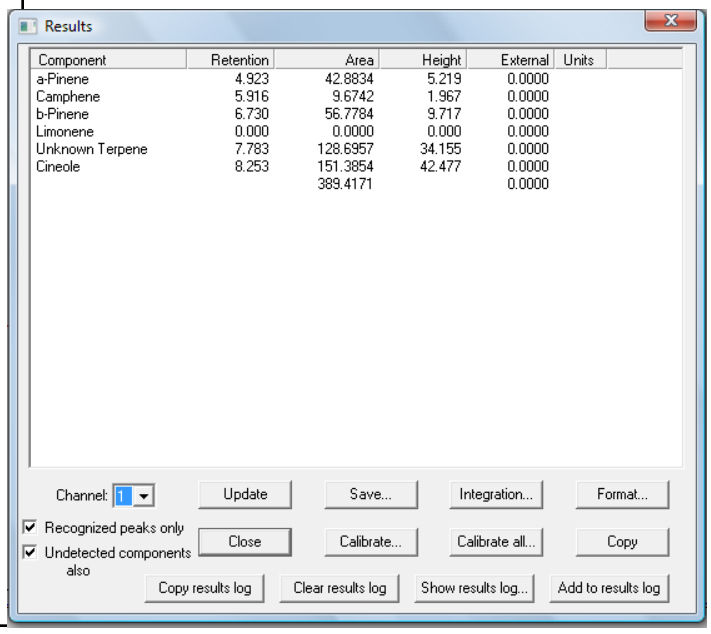
For this terpene standard we have five peaks. Identify the peaks so that each peak is defined by a “retention window”. See the PeakSimple tutorial describing the process of creating retention windows.



After qualitatively identifying the five terpene standards we can identify the same terpenes on subsequent sample runs of actual cannabis.



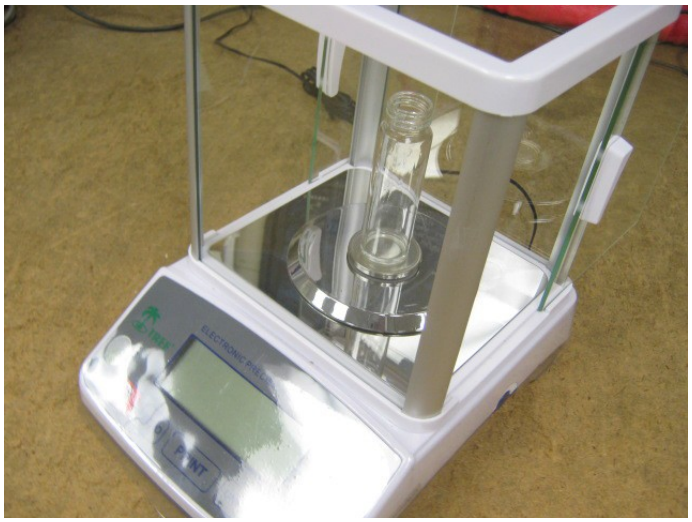
Navigate to the View/Results screen to see the report.



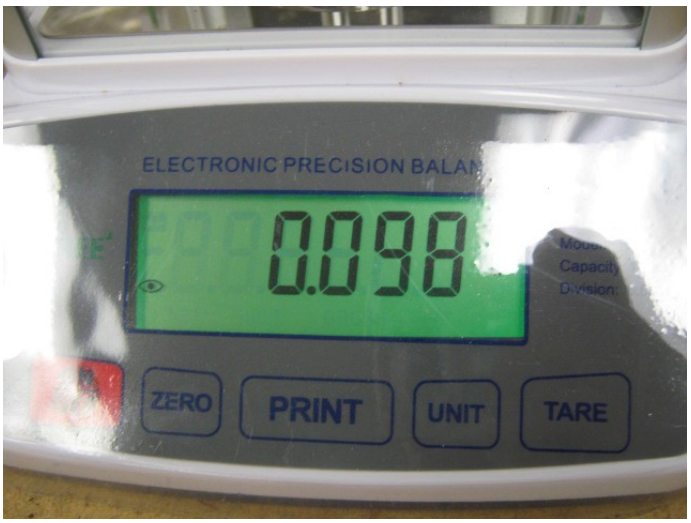
Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

Remove the cap from a 40mL vial and place it on a balance capable of reading 1 milligram (.001 gram). A balance like this can be purchased brand new for less than $300 on eBay.

With the 40mL vial on the balance, tare the reading (make the reading 0.000). Carefully add 100 milligrams of manicured cannabis to the vial. Drop the bits of cannabis into the vial slowly until the reading is close to 100 milligrams.



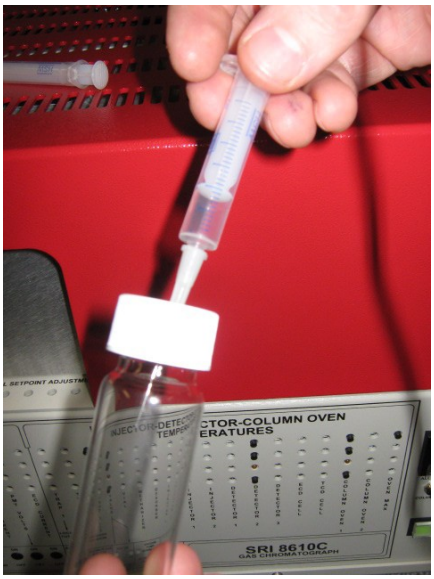
Don't worry if you are slightly under or above 100. In the photo at right, the reading is 98 milligrams which is close enough. Qualitative terpene analysis does not depend on an exact measurement of sample, but the operator may find it advantageous to use the same sample for a subsequent potency analysis. In this case, the reading on the scale will be important in properly measuring the cannabis sample. See the PeakSimple tutorial describing Medical Cannabis Potency.



Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

Seal the cap of the 40mL vial and let it sit for 30 minutes in the incubator.

Use a 3mL gas syringe to extract 1mL of gas from the “headspace” of the sample vial.

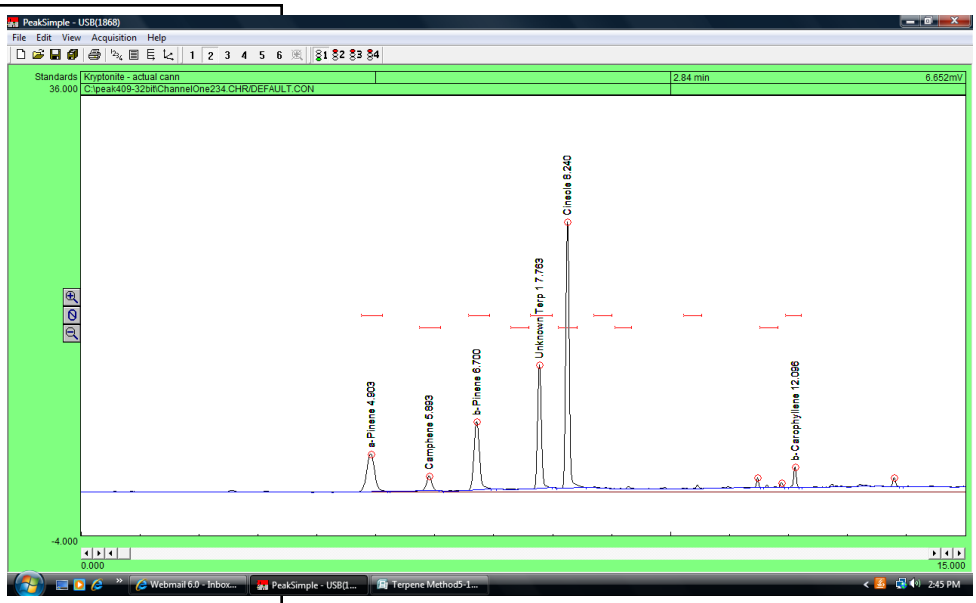


Inject the contents of the syringe into the injection port and start the run as shown previously.

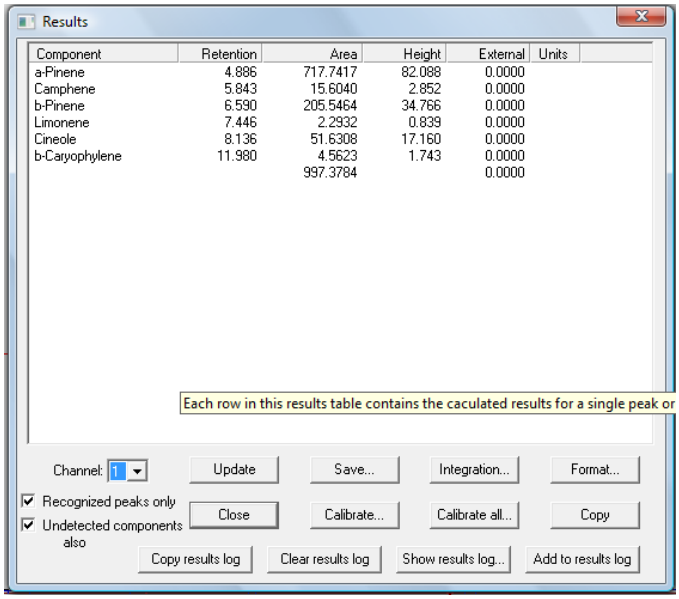
The picture at right shows a terpene sample vial filled to the neck with extraction solvent and ready to be injected for cannabis potency analysis. See the PeakSimple tutorial describing the process for Medical Cannabis Potency Testing.

Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

A real cannabis sample will look something like the chromatogram at right.



There may be several peaks under your known standard retention windows, there may be several unidentified terpenes without retention times.

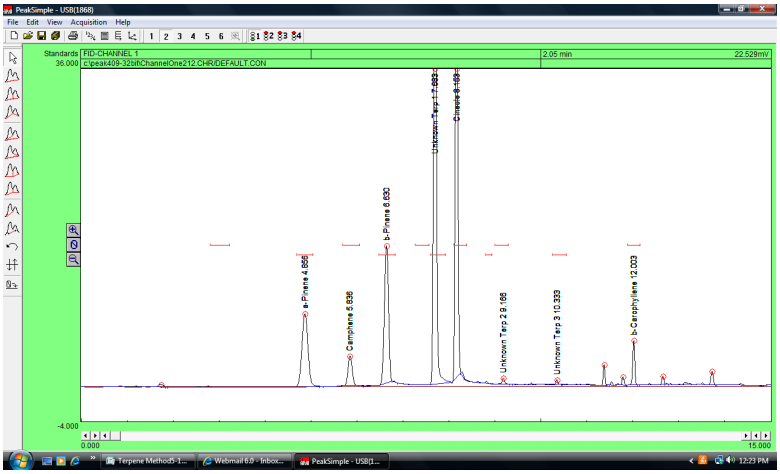


The Results screen will display the area counts of all peaks detected and identified with retention windows.

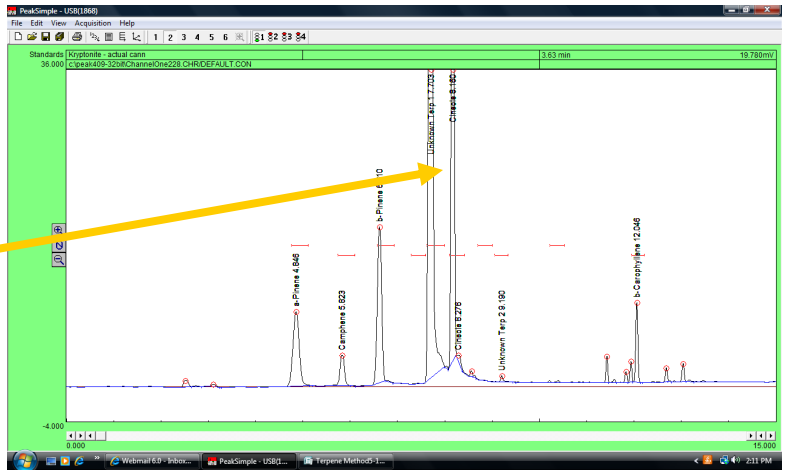
Print the chromatogram and results for a hardcopy record of the analysis.

Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

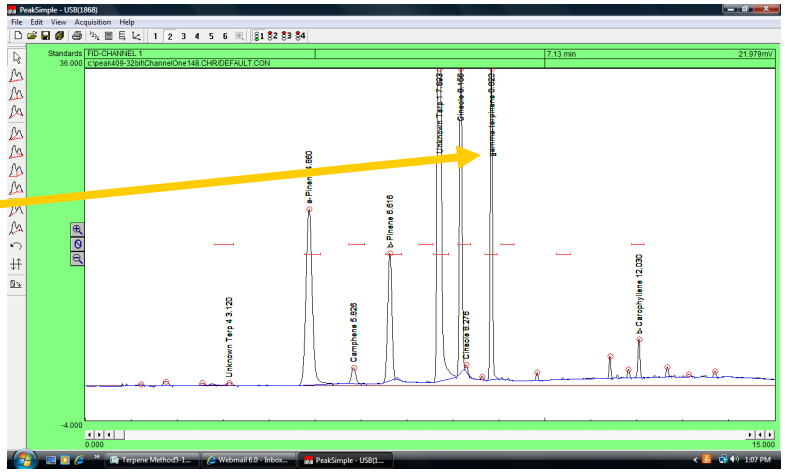
Here is what terpene analysis on the strain King Louie 13 OG looks like. Notice the presence of at least six terpenes: α– and β-Pinene, Camphene, Myrcene, Cineole, and β-Carophyllene.



This is a strain called Gush. Notice how, like many strains, it is highly concentrated in both myrcene and cineole. Also known as eucalyptol, cineole smells spicy, camphor-like, refreshing, and minty.

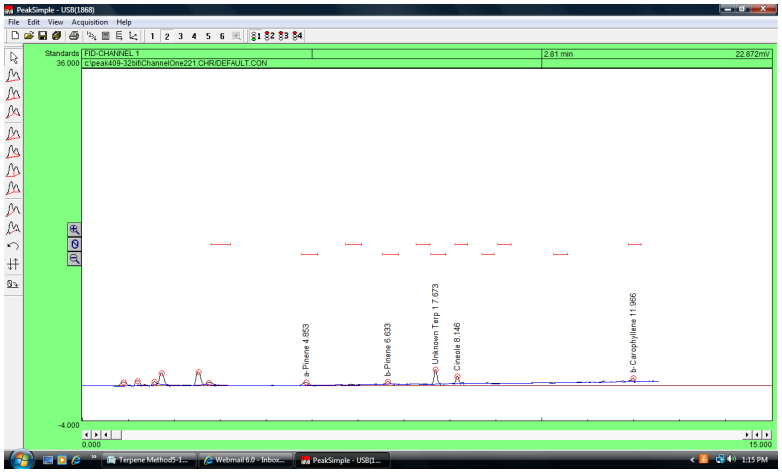


This sample of Green Crack has high concentrations of γ-terpinene. This terpene has a characteristic low-intensity lemon smell and is commonly used as an aromatic in foods, soaps, perfumes, and flavors.

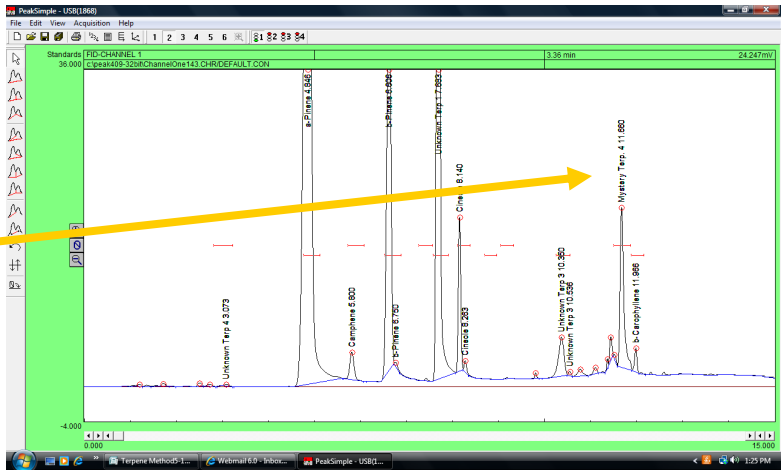


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

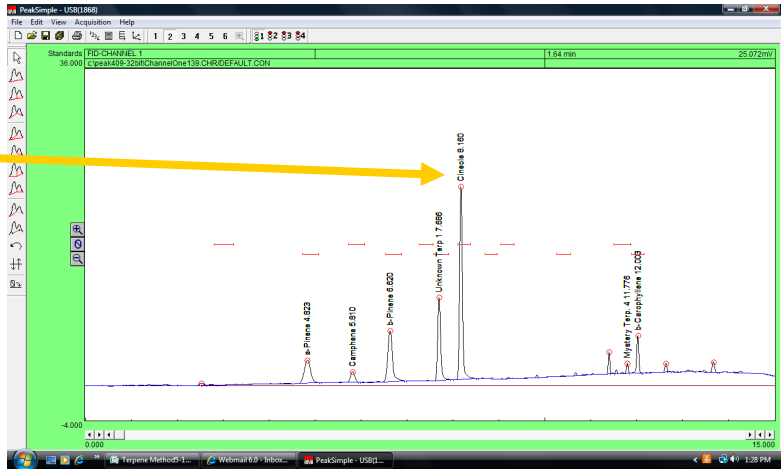
This sample was from visibly low-quality medical cannabis called Mango. Notice its overall low terpene concentrations.



This sample of Blue Dream was very high in overall terpene levels. Notice its high concentrations of an unknown terpene.

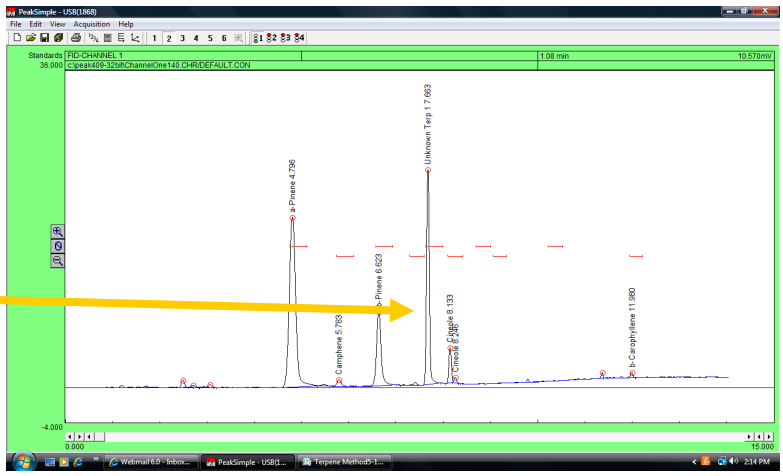


This is a strain called Super Sour Diesel. This chromatogram shows that it has the highest concentrations of the terpene cineole.

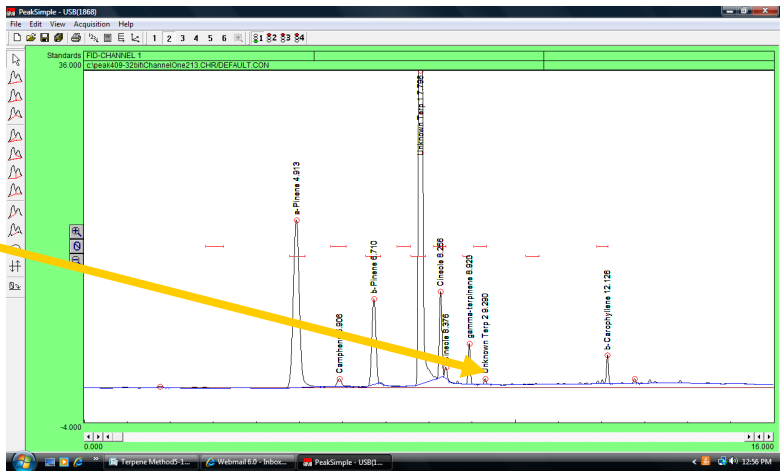


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

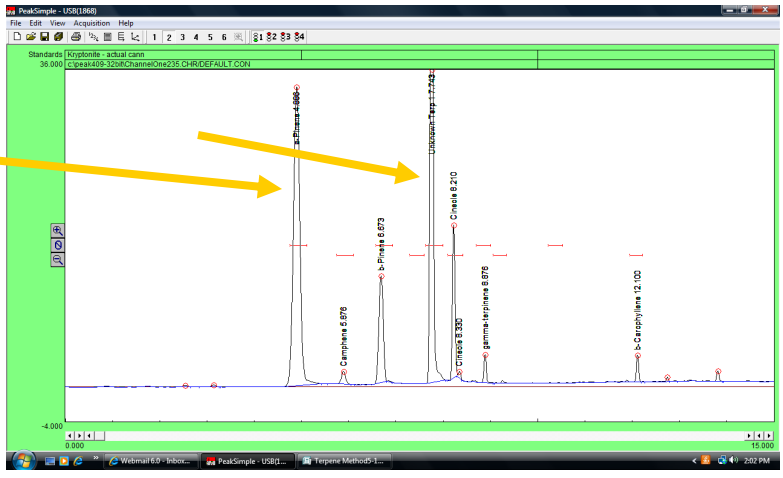
This is from a sample named Allen Wrench. Notice the high concentration of myrcene. This is typical of most strains as myrcene is the most common terpene in cannabis. Myrcene has a clove-like, earthy, vegetative, citrusy-mango smell.



This strain, AK-47, has a small concentration of an as yet unidentified terpene.

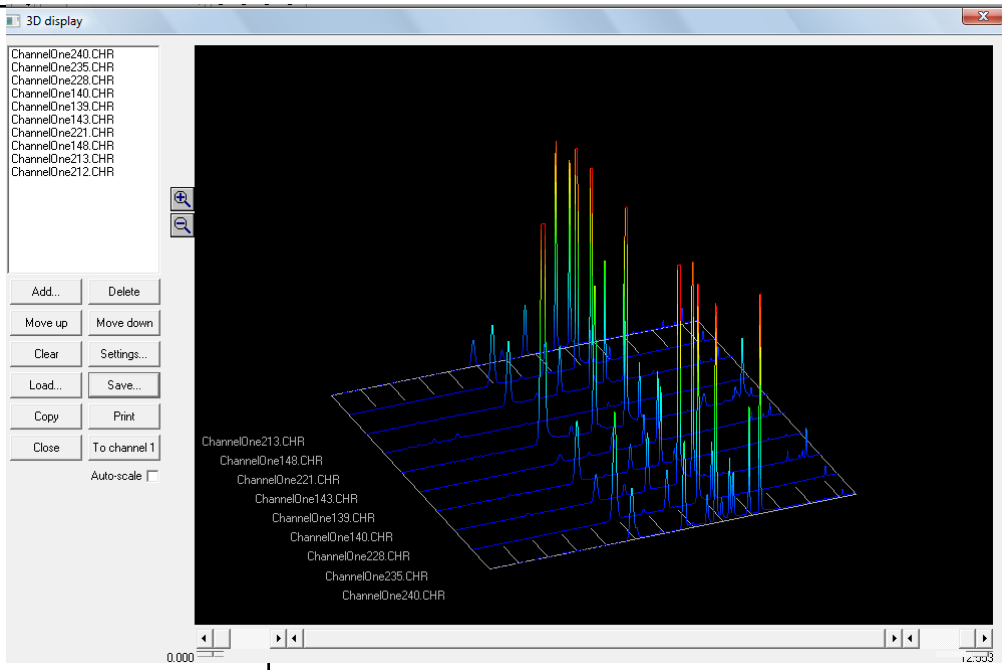


This is an outdoor variety of the strain Strawberry. Notice the nearly similar levels of α-pinene and myrcene. The terpene α-pinene has the characteristic odor of pine trees and is used in cleaning products like Pine-sol.



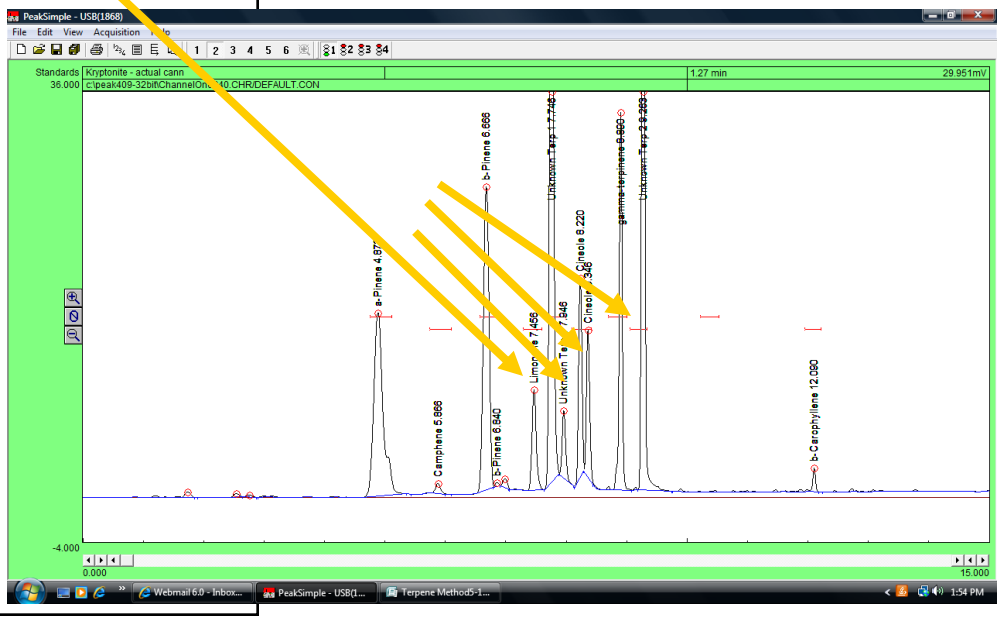
Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

The user can display multiple terpene analysis runs on PeakSimple's 3D display. This feature makes it easy to compare multiple cannabis strains and to look for patterns.



This last terpene analysis is from a strain called Blueberry Jack. Notice the number of significant peaks (well over ten) compared to the usual cannabis sample.

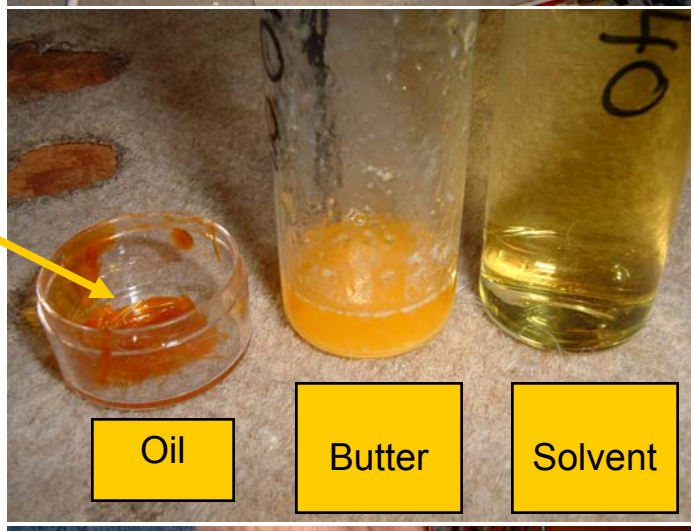
SRI Instruments welcomes your feedback, knowledge and experience with terpene analysis. Please contact us if you have any questions or information to provide.



easuring THC in Butter using the SRI C GC

The THC in butter analyses were performed using an SRI C GC configured for cannabinoid analysis.

milligrams of a cannabis oil was weighed into two identical ml vials. The oil was a CO ex-tract with an orange color. We used the oil for this test because it was very uniform in consistency.



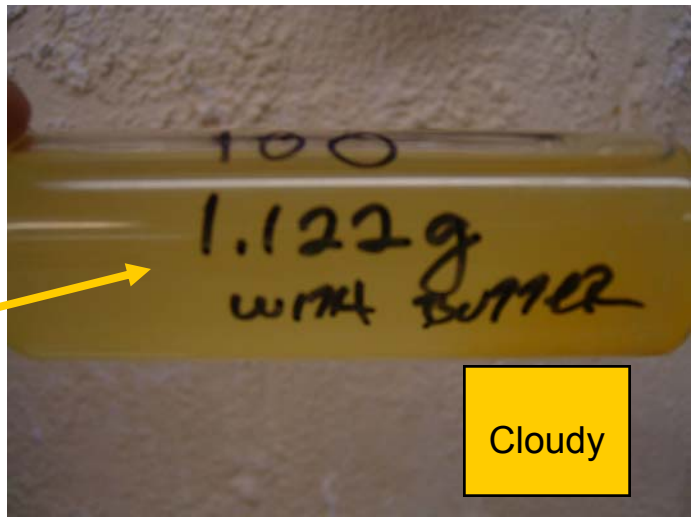
The first vial was filled with metha-nol and placed in the built-in sam-ple incubator which is part of this GC configuration. To the second vial was added gram of butter. The butter vial was placed in the incubator WITHOUT solvent until the butter melted and dissolved the cannabis oil. The cannabis oil could clearly be seen to dissolve in the butter. The incubator was set to C. A third vial with no oil was loaded with gram of butter for comparison.

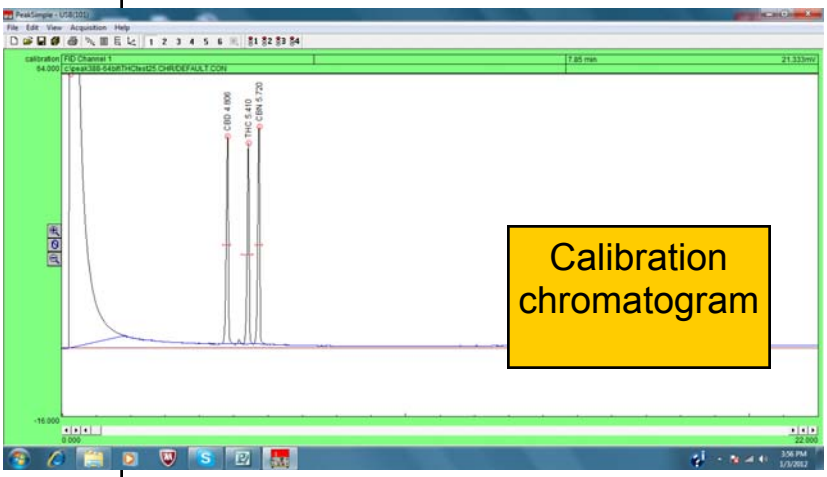
easuring THC in Butter using the SRI C GC

After minutes in the incubator the two butter vials were filled with methanol and placed back into the incubator. Once the methanol warmed to C the butter vials were shaken for seconds to disperse the butter into very fine droplets. This made a cloudy looking suspension The butter vials were again placed into the incubator for minutes.

After another minutes the butter solids dropped to the bottom of the vial leaving clear liquid in the top of the vial. Interestingly, the suspension did not clear at room temperature, only when heated in the incubator.

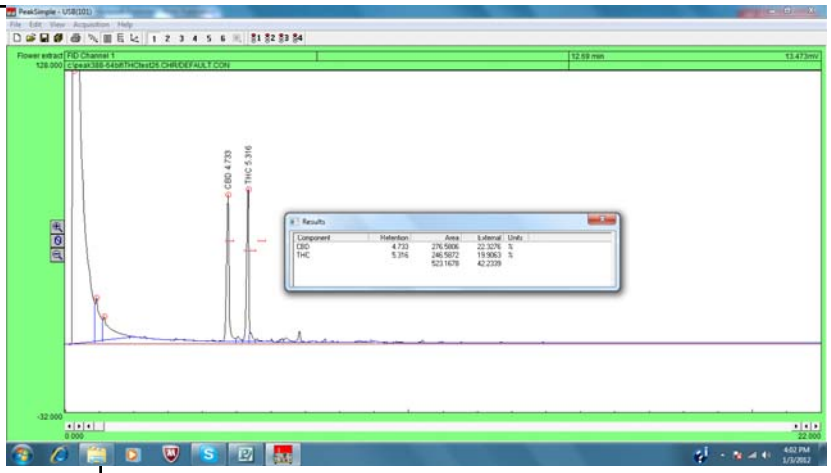
eanwhile the GC was calibrated with a mixture of CBD, delta THC and CBN each at a concentration of ng/ul. ul was injected on-column into a meter T capillary column with . mm id and a film thickness of . micron. The temperature program was set to start at C hold for . minutes , then ramp at degrees per minute to C then hold. The FID was set to C. Hydrogen carrier was used at psi or ml/min.



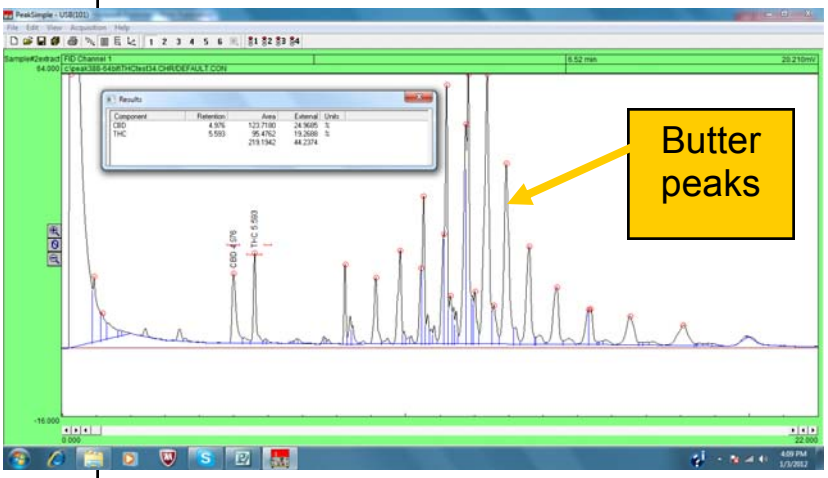


easuring THC in Butter using the SRI C GC

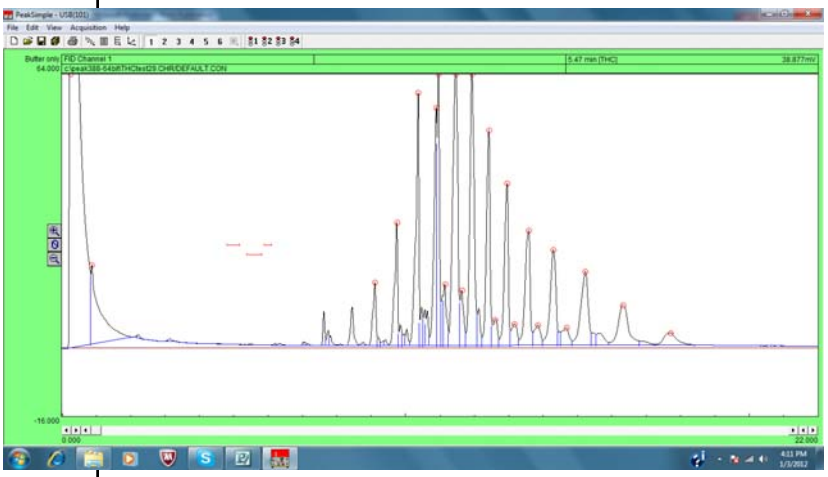
The oil only extract was injected and the results showed . for CBD and . for d THC. Presumably this particular oil was prepared from industrial hemp since the CBD was so high.



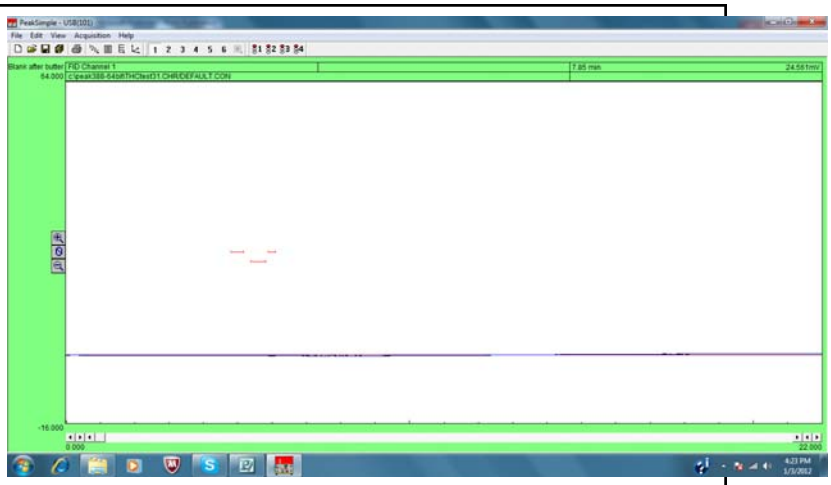
The vial with butter and oil was injected and the results showed . for CBD and . d THC. Some thickening of the CBD is apparent while the THC peak looks much the same as the non-butter vial.



The vial with butter only (no oil) was injected for comparison. No interfering peaks were observed at the CBD or THC times but the butter peaks appear identical.



easuring THC in Butter using the SRI C GC



A blank run was made after the butter chromatograms. No carryover peaks or residue from the butter was observed.

We did notice that the retention times of the CBD and THC were shifted about earlier with the gram butter injections, but returned to the normal time in subsequent injections of non-butter samples.

We made a more concentrated butter extract (grams butter in ml methanol) and saw the retention times move even earlier.
We suspect the butter temporarily covers the stationary phase of the column resulting in less retention.

Conclusion
This experiment shows that a simple methanol extraction completely transfers THC and CBD from butter into the methanol and avoids problems with the butter fats on the GC so long as the column is taken high enough in temperature during each analysis to elute the butter fats completely. The T column which was used is rated to over C which allows this high temperature operation. In addition the thin film promotes fast elution of the high boiling molecules. ven so, the analysis took minutes.

The peculiar shape of the CBD peak and the evidence that the butter increases the CBD number but not the d THC is not explained and requires further investigation.

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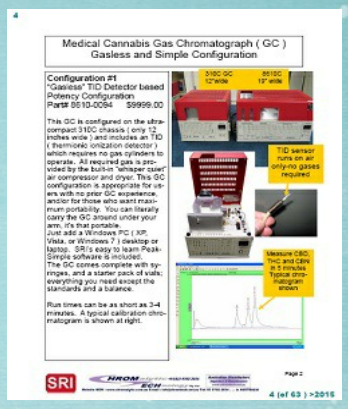
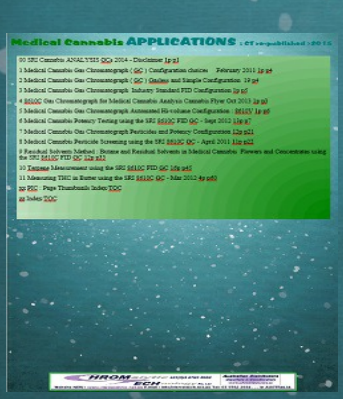
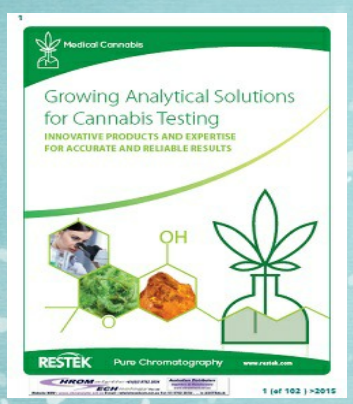
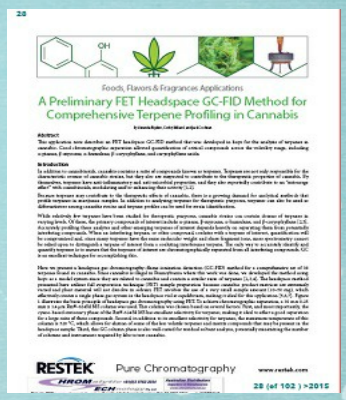


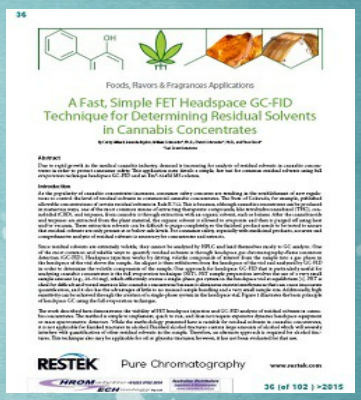


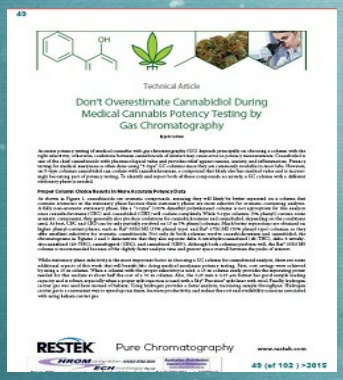
Medical Cannabis APPLICATIONS : CT re-published >2015

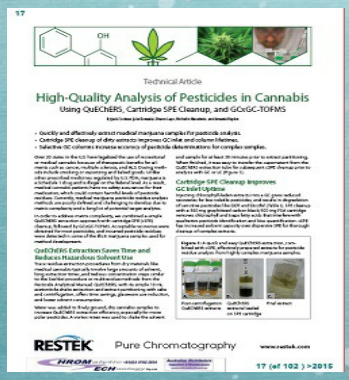
RESTEK

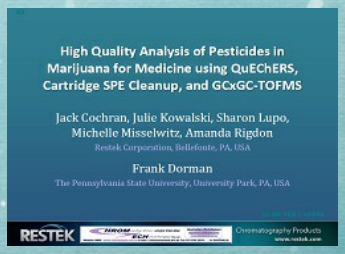


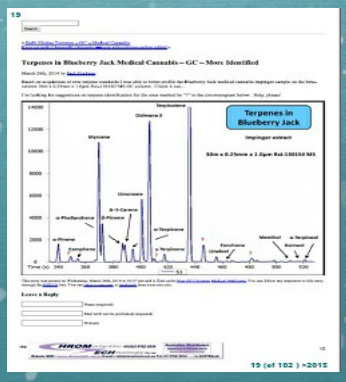












RESTEK

Restek APPLICATIONS : MEDICAL CANNABIS . . . 102p CT-republished >2015

Hints - DISCLAIMER

1 Growing Analytical Solutions for Cannabis Testing_Accurate and Reliable Results_1-16_FFBR2073A-UNV

TECHNICAL ARTICLE

2 A Preliminary FET Headspace GC-FID Method for Comprehensive Terpene Profiling in Cannabis_1-8_FFAN2045-UNV 8p p28

3 Solvents in Cannabis Concentrates_1-8_FFAN2009A-UNV p36

4 High Quality Analysis of Pesticides in Marijuana for Food and Medicine using Quechers, Cartridge SPE, GCxGC-TOFMS and LC-MS-MS_MJrafa2011_med_mj_1-1

5 Don't Overestimate Cannabidiol During Medical Cannabis Potency Testing by GC_FFAR1954-UNV_1-4

6 High-Quality Analysis of Pesticides in Cannabis Using Quechers Cartridge SPE Cleanup and GcxGC-TOFMS_FFAR1950-UNV_1-2 p49 2p

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7 High Quality Analysis of Pesticides in Marijuana for Medicine using Quechers, Cartridge SPE Cleanup and GCxGC-TOFMS_Cannabis_Pittcon2013_1-31

BLOGS . . . *& Ongoing !*

8 Accurate Quantification of Cannabinoid Acids and Neutrals by GC - Derivatices without Calculus - Blog

9 Terpenes in Impinger Extracts of Kryptonite and Blueberry Strains of Medical Cannabis.

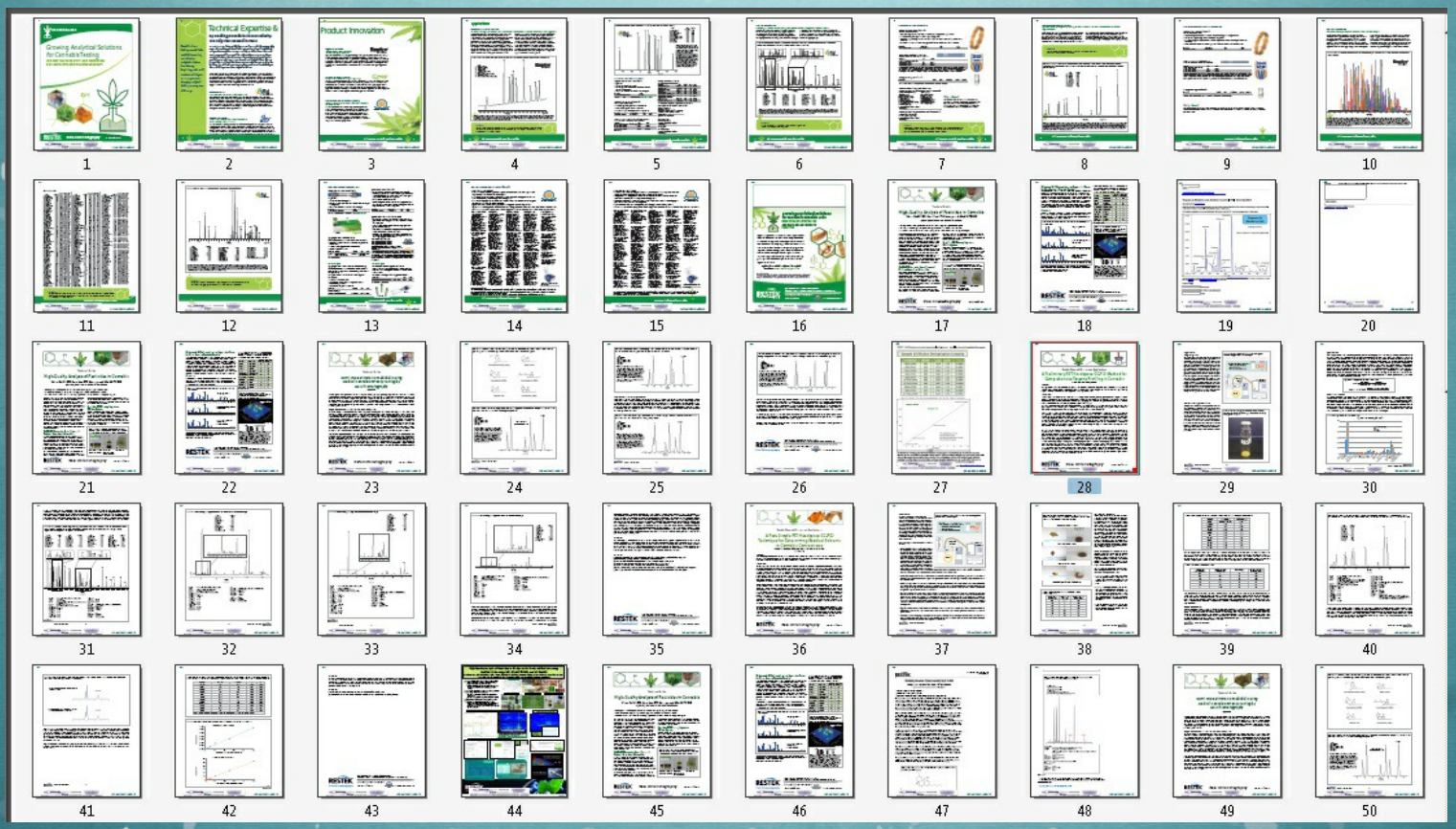
10 Terpenes in Blueberry Jack Medical Cannabis - GC - More Identified

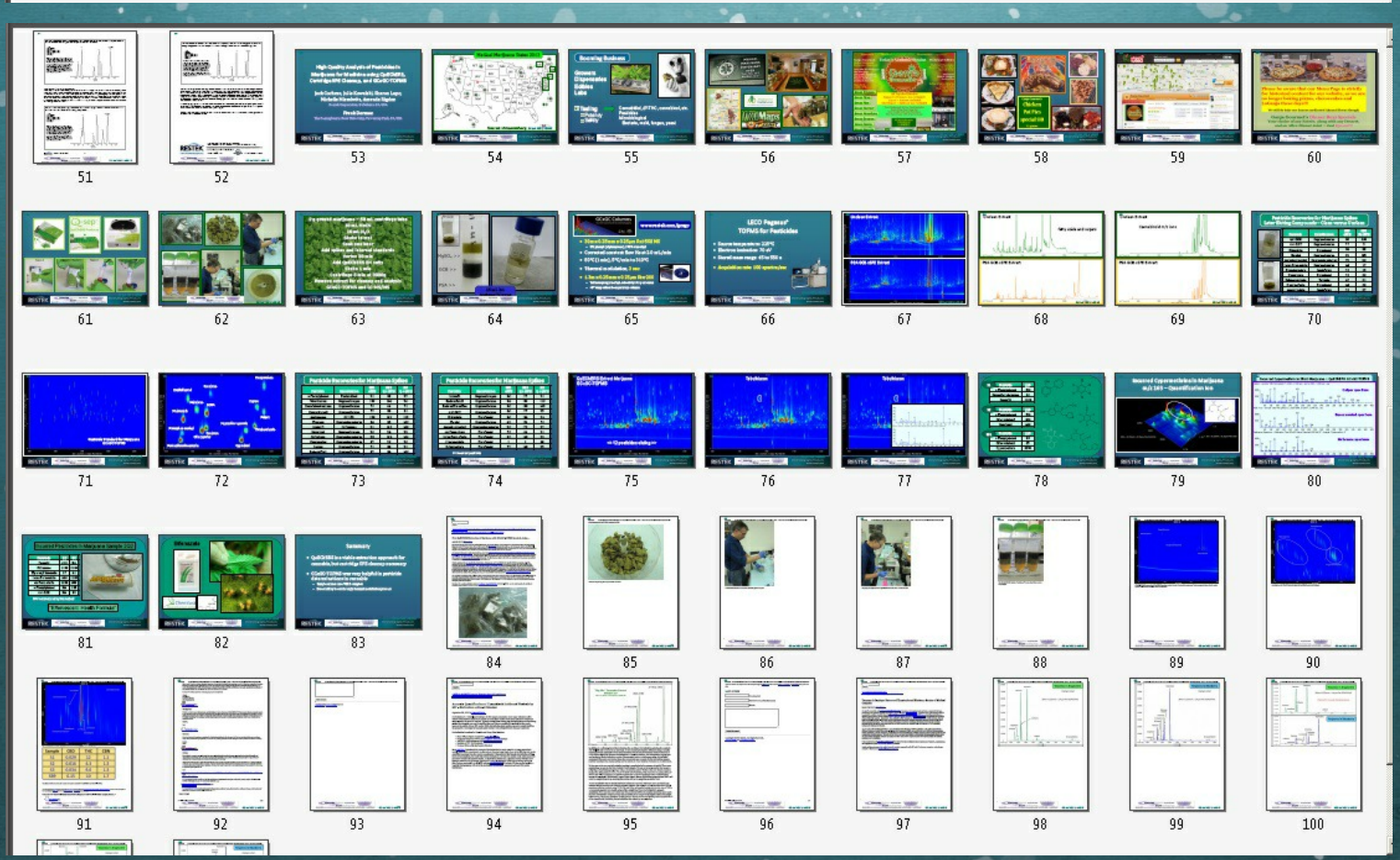
See SRI GCs-Cannabis - for some h'ware related Custom GCs and accessories

Restek prolific & on-going effort (& societies in general) " a work in progress" - and a potential "drug of least harm" and "potent"ial beneficial . . . even when / and for Australia when we wake up to reality R&D sure! but QC is the issue & Restek has (at least some of) THE answers ! Hints Disclaimer see flip.chromalytic.net.au/books/gydm/

Medical Cannabis APPLICATIONS : CT re-published >2015

RESTEK®

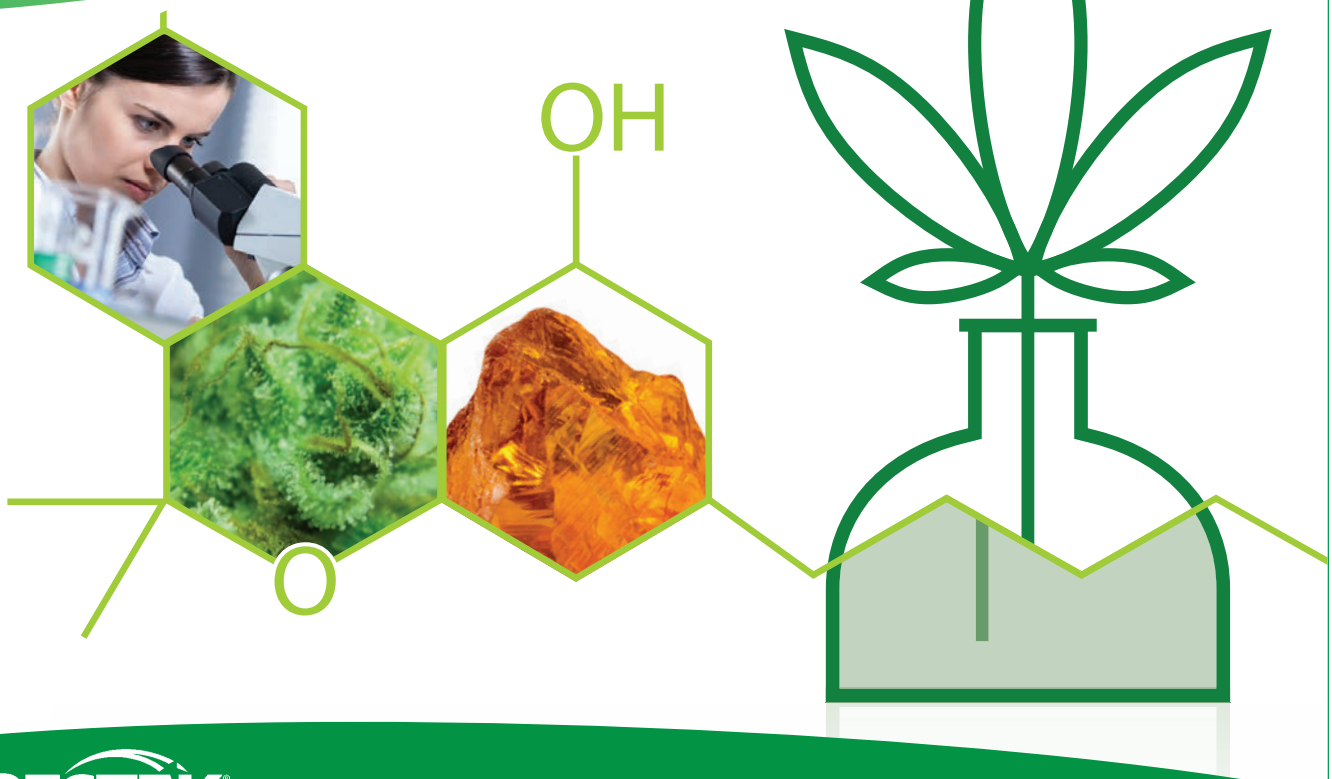




Medical Cannabis

Growing Analytical Solutions for Cannabis Testing

INNOVATIVE PRODUCTS AND EXPERTISE FOR ACCURATE AND RELIABLE RESULTS



RESTEK® Pure Chromatography www.restek.com

Technical Expertise &

By Breaking Boundaries in Our Industry, We Help You Succeed in Yours

Restek has been helping cannabis labs establish innovative, cost-effective analytical solutions from the very beginning, and we will continue to help you manage your ever-changing analytical challenges every step of the way.

We get it. Your market is quickly changing and you need a chromatography partner that understands that. Whether you are part of a well-established safety and potency lab or starting a new lab, Restek has the products and expertise you need for successful cannabis analyses. Being an employee-owned and independent chromatography company, every employee at Restek has a vested interest in your success. We design the best solutions for your lab, regardless of the instrumentation and techniques used. In this brochure, you will find innovative LC and GC products and methodologies designed to fit your toughest analytical problems.

We've been in your shoes. That's why we understand your challenges and focus on solving them. Using our expertise to develop innovative products that help chromatographers has always been, and continues to be, Restek's top priority. We strive to develop industry-leading technologies that fit the needs of today's analysts. When setting up a laboratory for cannabis testing, we realize that you need dependable products that deliver high quality data without considerable capital investment. We know you need to work with a company that understands the challenges of your market and supports you with tailored solutions and superior customer service.

Rxi® GC COLUMNS

Lower Costs With Rugged, Long-Lasting Rxi® Columns

The chemists at Restek have combined their analytical expertise and wide range of polymer chemistries to provide a solution for straightforward analysis of terpenes and residual solvents on a single Rxi® column platform, streamlining workflows for busy labs. Rxi® columns deliver more accurate, reliable results than any other fused silica column on the market. To ensure the highest level of performance, all Rxi® capillary columns for the cannabis industry are manufactured and individually tested to meet stringent requirements for exceptional inertness, low bleed, and unsurpassed column-to-column reproducibility.

Sky® GC INLET LINERS

True Blue Performance—State-of-the-Art Deactivation With a 100% Satisfaction Guarantee

Whether you're determining cannabinoids, residual solvents, pesticides, or terpenes by GC, the inertness of your inlet is crucial for the success of your analyses. Sky® inlet liners from Restek use a comprehensive, state-of-the-art deactivation and are the only blue liners on the market—making them an easy-to-recognize solution to common inlet problems. The innovative deactivation used for Sky® liners results in exceptional inertness for a wide range of analyte chemistries. In addition to improved data quality, you'll benefit from fewer liner changes and less downtime for maintenance.

Product Innovation

Raptor™ LC COLUMNS

Maximize Analytical Performance and Minimize Your Capital Investment

Raptor™ LC columns combine the speed of a superficially porous particle (SPP or "core-shell") with the separation power of optimized USLC phase chemistry. These columns are ideal for cannabis testing because they quickly separate your target compounds, providing higher sample throughput. Raptor™ LC columns maximize your instrument performance so you won't need to buy expensive UHPLC equipment or extend your capital investment when the sample volume increases. Build a solid analytical foundation on any instrument with fast, rugged Raptor™ LC columns.

Q-sep® SAMPLE PREP SUPPLIES

Everything You Need for Fast, Simple Sample Prep

Cannabis products present a broad array of challenging matrices, from foods, to plant materials, to concentrates. For pesticides analysis, a fast, easy cleanup method is required to remove the matrix background for accurate, reliable results. Restek's versatile line of Q-sep™ QuEChERS extraction and cleanup salts allows for the development of quick, easy, and affordable sample preparation methods without capital investment in extraction equipment. The friendly experts at Restek are always willing to help with method development questions, too.

CERTIFIED REFERENCE MATERIALS (CRMs)

Get Results You can Trust With World-Class CRMs Produced in ISO-Accredited Labs

In order to achieve accurate results, samples must be quantified using certified reference materials. Restek has the widest offering of cannabinoid standards in the industry, and we are continually expanding our product line in order to meet the evolving needs of the cannabis industry. Restek's certified reference materials are manufactured and QC tested under our ISO Guide 34 and ISO/IEC 17025 accreditations, helping ensure confidence in results and compliance with changing regulations.

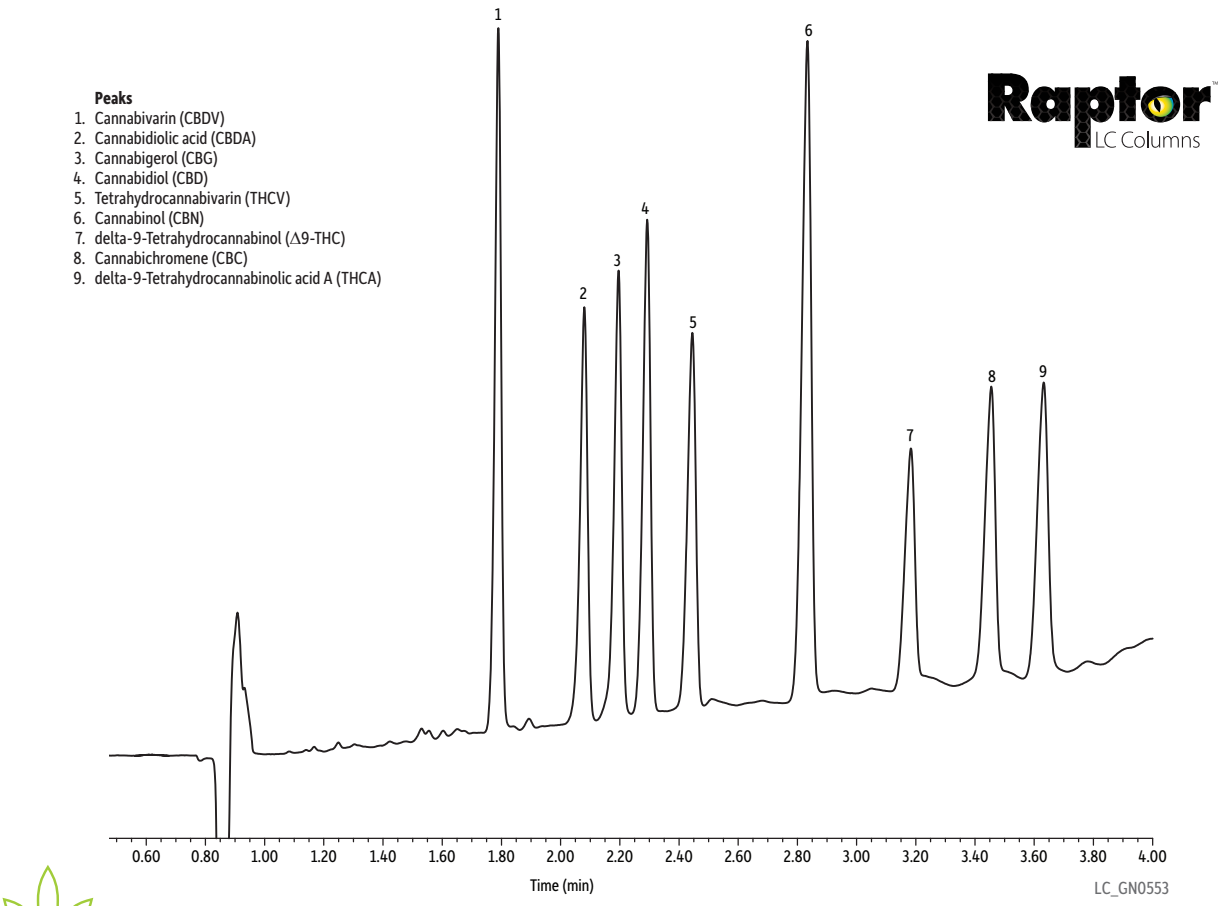
Applications

PRODUCT POTENCY TESTING

Our High-Throughput LC and GC Cannabinoids Methods Produce Results Quickly Without the Cost of New Equipment

When setting up a lab, often you just can't invest in the latest instrumentation, but you still need to get results fast. We understand that. That's why Restek has developed both LC and GC methods for cannabinoids that let you report potency results quickly. For LC, we created a fast analysis that can be performed on any LC instrument. By utilizing Raptor™ column technology, as shown in Figure 1, we developed a 3.7 minute analysis (7 minutes total cycle time) that is compatible with any HPLC instrument—so you get UHPLC speed on your existing equipment without the capital investment. Also, we specifically chose an easy-to-make mobile phase that can be directly transferred to LC-MS, if you ever need to move to MS due to regulation changes. For labs using GC equipment, you can analyze cannabinoids in just minutes using an Rxi®-35Sil MS column and the instrument conditions shown in Figure 2. We also offer a similar 35-type stationary phase on metal MXT® tubing for labs using SRI GC instruments. Why did we focus on fast cannabinoid analyses? Potency testing is the cornerstone of your lab. Building a fast method means your productivity increases and you can analyze more samples per day on the same instrument, delaying the need for expensive capital investments in new equipment.

Figure 1: Raptor™ LC columns give you fast analysis times for cannabinoids without the expense of UHPLC equipment.

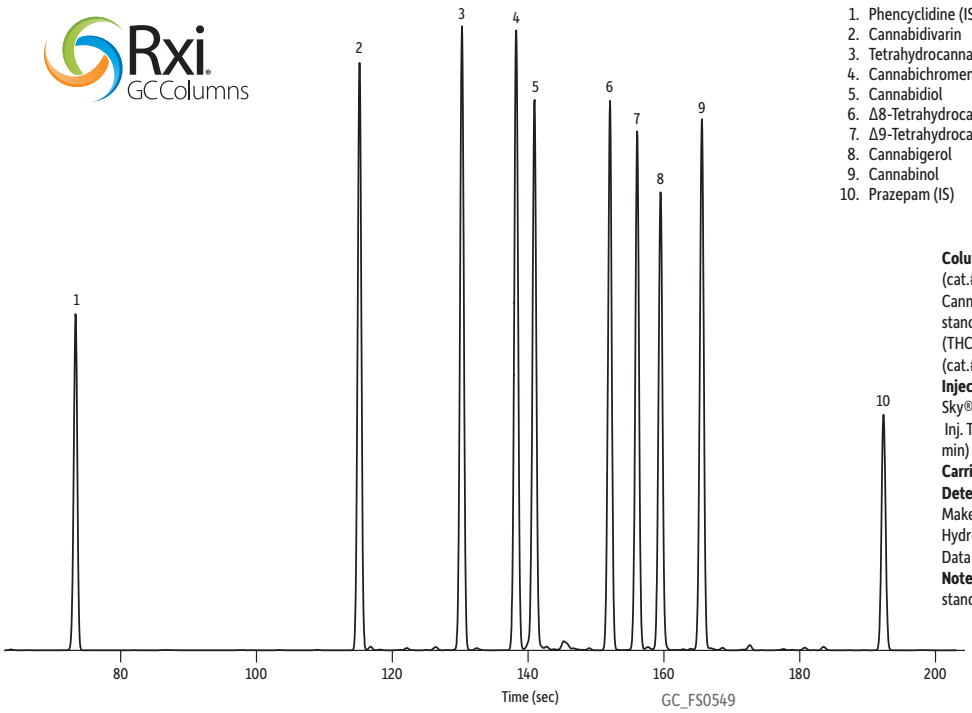


Column: Raptor™ ARC-18 (cat.# 9314A65), Dimensions: 150 mm x 4.6 mm ID, Particle Size: 2.7 µm, Temp.: 50 °C; **Sample:** Cannabidiolic acid (cat.# 34094), Cannabigerol (cat.# 34091), Cannabidiol (cat.# 34011), Cannabinol (cat.# 34010), delta-9-Tetrahydrocannabinol (THC) (cat.# 34067), Cannabichromene (cat.# 34092), delta-9-Tetrahydrocannabinolic acid A (THCA) (cat.# 34093), Diluent: 50:50 Methanol:water, Conc.: 50 µg/mL, Inj. Vol.: 5 µL; **Mobile Phase:** A: 0.1% Formic acid in water, B: 0.1% Formic acid in acetonitrile; **Gradient (%B):** 0.00 min (75%), 4.00 min (100%), 4.01 min (75%), 7.00 min (75%); **Flow:** 1.5 mL/min; **Detector:** UV/Vis @ 220 nm; **Instrument:** HPLC

TECH TIP

Using syringe filters is an economical way to remove particulate matter that could clog your column. Visit **www.restek.com/filters** to access our solvent/syringe filter compatibility guide and quickly find the best filter for your method.

Figure 2: Determine critical cannabinoids in minutes by GC using an Rxi®-35Sil MS column.



	Peaks	Conc. (µg/mL)	tR (sec)
1.	Phencyclidine (IS)	50	73.4
2.	Cannabidivarin	100	115.2
3.	Tetrahydrocannabivarin	100	130.3
4.	Cannabichromene	100	138.3
5.	Cannabidiol	100	150.0
6.	Δ8-Tetrahydrocannabinol	100	152.1
7.	Δ9-Tetrahydrocannabinol	100	156.1
8.	Cannabigerol	100	159.5
9.	Cannabinol	100	165.6
10.	Prazepam (IS)	50	192.4

Column: Rxi®-35Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13820), **Sample:** Phencyclidine (cat.# 34027); Cannabichromene (CBC) (cat.# 34092); Cannabinoids standard (cat.# 34014); delta-8-Tetrahydrocannabinol (THC) (cat.# 34090); Cannabigerol (CBG) (cat.# 34091); Prazepam (cat.# 34055); **Injection:** Inj. Vol.: 1 µL split (split ratio 20:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 190 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.9 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 2.5 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC; **Notes:** Cannabidivarin and tetrahydrocannabivarin standards were obtained from BOC Sciences.

POTENCY TESTING PRODUCTS

Raptor™ ARC-18 LC Columns (USP L1)

Properties:

- Well-balanced retention profile.
- Sterically protected and acid-resistant to resist harsh, low-pH mobile phases.
- Ideal for use with sensitive detectors like mass spec.

Description	cat.#
2.7 µm Columns 150 mm, 4.6 mm ID	9314A65

For guard cartridges, visit our website at **www.restek.com**

Rxi®-35Sil MS Columns (fused silica)

(midpolarity Crossbond® phase)

- Provides superior separation for cannabinoids.
- Very low-bleed phase for GC-MS analysis.
- Extended temperature range: 50 °C to 340/360 °C.

Description	temp. limits	qty.	cat.#
15 m, 0.25 mm ID, 0.25 µm	50 to 340/360 °C	ea.	13820

Sky® 4.0 mm ID Precision® Inlet Liner w/Wool

For Agilent GCs equipped with split/splitless inlets

ID x OD x L	qty.	cat.#
Precision, Sky Technology, Borosilicate Glass with Quartz Wool		
4.0 mm x 6.3 mm x 78.5 mm	ea.	23305.1
4.0 mm x 6.3 mm x 78.5 mm	5-pk.	23305.5
4.0 mm x 6.3 mm x 78.5 mm	25-pk.	23305.25

Patent pending

Medical Marijuana Singles

Concentration is µg/mL. Volume is 1 mL/ampul.

Compound	CAS #	Solvent	Conc.	cat.#
Cannabichromene (CBC)	20675-51-8	PTM	1,000	34092
Cannabidiol (CBD)	13956-29-1	PTM	1,000	34011
Cannabidiolic Acid (CBDA)	1244-58-2	ACN	1,000	34094
Cannabigerol	25654-31-3	PTM	1,000	34091
Cannabinol (CBN)	521-35-7	PTM	1,000	34010
delta-8-Tetrahydrocannabinol (THC)	5957-75-5	PTM	1,000	34090
delta-9-Tetrahydrocannabinol (THC)	1972-08-3	M	1,000	34067
delta-9-Tetrahydrocannabinolic acid A (THCA-A)	23978-85-0	PTM	1,000	34093
Tetrahydrofuran-d8	1693-74-9	PTM	2,000	30112
(±)11-nor-9-carboxy-Δ^9-THC	104874-50-2	M	100	34068

M = methanol; PTM = purge-and-trap grade methanol; ACN = acetonitrile

Cannabinoids Standard (3 components)

Cannabidiol (13956-29-1)
Cannabinol (521-35-7)
delta-9-Tetrahydrocannabinol (Δ^9-THC) (1972-08-3)

1,000 µg/mL each in P&T methanol, 1 mL/ampul
cat.# 34014 (ea.)

Quantity discounts not available.

Phencyclidine

Phencyclidine (956-90-1)

1,000 µg/mL in P&T methanol, 1 mL/ampul
cat.# 34027 (ea.)

Prazepam

Prazepam (2955-38-6)

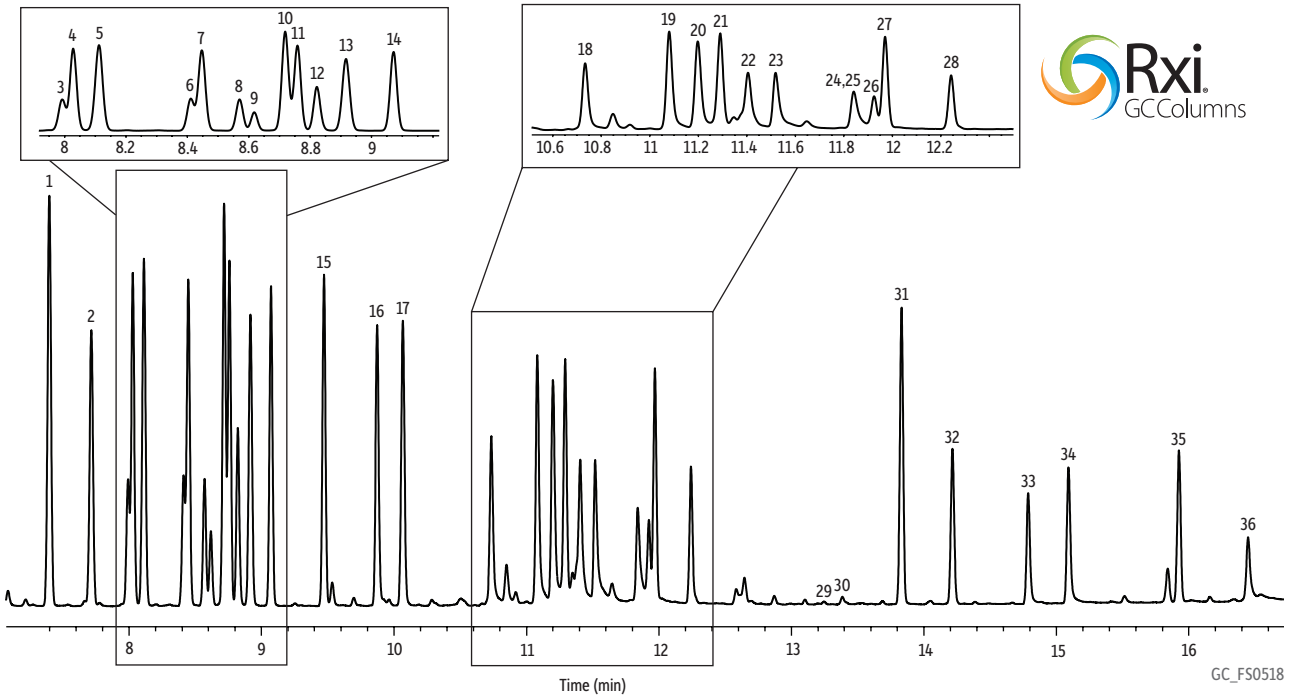
1,000 µg/mL in P&T methanol, 1 mL/ampul
cat.# 34055 (ea.)

TERPENE PROFILING

Reduce Capital Investments—Analyze Terpenes by GC on the Same Setup Used for Residual Solvents

Cannabis has a complex terpene profile, which is theorized to increase its therapeutic effects. Terpene profiling is used for both product quality testing and strain identification. These complex and sometimes problematic compounds are challenging to analyze, but the experts at Restek have developed GC methodology for terpene profiling that fits easily into required laboratory workflows. To keep things simple, the GC terpene profile analysis in Figure 3 can be performed on the same instrument and column that we recommend for residual solvent testing (see page 8).

Figure 3: Comprehensive terpene analysis by headspace GC-FID can be done on the same instrument and GC column as residual solvents analysis, which simplifies setup and improves lab productivity.



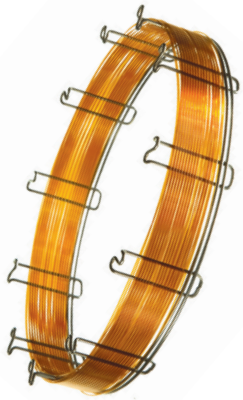
Peaks	tR (min)						
1. α-Pinene	7.39	10. Limonene	8.71	19. dl-Menthol	11.08	28. Citral 2	12.24
2. Camphene	7.71	11. *p*-Cymene	8.75	20. Borneol	11.19	29. Citral 3	13.19
3. β-Myrcene	7.98	12. β-Ocimene	8.82	21. α-Terpineol	11.29	30. Citral 4	13.43
4. Sabinene	8.02	13. Eucalyptol	8.91	22. Dihydrocarveol	11.40	31. β-caryophyllene	13.83
5. β-Pinene	8.11	14. γ-Terpinene	9.06	23. Citronellol	11.51	32. α-Humulene	14.21
6. α-Phellandrene	8.4	15. Terpinolene	9.47	24. Geraniol	11.82	33. Nerolidol 1	14.78
7. δ 3-Carene	8.44	16. Linalool	9.87	25. 2-Piperidinone	11.88	34. Nerolidol 2	15.08
8. α-Terpinene	8.57	17. Fenchone	10.06	26. Citral 1	11.92	35. Caryophyllene oxide	15.92
9. Ocimene	8.61	18. Isopulegol	10.73	27. Pulegone	11.97	36. α-Bisabolol	16.43

Column: Rxi® -624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868); **Sample:**Terpenes mix; Diluent: Isopropyl alcohol; Conc.: 200 ng/µL (0.02% wt/vol). The sample was prepared by placing 10 µL into the headspace vial.; **Injection:** headspace-loop split (split ratio 10:1); Liner: Sky® 1.0 mm ID straight inlet liner (cat.# 23333.1); **Headspace-Loop:** Inj. Port Temp.: 250 °C; Instrument: Tekmar HT-3; Inj. Time: 1.0 min; Transfer Line Temp.: 160 °C; Valve Oven Temp.: 160 °C; Needle Temp.: 140 °C; Sample Temp.: 140 °C; Sample Equil. Time: 30.0 min; Vial Pressure: 20 psi; Loop Pressure: 15 psi; **Oven:** Oven Temp.: 60 °C (hold 0.10 min) to 300 °C at 12.50 °C/min (hold 3.0 min); **Carrier Gas:** He, constant flow; Linear Velocity: 33 cm/sec; **Detector:** FID @ 320 °C; Make-up Gas Flow Rate: 45 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument** Agilent/HP6890 GC

TECH TIP

For full method details on headspace GC analysis of terpenes, visit **www.restek.com/cannabis_terpenes**

TERPENE TESTING PRODUCTS



Rxi®-624Sil MS Columns (fused silica)

(midpolarity Crossbond® phase)

- Low-bleed, high-thermal stability column—maximum temperatures up to 320 °C.
- Inert—excellent peak shape for a wide range of compounds.
- Selective—G43 phase highly selective for volatile organics and residual solvents, great choice for USP<467>.
- Manufactured for column-to-column reproducibility—well-suited for validated methods.

Description	temp. limits	qty.	cat.#
30 m, 0.25 mm ID, 1.40 µm	-20 to 300/320 °C	ea.	13868

Sky® 1.0 mm ID Straight Inlet Liner

for Agilent GCs equipped with split/splitless inlets

ID x OD x L	qty.	cat.#
Straight, Sky Technology, Borosilicate Glass		
1.0 mm x 6.3 mm x 78.5 mm	ea.	23333.1
1.0 mm x 6.3 mm x 78.5 mm	5-pk.	23333.5
1.0 mm x 6.3 mm x 78.5 mm	25-pk.	23333.25

* 100% SATISFACTION GUARANTEE: If your Sky® inlet liner does not perform to your expectations for any reason, simply contact Restek® Technical Service or your local Restek® representative and provide a sample chromatogram showing the problem. If our GC experts are not able to quickly and completely resolve the issue to your satisfaction, you will be given an account credit or replacement product (same cat.#) along with instructions for returning any unopened product. (Do not return product prior to receiving authorization.) For additional details about Restek's return policy, visit www.restek.com/warranty

Headspace Crimp Vials (20 mm)

Description	Volume	Color	Dimensions	100-pk.	1,000-pk.
Headspace Vial, Flat Bottom	20 mL	Clear	23 x 75 mm	24685	24686

Vial-to-instrument compatibility are designated in instrument reference chart on the product web page.

Medical Cannabis Terpenes Standards

Medical Cannabis Terpenes Standard #1 (19 components)

(-)-alpha-Bisabolol (23089-26-1)
Camphene (79-92-5)
delta-3-Carene (13466-78-9)
beta-Caryophyllene (87-44-5)
Geraniol (106-24-1)
(-)-Guaiol (489-86-1)
alpha-Humulene (6753-98-6)
p-Isopropyltoluene (*p*-cymene) (99-87-6)
(-)-Isopulegol (89-79-2)
d-Limonene (5989-27-5)
Linalool (78-70-6)
beta-Myrcene (123-35-3)
Nerolidol (7212-44-4)
Ocimene (13877-91-3)
alpha-Pinene (80-56-8)
(-)-beta-Pinene (18172-67-3)
alpha-Terpinene (99-86-5)
gamma-Terpinene (99-85-4)
Terpinolene (586-62-9)

2,500 µg/mL each in isopropanol, 1 mL/ampul
cat.# 34095 (ea.)

Medical Cannabis Terpenes Standard #2 (2 components)

(-)-Caryophyllene oxide (1139-30-6)
1,8-Cineole (Eucalyptol) (470-82-6)

2,500 µg/mL each in isopropanol, 1 mL/ampul
cat.# 34096 (ea.)

Did you **know**?

You'll save money ordering from Restek because we understand the need to control costs and build efficient workflows. We develop as many analyses as possible using the same columns and consumables, so you can minimize the number of products you need to stock.

TECH TIP

Did you know that headspace analysis eliminates the possibility of column contamination from nonvolatile matrix components? This results in an extremely clean chromatogram, minimal instrument maintenance, and longer column lifetimes.

RESIDUAL SOLVENT ANALYSIS

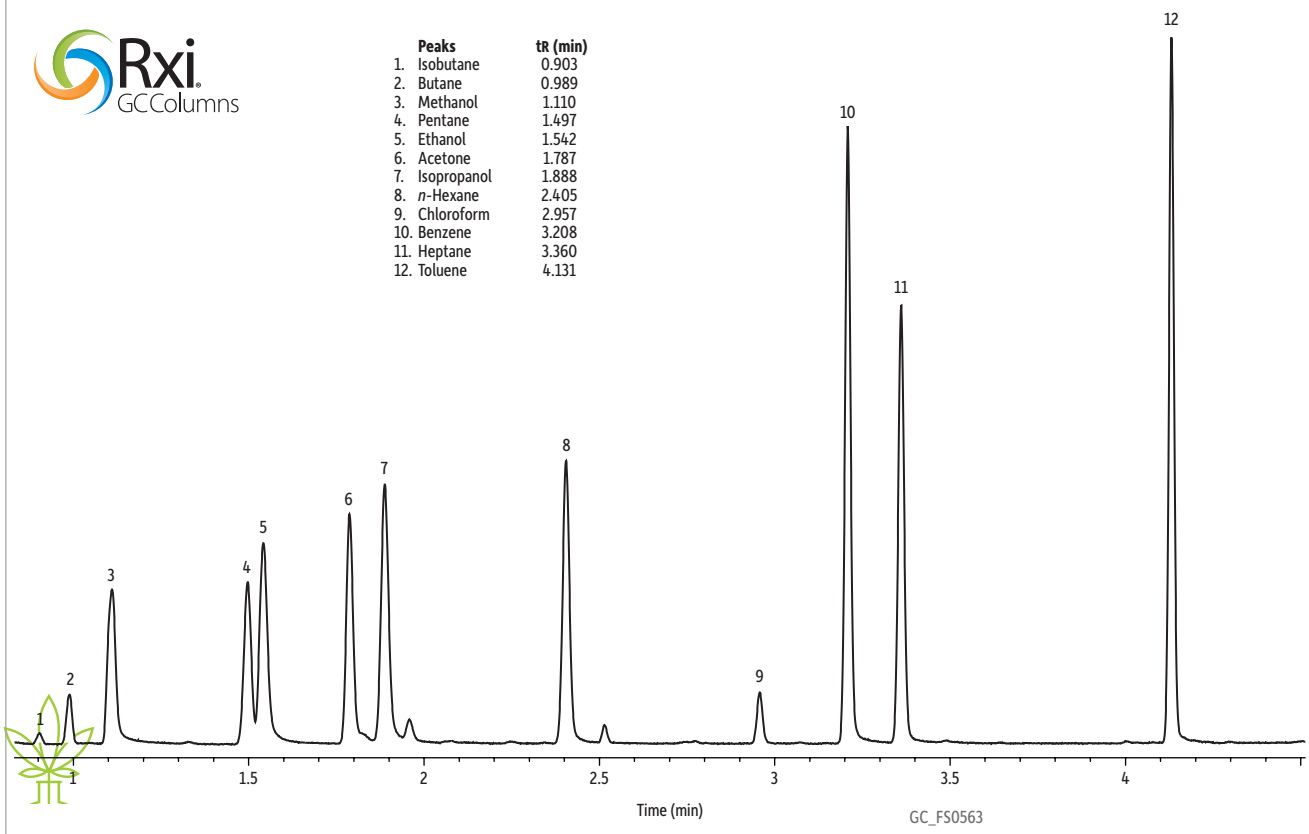
Improve Productivity—Keep Analyzing Samples Instead of Changing Columns Between Residual Solvent and Terpene Methods.

As the popularity of medical cannabis grows, so does concern over the safety of the drug products. Cannabis concentrates can contain residual solvents left over from manufacturing that can be harmful to human health. Because of this risk, many states will require residual solvent testing of cannabis concentrates. Due to their high volatility, residual solvents can only be analyzed using GC techniques. The chemists at Restek have developed a quick and easy method that allows for residual solvent analysis (Figure 4) and terpene profiling (Figure 3) on the same column and instrument platform with minimal sample preparation (see page 6 for terpene profiling).

TECH TIP

For full method details on headspace GC analysis of residual solvents, visit **www.restek.com/cannabis_solvents**

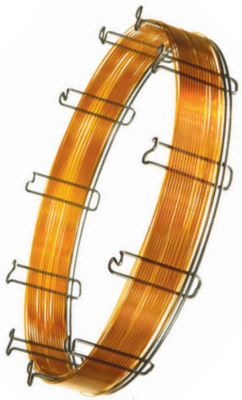
Figure 4: Improve productivity and reduce downtime for column changes—this sensitive headspace GC-FID analysis of residual solvents can be accomplished on the same instrument and Rxi®-624Sil MS column that is used in Restek's terpenes profiling method.



Column: Rxi®-624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868); **Sample**: Residual solvent mix; Diluent: Dimethyl sulfoxide (DMSO); Conc.: 25 ppm (For the HS-FET technique, 10 µL of a 50 µg/mL standard was placed into a 20 mL headspace vial to represent a 25 ppm sample concentration, assuming a 20 mg sample weight.); **Injection:** headspace-loop split (split ratio 10:1); Liner: Sky® 1.0 mm ID straight inlet liner (cat.# 23333.1); **Headspace-Loop:** Inj. Port Temp.: 250 °C; Instrument: Tekmar HT3; Inj. Time: 1.0 min; Transfer Line: Temp.: 160 °C; Valve Oven Temp.: 160 °C; Needle Temp.:140 °C; Sample Temp.: 140 °C; Platen temp equil. time: 1.0 min; Sample Equil. Time: 30.0 min; Vial Pressure: 20 psi; Pressurize Time: 5.0 min; Loop Pressure: 15 psi; Loop Fill Time: 2.0 min; Oven Temp.:35 °C (hold 1.5 min) to 300 °C at 30 °C/min (hold 2.0 min); **Carrier Gas**: He, constant flow; Linear Velocity: 80 cm/sec; **Detector:** FID @ 320 °C; Make-up Gas Flow Rate: 45 mL/min; Make-up Gas Type: N2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument**: Agilent/HP6890 GC; **Notes**: The butane used for standard preparation was a mixture of butane and isobutane in an unknown ratio. The concentrations should be considered approximate, but do not exceed 50 ppm for any component.

RESIDUAL SOLVENT TESTING PRODUCTS

Rxi®-624Sil MS Columns (fused silica)
(midpolarity Crossbond® phase)



- Low-bleed, high-thermal stability column—maximum temperatures up to 320 °C.
- Inert—excellent peak shape for a wide range of compounds.
- Selective—G43 phase highly selective for volatile organics and residual solvents, great choice for USP<467>.
- Manufactured for column-to-column reproducibility—well-suited for validated methods.

Description	temp. limits	qty.	cat.#
30 m, 0.25 mm ID, 1.40 µm	-20 to 300/320 °C	ea.	13868

Sky® 1.0 mm ID Straight Inlet Liner
for Agilent GCs equipped with split/splitless inlets

ID x OD x L	qty.	cat.#
Straight, Sky Technology, Borosilicate Glass		
1.0 mm x 6.3 mm x 78.5 mm	ea.	23333.1
1.0 mm x 6.3 mm x 78.5 mm	5-pk.	23333.5
1.0 mm x 6.3 mm x 78.5 mm	25-pk.	23333.25

* 100% SATISFACTION GUARANTEE: If your Sky® inlet liner does not perform to your expectations for any reason, simply contact Restek® Technical Service or your local Restek® representative and provide a sample chromatogram showing the problem. If our GC experts are not able to quickly and completely resolve the issue to your satisfaction, you will be given an account credit or replacement product (same cat.#) along with instructions for returning any unopened product. (Do not return product prior to receiving authorization.) For additional details about Restek's return policy, visit www.restek.com/warranty

Headspace Crimp Vials (20 mm)

Description	Volume	Color	Dimensions	100-pk.	1,000-pk.
Headspace Vial, Flat Bottom	20 mL	Clear	23 x 75 mm	24685	24686

Vial-to-instrument compatibility are designated in instrument reference chart on the product web page.

Did you **know**?

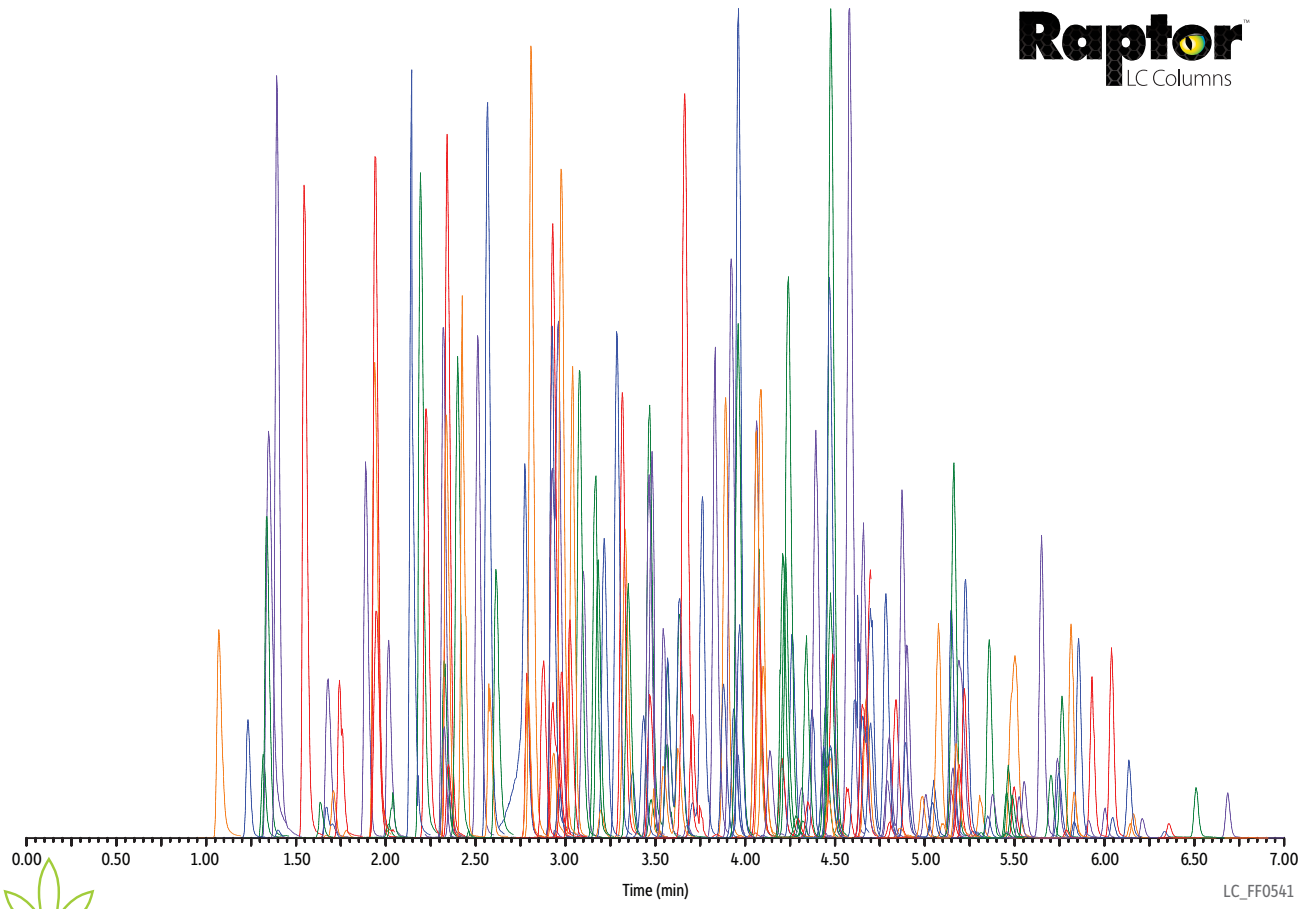
You'll save money ordering from Restek because we understand the need to control costs and build efficient workflows. We develop as many analyses as possible using the same columns and consumables, so you can minimize the number of products you need to stock.

PESTICIDE ANALYSIS

Ensure Product Safety With Fast, Selective Multiresidue Pesticide Analysis

In addition to residual solvents, cannabis products can contain residues of pesticides that were applied to cannabis plants during growth in order to control agricultural pests. These pesticides can be analyzed by LC-MS/MS, GC-MS/MS, and GC-MS. Regardless of the technique used, lists of target compounds can be extensive, so column selectivity is an important factor in achieving good separations. Both Raptor™ ARC-18 LC columns (Figure 5) and Rxi®-5ms GC columns (Figure 6) provide the selectivity needed for accurate and reliable multiresidue pesticides analysis. Removing matrix interferences while also recovering the analytes of interest is also crucial for a successful pesticide analysis using either LC or GC, and Restek's Q-sep® QuEChERS products allow for fast, easy, adaptable cleanup of a wide variety of matrices.

Figure 5: A high-throughput separation of 204 pesticides by LC-MS/MS can be achieved in only 7 minutes with the Raptor™ ARC-18 column.



Column: Raptor™ ARC-18 (cat.# 9314A12), Dimensions: 100 mm x 2.1 mm ID, Particle Size: 2.7 µm, Temp.: 50 °C; **Sample:** LC multiresidue pesticide kit (cat.# 31971), Diluent: Water, Conc.: 20 ng/mL, Inj. Vol.: 5 µL; **Mobile Phase:** A: Water + 2 mM ammonium formate + 0.2% formic acid, B: Methanol + 2 mM ammonium formate + 0.2% formic acid; **Gradient (%B):** 0.00 min (5%), 2.00 min (60%), 4.00 min (75%), 6.00 min (100%), 7.00 min (100%), 7.01 min (5%), 9.50 min (5%); **Flow:** 0.4 mL/min; Max Pressure: 525 bar; **Detector:** Waters Xevo TQ-S, Ion Source: Waters Zspray™ ESI, Ion Mode: ESI+, Mode: MRM, **Instrument:** Waters ACQUITY UPLC® I-Class; **Notes:** When combining a large number of compounds with different chemical functionalities, mix stability can be an issue. In formulating our LC multi-residue pesticide standard kit (cat.# 31971), we extensively studied the 204 compounds involved, then grouped them into as few mixes as possible while still ensuring maximum long-term stability and reliability. Several of these compounds are isomeric and separation of the isomers accounts for 216 peaks in the chromatogram compound list. For quantitative analysis, we recommend analyzing each mix separately to ensure accurate results for every compound.

visit **www.restek.com/cannabis**

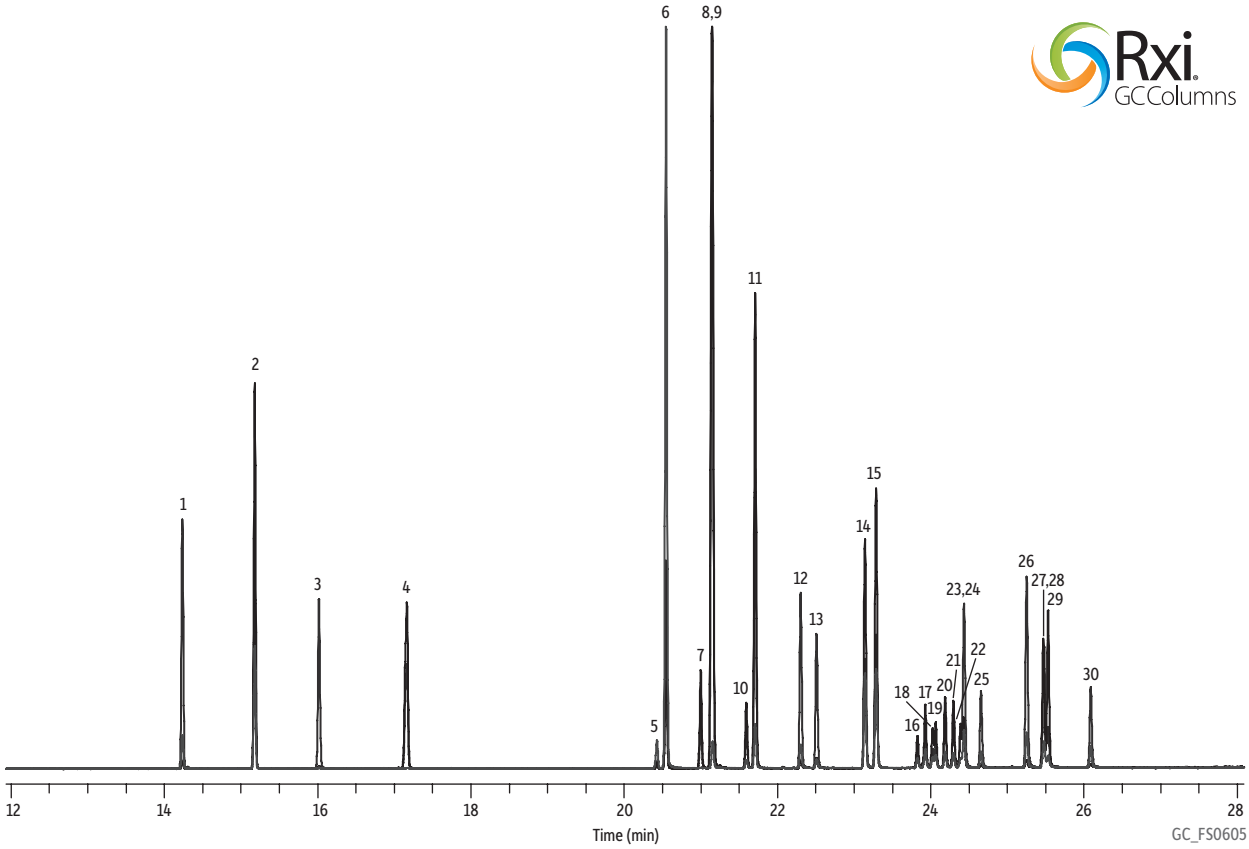
Figure 6: Peak List

	Peaks	t_R (min)	Precursor Ion	Product Ion 1	Product Ion 2
1.	Cyromazine	1.07	167.0	85.0	108.1
2.	Methamidophos	1.23	142.0	93.9	124.9
3.	Formetanate HCl	1.32	222.0	165.0	46.0
4.	Aminocarb	1.34	209.0	137.0	152.0
5.	Pymetrozine	1.35	218.0	105.0	79.0
6.	Acephate	1.40	184.1	143.0	125.1
7.	Propamocarb	1.40	189.1	102.0	144.0
8.	Omethoate	1.55	214.1	125.1	183.1
9.	Aldicarb sulfoxide	1.64	207.0	89.0	132.0
10.	Dinotefuran	1.64	203.0	129.0	157.0
11.	Butoxycarboxim	1.67	223.0	106.0	166.0
12.	Nitenpyram	1.68	271.1	125.9	224.9
13.	Aldicarb sulfone	1.71	240.0	148.0	86.0
14.	Carbendazim	1.74	192.1	160.1	132.1
15.	Oxamyl	1.78	237.0	72.0	90.0
16.	Flonicamid	1.89	230.0	203.1	174.1
17.	Methomyl	1.91	163.0	106.0	88.0
18.	Thiabendazole	1.94	202.0	175.0	131.0
19.	Thiamethoxam	1.94	292.0	211.0	181.0
20.	Mexacarbate	1.95	222.9	151.1	166.1
21.	Monocrotophos	2.02	224.1	127.1	98.1
22.	Fuberidazole	2.04	185.0	157.0	156.0
23.	Dicrotophos	2.14	238.0	112.0	193.0
24.	Imidacloprid	2.19	256.1	175.1	209.1
25.	Clothianidin	2.22	250.0	169.0	132.0
26.	Trichlorfon	2.32	257.0	109.0	79.0
27.	3-Hydroxycarbofuran	2.33	238.0	181.0	163.0
28.	Fenuron	2.33	165.0	71.9	45.9
29.	Dimethoate	2.34	230.1	125.0	199.0
30.	Vamidothion	2.34	288.0	146.0	118.0
31.	Dioxacarb	2.35	224.1	123.1	167.1
32.	Mevinphos isomer 1	2.36	225.1	127.1	193.1
33.	Acetamiprid	2.40	223.0	126.0	56.1
34.	Ethirimol	2.43	210.1	140.0	98.0
35.	Cymoxanil	2.46	199.0	128.0	111.0
36.	Pirimicarb	2.51	239.1	72.0	182.1
37.	Thiacloprid	2.56	253.0	126.0	90.1
38.	Mevinphos isomer 2	2.58	225.1	127.1	193.1
39.	Mesotrione	2.62	340.1	228.1	104.0
40.	Butocarboxim	2.68	213.0	156.0	116.0
41.	Aldicarb	2.71	213.1	89.1	116.1
42.	Oxadixyl	2.77	279.0	219.0	132.0
43.	Carbetamide	2.79	237.0	118.0	192.0
44.	Tricyclazole	2.79	190.0	163.0	136.0
45.	Simetryn	2.81	214.0	124.0	95.9
46.	Thiophanate-methyl	2.88	343.0	151.0	93.0
47.	Bendiocarb	2.93	224.1	109.0	167.0
48.	Prometon	2.93	226.0	184.3	86.3
49.	Secbumeton	2.93	226.2	100.2	170.2
50.	Thidiazuron	2.93	221.0	101.9	93.9
51.	Propoxur	2.95	210.0	111.0	168.0
52.	Metribuzin	2.96	215.0	131.0	89.0
53.	Terbumeton	2.96	226.1	114.1	170.1
54.	Carbofuran	2.98	222.1	123.0	165.1
55.	Imazalil	2.98	297.0	159.0	69.0
56.	Sulfentrazone	3.03	387.0	307.0	145.8
57.	Pyracarbolid	3.04	218.1	125.1	97.1
58.	Tebuthiuron	3.08	229.0	172.0	116.0
59.	Carbaryl	3.09	202.0	145.0	127.0
60.	Carboxin	3.10	236.0	143.0	87.0
61.	Monolinuron	3.17	215.0	126.0	99.0
62.	Fluometuron	3.18	233.2	72.2	46.4
63.	Ethiofencarb	3.20	226.1	107.0	164.0
64.	Ametryn	3.21	228.1	186.1	68.1
65.	Chlortoluron	3.29	213.0	72.0	46.0
66.	Metobromuron	3.32	259.1	170.0	148.1
67.	Methoprotryne	3.33	272.2	170.2	198.2
68.	Propham	3.33	180.0	138.0	120.1
69.	Flutriafol	3.35	302.1	123.1	70.2
70.	Isoprocarb	3.37	194.1	95.1	137.1
71.	Fenpropimorph	3.44	304.2	147.1	57.2
72.	Methabenzthiazuron	3.46	222.0	165.0	150.0
73.	Diuron	3.47	233.0	72.1	46.3
74.	Forchlorfenuron	3.47	248.1	129.0	93.0
75.	Isocarbophos	3.48	291.1	121.1	231.1
76.	Isoproturon	3.48	207.0	72.0	47.0
77.	Pyrimethanil	3.48	200.0	107.0	82.0
78.	Desmedipham	3.55	318.0	182.0	154.0
79.	Metalaxyl	3.56	280.1	220.1	192.1
80.	Spiroxamine isomer 1	3.57	298.0	144.0	100.0
81.	Phenmedipham	3.63	301.0	168.0	136.0
82.	Spiroxamine isomer 2	3.63	298.0	144.0	100.0
83.	Chlorantraniliprole	3.66	483.9	286.0	453.0
84.	Cycluron	3.68	199.0	89.1	69.2
85.	Prometryn	3.71	242.0	158.0	200.1
86.	Terbutryn	3.76	242.1	186.1	91.0
87.	Linuron	3.83	249.1	160.0	182.0
88.	Fenobucarb	3.84	208.0	94.9	152.0
89.	Diethofencarb	3.88	268.0	226.0	124.0
90.	Ethofumesate	3.89	287.1	121.1	259.1
91.	Azoxystrobin	3.92	404.1	372.0	329.0
92.	Ethriprole	3.94	396.9	350.9	255.2
93.	Fenamidone	3.96	312.1	236.1	92.0
94.	Methiocarb	3.96	226.0	121.0	169.0
95.	Siduron	3.96	233.0	93.8	137.0
96.	Fludioxonil	3.97	249.1	229.1	158.1
97.	Furalaxyl	3.97	302.1	270.1	242.2
98.	Halofenozide	3.99	331.1	104.9	275.0
99.	Acibenzolar-S-methyl	4.06	210.9	91.0	135.9
100.	Boscalid	4.06	342.9	307.0	139.9
101.	Dimethomorph isomer 1	4.06	388.1	300.9	165.0
102.	Nuarimol	4.08	315.0	252.0	81.1
103.	Mandipropamid	4.09	412.3	328.2	356.2
104.	Flutolanil	4.10	324.1	262.1	65.0
105.	Promecarb	4.10	208.1	151.0	109.0
106.	Paclobutrazol	4.14	294.1	125.1	70.2
107.	Thiofanox	4.19	219.1	172.9	129.0
108.	Cyproconazole isomer 1	4.21	292.2	125.1	70.2
109.	Mepronil	4.21	270.1	119.0	91.0
110.	Bupirimate	4.22	317.0	166.0	108.0
111.	Dimethomorph isomer 2	4.24	388.1	300.9	165.0
112.	Myclobutanil	4.26	289.1	70.2	125.1
113.	Clethodim isomer 1	4.28	360.0	164.0	268.1
114.	Methoxyfenozide	4.30	369.1	149.1	313.2
115.	Chloroxuron	4.31	291.1	164.1	111.0
116.	Cyprodinil	4.32	226.0	93.0	108.0
117.	Triadimefon	4.34	294.1	197.2	69.3
118.	Bifenazate	4.35	301.1	198.0	170.0
119.	Triadimenol	4.35	296.1	99.1	70.2
120.	Cyproconazole isomer 2	4.38	292.2	125.1	70.2
121.	Mefenacet	4.39	299.0	148.0	120.0
122.	Mepanipyrim	4.40	224.1	106.0	77.0
123.	Iprovalicarb isomer 1	4.44	321.1	119.1	203.1
124.	Fluquinconazole	4.45	376.0	348.8	306.9
125.	Fenhexamid	4.46	302.1	97.2	55.3
126.	Bromuconazole isomer 1	4.47	376.0	158.9	70.1
127.	Fluoxastrobin	4.47	459.0	427.0	188.0
128.	Iprovalicarb isomer 2	4.47	321.1	119.1	203.1
129.	Butafenacil	4.48	492.0	180.0	331.0
130.	Tetraconazole	4.48	372.0	159.0	70.1
131.	Flufenacet	4.49	364.0	152.1	194.1
132.	Triticonazole	4.52	318.1	70.1	124.9
133.	Cyazofamid	4.57	325.0	107.9	261.0
134.	Spirotetramat	4.58	374.2	330.3	302.2
135.	Diflubenzuron	4.63	311.1	141.0	158.1
136.	Epoxiconazole	4.66	330.0	121.0	101.0
137.	Etaconazole isomer 1	4.66	328.1	205.0	159.0
138.	Fenbuconazole	4.67	337.0	125.0	70.1
139.	Fenarimol	4.68	331.0	268.0	81.0
140.	Etaconazole isomer 2	4.70	328.1	205.0	159.0
141.	Fipronil	4.70	437.0	367.9	290.0
142.	Flusilazole	4.78	316.0	247.0	165.0
143.	Picoxystrobin	4.79	368.0	145.1	205.1
144.	Fenoxycarb	4.80	302.1	116.1	88.0
145.	Neburon	4.80	275.0	88.0	57.0
146.	Rotenone	4.84	395.0	213.1	192.1
147.	Tebufenozide	4.87	353.1	133.0	297.1
148.	Dimoxystrobin	4.88	327.1	116.1	205.2
149.	Bromuconazole isomer 2	4.89	376.0	158.9	70.1
150.	Flubendiamide	4.89	683.0	408.0	274.0
151.	Carfentrazone ethyl	4.90	412.0	346.0	266.0
152.	Diclobutrazol	4.91	328.0	70.0	59.1
153.	Kresoxim-methyl	4.92	314.1	206.0	116.0
154.	Tebuconazole	4.98	308.0	70.1	125.0
155.	Penconazole	5.00	284.0	70.1	159.0
156.	Spinosyn A	5.04	732.6	142.0	98.1
157.	Prothioconazole	5.05	344.0	326.0	189.0
158.	Alanycarb	5.06	400.0	238.2	254.1
159.	Zoxamide	5.08	336.0	187.1	159.0
160.	Famoxadone	5.10	392.2	331.1	238.0
161.	Prochloraz	5.15	376.0	308.0	70.1
162.	Triflumuron	5.15	359.0	156.1	139.1
163.	Benalaxyl	5.16	326.1	148.0	91.0
164.	Hexaconazole	5.16	314.0	70.1	159.0
165.	Hydramethylnon	5.17	495.1	323.2	151.1
166.	Metconazole	5.19	320.1	70.0	125.0
167.	Propiconazole isomer 1 & 2	5.19	342.0	159.0	69.0
168.	Clofentezine	5.22	303.0	138.0	102.0
169.	Pyraclostrobin	5.23	388.1	163.0	193.9
170.	Bitertanol	5.27	338.1	269.2	70.1
171.	Benzoximate	5.29	364.0	199.1	105.0
172.	Spinosyn D	5.31	746.5	142.0	98.1
173.	Thiobencarb	5.31	257.9	125.1	100.1
174.	Diniconazole	5.35	326.1	70.2	159.0
175.	Pencycuron	5.36	329.1	125.0	218.0
176.	Spinetoram	5.38	748.5	142.2	98.1
177.	Hexaflumuron	5.46	461.0	158.0	141.0
178.	Indoxacarb	5.46	528.0	203.0	218.0
179.	Ipconazole isomer 1	5.46	334.2	70.0	125.1
180.	Triflumizole	5.49	346.0	277.9	60.0
181.	Difenoconazole isomer 1 & 2	5.50	406.0	251.1	111.1
182.	Trifloxystrobin	5.50	409.0	186.0	145.0
183.	Novaluron	5.53	493.0	158.0	141.0
184.	Ipconazole isomer 2	5.56	334.2	70.0	125.1
185.	Emamectin benzoate B1b	5.57	872.4	158.2	126.1
186.	Clethodim isomer 2	5.65	360.0	164.0	268.1
187.	Buprofezin	5.70	306.1	201.0	57.4
188.	Teflubenzuron	5.74	380.9	158.0	140.9
189.	Emamectin benzoate B1a	5.75	886.5	158.1	126.1
190.	Benfuracarb	5.76	411.1	195.0	190.0
191.	Fluazinam	5.78	464.8	373.0	338.1
192.	Metaflumizone	5.79	507.0	287.2	267.1
193.	Furathiocarb	5.82	383.2	194.9	252.0
194.	Lufenuron	5.83	511.2	158.0	141.0
195.	Temephos	5.83	467.1	125.0	418.9
196.	Tebufenpyrad	5.86	334.0	117.0	145.0
197.	Pyriproxifen	5.91	322.1	96.0	227.1
198.	Piperonyl butoxide	5.93	356.3	176.9	119.0
199.	Hexythiazox	6.01	353.0	228.1	168.1
200.	Quinoxyfen	6.04	308.0	197.0	161.9
201.	Flufenoxuron	6.05	489.1	158.0	141.0
202.	Amitraz	6.14	294.0	163.0	122.0
203.	Propargite	6.14	368.2	175.0	231.1
204.	Etoxazole	6.16	360.2	304.2	177.2
205.	Spiromesifen	6.20	371.1	273.1	255.1
206.	Chlorfluazuron	6.21	539.8	382.9	158.0
207.	Spirodiclofen	6.33	411.1	313.0	71.2
208.	Fenpyroximate	6.36	422.2	366.1	138.1
209.	Abamectin B1b	6.48	876.6	553.4	291.0
210.	Pyridaben	6.51	365.1	147.1	309.1
211.	Eprinomectin	6.53	914.6	186.0	154.0
212.	Abamectin B1a	6.61	890.5	305.2	567.3
213.	Fenazaquin	6.69	307.2	161.0	57.2
214.	Doramectin	6.82	916.6	331.2	593.4
215.	Moxidectin	6.82	640.5	498.3	528.4
216.	Ivermectin	7.01	892.6	569.4	551.4

TECH TIP

Using syringe filters is an economical way to remove particulate matter that could clog your column. Visit **www.restek.com/filters** to access our solvent/syringe filter compatibility guide and quickly find the best filter for your method.

Figure 6: Rxi®-5ms GC columns reliably separate many commonly used pesticides.



Peaks	t_R (min)				
1. Tefluthrin	14.23	12. lambda-Cyhalothrin	22.30	23. Cypermethrin 4*	24.43
2. Transfluthrin	15.18	13. Acrinathrin	22.51	24. Flucythrinate 1*	24.43
3. Anthraquinone	16.02	14. *cis*-Permethrin	23.14	25. Flucythrinate 2*	24.66
4. Bioallethrin	17.17	15. *trans*-Permethrin	23.29	26. Fenvalerate 1*	25.25
5. Resmethrin 1*	20.43	16. Cyfluthrin 1*	23.83	27. tau-Fluvalinate 1*	25.47
6. Resmethrin 2*	20.55	17. Cyfluthrin 2*	23.93	28. Fenvalerate 2*	25.48
7. Tetramethrin 1*	21.00	18. Cyfluthrin 3*	24.02	29. tau-Fluvalinate 2*	25.53
8. Tetramethrin 2*	21.14	19. Cyfluthrin 4*	24.06	30. Deltamethrin	26.09
9. Bifenthrin	21.15	20. Cypermethrin 1*	24.19	*Isomers numbered according to elution order.	
10. Phenothrin 1*	21.59	21. Cypermethrin 2*	24.30		
11. Phenothrin 2*	21.71	22. Cypermethrin 3*	24.39		

Column: Rxi®-5ms, 30 m, 0.25 mm ID, 0.25 µm (cat.# 13423); **Sample:** GC multiresidue pesticide standard #6-SPP (cat.# 32568); Diluent: Toluene; Conc.: 100 µg/mL; **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4.0 mm ID Precision® inlet liner w/wool (cat.# 23305.1); Inj. Temp.: 250 °C; **Oven:** 90 °C (hold 1 min) to 330 °C at 8.5 °C/min (hold 5 min); **Carrier Gas:** He, constant flow; Flow Rate: 1.4 mL/min; **Detector:** MS; Mode: Scan; Start Time: 5 min; Scan Range: 55–550 amu; Scan Rate: 7 scans/sec; Transfer Line Temp.: 290 °C; Analyzer Type: Quadrupole; Source Temp.: 325 °C; Electron Energy: 70 eV; Solvent Delay Time: 5 min; Ionization Mode: EI; **Instrument:** Thermo Scientific TSQ 8000 Triple Quadrupole GC-MS; **Notes:** Bioallethrin isomers are only slightly resolved with this method, so they are treated as one peak. Chromatogram is reconstructed from select ions.

TECH TIP

Struggling with matrix interferences or high back pressures? Contact Restek's Technical Service team at **support@restek.com** for guard column recommendations.

PESTICIDE ANALYSIS PRODUCTS

Raptor™ ARC-18 LC Columns (USP L1)

Properties:

- Well-balanced retention profile.
- Sterically protected and acid-resistant to resist harsh, low-pH mobile phases.
- Ideal for use with sensitive detectors like mass spec.

Description	cat.#
2.7 µm Columns 100 mm, 2.1 mm ID	9314A12

For guard cartridges, visit our website at **www.restek.com**

26237

Q-sep® QuEChERS Extraction Salts

Fast, Simple Sample Prep for Multiresidue Pesticide Analysis

- Salt packets eliminate the need for a second empty tube to transfer salts.
- Go green by using packets with reusable tubes.
- Convenient and easy to use.

Description	Material	Methods	qty.	cat.#
Q-sep Kit	6 g $MgSO_4$, 1.5 g NaOAc with 50 mL Centrifuge Tube	AOAC 2007.01	50 packets & 50 tubes	26237

NaOAc—sodium acetate

Rxi®-5ms Columns (fused silica)

(low-polarity phase; Crossbond® diphenyl dimethyl polysiloxane)

- General-purpose columns for semivolatiles, phenols, amines, residual solvents, drugs of abuse, pesticides, PCB congeners (e.g., Aroclor mixes), solvent impurities.
- Most inert column on the market.
- Tested and guaranteed for ultra-low bleed; improved signal-to-noise ratio for better sensitivity and mass spectral integrity.
- Equivalent to USP G27 and G36 phases.

Description	temp. limits	qty.	cat.#
30 m, 0.25 mm ID, 0.25 µm	-60 to 330/350 °C	ea.	13423

QuEChERS Performance Standards Kit

- Kit contains organochlorine, organonitrogen, organophosphorus, and carbamate pesticides commonly used on fruits and vegetables.
- Ideal for initial method evaluations and ongoing method performance validations.
- Analytes are divided into three ampuls based on compatibility for maximum stability and shelf life.*
- Precise formulations improve data quality and operational efficiency; spend more time running samples and less time sourcing and preparing standards.

Contains 1 mL each of these mixtures.
31153: QuEChERS Performance Standard A
31154: QuEChERS Performance Standard B
31155: QuEChERS Performance Standard C

300 µg/mL each in acetonitrile/acetic acid (99.9:0.1), 1 mL/ampul.
Blend equal volumes of all three ampuls for a 100 µg/mL final solution.

cat.# 31152 (kit)

kit

*When combining compounds with different functionalities, chemical stability can be an issue. The analytes in this kit are separated into three mixes to ensure maximum long-term storage stability. For analysis, a fresh working standard should be prepared by combining the three kit mixes in a 1:1:1 ratio to prepare a 100 µg/mL working standard solution. Once blended, Restek does not recommend storing working standards or subsequent dilutions for future use.

For LC Analysis

Q-sep® QuEChERS dSPE Tubes for Extract Cleanup

Fast, Simple Sample Prep for Multiresidue Pesticide Analysis

Packaged in foil subpacks of 10 for enhanced protection and storage stability.

Multiple sorbents are used to extract different types of interferences.

- $MgSO_4$ removes excess water
- PSA removes sugars, fatty acids, organic acids, and anthocyanine pigments
- C18 removes nonpolar interferences

Description	Methods	qty.	cat.#
2 mL Micro-Centrifuge Tubes for dSPE (cleanup of 1 mL extract)			
150 mg $MgSO_4$, 50 mg PSA, 50 mg C18	AOAC 2007.01	100-pk.	26125

PSA—primary and secondary amine

26125

For GC Analysis

Pesticide Residue Cleanup SPE Cartridges

- Convenient, multiple adsorbent beds in a single cartridge.
- For use in multiresidue pesticide analysis to remove matrix interferences.
- Excellent for cleanup of dietary supplement extracts.

SPE Cartridge	qty.	cat.#
6 mL Combo SPE Cartridge Packed with 500 mg CarboPrep 90/500 mg PSA, Polyethylene Frits	30-pk.	26194

PSA–primary and secondary amine

26194

PESTICIDE ANALYSIS PRODUCTS (CONT.)

LC Multiresidue Pesticide Kit

- Accurately detect and quantify pesticides of global food safety concern in a wide range of fruits, vegetables, and other commodities by LC-MS/MS.
- Full kit contains 204 compounds of interest, covering many LC-determined pesticides listed by government agencies; individual ampuls also sold separately.
- Formulated and grouped for maximum long-term stability* and well-balanced chromatographic performance, even for early eluting compounds.
- Quantitatively tested to confirm composition; detailed support documentation provided.
- Optimized multiresidue pesticide method is offered free of charge; downloadable XLS file includes conditions and transition tables.
- Certified reference material (CRM) manufactured and QC-tested in Restek's ISO-accredited labs satisfies your ISO requirements.

Restek® Certified Reference Materials

Cat.# 31972: LC Multiresidue Pesticide Standard #1 (13 components)
Organophosphorus Compounds
Acephate (30560-19-1)
Carbaryl (Sevin) (63-25-2)
Dicrotophos (141-66-2)
Dimethoate (60-51-5)
Dimethomorph (110488-70-5)
Isocarbophos (24353-61-5)
Methamidophos (10265-92-6)
Mevinphos (7786-34-7)
Monocrotophos (6923-22-4)
Omethoate (1113-02-6)
Temephos (Abate) (3383-96-8)
Trichlorfon (Dylox) (52-68-6)
Vamidothion (Vamidoate) (2275-23-2)

Cat.# 31973: LC Multiresidue Pesticide Standard #2 (16 components)
Carbamate/Uron Compounds
Alanycarb (83130-01-2)
Aldicarb (116-06-3)
Aldicarb sulfone (1646-88-4)
Aldicarb sulfoxide (1646-87-3)
Benfuracarb (82560-54-1)
Butocarboxim (34681-10-2)
Butoxycarboxim (34681-23-7)
Ethiofencarb (29973-13-5)
Furathiocarb (65907-30-4)
Methabenzthiazuron (18691-97-9)
Methiocarb (2032-65-7)
Methomyl (16752-77-5)
Oxamyl (23135-22-0)
Tebuthiuron (34014-18-1)
Thidiazuron (51707-55-2)
Thiophanate-methyl (23564-05-8)

Cat.# 31974: LC Multiresidue Pesticide Standard #3 (38 components)
Carbamate/Uron Compounds
Bendiocarb (22781-23-3)
Bifenazate (149877-41-8)
Carbofuran (1563-66-2)
Chlorfluazuron (71422-67-8)
Chloroxuron (1982-47-4)
Chlortoluron (15545-48-9)
Cycluron (2163-69-1)
Diethofencarb (87130-20-9)
Diflubenzuron (35367-38-5)
Dioxacarb (6988-21-2)
Diuron (330-54-1)
Fenobucarb (BPMC) (3766-81-2)
Fenoxycarb (79127-80-3)
Fenuron (101-42-8)
Flufenoxuron (101463-69-8)
Fluometuron (2164-17-2)
Forchlorfenuron (68157-60-8)
Hexaflumuron (86479-06-3)
3-Hydroxycarbofuran (16655-82-6)
Indoxacarb (173584-44-6)
Iprovalicarb (140923-17-7)
Isoprocarb (2631-40-5)
Isoproturon (34123-59-6)
Linuron (330-55-2)
Lufenuron (103055-07-8)
Metobromuron (3060-89-7)
Monolinuron (1746-81-2)
Neburon (555-37-3)
Novaluron (116714-46-6)
Pirimicarb (23103-98-2)
Promecarb (2631-37-0)
Propham (122-42-9)
Propoxur (Baygon) (114-26-1)
Pyraclostrobin (175013-18-0)
Siduron (1982-49-6)
Teflubenzuron (83121-18-0)
Thiobencarb (28249-77-6)
Triflumuron (64628-44-0)

Cat.# 31975: LC Multiresidue Pesticide Standard #4 (63 components)
Organonitrogen Compounds
Abamectin (71751-41-2)
Acetamiprid (135410-20-7)
Ametryn (834-12-8)
Amitraz (33089-61-1)
Azoxystrobin (131860-33-8)
Benalaxyl (71626-11-4)
Benzoximate (29104-30-1)
Boscalid (188425-85-6)
Butafenacil (134605-64-4)
Carbetamide (16118-49-3)
Carfentrazone-ethyl (128639-02-1)
Chlorantraniliprole (500008-45-7)
Clofentezine (74115-24-5)
Cymoxanil (57966-95-7)
Cyprodinil (121552-61-2)
Cyromazine (66215-27-8)
Dimoxystrobin (149961-52-4)
Dinotefuran (165252-70-0)
Doramectin (117704-25-3)
Eprinomectin (123997-26-2)
Famoxadon (131807-57-3)
Fenazaquin (120928-09-8)
Fenhexamid (126833-17-8)
Fenpyroximate (111812-58-9)
Flonicamid (158062-67-0)
Fluazinam** (79622-59-6)
Fludioxonil (131341-86-1)
Fluoxastrobin (361377-29-9)
Flutolanil (66332-96-5)
Furalaxyl (57646-30-7)
Halofenozide (112226-61-6)
Imazalil (35554-44-0)
Imidacloprid (138261-41-3)
Ivermectin (70288-86-7)
Kresoxim-methyl (143390-89-0)
Mandipropamid (374726-62-2)
Mepanipyrim (110235-47-7)
Mepronil (55814-41-0)
Metaflumizone (139968-49-3)
Metalaxyl (57837-19-1)
Methoxyfenozide (161050-58-4)
Moxidectin (113507-06-5)
Myclobutanil (88671-89-0)
Nitenpyram (120738-89-8)
Oxadixyl (77732-09-3)
Picoxystrobin (117428-22-5)
Piperonyl butoxide (51-03-6)
Prochloraz (67747-09-5)
Prometon (1610-18-0)
Pymetrozine (123312-89-0)
Pyracarbolid (24691-76-7)
Pyrimethanil (53112-28-0)
Pyriproxyfen (95737-68-1)
Quinoxyfen (124495-18-7)
Rotenone (83-79-4)
Secbumeton (26259-45-0)
Spiroxamine (118134-30-8)
Tebufenozide (112410-23-8)
Tebufenpyrad (119168-77-3)
Terbumeton (33693-04-8)
Triadimefon (43121-43-3)
Trifloxystrobin (141517-21-7)
Zoxamide (156052-68-5)

Cat.# 31976: LC Multiresidue Pesticide Standard #5 (30 components)
Organonitrogen Compounds
Acibenzolar-S-methyl (135158-54-2)
Bupirimate (41483-43-6)
Buprofezin (69327-76-0)
Carboxin (5234-68-4)
Clethodim (99129-21-2)
Clothianidin (210880-92-5)
Cyazofamid (120116-88-3)
Ethiprole (181587-01-9)
Ethofumesate (26225-79-6)
Fenamidone (161326-34-7)
Fipronil (120068-37-3)
Flubendimide (272451-65-7)
Flufenacet (Fluthiamide) (142459-58-3)
Hexythiazox (78587-05-0)
Mefenacet (73250-68-7)
Mesotrione (104206-82-8)
Methoprotryne (841-06-5)
Metribuzin (21087-64-9)
Prometryne (7287-19-6)
Propargite (2312-35-8)
Prothioconazole (178928-70-6)
Pyridaben (96489-71-3)
Simetryn (1014-70-6)
Sulfentrazone (122836-35-5)
Terbutryn (886-50-0)
Thiabendazole (148-79-8)
Thiacloprid (111988-49-9)
Thiamethoxam (153719-23-4)
Thiofanox (39196-18-4)
Tricyclazole (Beam) (41814-78-2)

Cat.# 31977: LC Multiresidue Pesticide Standard #6 (28 components)
Organonitrogen Compounds
Baycor (Bitertanol) (55179-31-2)
Bromuconazole (116255-48-2)
Cyproconazole (113096-99-4)
Diclobutrazol (75736-33-3)
Difenoconazole (119446-68-3)
Diniconazole (83657-24-3)
Epoxiconazole (133855-98-8)
Etaconazole (60207-93-4)
Ethirimol (23947-60-6)
Etoxazole (153233-91-1)
Fenarimol (60168-88-9)
Fenbuconazole (114369-43-6)
Fluquinconazole (136426-54-5)
Flusilazole (85509-19-9)
Flutriafol (76674-21-0)
Fuberidazole (3878-19-1)
Hexaconazole (79983-71-4)
Ipconazole (125225-28-7)
Metconazole (125116-23-6)
Nuarimol (63284-71-9)
Paclobutrazol (76738-62-0)
Penconazole (66246-88-6)
Propiconazole (Tilt) (60207-90-1)
Tebuconazole (107534-96-3)
Tetraconazole (112281-77-3)
Triadimenol (55219-65-3)
Triflumizole (68694-11-1)
Triticonazole (131983-72-7)

Cat.# 31978: LC Multiresidue Pesticide Standard #7 (7 components)
Organonitrogen Compounds
Emamectin-benzoate (155569-91-8)
Fenpropimorph (67564-91-4)
Spirodiclofen (148477-71-8)
Spinosad (168316-95-8)
Spirotetramat (203313-25-1)
Spinetoram (J&L) (187166-40-1)
Spiromesifen (283594-90-1)

Cat.# 31979: LC Multiresidue Pesticide Standard #8
Organonitrogen Compounds
Hydramethylnon (67485-29-4)

Cat.# 31980: LC Multiresidue Pesticide Standard #9 (7 components)
Carbamate/Uron Compounds
Aminocarb (2032-59-9)
Desmedipham (13684-56-5)
Formetanate HCL (23422-53-9)
Mexacarbate (Zectran) (315-18-4)
Monceren (Pencycuron) (66063-05-6)
Phenmedipham (13684-63-4)
Propamocarb free base (24579-73-5)

Cat.# 31981: LC Multiresidue Pesticide Standard #10
Carbamate/Uron Compounds
Carbendazim (10605-21-7)

Contains 1 mL each of these mixtures.
cat.# 31971 (kit)

Quantity discounts not available.

* NOTE: When combining a large number of compounds with different chemical functionalities, mix stability can be an issue. In formulating these standards, we extensively studied the 204 compounds involved, then grouped them into as few mixes as possible while still ensuring maximum long-term stability and reliability. For quantitative analysis, we recommend analyzing each mix separately to ensure accurate results for every compound.

** NOTE: In this standard, fluazinam should only be used for qualitative analysis. A single-component standard (cat.# 31982) is available for quantitative analysis.

GC Multiresidue Pesticide Kit

- Accurately identify and quantify pesticide residues by GC-MS/MS in fruits, vegetables, botanicals, and herbals like tea, ginseng, ginger, Echinacea, and dietary supplements.
- Comprehensive 203-compound kit covers food safety lists by the FDA, USDA, and other global governmental agencies; individual ampuls also sold separately.
- Formulated and grouped for maximum long-term stability* and well-balanced chromatographic performance, even for early eluting compounds.
- Quantitatively tested to confirm composition; detailed support documentation provided.
- Certified reference material (CRM) manufactured and QC-tested in Restek's ISO-accredited labs satisfies your ISO requirements.

Restek® Certified Reference Materials

Cat.# 32563: GC Multiresidue Pesticide Standard #1 (16 components)
Organophosphorus Compounds
Azinphos ethyl (2642-71-9)
Azinphos-methyl (86-50-0)
Chlorpyrifos (2921-88-2)
Chlorpyrifos methyl (5598-13-0)
Diazinon (333-41-5)
EPN (2104-64-5)
Fenitrothion (122-14-5)
Isazophos (42509-80-8)
Phosalone (2310-17-0)
Phosmet (732-11-6)
Pirimiphos ethyl (23505-41-1)
Pirimiphos methyl (29232-93-7)
Pyraclofos (77458-01-6)
Pyrazophos (13457-18-6)
Pyridaphenthion (119-12-0)
Quinalphos (13593-03-8)

Cat.# 32564: GC Multiresidue Pesticide Standard #2 (40 components)
Organochlorine Compounds
Aldrin (309-00-2)
alpha-BHC (319-84-6)
beta-BHC (319-85-7)
delta-BHC (319-86-8)
gamma-BHC (Lindane) (58-89-9)
Chlorbenside (103-17-3)
cis-Chlordane (5103-71-9)
trans-Chlordane (5103-74-2)
Chlorfenson (Ovex) (80-33-1)
Chloroneb (2675-77-6)
2,4'-DDD (53-19-0)
4,4'-DDD (72-54-8)
2,4'-DDE (3424-82-6)
4,4'-DDE (72-55-9)
2,4'-DDT (789-02-6)
4,4'-DDT (50-29-3)
4,4'-Dichlorobenzophenone (90-98-2)
Dieldrin (60-57-1)
Endosulfan I (959-98-8)
Endosulfan II (33213-65-9)
Endosulfan ether (3369-52-6)
Endosulfan sulfate (1031-07-8)
Endrin (72-20-8)
Endrin aldehyde (7421-93-4)
Endrin ketone (53494-70-5)
Ethylan (Perthane) (72-56-0)
Fenson (80-38-6)
Heptachlor (76-44-8)
Heptachlor epoxide (Isomer B) (1024-57-3)
Hexachlorobenzene (118-74-1)
Isodrin (465-73-6)
2,4'-Methoxychlor (30667-99-3)
4,4'-Methoxychlor olefin (2132-70-9)
Mirex (2385-85-5)
cis-Nonachlor (5103-73-1)
trans-Nonachlor (39765-80-5)
Pentachloroanisole (1825-21-4)
Pentachlorobenzene (608-93-5)
Pentachlorothioanisole (1825-19-0)
Tetradifon (116-29-0)

Cat.# 32565: GC Multiresidue Pesticide Standard #3 (25 components)
Organonitrogen Compounds
Benfluralin (1861-40-1)
Biphenyl (92-52-4)
Chlorothalonil (1897-45-6)
Dichlofluanid (1085-98-9)
Dichloran (99-30-9)
3,4-Dichloroaniline (95-76-1)
2,6-Dichlorobenzonitrile (Dichlobenil) (1194-65-6)
Diphenylamine (122-39-4)
Ethalfluralin (55283-68-6)
Fluchloralin (33245-39-5)
Isopropalin (33820-53-0)
Nitralin (4726-14-1)
Nitrofen (1836-75-5)
Oxyfluorfen (42874-03-3)
Pendimethalin (40487-42-1)
Pentachloroaniline (527-20-8)
Pentachlorobenzonitrile (20925-85-3)
Pentachloronitrobenzene (Quintozene) (82-68-8)
Prodiamine (29091-21-2)
Profluralin (26399-36-0)
2,3,5,6-Tetrachloroaniline (3481-20-7)
Tetrachloronitrobenzene (Tecnazene) (117-18-0)
THPI (Tetrahydrophthalimide) (1469-48-3)
Tolylfluanid (731-27-1)
Trifluralin (1582-09-8)

Cat.# 32566: GC Multiresidue Pesticide Standard #4 (28 components)
Organonitrogen Compounds
Acetochlor (34256-82-1)
Alachlor (15972-60-8)
Allidochlor (93-71-0)
Clomazone (Command) (81777-89-1)
Cycloate (1134-23-2)
Diallate (*cis* and *trans*) (2303-16-4)
Dimethachlor (50563-36-5)
Diphenamid (957-51-7)
Fenpropathrin (39515-41-8)
Fluquinconazole (136426-54-5)
Flutolanil (66332-96-5)
Linuron (330-55-2)
Metazachlor (67129-08-2)
Methoxychlor (72-43-5)
Metolachlor (51218-45-2)
N-(2,4-Dimethylphenyl) formamide (60397-77-5)
Norflurazon (27314-13-2)
Oxadiazon (19666-30-9)
Pebulate (1114-71-2)
Pretilachlor (51218-49-6)
Prochloraz (67747-09-5)
Propachlor (1918-16-7)
Propanil (709-98-8)
Propisochlor (86763-47-5)
Propyzamide (23950-58-5)
Pyridaben (96489-71-3)
Tebufenpyrad (119168-77-3)
Triallate (2303-17-5)

Cat.# 32567: GC Multiresidue Pesticide Standard #5 (34 components)
Organonitrogen Compounds
Atrazine (1912-24-9)
Bupirimate (41483-43-6)
Captafol (2425-06-1)
Captan (133-06-2)
Chlorfenapyr (122453-73-0)
Cyprodinil (121552-61-2)
Etofenprox (80844-07-1)
Etridiazole (2593-15-9)
Fenarimol (60168-88-9)
Fipronil (120068-37-3)
Fludioxonil (131341-86-1)
Fluridone (Sonar) (59756-60-4)
Flusilazole (85509-19-9)
Flutriafol (76674-21-0)
Folpet (133-07-3)
Hexazinone (Velpar) (51235-04-2)
Iprodione (36734-19-7)
Lenacil (2164-08-1)
MGK-264 (113-48-4)
Myclobutanil (88671-89-0)
Paclobutrazol (76738-62-0)
Penconazole (66246-88-6)
Procymidone (32809-16-8)
Propargite (2312-35-8)
Pyrimethanil (53112-28-0)
Pyriproxyfen (95737-68-1)
Tebuconazole (107534-96-3)
Terbacil (5902-51-2)
Terbuthylazine (5915-41-3)
Triadimefon (43121-43-3)
Triadimenol (55219-65-3)
Tricyclazole (Beam) (41814-78-2)
Triflumizole (68694-11-1)
Vinclozolin (50471-44-8)

Cat.# 32568: GC Multiresidue Pesticide Standard #6 (18 components)
Synthetic Pyrethroid Compounds
Acrinathrin (101007-06-1)
Anthraquinone (84-65-1)
Bifenthrin (82657-04-3)
Bioallethrin (584-79-2)
Cyfluthrin (68359-37-5)
lambda-Cyhalothrin (91465-08-6)
Cypermethrin (52315-07-8)
Deltamethrin (52918-63-5)
Fenvalerate (51630-58-1)
Flucythrinate (70124-77-5)
tau-Fluvalinate (102851-06-9)
cis-Permethrin (61949-76-6)
trans-Permethrin (61949-77-7)
Phenothrin (*cis* & *trans*) (26002-80-2)
Resmethrin (10453-86-8)
Tefluthrin (79538-32-2)
Tetramethrin (7696-12-0)
Transfluthrin (118712-89-3)

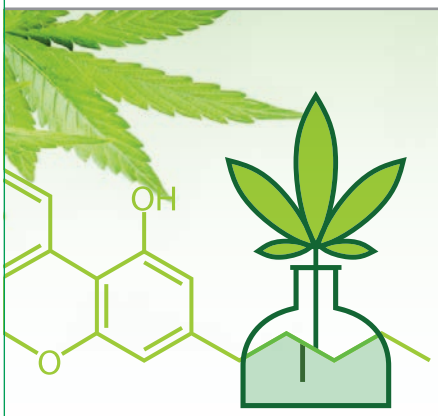
Cat.# 32569: GC Multiresidue Pesticide Standard #7 (10 components)
Herbicide Methyl Esters
Acequinocyl (57960-19-7)
Bromopropylate (18181-80-1)
Carfentrazone ethyl (128639-02-1)
Chlorobenzilate (510-15-6)
Chlorpropham (101-21-3)
Chlozolinate (84332-86-5)
DCPA methyl ester (Chlorthal-dimethyl) (1861-32-1)
Fluazifop-*p*-butyl (79241-46-6)
Metalaxyl (57837-19-1)
2-Phenylphenol (90-43-7)

Cat.# 32570: GC Multiresidue Pesticide Standard #8 (24 components)
Organophosphorus Compounds
Bromfenvinfos-methyl (13104-21-7)
Bromfenvinphos (33399-00-7)
Bromophos ethyl (4824-78-6)
Bromophos methyl (2104-96-3)
Carbophenothion (786-19-6)
Chlorfenvinphos (470-90-6)
Chlorthiophos (60238-56-4)
Coumaphos (56-72-4)
Edifenphos (17109-49-8)
Ethion (563-12-2)
Fenamiphos (22224-92-6)
Fenchlorphos (Ronnel) (299-84-3)
Fenthion (55-38-9)
Iodofenphos (18181-70-9)
Leptophos (21609-90-5)
Malathion (121-75-5)
Methacrifos (62610-77-9)
Profenofos (41198-08-7)
Prothiofos (34643-46-4)
Sulfotepp (3689-24-5)
Sulprofos (35400-43-2)
Terbufos (13071-79-9)
Tetrachlorvinfos (22248-79-9)
Tolclofos-methyl (57018-04-9)

Cat.# 32571: GC Multiresidue Pesticide Standard #9 (8 components)
Organophosphorus Compounds
Disulfoton (298-04-4)
Fonofos (944-22-9)
Methyl parathion (298-00-0)
Mevinphos (7786-34-7)
Parathion (Ethyl parathion) (56-38-2)
Phorate (298-02-2)
Piperonyl butoxide (51-03-6)
Triazophos (24017-47-8)

Contains 1 mL each of these mixtures.
cat.# 32562 (kit)

* NOTE: When combining a large number of compounds with different chemical functionalities, mix stability can be an issue. In formulating these standards, we extensively studied the 203 compounds involved, then grouped them into as few mixes as possible while still ensuring maximum long-term stability and reliability. For quantitative analysis, we recommend analyzing each mix separately to ensure accurate results for every compound.



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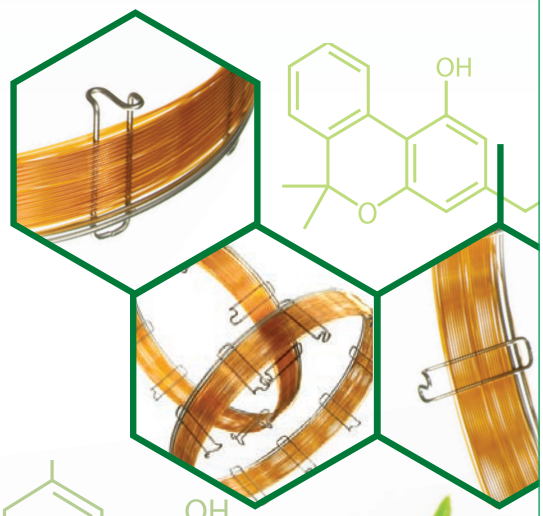

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Technical Article

High-Quality Analysis of Pesticides in Cannabis

Using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS

By Jack Cochran, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, and Amanda Rigdon

- Quickly and effectively extract medical marijuana samples for pesticide analysis.
- Cartridge SPE cleanup of dirty extracts improves GC inlet and column lifetimes.
- Selective GC columns increase accuracy of pesticide determinations for complex samples.

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In order to address matrix complexity, we combined a simple QuEChERS extraction approach with cartridge SPE (cSPE) cleanup, followed by GCxGC-TOFMS. Acceptable recoveries were obtained for most pesticides, and incurred pesticide residues were detected in some of the illicit marijuana samples used for method development.

QuEChERS Extraction Saves Time and Reduces Hazardous Solvent Use

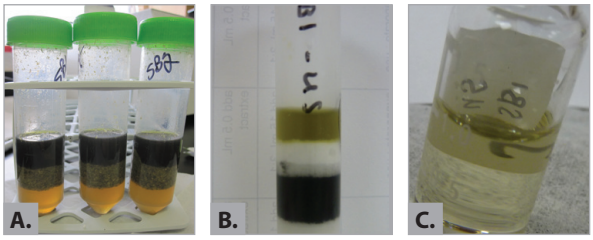
Trace residue extraction procedures from dry materials like medical cannabis typically involve large amounts of solvent, long extraction times, and tedious concentration steps similar to the Soxhlet procedure or multiresidue methods from the Pesticide Analytical Manual. QuEChERS, with its simple 10 mL acetonitrile shake extraction and extract partitioning with salts and centrifugation, offers time savings, glassware use reduction, and lower solvent consumption.

Water was added to finely ground, dry cannabis samples to increase QuEChERS extraction efficiency, especially for more polar pesticides. A vortex mixer was used to shake the solvent and sample for at least 30 minutes prior to extract partitioning. When finished, it was easy to transfer the supernatant from the QuEChERS extraction tube for subsequent cSPE cleanup prior to analysis with GC or LC (Figure 1).

Cartridge SPE Cleanup Improves GC Inlet Uptime

Injecting chlorophyll-laden extracts into a GC gives reduced recoveries for less volatile pesticides, and results in degradation of sensitive pesticides like DDT and Dicofol (Table I). SPE cleanup with a 500 mg graphitized carbon black/500 mg PSA cartridge removes chlorophyll and traps fatty acids that interfere with qualitative pesticide identification and bias quantification. cSPE has increased sorbent capacity over dispersive SPE for thorough cleanup of complex extracts.

Figure 1: A quick and easy QuEChERS extraction, combined with cSPE, effectively prepared extracts for pesticide residue analysis from highly complex marijuana samples.



Post-centrifugation QuEChERS extracts | QuEChERS extracts loaded on SPE cartridge | Final extract

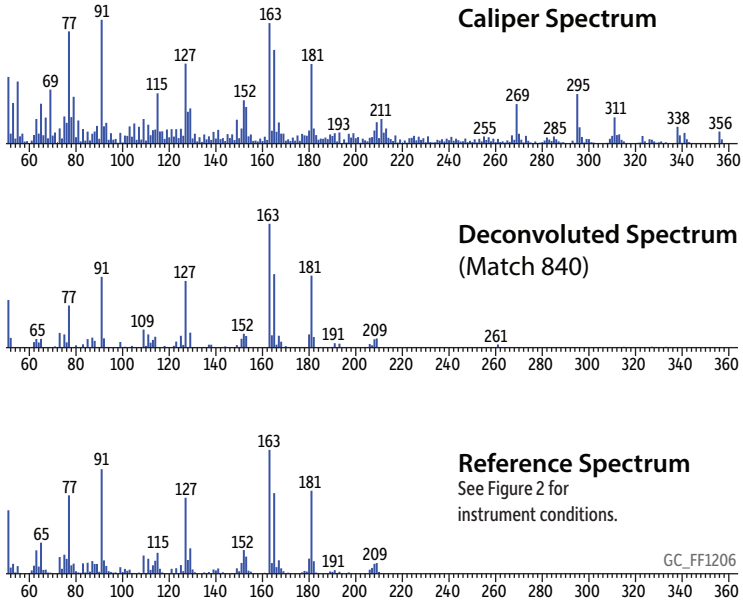
Orthogonal GC Columns Increase Separation Power for More Accurate Pesticide Results

GCxGC is a powerful multidimensional approach that gives two independent separations in one instrumental analysis. An Rxi®-5Sil MS and Rtx®-200 column combination distributes pesticides broadly in both dimensions, providing a highly orthogonal GCxGC system. More important though is separating pesticides from potential isobaric matrix interferences, as seen in the surface plot for the insecticide cypermethrin (Figure 2). Cypermethrin gas chromatographs as four isomers, and all would have experienced qualitative interference and quantitative bias from peaks in the foreground of the surface plot had only 1-dimensional GC been used. With GCxGC-TOFMS, cypermethrin was unequivocally identified in a marijuana sample at a low ppm level (Figure 3).

Summary

QuEChERS and cSPE produced usable extracts from highly complex cannabis samples for high-quality pesticide residue analysis. The multidimensional separation power of GCxGC-TOFMS was then used to correctly identify and quantify pesticides in these complex extracts.

Figure 3: Positive mass spectral identification of incurred cypermethrin in illicit marijuana.



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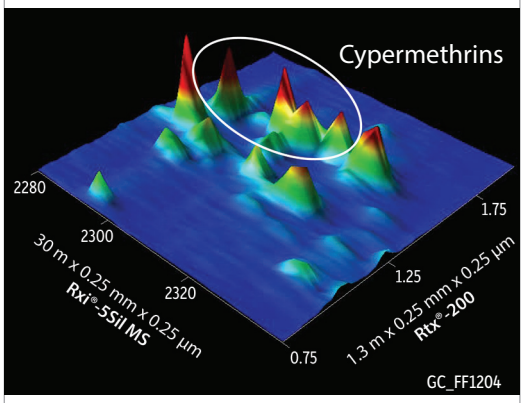
Dicofol	Organochlorine	84	ND
Azinphos methyl	Organophosphorus	79	53
trans-Permethrin	Organochlorine	68	17
Pyraclostrobin	Strobilurin	73	19
Fluvalinate	Pyrethroid	72	23
Difenoconazole	Triazole	67	21
Deltamethrin	Pyrethroid	68	20
Azoxystrobin	Strobilurin	72	27

ND = no peak detected

Figure 2: GCxGC-TOFMS and orthogonal Rxi®-5Sil MS and Rtx®-200 columns allow incurred cypermethrins in a marijuana extract to be separated from interferences (m/z 163 quantification ion).



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Column: Rxi®-5Sil MS 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623), Rtx®-200 1.3 m, 0.25 mm ID, 0.25 µm (cat.# 15124); **Sample:** Diluent: Toluene; **Injection:** Inj. Vol.: 1 µL splitless (hold 1 min); Liner: Sky® 4mm single taper w/wool (cat.# 23303.1); Inj. Temp.: 250 °C; Purge Flow: 40 mL/min; **Oven:** Oven Temp: Rxi®-5Sil MS: 80 °C (hold 1 min) to 310 °C at 5 °C/min, Rtx®-200: 85 °C (hold 1 min) to 315 °C at 5 °C/min; **Carrier Gas:** He, corrected constant flow (2 mL/min); **Modulation**: Modulator Temp. Offset: 20 °C; Second Dimension Separation Time: 3 sec.; Hot Pulse Time: 0.9 sec.; Cool Time between Stages: 0.6 sec.; **Instrument**: LECO Pegasus 4D GCxGC-TOFMS; For complete conditions, visit www.restek.com and enter GC_FF1204 in the search.

Search

« Early Eluting Terpenes – GC – Medical Cannabis
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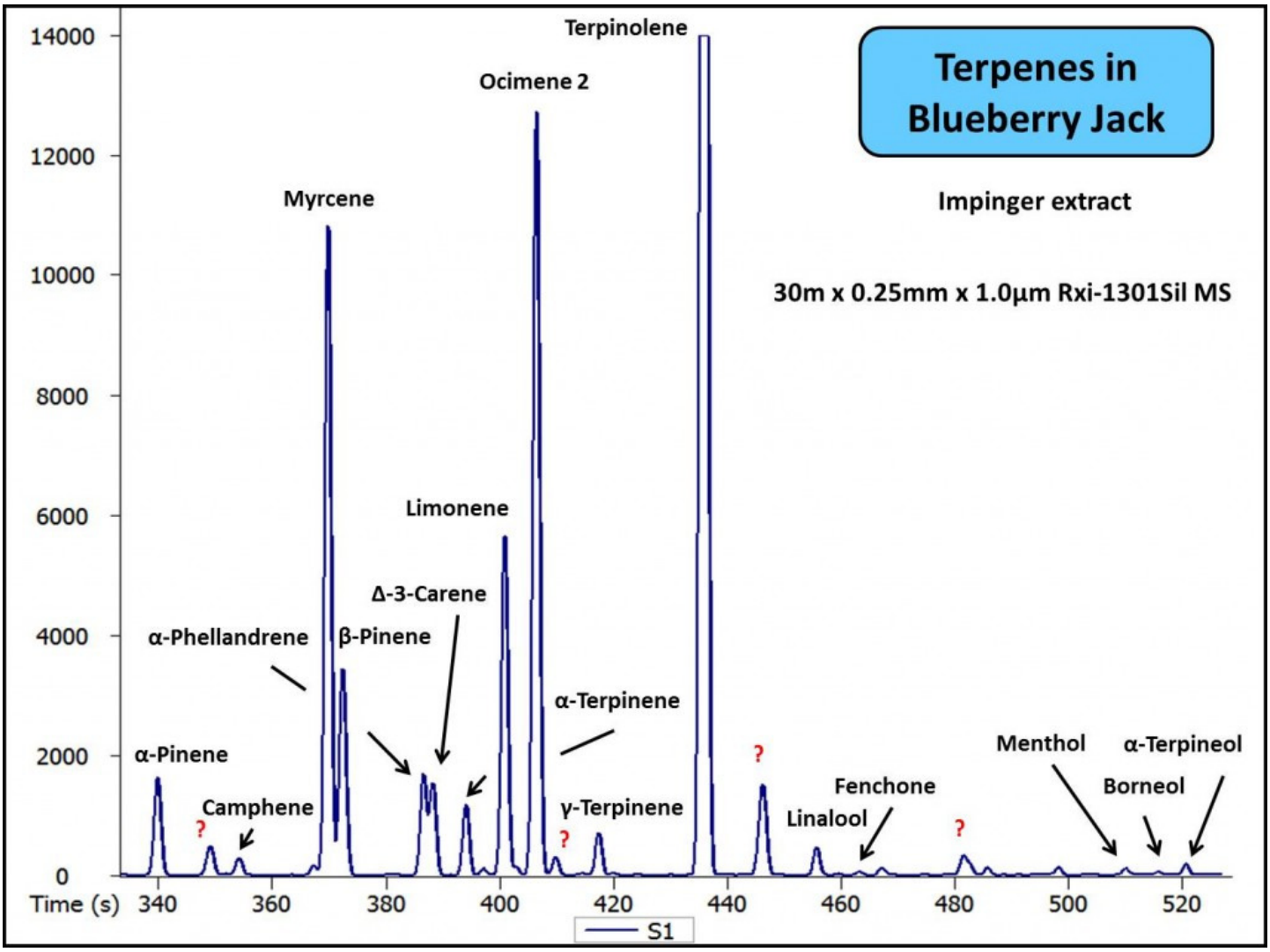
Terpenes in Blueberry Jack Medical Cannabis – GC – More Identified

March 26th, 2014 by Jack Cochran

Based on acquisition of new terpene standards I was able to better profile the Blueberry Jack medical cannabis impinger sample on the beta-version 30m x 0.25mm x 1.0µm Rxi-1301Sil MS GC column. Check it out…

I'm looking for suggestions on terpene identification for the ones marked by "?" in the chromatogram below. Help, please!



This entry was posted on Wednesday, March 26th, 2014 at 10:37 pm and is filed under New GC Columns, Medical Marijuana. You can follow any responses to this entry through the RSS 2.0 feed. You can leave a response, or trackback from your own site.

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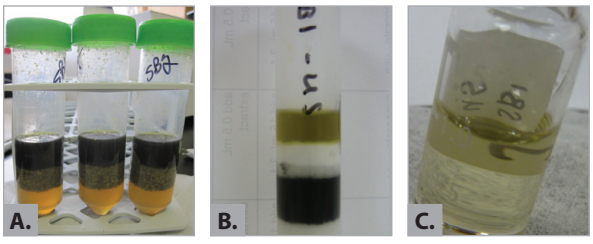

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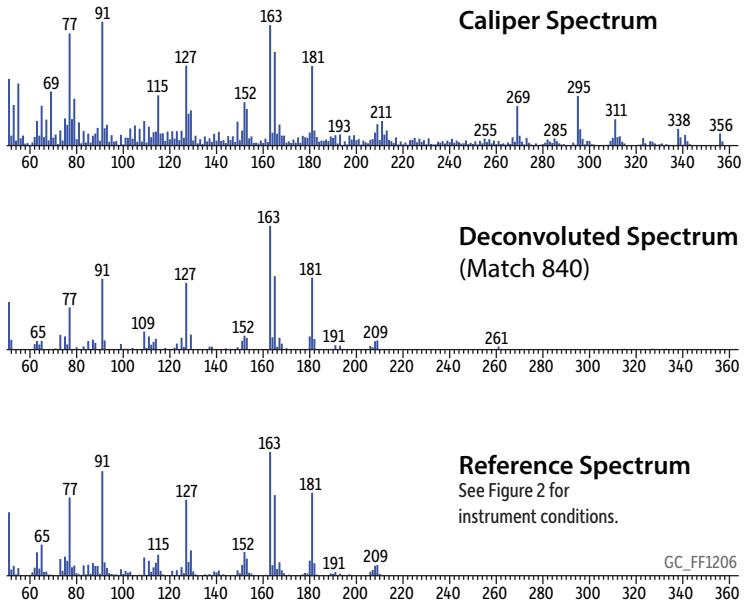

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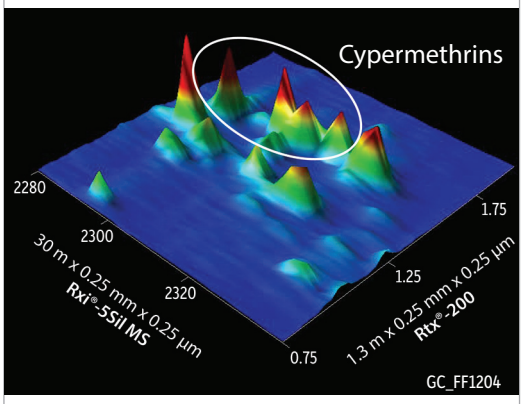

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Technical Article

Don't Overestimate Cannabidiol During Medical Cannabis Potency Testing by Gas Chromatography

By Jack Cochran

Accurate potency testing of medical cannabis with gas chromatography (GC) depends principally on choosing a column with the right selectivity; otherwise, coelutions between cannabinoids of interest may cause error in potency measurements. Cannabidiol is one of the chief cannabinoids with pharmacological value and provides relief against nausea, anxiety, and inflammation. Potency testing for medical marijuana is often done using "5-type" GC columns since they are commonly available in most labs. However, on 5-type columns cannabidiol can coelute with cannabichromene, a compound that likely also has medical value and is increasingly becoming part of potency testing. To identify and report both of these compounds accurately, a GC column with a different stationary phase is needed.

Proper Column Choice Results in More Accurate Potency Data

As shown in Figure 1, cannabinoids are aromatic compounds, meaning they will likely be better separated on a column that contains aromatics in the stationary phase because these stationary phases are more selective for aromatic-containing analytes. A fully non-aromatic stationary phase, like a "1-type" (100% dimethyl polysiloxane) column is not appropriate for this analysis since cannabichromene (CBC) and cannabidiol (CBD) will coelute completely. While 5-type columns (5% phenyl) contain some aromatic component, they generally also produce coelutions for cannabichromene and cannabidiol, depending on the conditions used. At best, CBC and CBD can be only partially resolved on 15 m 5% phenyl columns. Much better separations are obtained on higher phenyl-content phases, such as Rxi®-35Sil MS (35% phenyl type) and Rxi®-17Sil MS (50% phenyl type) columns, as they offer excellent selectivity for aromatic cannabinoids. Not only do both columns resolve cannabichromene and cannabidiol, the chromatograms in Figures 2 and 3 demonstrate that they also separate delta-8-tetrahydrocannabinol (d8-THC), delta-9-tetrahydrocannabinol (d9-THC), cannabigerol (CBG), and cannabinol (CBN). Although both columns perform well, the Rxi®-35Sil MS column is recommended because of the slightly faster analysis time and greater space overall between the peaks of interest.

While stationary phase selectivity is the most important factor in choosing a GC column for cannabinoid analysis, there are some additional aspects of this work that will benefit labs doing medical marijuana potency testing. First, cost savings were achieved by using a 15 m column. When a column with the proper selectivity is used, a 15 m column easily provides the separating power needed for this analysis at about half the cost of a 30 m column. Also, the 0.25 mm x 0.25 µm format has good sample loading capacity and is robust, especially when a proper split injection is used with a Sky® Precision® split liner with wool. Finally, hydrogen carrier gas was used here instead of helium. Using hydrogen provides a faster analysis, increasing sample throughput. Hydrogen carrier gas is a convenient way to speed up run times, increase productivity, and reduce the cost and availability concerns associated with using helium carrier gas.

Figure 1: Since cannabinoids are aromatic compounds, a GC column that contains aromatics in the stationary phase will provide much better separations than a column with a non-aromatic phase.

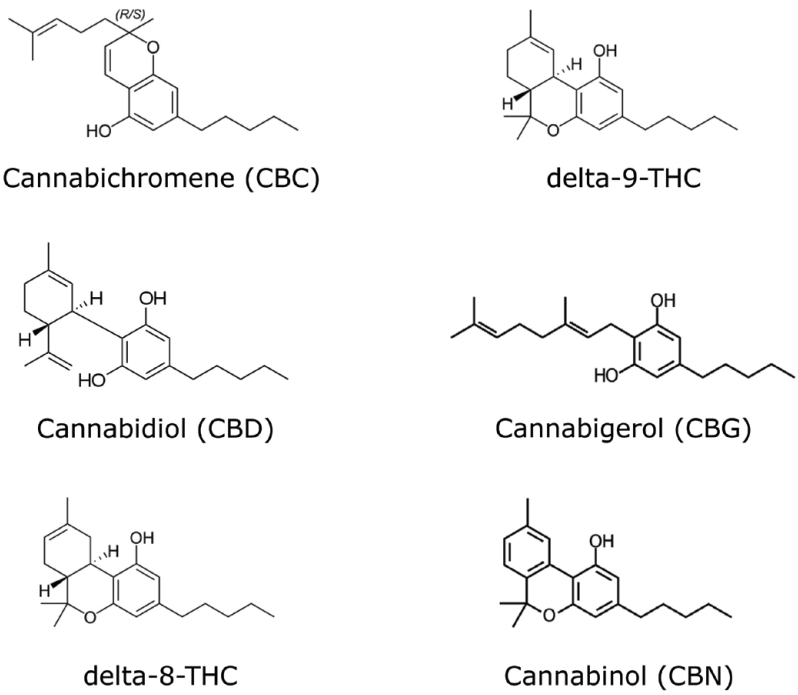


Figure 2: The Rxi®-35Sil MS column provides both the best separation and the fastest analysis time, making it the ideal GC column choice for medical cannabis potency testing.

Peaks
1. Cannabichromene
2. Cannabidiol
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-35Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13820); **Sample:** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 225 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.9 min); **Carrier Gas;** H_2, constant flow; Flow Rate: 2.5 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC

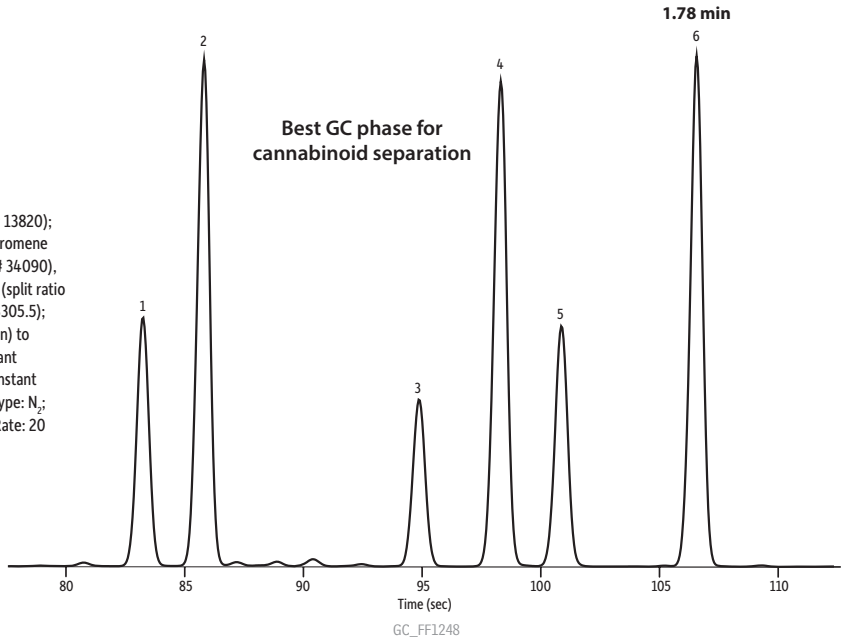
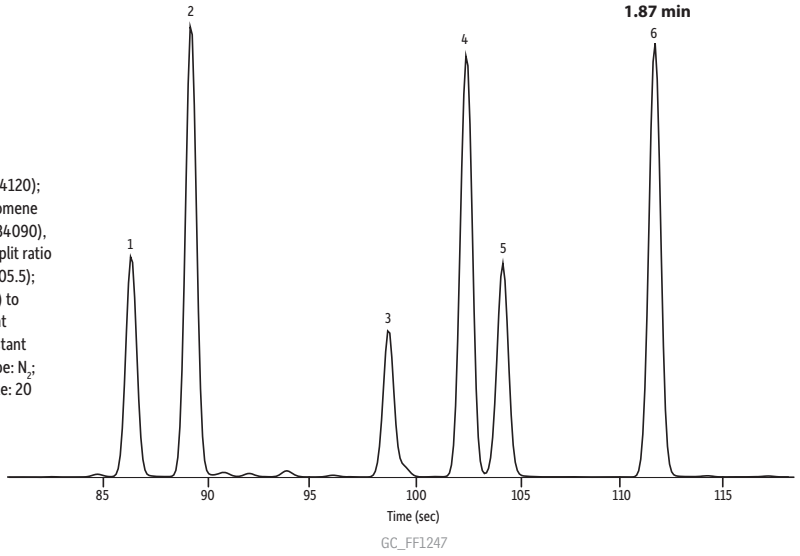


Figure 3: Cannabinoids can be effectively separated on an Rxi® 17Sil MS column, but with slightly less resolution than that obtained with the optimal selectivity of the Rxi®-35Sil MS column.

Peaks
1. Cannabichromene
2. Cannabidiol
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-17Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 14120); **Sample;** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 225 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.9 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 2.5 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC



Adjusting Conditions for 5-Type Columns

While using an Rxi®-35Sil MS column provides the best selectivity and speed for cannabinoid analysis, cannabidiol potency can be determined in medical cannabis using a 5-type column under certain conditions. If you already have a 5-type column for this work, you can vary the GC conditions, especially carrier flow and oven temperature program, and still separate cannabichromene and cannabidiol, just not as quickly or easily as with the Rxi®-35Sil MS column. Figures 4 and 5 show this analysis on Rxi®-5ms and Rxi®-5Sil MS columns, respectively. Again, the 0.25 mm x 0.25 µm format was used here because it offers better efficiency than wider bore columns (e.g., 0.32 mm and 0.53 mm IDs), which may not separate cannabichromene and cannabidiol under any operational conditions.

Figure 4: The selectivity of a 5-type column is not sufficient to fully separate cannabichromene and cannabidiol, resulting in less accurate medical marijuana potency testing.

Peaks
1. Cannabichromene
2. Cannabidiol
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-5ms, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13420); **Sample;** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 250 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.6 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 1.6 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC

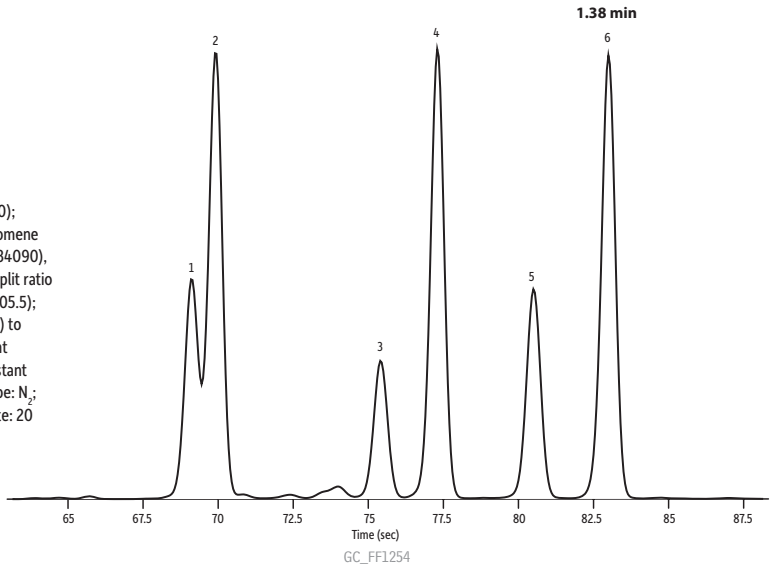
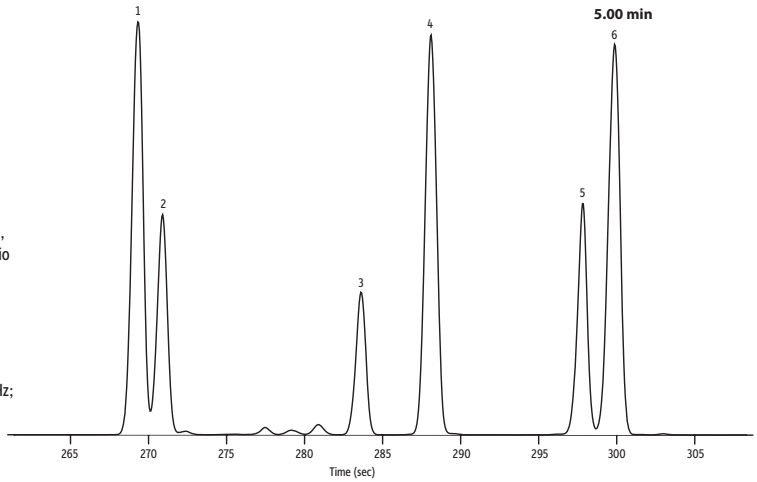


Figure 5: Rxi®-5Sil MS columns offer better resolution of key cannabinoids than standard 5-type columns, but the incomplete separation and longer analysis time mean further optimization is needed for accurate reporting.

Peaks
1. Cannabidiol
2. Cannabichromene
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-5Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13620); **Sample;** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 150 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.7 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 1.6 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC



Note that even though these are both 5-type columns, the elution order of cannabichromene and cannabidiol changed. This is due to two things. The first is that Rxi®-5ms and Rxi®-5Sil MS columns differ slightly in selectivity for certain compounds; even though they are both considered 5-type columns, they contain different stationary phases that retain some compounds differently. The second reason is that the GC oven programs are different for the columns, which means that the compounds are eluting at different temperatures. You may be able to further optimize the separation of cannabichromene and cannabidiol on a 5-type column, but the selectivity and faster analysis that can be obtained using a high-phenyl content Rxi®-35Sil MS column make it ideal for potency determinations in medical cannabis.

To sum things up, proper column choice is essential for accurate and robust cannabis potency testing. Using the right column not only gives you more confidence in your potency values, but it also saves you time and money. Switching to hydrogen carrier gas can reduce your costs even further, while increasing sample throughput.

Visit www.restek.com/medical-cannabis for Restek® GC and LC columns, accessories, reference standards, and other products and resources for medical marijuana analysis.

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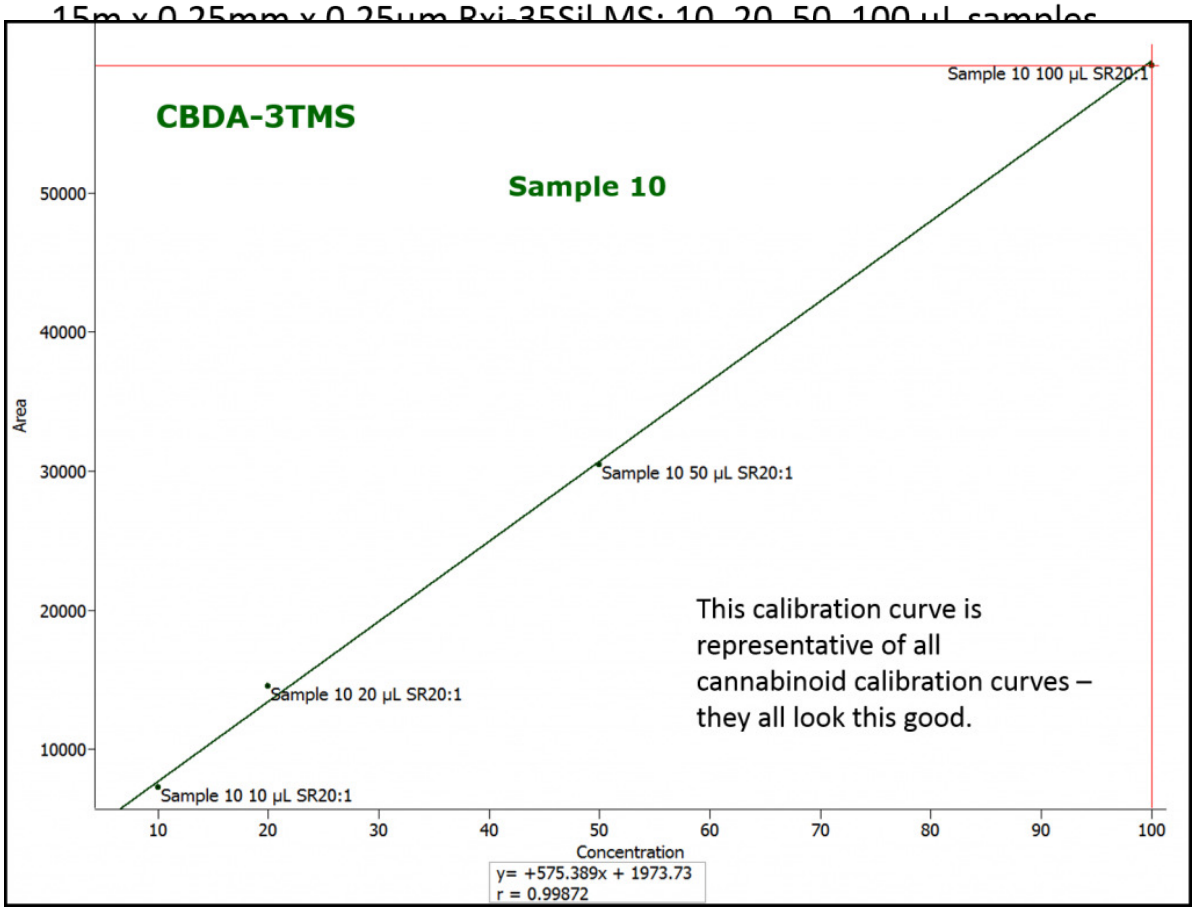
Sample 10 Dilution Derivatization Linearity

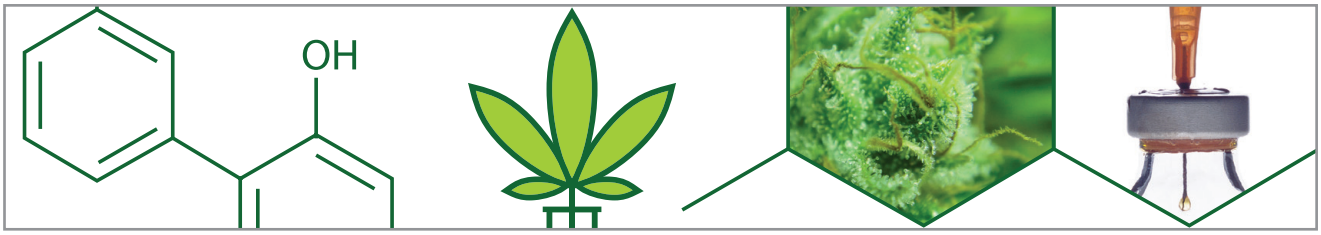
Compound	Mol Wt		t_R sec	t_R min		r
CBDV-2TMS	430		94.6	1.58		0.9994
CBD-2TMS	458		121.0	2.02		0.9995
THCV-1TMS	358		128.2	2.14		0.9991
CBC-1TMS	386		148.2	2.47		0.9989
CBG-2TMS	460		149.9	2.50		0.9993
Δ^8-THC-1TMS	386		156.4	2.61		0.9993
Δ^9-THC-1TMS	386		160.7	2.68		0.9990
CBDA-3TMS	574		164.8	2.75		0.9987
CBN-1TMS	382		178.7	2.98		0.9988
Δ^9-THCA-2TMS	502		212.3	3.54		0.9992

15m x 0.25mm x 0.25um Rxi-35Sil MS; 10, 20, 50, 100 µL samples



In addition to verifying that the derivatization reaction goes to completion in the presence of plant matrix, we also verified the procedure using several different samples which were generated at the same time as the sample shown in the figure above. Our preliminary work is still looking good, which is exciting, but what about all of the other matrices cannabis chemists have to work with? Well, we're planning on moving the work forward into edible matrices next, so stay tuned for an update!

Th… under Tips & Tricks, Medical Marijuana.



Foods, Flavors & Fragrances Applications

A Preliminary FET Headspace GC-FID Method for Comprehensive Terpene Profiling in Cannabis

By Amanda Rigdon, Corby Hilliard, and Jack Cochran

Abstract

This application note describes an FET headspace GC-FID method that was developed in hops for the analysis of terpenes in cannabis. Good chromatographic separation allowed quantification of critical compounds across the volatility range, including α-pinene, β-myrcene, α-humulene, β-caryophyllene, and caryophyllene oxide.

Introduction

In addition to cannabinoids, cannabis contains a suite of compounds known as terpenes. Terpenes are not only responsible for the characteristic aromas of cannabis strains, but they also are suspected to contribute to the therapeutic properties of cannabis. By themselves, terpenes have anti-inflammatory and anti-microbial properties, and they also reportedly contribute to an "entourage effect" with cannabinoids, modulating and/or enhancing their activity [1,2].

Because terpenes may contribute to the therapeutic effects of cannabis, there is a growing demand for analytical methods that profile terpenes in marijuana samples. In addition to analyzing terpenes for therapeutic purposes, terpenes can also be used as differentiators among cannabis strains and terpene profiles can be used for strain identification.

While relatively few terpenes have been studied for therapeutic purposes, cannabis strains can contain dozens of terpenes in varying levels. Of these, the primary compounds of interest include α-pinene, β-myrcene, α-humulene, and β-caryophyllene [2,3]. Accurately profiling these analytes and other emerging terpenes of interest depends heavily on separating them from potentially interfering compounds. When an interfering terpene, or other compound, coelutes with a terpene of interest, quantification will be compromised and, since many terpenes have the same molecular weight and share fragment ions, mass spectrometry cannot be relied upon to distinguish a terpene of interest from a coeluting interference terpene. The only way to accurately identify and quantify terpenes is to ensure that the terpenes of interest are chromatographically separated from all interfering compounds. GC is an excellent technique for accomplishing this.

Here we present a headspace gas chromatography–flame ionization detection (GC-FID) method for a comprehensive set of 38 terpenes found in cannabis. Since cannabis is illegal in Pennsylvania where this work was done, we developed the method using hops as a model system since they are related to cannabis and contain a similar suite of terpenes [2,3,4]. The headspace method presented here utilizes full evaporation technique (FET) sample preparation because cannabis product matrices are extremely varied and plant material will not dissolve in solvent. FET involves the use of a very small sample amount (10–50 mg), which effectively creates a single phase gas system in the headspace vial at equilibrium, making it ideal for this application [5,6,7]. Figure 1 illustrates the basic principle of headspace gas chromatography using FET. To achieve chromatographic separation, a 30 m x 0.25 mm x 1.4 µm Rxi®-624Sil MS column was used. This column was chosen based on several factors. First, and most importantly, the cyano-based stationary phase of the Rxi®-624Sil MS has excellent selectivity for terpenes, making it ideal to effect a good separation for a large suite of these compounds. Second, in addition to its excellent selectivity for terpenes, the maximum temperature of this column is 320 °C, which allows for elution of some of the less volatile terpenes and matrix compounds that may be present in the headspace sample. Third, this GC column phase is also well-suited for residual solvent analysis, potentially minimizing the number of columns and instruments required by labs to test cannabis.

Experimental

Sample Preparation

Pelletized hops from three strains (UK East Kent Golding, Citra, and Cascade) were purchased from HopUnion. The pelletized hops were first ground to a fine powder using an IKA® mill. Because the hops were already ground and pelletized, very little grinding was necessary. For cannabis plant material, it is recommended that samples be frozen prior to grinding or that grinding occur under liquid nitrogen. This keeps the samples cold during the grinding process, reducing loss of the more volatile terpenes such as α-pinene. 10 mg samples of each strain were then placed in headspace vials (Figure 2). An incubation temperature of 140 °C was used to ensure volatilization of all terpenes and terpenoids in the sample. This temperature was chosen because it is also sufficient to melt samples of cannabis concentrates. An incubation time of 30 minutes was used to ensure the establishment of equilibrium during incubation, which is required for reproducible, quantitative results.

Gas Chromatographic Conditions

Samples were analyzed on an Agilent® 6890 gas chromatograph equipped with a Tekmar® HT-3 headspace autosampler. A 30 m x 0.25 mm x 1.4 µm Rxi®-624Sil MS column was installed based on its selectivity for terpenes and because it could also be used for analysis of residual solvents in cannabis concentrates. A 1 mm straight Sky® inlet liner was used to limit the volume in the GC inlet. For headspace instruments, reducing the inlet volume increases efficiency by reducing band broadening during sample introduction. Greater efficiency maximizes peak separation, which is essential for this analysis. Complete chromatographic conditions are presented in Figure 4.

Figure 1: Setup and Basic Principle of FET Headspace Injection Coupled With GC-FID Analysis

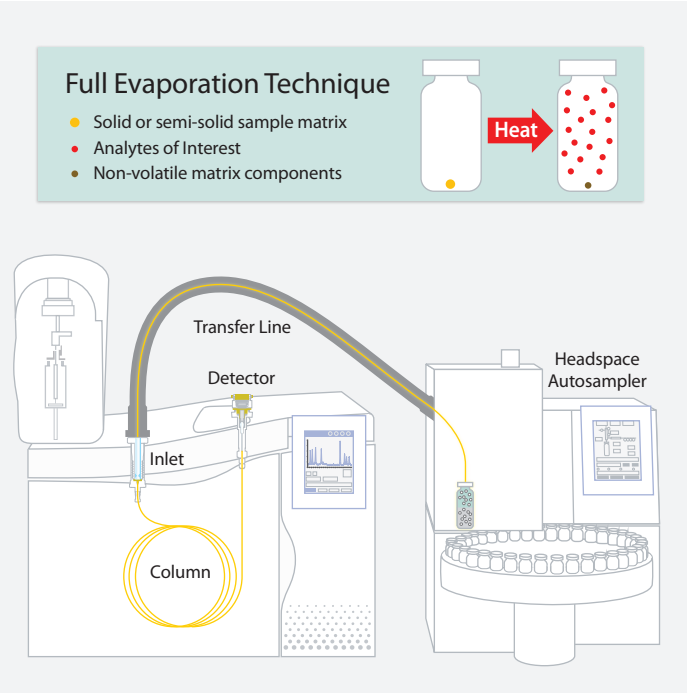


Figure 2: Grinding samples maximizes and normalizes surface area from sample to sample, increasing sensitivity and reproducibility.

Quantification

To aid in peak identification, a multi-component terpene standard was prepared with each compound at approximately 0.02% wt/vol. 10 µL of this standard solution was injected into a capped headspace vial and analyzed by FET headspace GC-FID. Standards were analyzed under the same conditions as the samples in order to eliminate the potential for discrimination across the volatility range (e.g., more volatile terpenes may show higher responses than less volatile terpenes). Since any discrimination effect would be the same in both the sample and standard, analytes were quantified based on their relative response factor compared to the standard as shown in Equation 1. This normalizes the values between sample and standard, ensuring accurate quantification across the full range of volatility for terpenes. Note that while the relative response factor technique improves accuracy, the semi-quantitative preparation of the standard and lack of well-characterized certified reference materials for terpenes limits the overall quantitative accuracy that can be obtained for this analysis. Additionally, the lack of pure, neat standards available to prepare a more concentrated standard resulted in a standard well below the level of many of the terpenes detected in this work. For accurate quantification, a calibration curve encompassing the expected concentration range of all analytes is required. The data presented in this article should be considered semi-quantitative.

Equation 1: Sample Concentration Calculation

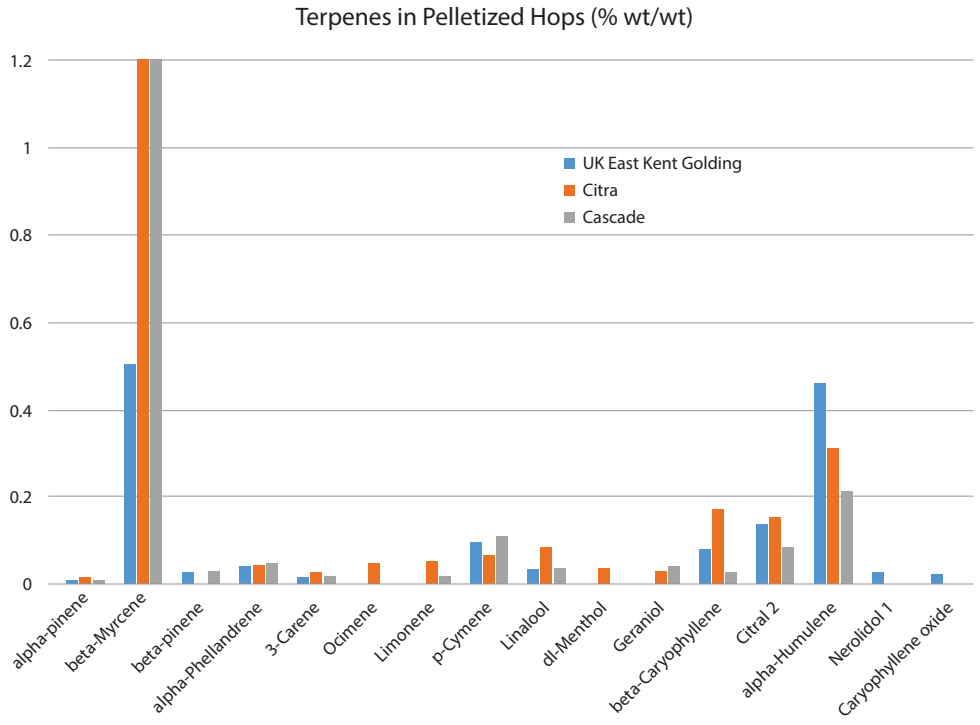
$$\text{Given: } \frac{\text{Standard Area}}{\text{Standard Concentration}} = \frac{\text{Sample Area}}{\text{Sample Concentration}}$$

$$\text{Sample Concentration} = \frac{(\text{Sample Area} \times \text{Standard Concentration})}{\text{Standard Area}}$$

Results and Discussion

The purpose of this study was to develop an FET headspace GC-FID method for the analysis of terpenes in cannabis using hops as a model system. The terpenes found in our samples matched well with literature descriptions of the terpenes present in hops [4]. High levels of terpenes were found across the volatility range, indicating that the FET headspace GC-FID technique was appropriate and that analysis of the standard adequately normalized any discrimination between the more and less volatile terpenes (Figure 3). Due to the starting concentration of some of the commercially available terpene standards, the maximum concentration at which the mixed terpene standard used for quantification could be prepared was 0.02% wt/vol, which is significantly lower than the concentration of some of the more prevalent terpenes in hops and cannabis. The use of a more concentrated standard solution is recommended to improve quantification of the higher concentrations found in these samples.

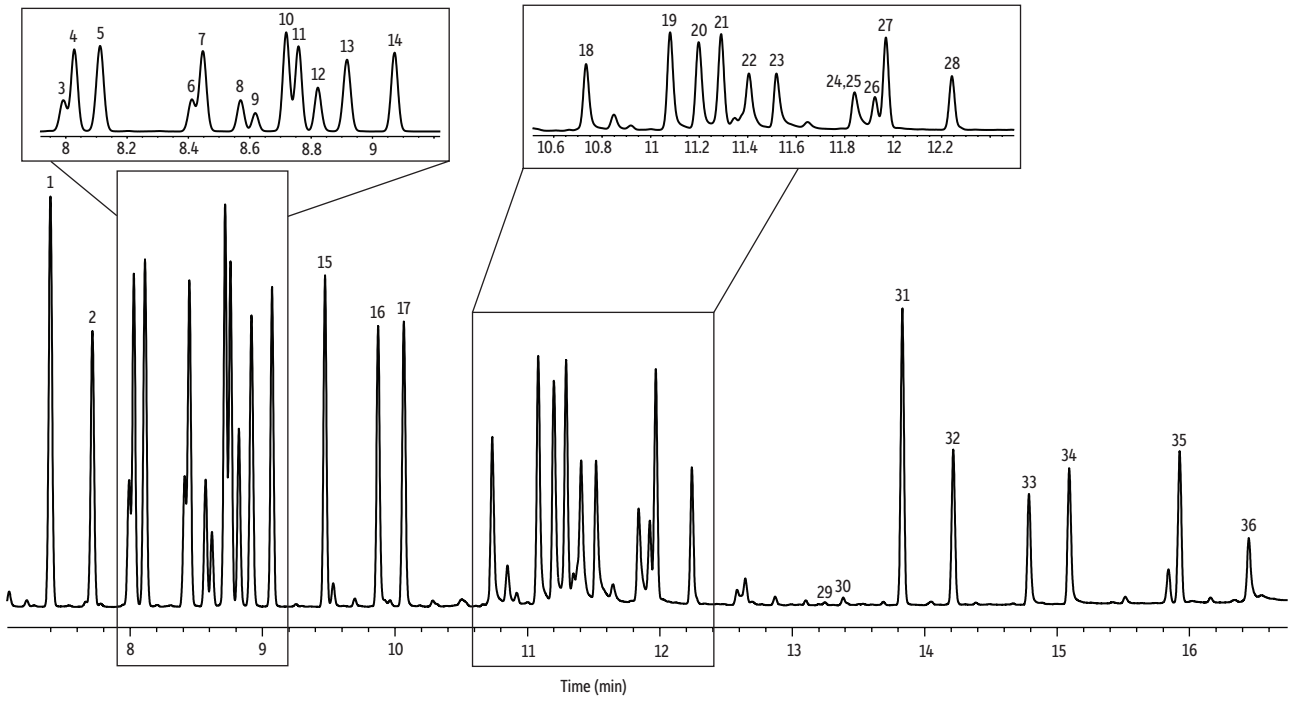
Figure 3: Terpene Profiles of Pelletized Hops



Figures 4–7 show individual chromatograms for the standard and each sample profiled for terpenes. Note that α-pinene, β-myrcene, α-humulene, β-caryophyllene, and caryophyllene oxide are well separated from interferences. For complex matrices, such as hops and marijuana, excellent chromatographic efficiency and selectivity are required to separate terpenes from one another and from other volatile matrix components in order to obtain accurate quantification. The selectivity of the Rxi®-624Sil MS column used here provided good separation of most terpenes and the small bore configuration (0.25 mm internal diameter) improved column efficiency, ultimately resulting in greater resolution between closely eluting terpenes than would be obtained using a wider bore column.

Figure 4: A 0.02% wt/vol multi-component terpenes standard analyzed on an Rxi®-624Sil MS column (30 m x 0.25 mm x 1.4 µm) demonstrates that this column provides the selectivity and efficiency needed to separate key terpenes using a simple FET headspace GC-FID method.

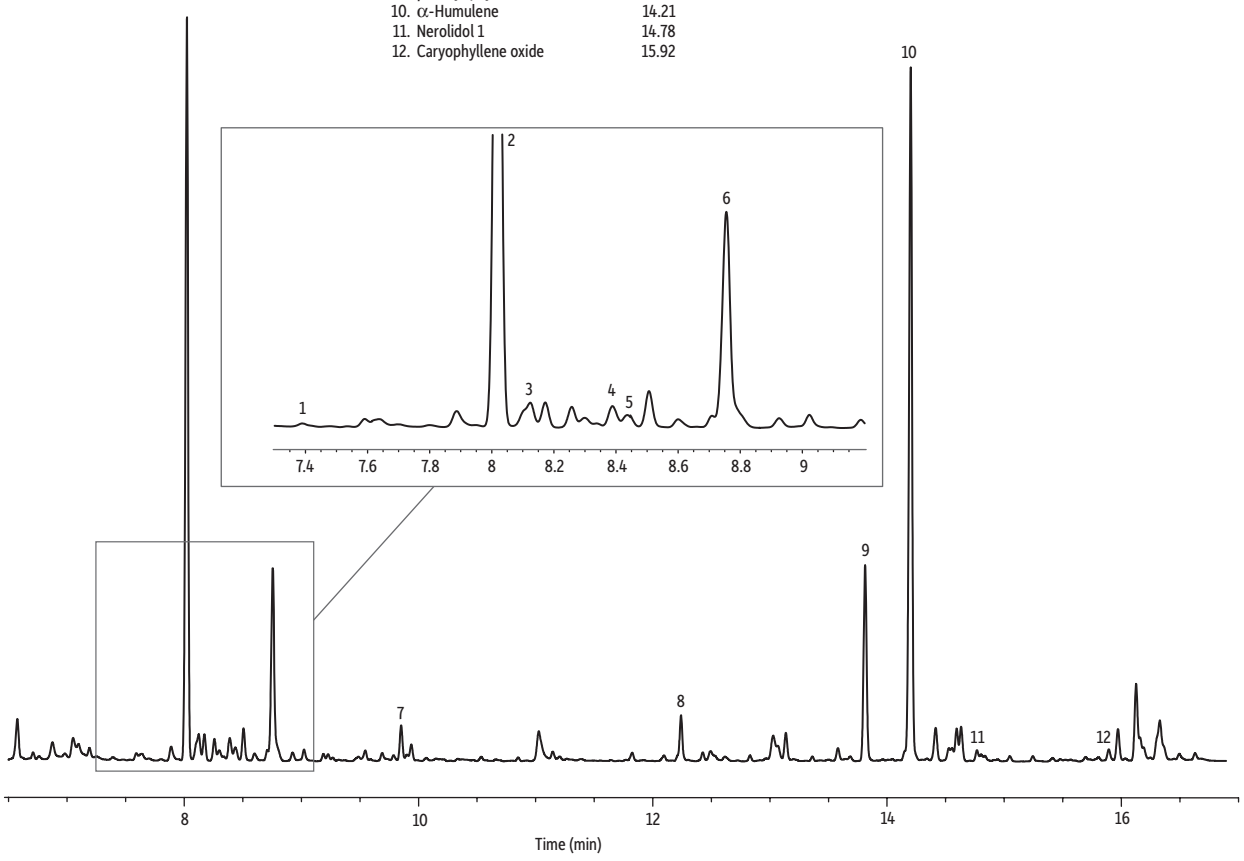
Peaks	t_R (min)	Peaks	t_R (min)	Peaks	t_R (min)	Peaks	t_R (min)
1. α-Pinene	7.39	10. Limonene	8.71	19. dl-Menthol	11.08	28. Citral 2	12.24
2. Camphene	7.71	11. *p*-Cymene	8.75	20. Borneol	11.19	29. Citral 3	13.19
3. β-Myrcene	7.98	12. β-Ocimene	8.82	21. α-Terpineol	11.29	30. Citral 4	13.43
4. Sabinene	8.02	13. Eucalyptol	8.91	22. Dihydrocarveol	11.40	31. β-caryophyllene	13.83
5. β-Pinene	8.11	14. γ-Terpinene	9.06	23. Citronellol	11.51	32. α-Humulene	14.21
6. α-Phellandrene	8.4	15. Terpinolene	9.47	24. Geraniol	11.82	33. Nerolidol 1	14.78
7. δ 3-Carene	8.44	16. Linalool	9.87	25. 2-Piperidinone	11.88	34. Nerolidol 2	15.08
8. α-Terpinene	8.57	17. Fenchone	10.06	26. Citral 1	11.92	35. Caryophyllene oxide	15.92
9. Ocimene	8.61	18. Isopulegol	10.73	27. Pulegone	11.97	36. α-Bisabolol	16.43



Column	Rxi® -624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868)
Sample	Terpenes mix
Diluent:	Isopropyl alcohol
Conc.:	200 ng/µL (0.02% wt/vol). The sample was prepared by placing 10 µL into the headspace vial.
Injection	headspace-loop split (split ratio 10:1)
Liner:	Sky® 1.0 mm ID straight inlet liner (cat.# 23333.1)
Headspace-Loop	
Inj. Port Temp.:	250 °C
Instrument:	Tekmar HT-3
Inj. Time:	1.0 min
Transfer Line Temp.:	160 °C
Valve Oven Temp.:	160 °C
Needle Temp.:	140 °C
Sample Temp.:	140 °C
Sample Equil. Time:	30.0 min
Vial Pressure:	20 psi
Loop Pressure:	15 psi
Oven	
Oven Temp.:	60 °C (hold 0.10 min) to 300 °C at 12.50 °C/min (hold 3.0 min)
Carrier Gas	He, constant flow
Linear Velocity:	33 cm/sec
Detector	FID @ 320 °C
Make-up Gas Flow Rate:	45 mL/min
Make-up Gas Type:	N_2
Hydrogen flow:	40 mL/min
Air flow:	450 mL/min
Data Rate:	20 Hz
Instrument	Agilent/HP6890 GC

Figure 5: Chromatographic Terpene Profile of Pelletized UK East Kent Golding Hops

Peaks	t_R (min)
1. α-Pinene	7.39
2. β-Myrcene	8.02
3. β-Pinene	8.11
4. α-Phellandrene	8.40
5. δ 3-Carene	8.44
6. *p*-Cymene	8.75
7. Linalool	9.87
8. Citral-2	12.24
9. β-Caryophyllene	13.83
10. α-Humulene	14.21
11. Nerolidol 1	14.78
12. Caryophyllene oxide	15.92



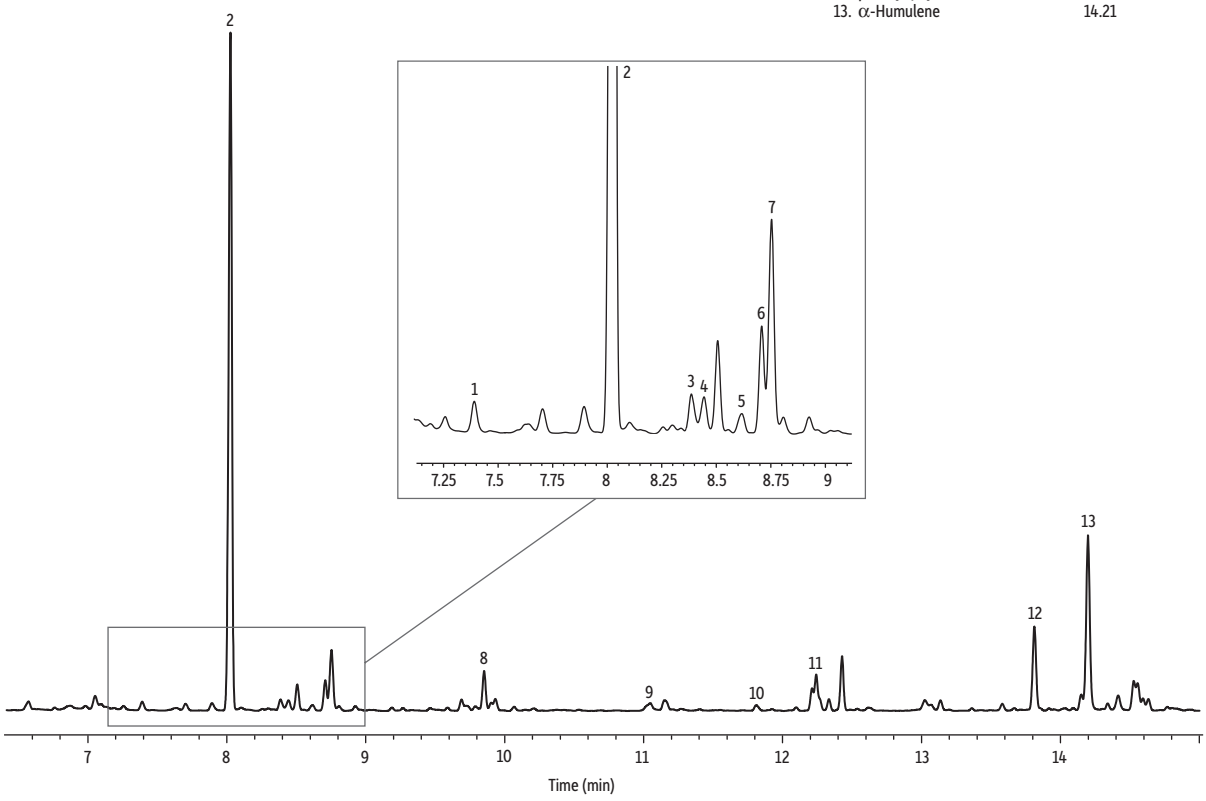
GC_FS0522

Column	Rxi®-624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868)
Sample	
Conc.:	10 mg of ground UK East Kent Goldings hops
Injection	headspace-loop split (split ratio 10:1)
Liner:	Sky® 1.0 mm ID straight inlet liner (cat.# 23333.1)
Headspace-Loop	
Inj. Port Temp.:	250 °C
Instrument:	Tekmar HT-3
Inj. Time:	1.0 min
Transfer Line Temp.:	160 °C
Valve Oven Temp.:	160 °C
Needle Temp.:	140 °C
Sample Temp.:	140 °C
Sample Equil. Time:	30.0 min
Vial Pressure:	20 psi
Loop Pressure:	15 psi

Oven	
Oven Temp.:	60 °C (hold 0.10 min) to 300 °C at 12.50 °C/min (hold 3.0 min)
Carrier Gas	He, constant flow
Linear Velocity:	33 cm/sec
Detector	FID @ 320 °C
Make-up Gas Flow Rate:	45 mL/min
Make-up Gas Type:	N_2
Hydrogen flow:	40 mL/min
Air flow:	450 mL/min
Data Rate:	20 Hz
Instrument	Agilent/HP6890 GC

Figure 6: Chromatographic Terpene Profile of Pelletized Citra Hops

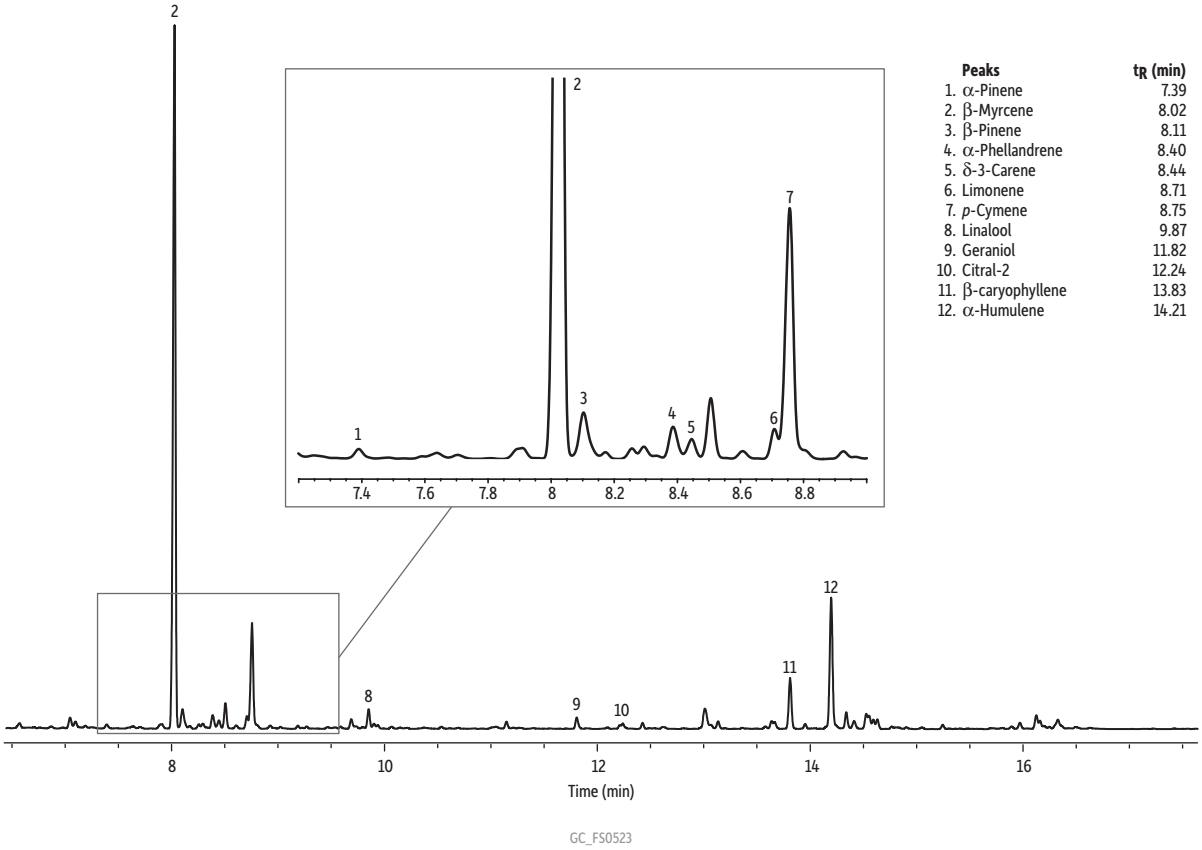
Peaks	t_R (min)
1. α-Pinene	7.39
2. β-Myrcene	8.02
3. α-Phellandrene	8.40
4. δ 3-Carene	8.44
5. Ocimene	8.61
6. Limonene	8.71
7. *p*-Cymene	8.75
8. Linalool	9.87
9. dl-Menthol	11.08
10. Geraniol	11.82
11. Citral-2	12.24
12. β-Caryophyllene	13.83
13. α-Humulene	14.21



GC_FS0525

Column	Rxi®-624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868)
Sample	
Conc.:	10 mg of ground Citra hops
Injection	headspace-loop split (split ratio 10:1)
Liner:	Sky® 1.0 mm ID straight inlet liner (cat.# 23333.1)
Headspace-Loop	
Inj. Port Temp.:	250 °C
Instrument:	Tekmar HT-3
Inj. Time:	1.0 min
Transfer Line Temp.:	160 °C
Valve Oven Temp.:	160 °C
Needle Temp.:	140 °C
Standby flow rate:	50 mL/min
Sample Temp.:	140 °C
Platen temp equil. time:	1.0 min
Sample Equil. Time:	30.0 min
Vial Pressure:	20 psi
Pressurize Time:	5.0 min
Pressure Equilibration Time:	0.20 min
Loop Pressure:	15 psi
Loop Fill Time:	2.0 min
Oven	
Oven Temp.:	60 °C (hold 0.10 min) to 300 °C at 12.50 °C/min (hold 3.0 min)
Carrier Gas	He, constant flow
Flow Rate:	1.4 mL/min
Linear Velocity:	33 cm/sec
Detector	FID @ 320 °C
Make-up Gas Flow Rate:	45 mL/min
Make-up Gas Type:	N_2
Hydrogen flow:	40 mL/min
Air flow:	450 mL/min
Data Rate:	20 Hz
Instrument	Agilent/HP6890 GC

Figure 7: Chromatographic Terpene Profile of Pelletized Cascade Hops



Peaks	t_R (min)
1. α-Pinene	7.39
2. β-Myrcene	8.02
3. β-Pinene	8.11
4. α-Phellandrene	8.40
5. δ-3-Carene	8.44
6. Limonene	8.71
7. *p*-Cymene	8.75
8. Linalool	9.87
9. Geraniol	11.82
10. Citral-2	12.24
11. β-caryophyllene	13.83
12. α-Humulene	14.21

Column	Rxi®-624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868)
Sample	
Conc.:	10 mg of ground Cascade hops
Injection	headspace-loop split (split ratio 10:1)
Liner:	Sky® 1.0 mm ID straight inlet liner (cat.# 23333.1)
Headspace-Loop	
Inj. Port	
Temp.:	250 °C
Instrument:	Tekmar HT-3
Inj. Time:	1.0 min
Transfer Line	
Temp.:	160 °C
Valve Oven	
Temp.:	160 °C
Needle Temp.:	140 °C
Sample Temp.:	140 °C
Sample Equil.	
Time:	30.0 min
Vial Pressure:	20 psi
Loop Pressure:	15 psi

Oven	
Oven Temp.:	60 °C (hold 0.10 min) to 300 °C at 12.50 °C/min (hold 3.0 min)
Carrier Gas	He, constant flow
Linear Velocity:	33 cm/sec
Detector	FID @ 320 °C
Make-up Gas	
Flow Rate:	45 mL/min
Make-up Gas	
Type:	N_2
Hydrogen flow:	40 mL/min
Air flow:	450 mL/min
Data Rate:	20 Hz
Instrument	Agilent/HP6890 GC

While many cyano-based columns are commercially available, the Rxi®-624Sil MS column is recommended for terpene analysis because, in addition to offering optimized selectivity, the stationary phase is stabilized with silarylene, which significantly increases the operational temperature range of the column and improves its robustness. This is important for terpene analysis because some of the less-volatile terpenes require relatively high elution temperatures that would tax non-silarylene cyano stationary phases, resulting in shorter column lifetimes.

Although the Rxi®-624Sil MS column performs exceptionally well for the analysis of terpenes and residual solvents, it is too retentive for cannabinoids. In fact, cannabinoids do not elute from the Rxi®-624Sil MS column even at its 320 °C maximum

temperature. Injection of cannabinoids on this column can potentially result in reduced column lifetime, selectivity changes, or baseline disturbances due to cannabinoids "bleeding" off of the stationary phase over time. Since both cannabinoids and terpenes will be present in cannabis samples, the sample preparation method must minimize the introduction of cannabinoids onto the analytical column. The full evaporation technique headspace sampling approach used here is ideal for terpene profiling because it introduces the volatile terpenes onto the GC column while eliminating the introduction of less volatile cannabinoids and nonvolatile matrix components into the system. This results in longer column lifetime and reduced inlet maintenance. Headspace sampling in general is simple to perform and requires no extraction or cleanup. While other methods exist that could remove cannabinoids from the sample while leaving the terpenes behind, these sample preparation methods are more time- and labor-intensive, and the increased amount of sample handling could result in loss of some of the more volatile terpenes, such as α-pinene. Grinding samples under dry ice is an additional measure that could be taken to minimize the loss of more volatile terpenes as it reduces the heat generated during the grinding process.

Conclusion

An FET headspace GC-FID method was used to analyze a comprehensive suite of terpenes in hops that are also found in cannabis samples. Compounds of interest across the volatility range were chromatographically separated and quantified. This method utilizes straightforward FET sample preparation, which minimizes manual labor and sample handling time. In addition, because it prevents nonvolatile material from entering the GC system, using the FET approach can increase column lifetime and reduce inlet maintenance. This technique, column, and instrument setup can also be used to analyze residual solvents in cannabis concentrates, eliminating the need for additional capital investment for different instrumentation and/or columns.

References

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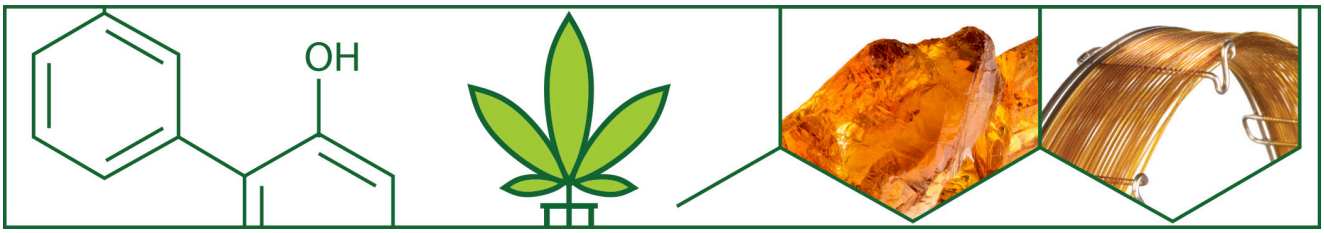


Foods, Flavors & Fragrances Applications

A Fast, Simple FET Headspace GC-FID Technique for Determining Residual Solvents in Cannabis Concentrates

By Corby Hilliard; Amanda Rigdon; William Schroeder*, Ph.D.; Christi Schroeder*, Ph.D.; and Theo Flood*

*Cal-Green Solutions

Abstract

Due to rapid growth in the medical cannabis industry, demand is increasing for analysis of residual solvents in cannabis concentrates in order to protect consumer safety. This application note details a simple, fast test for common residual solvents using full evaporation technique headspace GC-FID and an Rxi®-624Sil MS column.

Introduction

As the popularity of cannabis concentrates increases, consumer safety concerns are resulting in the establishment of new regulations to control the level of residual solvents in commercial cannabis concentrates. The State of Colorado, for example, published allowable concentrations of certain residual solvents in Rule R 712. This is because, although cannabis concentrates can be produced in numerous ways, one of the most common means of extracting therapeutic compounds, like tetrahydrocannabinol (THC), cannabidiol (CBD), and terpenes, from cannabis is through extraction with an organic solvent, such as butane. After the cannabinoids and terpenes are extracted from the plant material, the organic solvent is allowed to evaporate and then is purged off using heat and/or vacuum. These extraction solvents can be difficult to purge completely, so the finished product needs to be tested to ensure that residual solvents are only present at or below safe levels. For consumer safety, especially with medicinal products, accurate and comprehensive analysis of residual solvents is necessary for concentrates and extracts.

Since residual solvents are extremely volatile, they cannot be analyzed by HPLC and lend themselves nicely to GC analysis. One of the most common and reliable ways to quantify residual solvents is through headspace gas chromatography–flame ionization detection (GC-FID). Headspace injection works by driving volatile compounds of interest from the sample into a gas phase in the headspace of the vial above the sample. An aliquot is then withdrawn from the headspace of the vial and analyzed by GC-FID in order to determine the volatile components of the sample. One approach for headspace GC-FID that is particularly useful for analyzing cannabis concentrates is the full evaporation technique (FET). FET sample preparation involves the use of a very small sample amount (e.g., 20–50 mg), which effectively creates a single-phase gas system in the headspace vial at equilibrium [1]. FET is ideal for difficult and varied matrices like cannabis concentrates because it eliminates matrix interferences that can cause inaccurate quantification, and it also has the advantages of little to no manual sample handling and a very small sample size. Additionally, high sensitivity can be achieved through the creation of a single-phase system in the headspace vial. Figure 1 illustrates the basic principle of headspace GC using the full evaporation technique.

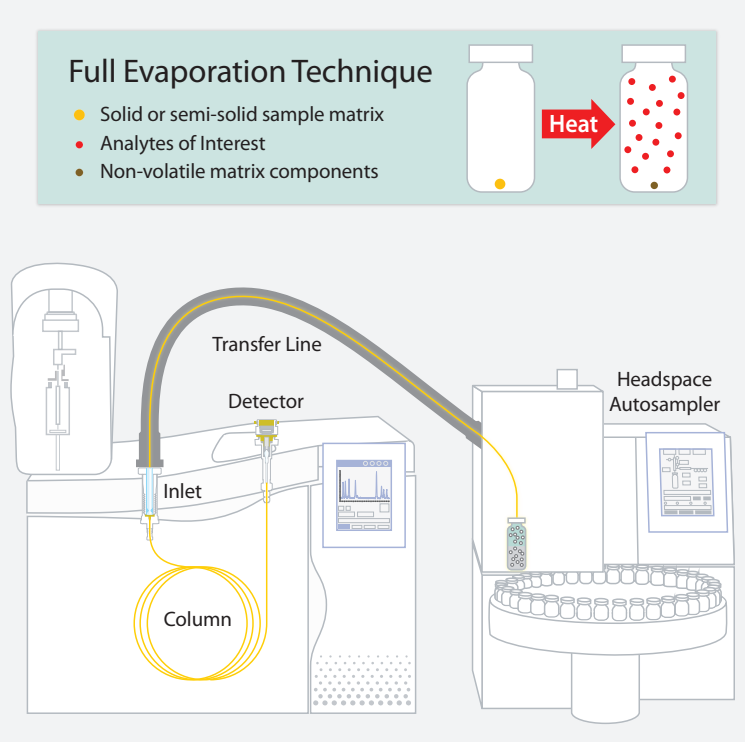
The work described here demonstrates the viability of FET headspace injection and GC-FID analysis of residual solvents in cannabis concentrates. The method is simple to implement, quick to run, and does not require expensive dynamic headspace equipment or mass spectrometric detectors. While the methodology presented here is suitable for residual solvents in cannabis concentrates, it is not applicable for finished tinctures in alcohol. Finished alcohol tinctures contain large amounts of alcohol which will severely interfere with quantification of other residual solvents in the sample. Therefore, an alternate approach is required for alcohol tinctures. This technique also may be applicable for oil or glycerin tinctures; however, it has not been evaluated for that use.

Experimental

Headspace and GC Method Optimization

An Rxi®-624Sil MS column was selected for this work as it is designed specifically for volatiles analysis and is widely used for the analysis of residual solvents in pharmaceutical products. Final FET headspace injector and GC-FID operating conditions are presented in Figure 3. Initially, modeled conditions for analyzing the specific compounds of interest were generated using Restek's *EZ*GC™ chromatogram modeler. The method from the modeler was then optimized to account for headspace analysis employing a headspace instrument with a transfer line.

Figure 1: Setup and Basic Principle of FET Headspace Injection Coupled With GC-FID Analysis



The following parameters were optimized for this method:

- *Linear velocity:* Linear velocity was increased to 80 cm/sec to allow for fast sample transfer through the headspace instrument transfer line. Fast sample transfer minimizes band broadening, which maximizes efficiency, resolution, and sensitivity. The original GC oven program generated by the *EZ*GC™ chromatogram modeler was translated using the *EZ*GC™ method translator to give a new oven program optimized for the new carrier flow. Method translation is required when changing flow rates in order to keep elution temperatures constant. Changes in elution temperatures between the original and the translated method will sometimes result in drastically different separations or even coelutions, especially on highly selective phases like the Rxi®-624Sil MS column.

- *GC inlet liner choice:* The liner used for this work was a 1 mm straight Sky® inlet liner (cat.# 23333.1). The use of a small internal diameter liner minimizes band broadening by reducing the overall volume of the inlet, again resulting in higher efficiency, resolution, and sensitivity.

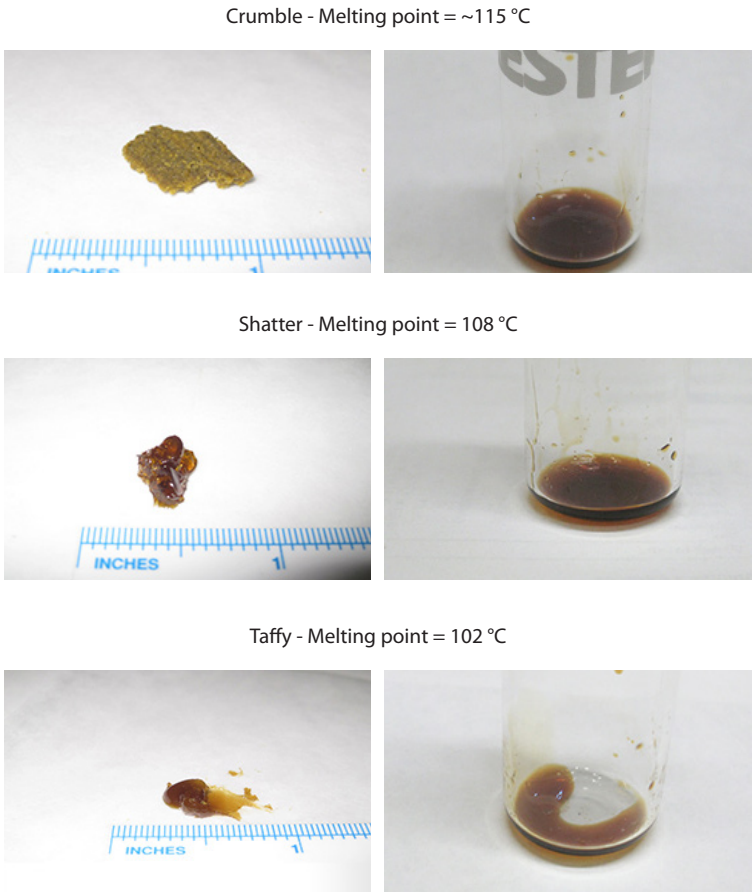
- *Split ratio:* A split ratio of 10:1 was used for this work. Although maximum sensitivity is required due to very low expected levels of target analytes, using a split ratio of at least 10:1 ensures high sample velocity through the GC inlet, which minimizes band broadening, increasing resolution without compromising sensitivity. Sharper peaks are taller peaks, so any loss in sensitivity is mitigated through an increase in signal-to-noise ratio.

- *Equilibration temperature:* Samples were equilibrated at 140 °C to encourage complete melting of waxy concentrates. By melting the extracts, the ratio of surface area to volume is maximized, ensuring 100% transfer of the analytes of interest into the headspace. The use of a larger sample size will compromise this ratio; therefore, sample sizes should be kept as small as possible to ensure accurate quantification (20 mg is recommended for this application). Representative concentrates are shown in Figure 2. Small samples (20–25 mg) of each concentrate type were placed in a capped headspace vial and incubated for 30 minutes at 140 °C. All concentrates melted completely at the 140 °C incubation temperature, forming a thin film at the bottom of the headspace vial.

- *Equilibration time:* The equilibration time for this method was 30 minutes. This allows enough time for waxy concentrates to melt completely and ensures equilibrium is reached in the headspace vial. Equilibrium is required for accurate and reproducible quantification.

- *Oven program:* The oven program was optimized for speed for this application. In samples that contain terpenes, it is recommended that the oven ramp be extended to 320 °C and the isothermal hold time be extended to 5 minutes in order to ensure complete elution of any terpenes that may be present in the sample.

Figure 2: Cannabis concentrate samples are solid before FET incubation (left) and then melt completely into a thin liquid layer after a 30-minute incubation at 140 °C (right).



Photos and melting point data courtesy Cal-Green Solutions

Table I: Commodity and Calibration Standard Curve Equivalency Levels

Concentration in Commodity (ppm)	Amount in 20 mg Sample (µg)	Concentration in 10 µL Standard (µg/mL)
500	10	1,000
250	5	500
100	2	200
50	1	100
25	0.5	50
10	0.2	20
5	0.1	10

Calibration Curve Preparation

When preparing standards for FET headspace GC-FID, it is necessary to calculate the total mass of analyte that will be present in a representative sample, since the equilibrium state results in a single-phase system. For example, a 20 mg sample containing a residual solvent at 50 ppm contains 1 µg of that residual solvent. Therefore, the 50 ppm point in the calibration curve should contain 1 µg of each compound of interest. Since FET headspace GC-FID depends on the establishment of a single phase system, very small volumes are required for standards. The volume used for standards in this application was 10 µL, which was placed directly into a capped headspace vial by injecting it through the vial septum with a clean syringe. Table I presents the 7-point calibration curve standards and their corresponding concentrations in commodity samples.

Standards were prepared in dimethyl sulfoxide (DMSO), which is a less-volatile, later-eluting solvent that does not interfere with the residual solvents of interest. Because FET establishes a single-phase system in the headspace vial without partitioning, it is not necessary to matrix-match standards and samples, which simplifies standard preparation for varied matrices.

The calibration curve was prepared by first making a 1,000 µg/mL stock solution for dilution. The stock solution was prepared as follows:

- Prepare a 5,000 µg/mL stock solution of butane by bubbling butane standard through DMSO on a balance in a fume hood. The butane used for this work was a mixture of butane and isobutane.
- Prepare a 1,000 µg/mL stock solution by adding 2 mL of 5,000 µg/mL butane stock to a 10 mL volumetric flask, adding ~4 mL DMSO, and then volumetrically adding each neat solvent to the flask using a syringe. Volumes required for the 1,000 µg/mL stock standard were adjusted to account for the density of each solvent as shown in Table II.
- After the addition of neat solvents, fill the flask to the line with DMSO and mix by gently inverting the flask three times and rotating to swirl the contents between inversions.

Table II: Density-Adjusted Volumes Used to Prepare 10 mL of the 1,000 µg/mL Stock Solution

Compound	Density (g/mL)	Volume Required (µL)
Butane	measured gravimetrically	2,000
Chloroform	1.48	6.7
Isobutane	NA	2,000
Acetone	0.79	12.6
Methanol	0.79	12.6
Ethanol	0.79	12.7
IPA	0.79	12.7
Benzene	0.88	11.4
Toluene	0.87	11.5
Pentane	0.63	16.0
Hexane	0.65	15.3
Heptane	0.68	14.7

The 1,000 µg/mL stock solution prepared using Table II was used as the highest calibration standard. All other calibration points were prepared in 5 mL volumetric flasks with separate dilutions of the 1,000 µg/mL stock solution. Serial dilution was not used for this work in order to minimize time-consuming syringe rinsing during calibration curve preparation. Because the compounds used here are volatile, work needed to be completed as quickly as possible to prepare the calibration standards. In addition, volumetric flasks were kept capped to minimize evaporative loss. Table III details the preparation of the calibration curve standards.

Table III: Calibration Curve Preparation

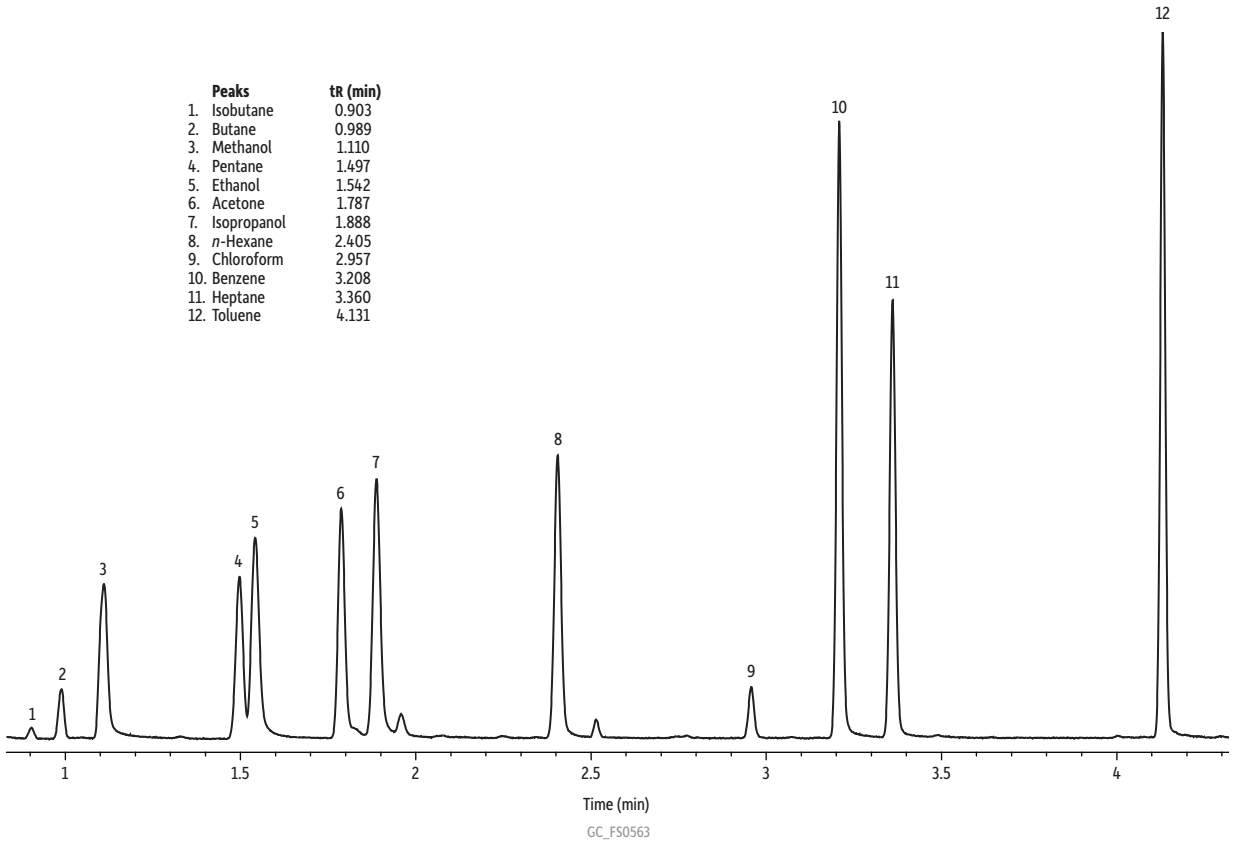
Calibration Level (ppm in Commodity)	Volume of 1,000 µg/mL Stock Solution (mL)	Final Volume (mL)	Final Calibration Standard Concentration (µg/mL)
500	5	5	1,000
250	2.5	5	500
100	1	5	200
50	0.5	5	100
25	0.25	5	50
10	0.1	5	20
5	0.05	5	10

After preparation, all calibration standards were divided into 2.5 mL aliquots and stored in the refrigerator at 5 °C. Since DMSO freezes under refrigeration, calibration standards were allowed to thaw completely prior to use. By aliquoting the calibration standards into separate vials, freeze/thaw cycles were reduced for the entire volume of the calibration solution, allowing for longer storage life of calibration and stock solutions. If desired, calibration standards may be split into aliquots smaller than 2.5 mL to further reduce freeze/thaw cycles. This can be accomplished by pipetting aliquots into gas-tight vials using a glass pipet and immediately capping the vials.

Results and Discussion

Good chromatographic peak shape, separation, and sensitivity were achieved for all analytes of interest. Figure 3 shows the 25 ppm calibration standard. Use of the Restek® Rxi®-624Sil MS column allowed for the separation of the wide variety of solvents that may be present in cannabis concentrates in a short analysis time, while retaining and resolving highly volatile butane isomers. This column was selected for the FET headspace GC-FID method because it was designed specifically for volatiles analysis and is widely used for the analysis of residual solvents in pharmaceutical products. Additionally, the column's unique selectivity also resolves dozens of terpenes [2]. This allows cannabis terpene profiling to be done without changing columns or injection technique, which decreases downtime between methods and improves lab productivity.

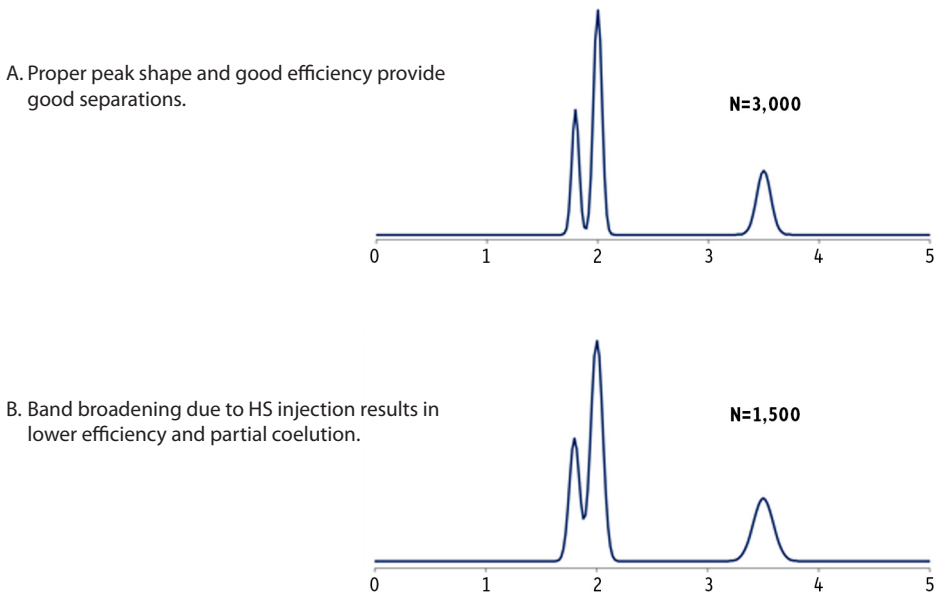
Figure 3: Calibration standard corresponding to a 20 mg cannabis concentrate sample containing 25 ppm of residual solvents. Good chromatographic separation and sensitivity were achieved for common residual solvents.



Column	Rxi®-624Sil MS, 30 m, 0.25 mm ID, 1.40 µm (cat.# 13868)
Sample	Residual solvent mix
Diluent:	Dimethyl sulfoxide (DMSO)
Conc.:	25 ppm (For the HS-FET technique, 10 µL of a 50 µg/mL standard was placed into a 20 mL headspace vial to represent a 25 ppm sample concentration, assuming a 20 mg sample weight.)
Injection	headspace-loop split (split ratio 10:1)
Liner:	Sky® 1.0 mm ID straight inlet liner (cat.# 23333.1)
Headspace-Loop	
Inj. Port Temp.:	250 °C
Instrument:	Tekmar HT3
Inj. Time:	1.0 min
Transfer Line Temp.:	160 °C
Valve Oven Temp.:	160 °C
Needle Temp.:	140 °C
Sample Temp.:	140 °C
Platen temp equil. time:	1.0 min
Sample Equil. Time:	30.0 min
Vial Pressure:	20 psi
Pressurize Time:	5.0 min
Loop Pressure:	15 psi
Loop Fill Time:	2.0 min
Oven	
Oven Temp.:	35 °C (hold 1.5 min) to 300 °C at 30 °C/min (hold 2.0 min)
Carrier Gas	He, constant flow
Linear Velocity:	80 cm/sec
Detector	FID @ 320 °C
Make-up Gas Flow Rate:	45 mL/min
Make-up Gas Type:	N_2
Hydrogen flow:	40 mL/min
Air flow:	450 mL/min
Data Rate:	20 Hz
Instrument	Agilent/HP6890 GC
Notes	The butane used for standard preparation was a mixture of butane and isobutane in an unknown ratio. The concentrations should be considered approximate, but do not exceed 50 ppm for any component.

In addition to using a highly efficient, selective Rxi®-624Sil MS column, it is critical to optimize several GC parameters for headspace analyses in order to prevent band broadening. Early-eluting compounds such as isobutane and butane do not focus on the head of the analytical column, so band broadening through the headspace system and injection port can reduce efficiency, severely impacting sensitivity and resolution for these compounds (Figure 4). As detailed in the Experimental section, band broadening was controlled by using a fast linear velocity, narrow bore inlet liner, and a 10:1 split ratio. This approach speeds up sample transfer and ensures good chromatographic peak shape and response.

Figure 4: Lower efficiency (N) due to band broadening during headspace sample introduction can reduce both resolution and sensitivity (modeled chromatogram).



Analysis of calibration standards resulted in good sensitivity and linear responses for all analytes of interest. Table IV shows the signal-to-noise ratios at 10 ppm and 50 ppm (current Colorado regulatory cutoff values), as well as the correlation coefficients (r values) and coefficients of determination (r^2 values) for all analytes. All compounds exhibited adequate signal-to-noise ratios (> 10:1) at their respective Colorado state regulatory limits. Signal-to-noise ratios were > 10:1 for all compounds at 10 ppm, with the exception of isobutane. The Colorado cutoff for isobutane was 50 ppm at the time of this study; however, prior to publication, Colorado changed the limits and solvents of interest for residual solvent testing. This method will be suitable for the new regulations as well as the older ones.

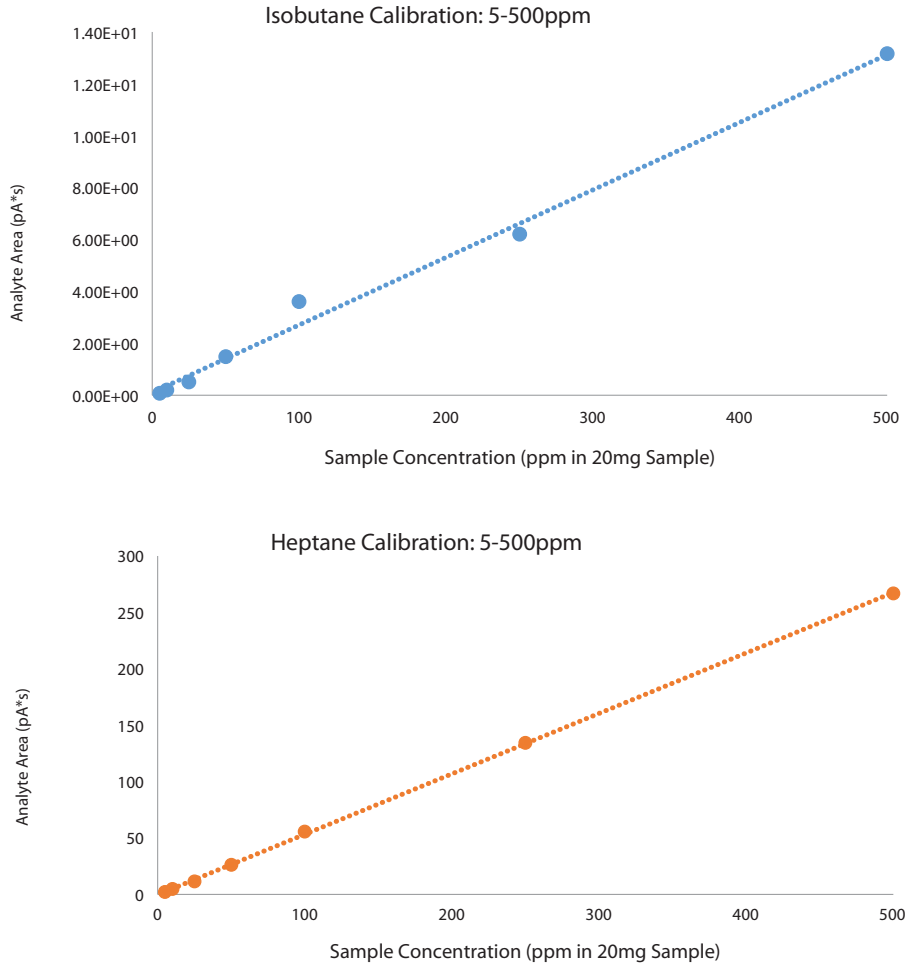
Figure 5 shows plots of the most linear (heptane) and least linear (isobutane) calibration curves. All calibration curves exhibited acceptable linearity without the use of an internal standard. The use of an internal standard may improve linearity and reproducibility, if desired.

Table IV: Using full evaporation technique sample introduction for headspace GC-FID resulted in good sensitivity and linearity for all residual solvents as shown by peak response and correlation data for the calibration standards.

Compound	S:N 10 ppm	S:N 50 ppm	r	r^2
Isobutane	5.30	30.7	0.996	0.992
Butane	18.8	119	0.997	0.994
Methanol	48.1	189	0.999	0.999
Pentane	19.0	50.0	0.998	0.995
Ethanol	45.2	88.1	0.999	0.998
Acetone	49.9	97.0	0.999	0.999
Isopropanol	56.4	107	0.998	0.996
Hexane	45.6	109	0.999	0.998
Chloroform	11.5	22.5	0.999	0.998
Benzene	150	293	0.999	0.998
Heptane	88.4	193	1.00	1.00
Toluene	166	317	0.999	0.998

*Signal-to-noise ratios were calculated using Chemstation® software. Noise ranges were set at 0.2–0.6 minutes and 2.1–2.3 minutes.

Figure 5: Representative Calibration Curves from 5–500 ppm for Heptane and Isobutane



Conclusion

By combining a selective Rxi®-624Sil MS GC column with the FET headspace GC-FID technique, excellent sensitivity and linearity were achieved for residual solvent compounds applicable to cannabis concentrates. The use of FET headspace GC-FID should allow quantification without the use of matrix-matched standards by creating a single non-partitioning phase system in the headspace vial. This technique also has the added benefit of needing very little sample and is applicable for the analysis of other volatile compounds, such as terpenes, in cannabis products.

References

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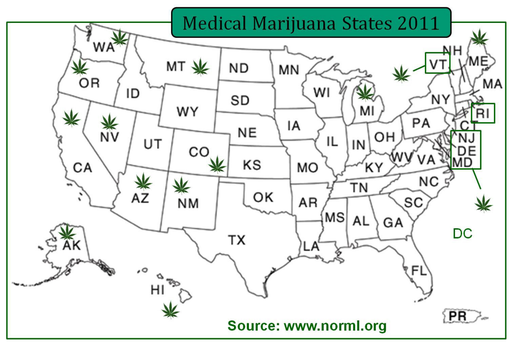
High Quality Analysis of Pesticides in Marijuana for Food and Medicine using QuEChERS, Cartridge SPE, GCxGC-TOFMS, and LC-MS/MS

Jack Cochran, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, Amanda Rigdon, Jason Thomas, Restek Corporation
Frank Dorman, Jessica Westland, Amanda Leffler, The Pennsylvania State University

- Over 15 states in the USA have medical marijuana laws.
  - Therapeutic benefits include pain relief, nausea control, appetite stimulation, and muscle relaxation.
  - Marijuana is illegal on the federal level so patients have no assurances on medicine safety, including for pesticide residues.
- We used the QuEChERS sample preparation approach for extracting pesticides from marijuana.
  - But dispersive SPE did not have the cleanup capacity for GCxGC work.
  - Instead, we employed cartridge SPE for cleanup for GCxGC.
- GCxGC-TOFMS and LC-MS/MS were used for pesticide determinations in cleaned up QuEChERS extracts.
  - The selectivity of advanced techniques was needed due to sample extract complexity, even after dilution/cleanup.
  - LC-MS/MS was necessary for abamectin because it does not gas chromatograph.

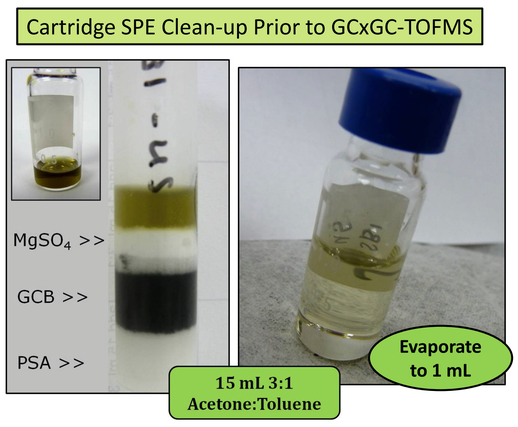


Preparing Marijuana Samples at PSU

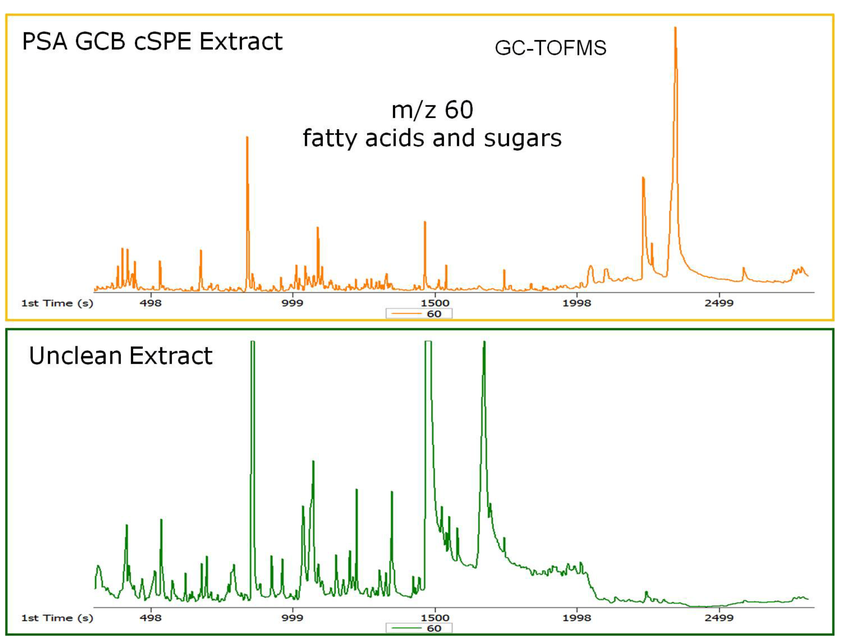
QuEChERS Procedure for Marijuana

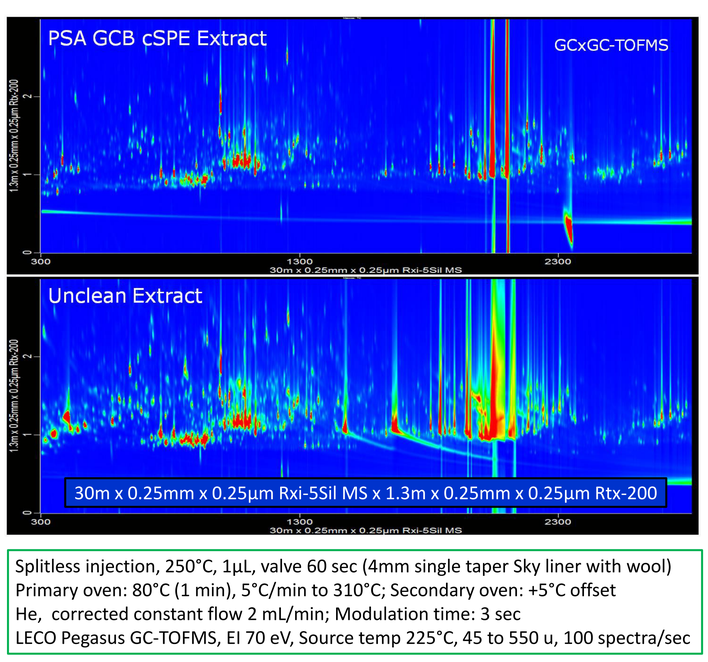
Up to 2 g ground marijuana – 50 mL centrifuge tube
10 mL MeCN
10 mL H_2O
Shake to wet
Soak one hour
Add spikes and internal standards
Vortex 30 min
Add QuEChERS EN salts
Shake 1 min
Centrifuge 5 min at 3000g
Remove extract for cleanup and analysis
GCxGC-TOFMS and LC-MS/MS

Cartridge SPE Clean-up Prior to GCxGC-TOFMS



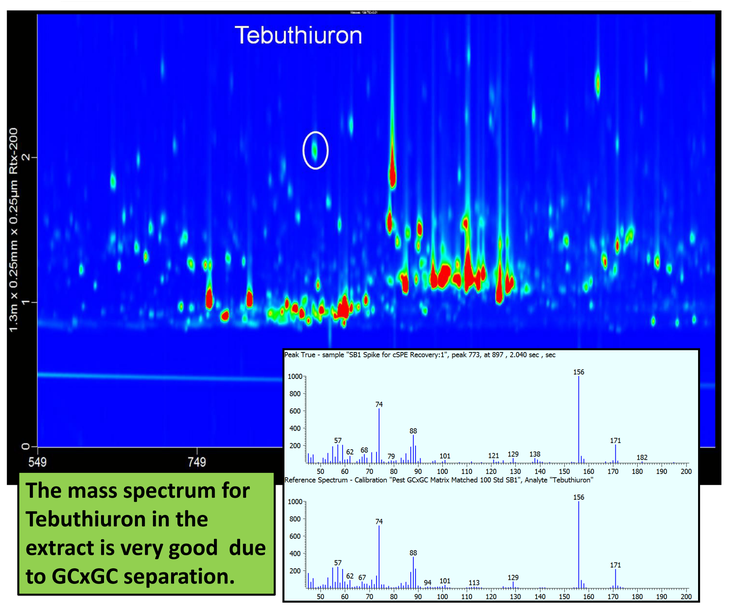
High Capacity Cartridge SPE Produces a Cleaner Extract by Removing Interfering Fatty Acids and other Matrix Co-extractives





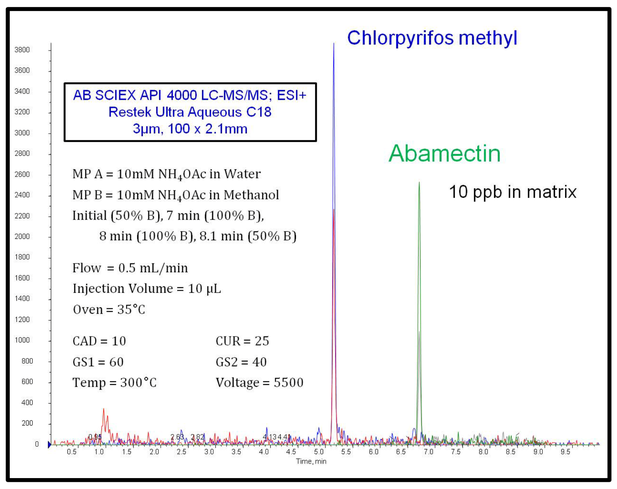
Splitless injection, 250°C, 1µL, valve 60 sec (4mm single taper Sky liner with wool)
Primary oven: 80°C (1 min), 5°C/min to 310°C; Secondary oven: +5°C offset
He, corrected constant flow 2 mL/min; Modulation time: 3 sec
LECO Pegasus GC-TOFMS, EI 70 eV, Source temp 225°C, 45 to 550 u, 100 spectra/sec

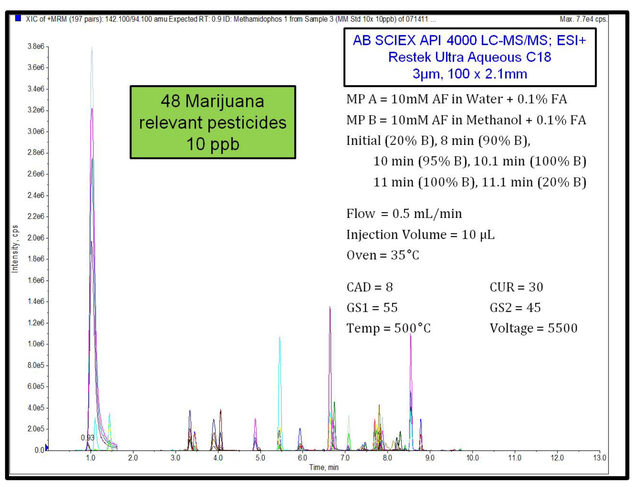
GCxGC Separates Pesticides from Remaining Matrix Co-extractives in a Marijuana Extract



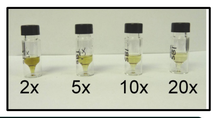
The mass spectrum for Tebuthiuron in the extract is very good due to GCxGC separation.

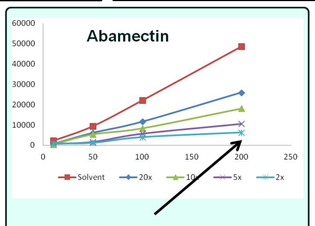
LC-MS/MS of Marijuana Pesticides – Abamectin Required a Single Analyte Method Approach

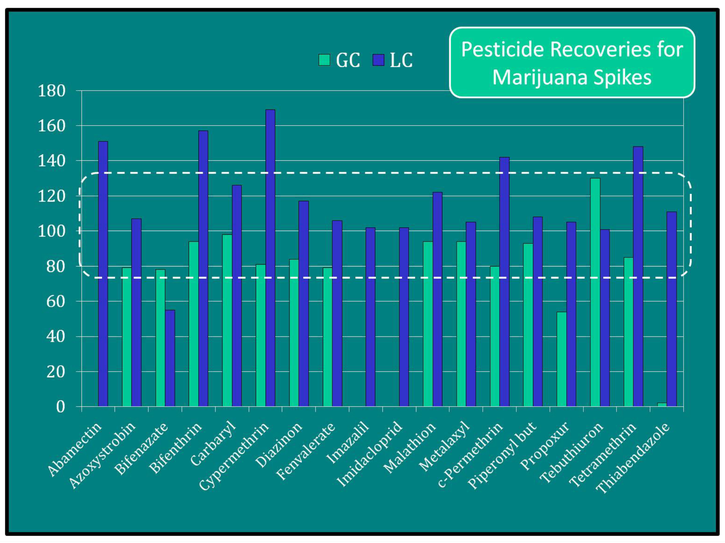




Dilute and shoot?







Incurred Pesticides in Marijuana Sample 2Q2

Pesticide	LC ppb	GC
Imazalil	410	NA
Bifenazate	1100	2180
Piperonyl butoxide	37	41
trans-Permethrin	660	1100
cis-Permethrin	1200	690
o-Phenylphenol	NA	280
4,4'-DDE	NA	30

NA = not analyzed by this method

"Effervescent Health Formula"

Technical Article

High-Quality Analysis of Pesticides in Cannabis

Using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS

By Jack Cochran, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, and Amanda Rigdon

- Quickly and effectively extract medical marijuana samples for pesticide analysis.
- Cartridge SPE cleanup of dirty extracts improves GC inlet and column lifetimes.
- Selective GC columns increase accuracy of pesticide determinations for complex samples.

Over 20 states in the U.S. have legalized the use of recreational or medical cannabis because of therapeutic benefits for ailments such as cancer, multiple sclerosis, and ALS. Dosing methods include smoking or vaporizing and baked goods. Unlike other prescribed medicines regulated by U.S. FDA, marijuana is a Schedule 1 drug and is illegal on the federal level. As a result, medical cannabis patients have no safety assurances for their medication, which could contain harmful levels of pesticide residues. Currently, medical marijuana pesticide residue analysis methods are poorly defined and challenging to develop due to matrix complexity and a long list of potential target analytes.

In order to address matrix complexity, we combined a simple QuEChERS extraction approach with cartridge SPE (cSPE) cleanup, followed by GCxGC-TOFMS. Acceptable recoveries were obtained for most pesticides, and incurred pesticide residues were detected in some of the illicit marijuana samples used for method development.

QuEChERS Extraction Saves Time and Reduces Hazardous Solvent Use

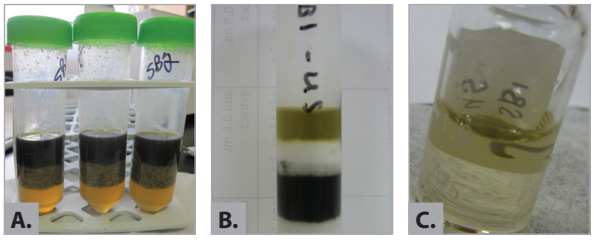
Trace residue extraction procedures from dry materials like medical cannabis typically involve large amounts of solvent, long extraction times, and tedious concentration steps similar to the Soxhlet procedure or multiresidue methods from the Pesticide Analytical Manual. QuEChERS, with its simple 10 mL acetonitrile shake extraction and extract partitioning with salts and centrifugation, offers time savings, glassware use reduction, and lower solvent consumption.

Water was added to finely ground, dry cannabis samples to increase QuEChERS extraction efficiency, especially for more polar pesticides. A vortex mixer was used to shake the solvent and sample for at least 30 minutes prior to extract partitioning. When finished, it was easy to transfer the supernatant from the QuEChERS extraction tube for subsequent cSPE cleanup prior to analysis with GC or LC (Figure 1).

Cartridge SPE Cleanup Improves GC Inlet Uptime

Injecting chlorophyll-laden extracts into a GC gives reduced recoveries for less volatile pesticides, and results in degradation of sensitive pesticides like DDT and Dicofol (Table I). SPE cleanup with a 500 mg graphitized carbon black/500 mg PSA cartridge removes chlorophyll and traps fatty acids that interfere with qualitative pesticide identification and bias quantification. cSPE has increased sorbent capacity over dispersive SPE for thorough cleanup of complex extracts.

Figure 1: A quick and easy QuEChERS extraction, combined with cSPE, effectively prepared extracts for pesticide residue analysis from highly complex marijuana samples.



A. Post-centrifugation QuEChERS extracts

B. QuEChERS extracts loaded on SPE cartridge

C. Final extract

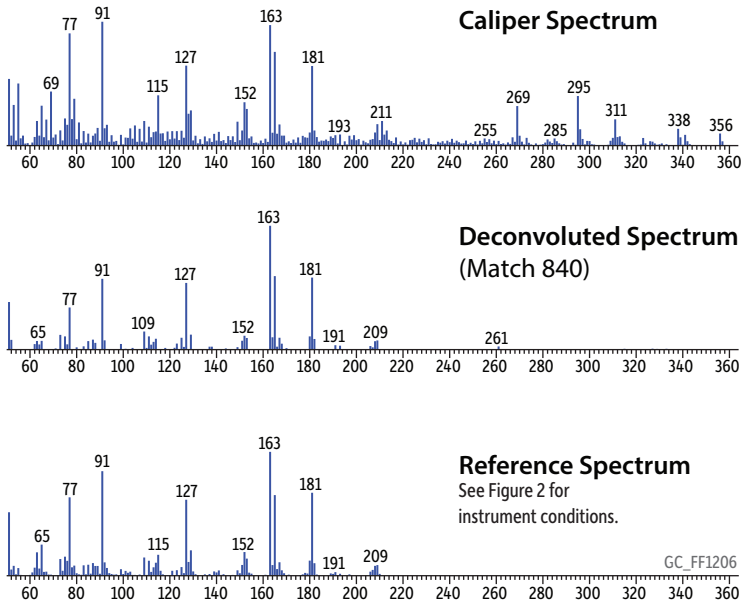
Orthogonal GC Columns Increase Separation Power for More Accurate Pesticide Results

GCxGC is a powerful multidimensional approach that gives two independent separations in one instrumental analysis. An Rxi®-5Sil MS and Rtx®-200 column combination distributes pesticides broadly in both dimensions, providing a highly orthogonal GCxGC system. More important though is separating pesticides from potential isobaric matrix interferences, as seen in the surface plot for the insecticide cypermethrin (Figure 2). Cypermethrin gas chromatographs as four isomers, and all would have experienced qualitative interference and quantitative bias from peaks in the foreground of the surface plot had only 1-dimensional GC been used. With GCxGC-TOFMS, cypermethrin was unequivocally identified in a marijuana sample at a low ppm level (Figure 3).

Summary

QuEChERS and cSPE produced usable extracts from highly complex cannabis samples for high-quality pesticide residue analysis. The multidimensional separation power of GCxGC-TOFMS was then used to correctly identify and quantify pesticides in these complex extracts.

Figure 3: Positive mass spectral identification of incurred cypermethrin in illicit marijuana.



Acknowledgment: Randy Hoffman, a Police Evidence Technician at The Pennsylvania State University (PSU), supplied the seized marijuana samples while overseeing their handling. Frank Dorman at PSU assisted with QuEChERS extractions.

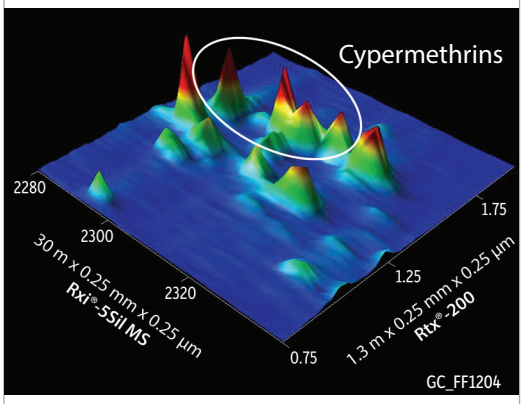
Initially published in *Restek® Advantage*.

Table I: Pesticide recoveries for a QuEChERS extract of cannabis give higher results when cSPE is used for cleanup. Dicofol and DDT are degraded in the inlet for the dirtier extract, yielding high DDD results.

Pesticide	Classification	With cSPE Cleanup (%)	Without cSPE Cleanup (%)
4,4'-DDD	Organochlorine	83	230
4,4'-DDT	Organochlorine	77	9
Bifenthrin	Pyrethroid	86	89
Dicofol	Organochlorine	84	ND
Azinphos methyl	Organophosphorus	79	53
trans-Permethrin	Organochlorine	68	17
Pyraclostrobin	Strobilurin	73	19
Fluvalinate	Pyrethroid	72	23
Difenoconazole	Triazole	67	21
Deltamethrin	Pyrethroid	68	20
Azoxystrobin	Strobilurin	72	27

ND = no peak detected

Figure 2: GCxGC-TOFMS and orthogonal Rxi®-5Sil MS and Rtx®-200 columns allow incurred cypermethrins in a marijuana extract to be separated from interferences (m/z 163 quantification ion).



Peaks	RT 1 (sec.)	RT 2 (sec.)
1. Cypermethrin 1	2292	1.50
2. Cypermethrin 2	2304	1.54
3. Cypermethrin 3	2310	1.53
4. Cypermethrin 4	2313	1.58

Column: Rxi®-5Sil MS 30 m, 0.25 mm ID, 0.25 µm (cat.# 13623), Rtx®-200 1.3 m, 0.25 mm ID, 0.25 µm (cat.# 15124); **Sample:** Diluent: Toluene; **Injection:** Inj. Vol.: 1 µL splitless (hold 1 min); Liner: Sky® 4mm single taper w/wool (cat.# 23303.1); Inj. Temp.: 250 °C; Purge Flow: 40 mL/min; **Oven:** Oven Temp: Rxi®-5Sil MS: 80 °C (hold 1 min) to 310 °C at 5 °C/min, Rtx®-200: 85 °C (hold 1 min) to 315 °C at 5 °C/min; **Carrier Gas:** He, corrected constant flow (2 mL/min); **Modulation**: Modulator Temp. Offset: 20 °C; Second Dimension Separation Time: 3 sec.; Hot Pulse Time: 0.9 sec.; Cool Time between Stages: 0.6 sec.; **Instrument**: LECO Pegasus 4D GCxGC-TOFMS; For complete conditions, visit www.restek.com and enter GC_FF1204 in the search.

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Reliably Confirm Cannabinoids by GC-MS

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

by Kristi Sellers, Clinical/Forensic Innovations Chemist

- 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-Δ9-tetrahydrocannabinol (Δ9-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. Δ9-carboxy-THC is the primary target in GC/MS confirmation analysis, but other marijuana metabolites present in samples include cannabinol, cannabidiol, 11-hydroxy-Δ9-tetrahydrocannabinol (Δ9-hydroxy-THC), Δ9-tetrahydrocannabinol (Δ9-THC), and Δ8-tetrahydrocannabinol (Δ8-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, Δ9-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.

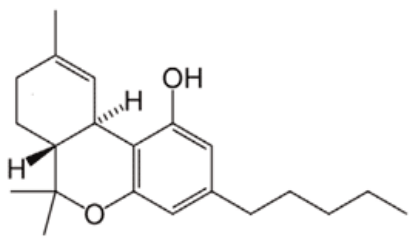
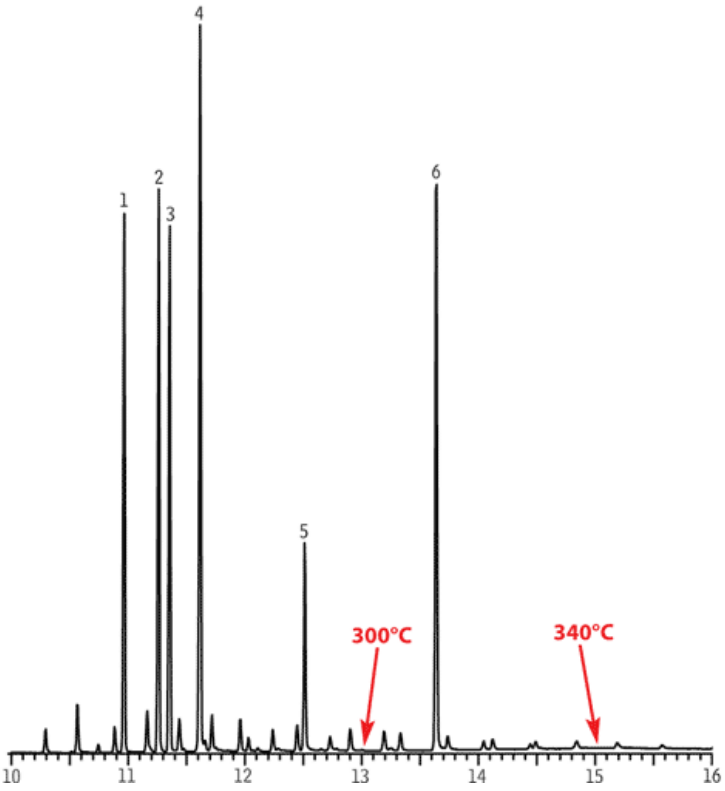


Figure 2 A 12m x 0.20mm ID x 0.33µm Rxi™-5ms column provides baseline resolution and short analysis time for cannabinoids.

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*

Technical Article

Don't Overestimate Cannabidiol During Medical Cannabis Potency Testing by Gas Chromatography

By Jack Cochran

Accurate potency testing of medical cannabis with gas chromatography (GC) depends principally on choosing a column with the right selectivity; otherwise, coelutions between cannabinoids of interest may cause error in potency measurements. Cannabidiol is one of the chief cannabinoids with pharmacological value and provides relief against nausea, anxiety, and inflammation. Potency testing for medical marijuana is often done using "5-type" GC columns since they are commonly available in most labs. However, on 5-type columns cannabidiol can coelute with cannabichromene, a compound that likely also has medical value and is increasingly becoming part of potency testing. To identify and report both of these compounds accurately, a GC column with a different stationary phase is needed.

Proper Column Choice Results in More Accurate Potency Data

As shown in Figure 1, cannabinoids are aromatic compounds, meaning they will likely be better separated on a column that contains aromatics in the stationary phase because these stationary phases are more selective for aromatic-containing analytes. A fully non-aromatic stationary phase, like a "1-type" (100% dimethyl polysiloxane) column is not appropriate for this analysis since cannabichromene (CBC) and cannabidiol (CBD) will coelute completely. While 5-type columns (5% phenyl) contain some aromatic component, they generally also produce coelutions for cannabichromene and cannabidiol, depending on the conditions used. At best, CBC and CBD can be only partially resolved on 15 m 5% phenyl columns. Much better separations are obtained on higher phenyl-content phases, such as Rxi®-35Sil MS (35% phenyl type) and Rxi®-17Sil MS (50% phenyl type) columns, as they offer excellent selectivity for aromatic cannabinoids. Not only do both columns resolve cannabichromene and cannabidiol, the chromatograms in Figures 2 and 3 demonstrate that they also separate delta-8-tetrahydrocannabinol (d8-THC), delta-9-tetrahydrocannabinol (d9-THC), cannabigerol (CBG), and cannabinol (CBN). Although both columns perform well, the Rxi®-35Sil MS column is recommended because of the slightly faster analysis time and greater space overall between the peaks of interest.

While stationary phase selectivity is the most important factor in choosing a GC column for cannabinoid analysis, there are some additional aspects of this work that will benefit labs doing medical marijuana potency testing. First, cost savings were achieved by using a 15 m column. When a column with the proper selectivity is used, a 15 m column easily provides the separating power needed for this analysis at about half the cost of a 30 m column. Also, the 0.25 mm x 0.25 µm format has good sample loading capacity and is robust, especially when a proper split injection is used with a Sky® Precision® split liner with wool. Finally, hydrogen carrier gas was used here instead of helium. Using hydrogen provides a faster analysis, increasing sample throughput. Hydrogen carrier gas is a convenient way to speed up run times, increase productivity, and reduce the cost and availability concerns associated with using helium carrier gas.

Figure 1: Since cannabinoids are aromatic compounds, a GC column that contains aromatics in the stationary phase will provide much better separations than a column with a non-aromatic phase.

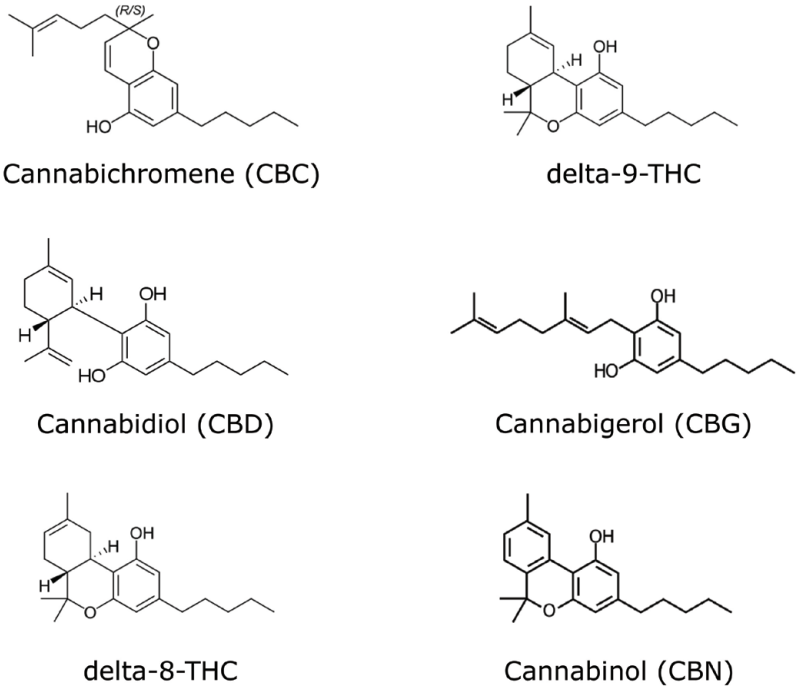


Figure 2: The Rxi®-35Sil MS column provides both the best separation and the fastest analysis time, making it the ideal GC column choice for medical cannabis potency testing.

Peaks
1. Cannabichromene
2. Cannabidiol
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-35Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13820); **Sample:** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 225 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.9 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 2.5 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC

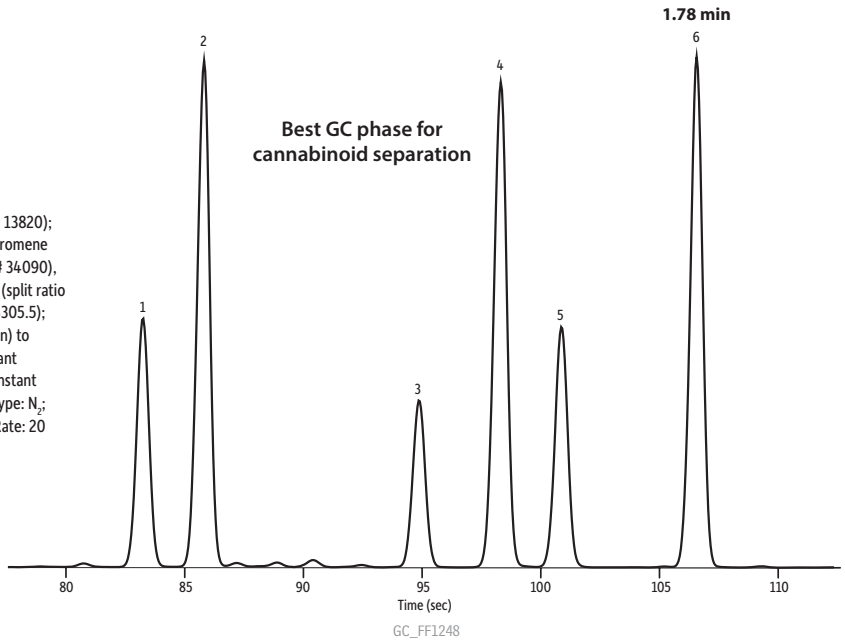
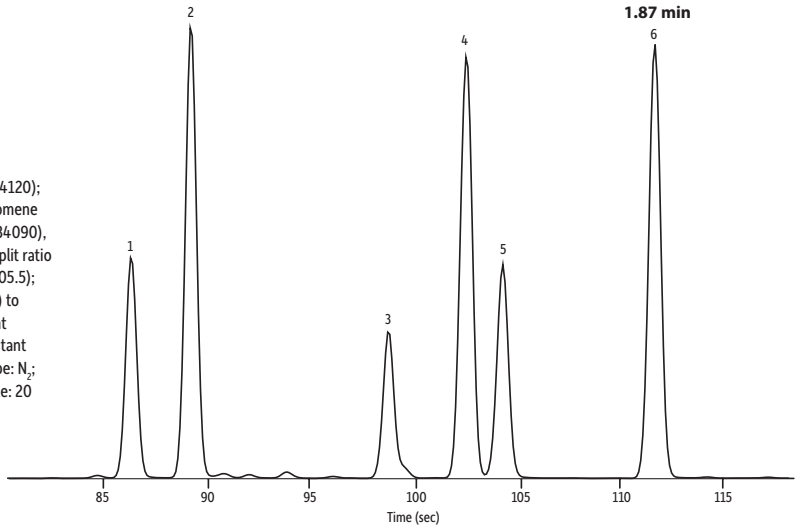


Figure 3: Cannabinoids can be effectively separated on an Rxi® 17Sil MS column, but with slightly less resolution than that obtained with the optimal selectivity of the Rxi®-35Sil MS column.

Peaks
1. Cannabichromene
2. Cannabidiol
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-17Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 14120); **Sample;** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 225 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.9 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 2.5 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC



Adjusting Conditions for 5-Type Columns

While using an Rxi®-35Sil MS column provides the best selectivity and speed for cannabinoid analysis, cannabidiol potency can be determined in medical cannabis using a 5-type column under certain conditions. If you already have a 5-type column for this work, you can vary the GC conditions, especially carrier flow and oven temperature program, and still separate cannabichromene and cannabidiol, just not as quickly or easily as with the Rxi®-35Sil MS column. Figures 4 and 5 show this analysis on Rxi®-5ms and Rxi®-5Sil MS columns, respectively. Again, the 0.25 mm x 0.25 µm format was used here because it offers better efficiency than wider bore columns (e.g., 0.32 mm and 0.53 mm IDs), which may not separate cannabichromene and cannabidiol under any operational conditions.

Figure 4: The selectivity of a 5-type column is not sufficient to fully separate cannabichromene and cannabidiol, resulting in less accurate medical marijuana potency testing.

Peaks
1. Cannabichromene
2. Cannabidiol
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-5ms, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13420); **Sample;** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 250 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.6 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 1.6 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC

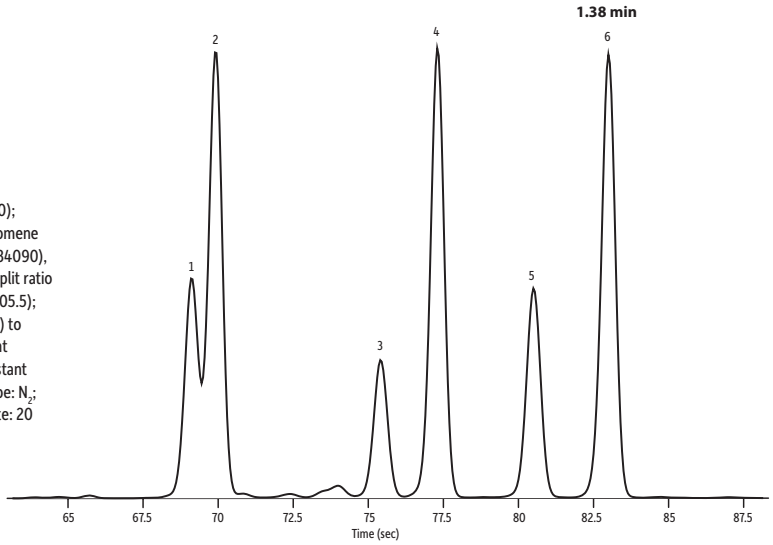
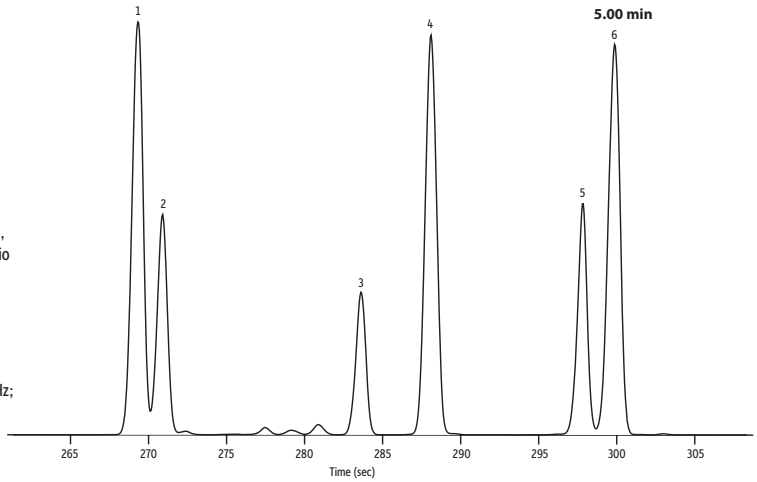


Figure 5: Rxi®-5Sil MS columns offer better resolution of key cannabinoids than standard 5-type columns, but the incomplete separation and longer analysis time mean further optimization is needed for accurate reporting.

Peaks
1. Cannabidiol
2. Cannabichromene
3. delta-8-Tetrahydrocannabinol
4. delta-9-Tetrahydrocannabinol
5. Cannabigerol
6. Cannabinol

Column: Rxi®-5Sil MS, 15 m, 0.25 mm ID, 0.25 µm (cat.# 13620); **Sample;** Cannabinoids standard (cat.# 34014), Cannabichromene (cat.# 34092), delta-8-Tetrahydrocannabinol (THC) (cat.# 34090), Cannabigerol (cat.# 34091); **Injection:** Inj. Vol.: 1 µL split (split ratio 50:1); Liner: Sky® 4 mm Precision® liner w/wool (cat.# 23305.5); Inj. Temp.: 250 °C; **Oven:** Oven Temp.: 150 °C (hold 0.1 min) to 330 °C at 35 °C/min (hold 0.7 min); **Carrier Gas:** H_2, constant flow; Flow Rate: 1.6 mL/min; **Detector:** FID @ 350 °C; Constant Column + Constant Make-up: 50 mL/min; Make-up Gas Type: N_2; Hydrogen flow: 40 mL/min; Air flow: 450 mL/min; Data Rate: 20 Hz; **Instrument:** Agilent/HP6890 GC



Note that even though these are both 5-type columns, the elution order of cannabichromene and cannabidiol changed. This is due to two things. The first is that Rxi®-5ms and Rxi®-5Sil MS columns differ slightly in selectivity for certain compounds; even though they are both considered 5-type columns, they contain different stationary phases that retain some compounds differently. The second reason is that the GC oven programs are different for the columns, which means that the compounds are eluting at different temperatures. You may be able to further optimize the separation of cannabichromene and cannabidiol on a 5-type column, but the selectivity and faster analysis that can be obtained using a high-phenyl content Rxi®-35Sil MS column make it ideal for potency determinations in medical cannabis.

To sum things up, proper column choice is essential for accurate and robust cannabis potency testing. Using the right column not only gives you more confidence in your potency values, but it also saves you time and money. Switching to hydrogen carrier gas can reduce your costs even further, while increasing sample throughput.

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High Quality Analysis of Pesticides in Marijuana for Medicine using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS

Jack Cochran, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, Amanda Rigdon

Restek Corporation, Bellefonte, PA, USA

Frank Dorman

The Pennsylvania State University, University Park, PA, USA

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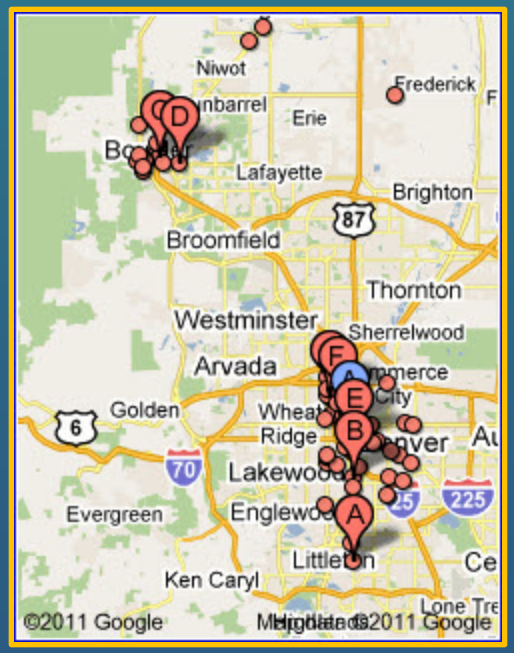

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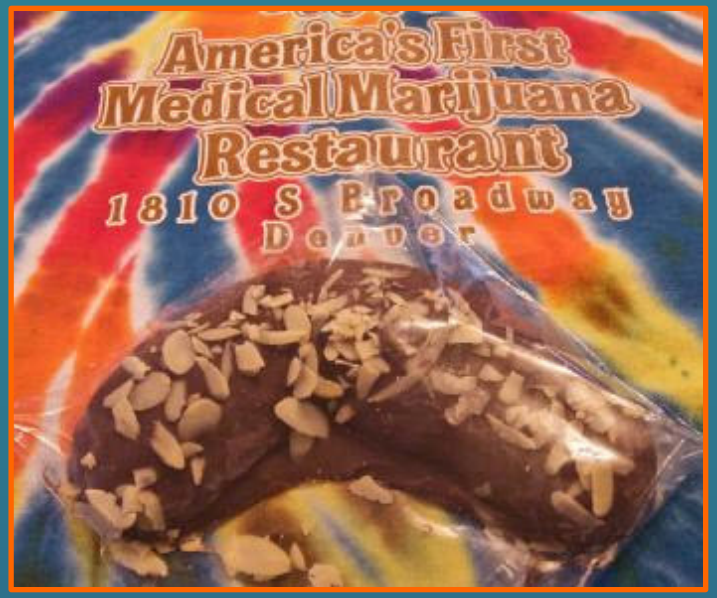

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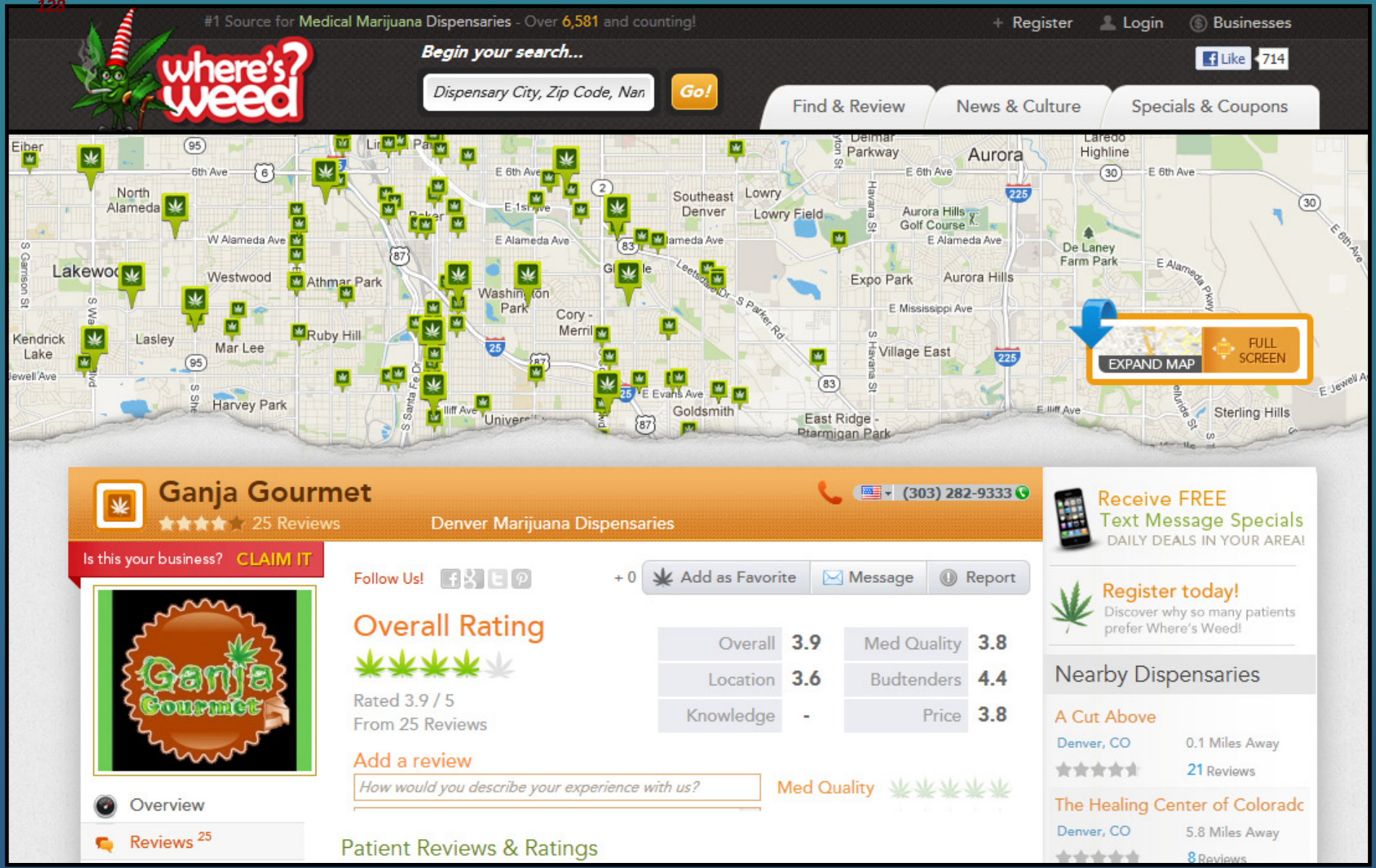
1.5 grams



Please be aware that our Menu Page is strictly for historical content for our website, as we are no longer baking pizzas, cheesecakes and LaGanja these days!!!

We still do bake our famous medicated Almond Horns though!

Ganja Gourmet's Dinner Buzz Specials

Your choice of any Entrée, along with any Dessert, and an After-Dinner Joint = Just $30.00!!!


Q-sep
RESTEK


Q-sep™
QuEChERS Products
RESTEK


Q-sep™ 3000
RUN
START
OPEN
RESTEK


Figure 4
Q-sep


Figure 5
Q-sep
RESTEK


Figure 6

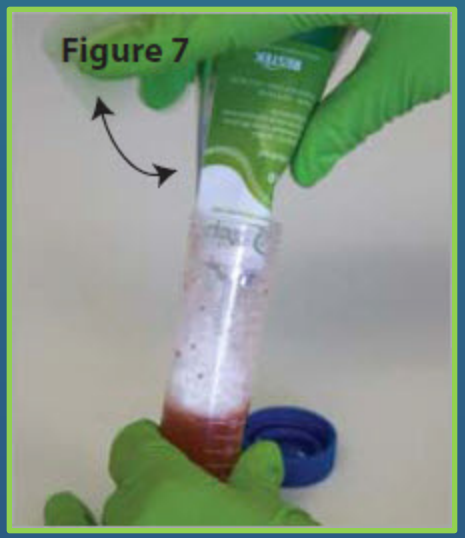
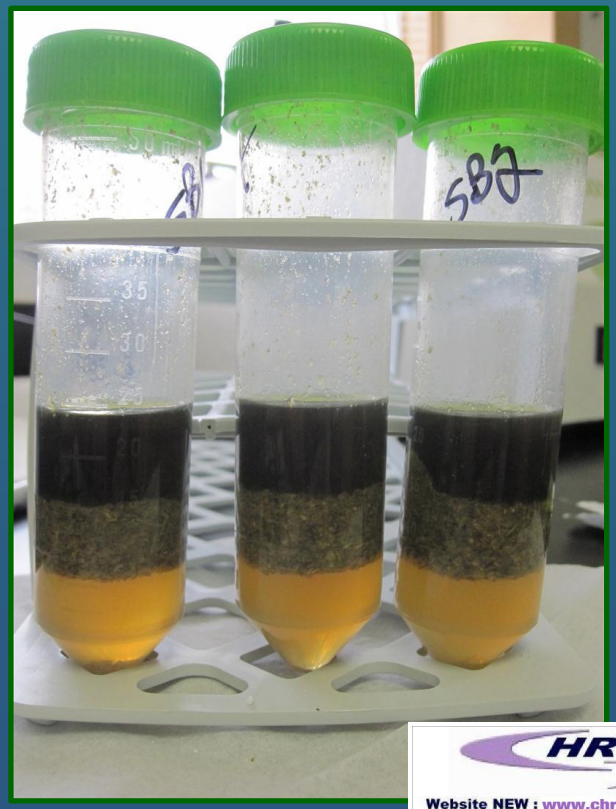
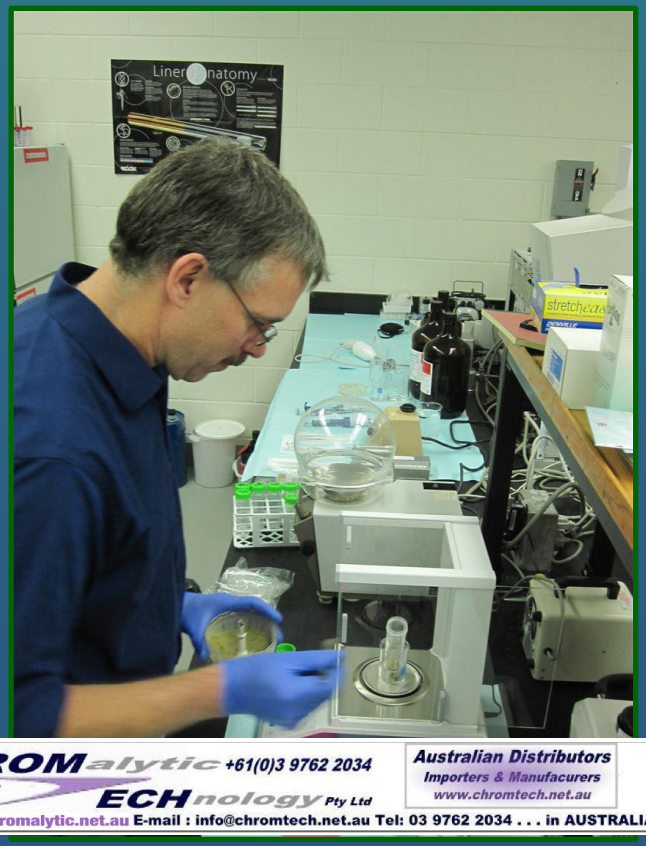


Figure 7





2 g ground marijuana – 50 mL centrifuge tube

10 mL MeCN

10 mL H_2O

Shake to wet

Soak one hour

Add spikes and internal standards

Vortex 30 min

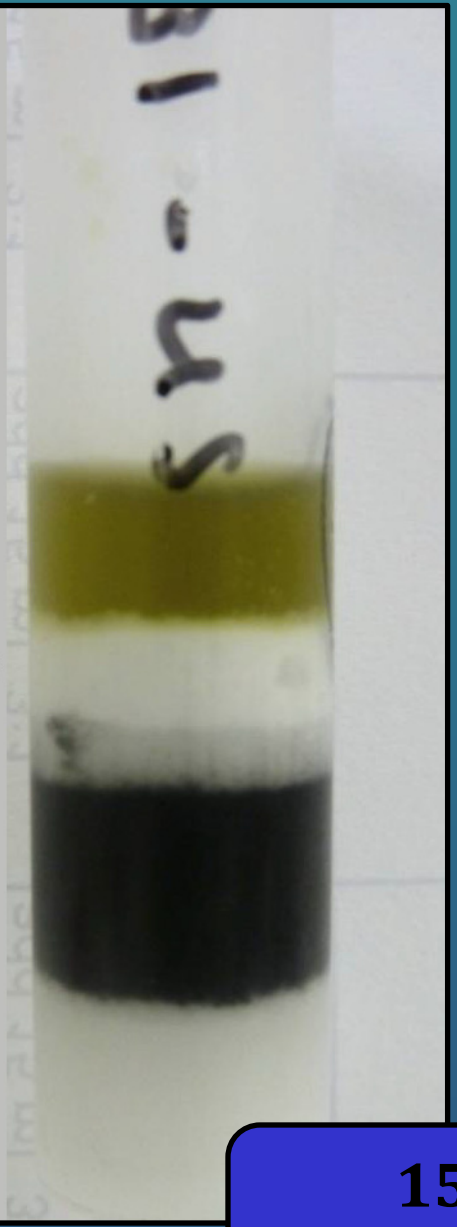
Add QuEChERS EN salts

Shake 1 min

Centrifuge 5 min at 3000g

Remove extract for cleanup and analysis

GCxGC-TOFMS and LC-MS/MS



$MgSO_4$ >>

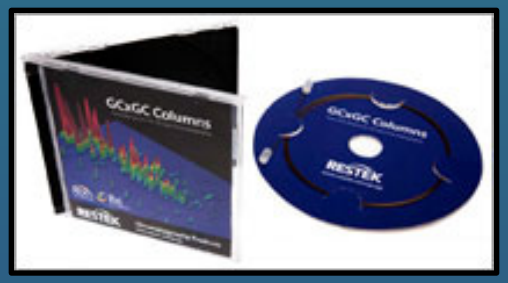
GCB >>

PSA >>

15 mL 3:1 Acetone:Toluene

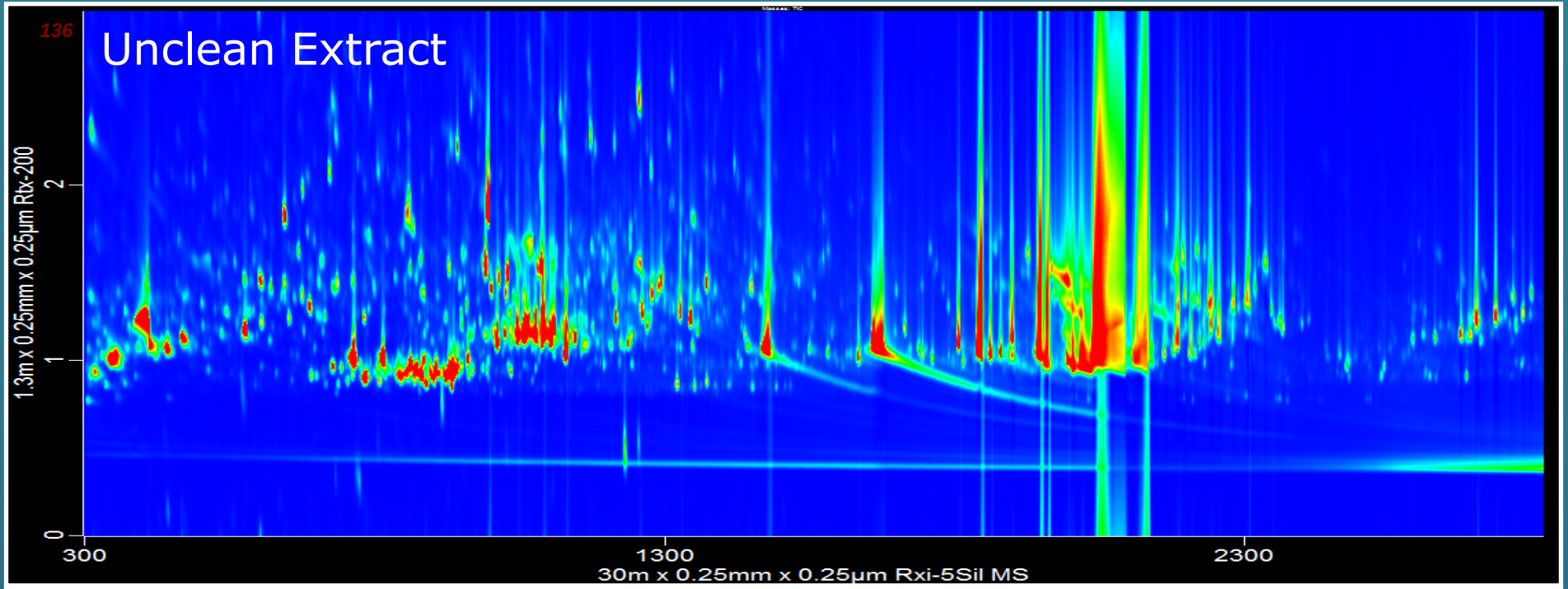
www.restek.com/gcxgc

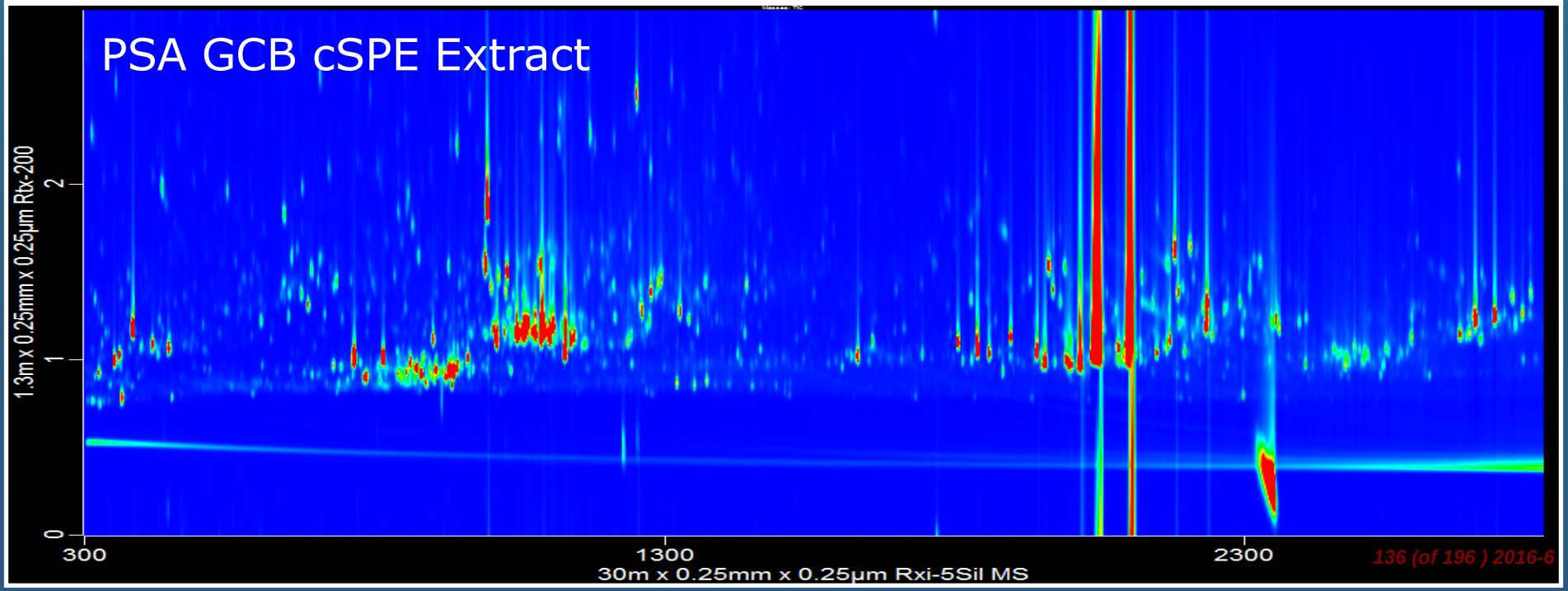
- 30m x 0.25mm x 0.25µm Rxi-5Sil MS
  - 5% phenyl (silphenylene) / 95% dimethyl
- Corrected constant flow He at 2.0 mL/min
- 80°C (1min), 5°C/min to 310°C
- Thermal modulation, 3 sec
- 1.3m x 0.25mm x 0.25µm Rtx-200
  - Trifluoropropylmethyl, selectivity for pesticides
  - +5° temp offset from primary column



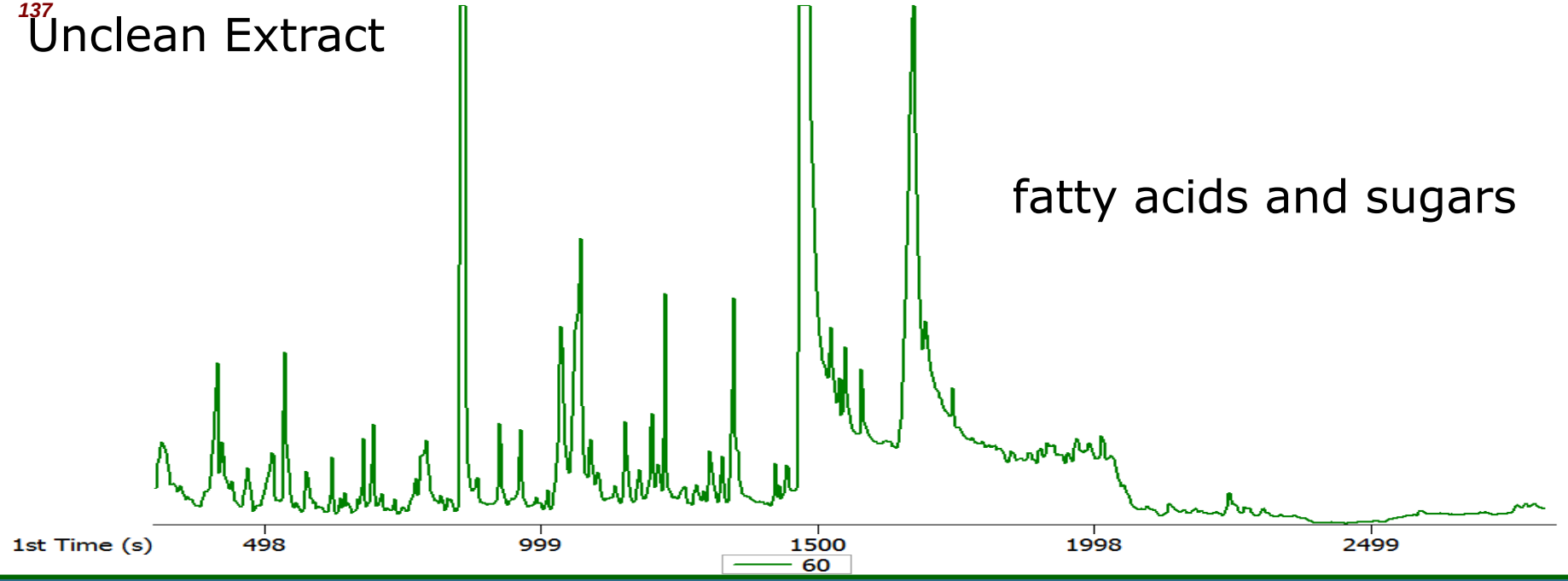
LECO Pegasus®
TOFMS for Pesticides

- Source temperature: 225°C
- Electron ionization: 70 eV
- Stored mass range: 45 to 550 u
- Acquisition rate: 100 spectra/sec

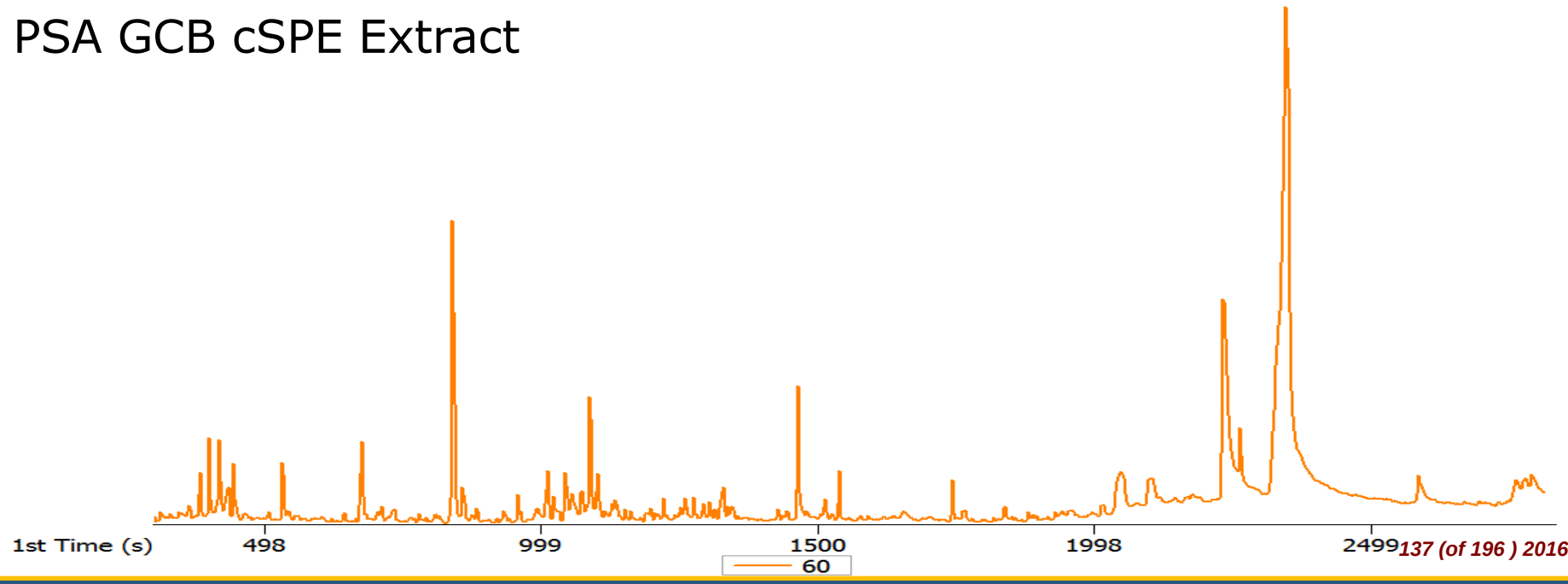

136
Unclean Extract
1.3m x 0.25mm x 0.25µm Rtx-200
0
1
2
300
1300
2300
30m x 0.25mm x 0.25µm Rxi-5Sil MS


PSA GCB cSPE Extract
1.3m x 0.25mm x 0.25µm Rtx-200
0
1
2
300
1300
2300
30m x 0.25mm x 0.25µm Rxi-5Sil MS
136 (of 196) 2016-6

Unclean Extract

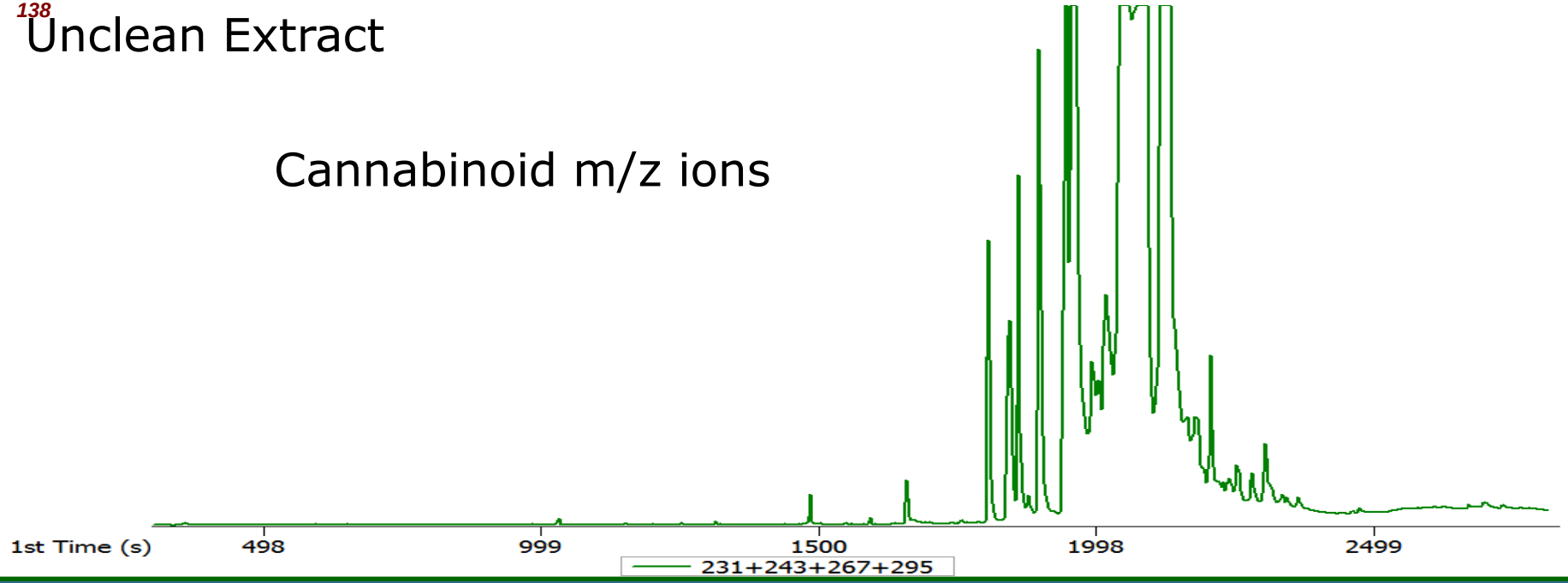


PSA GCB cSPE Extract

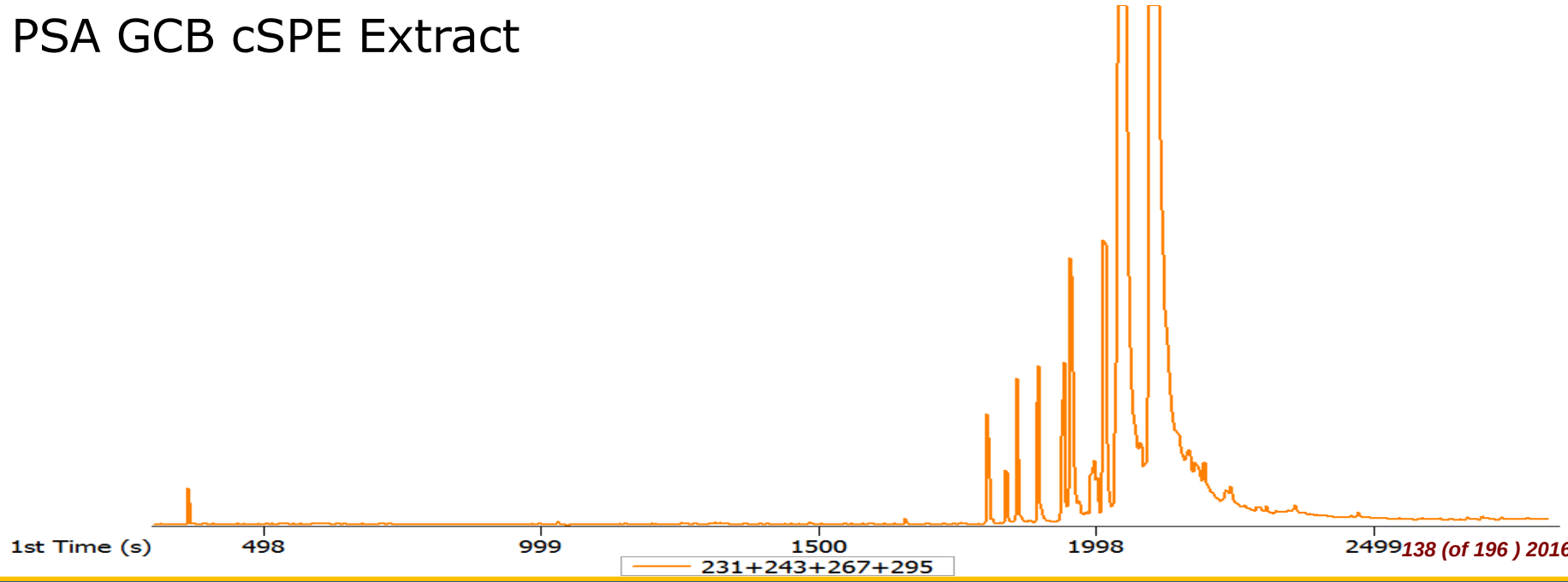


Unclean Extract

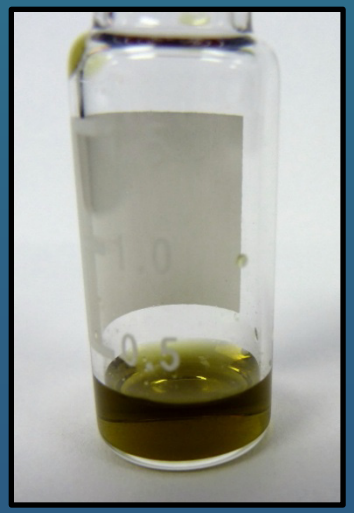
Cannabinoid m/z ions



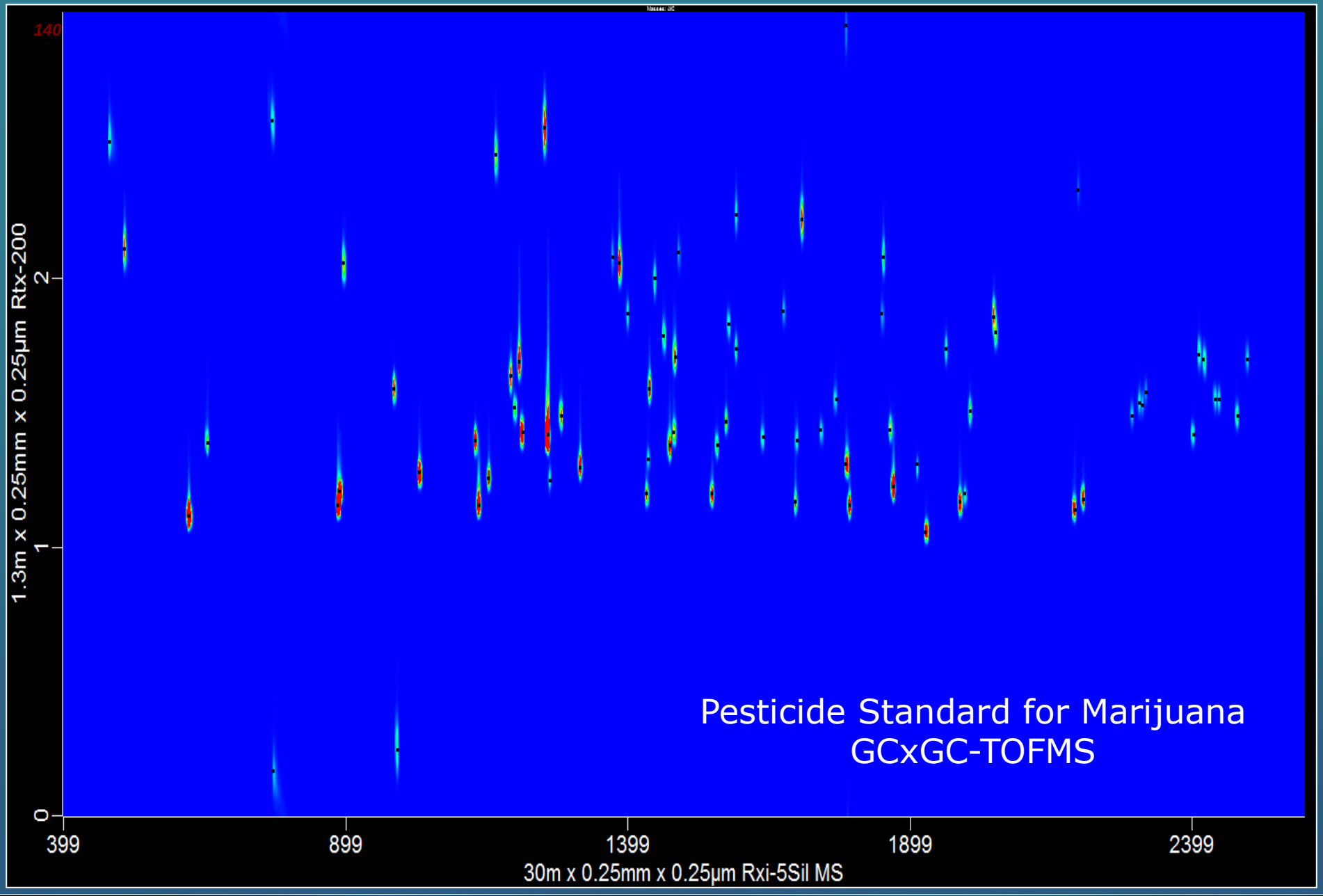
PSA GCB cSPE Extract

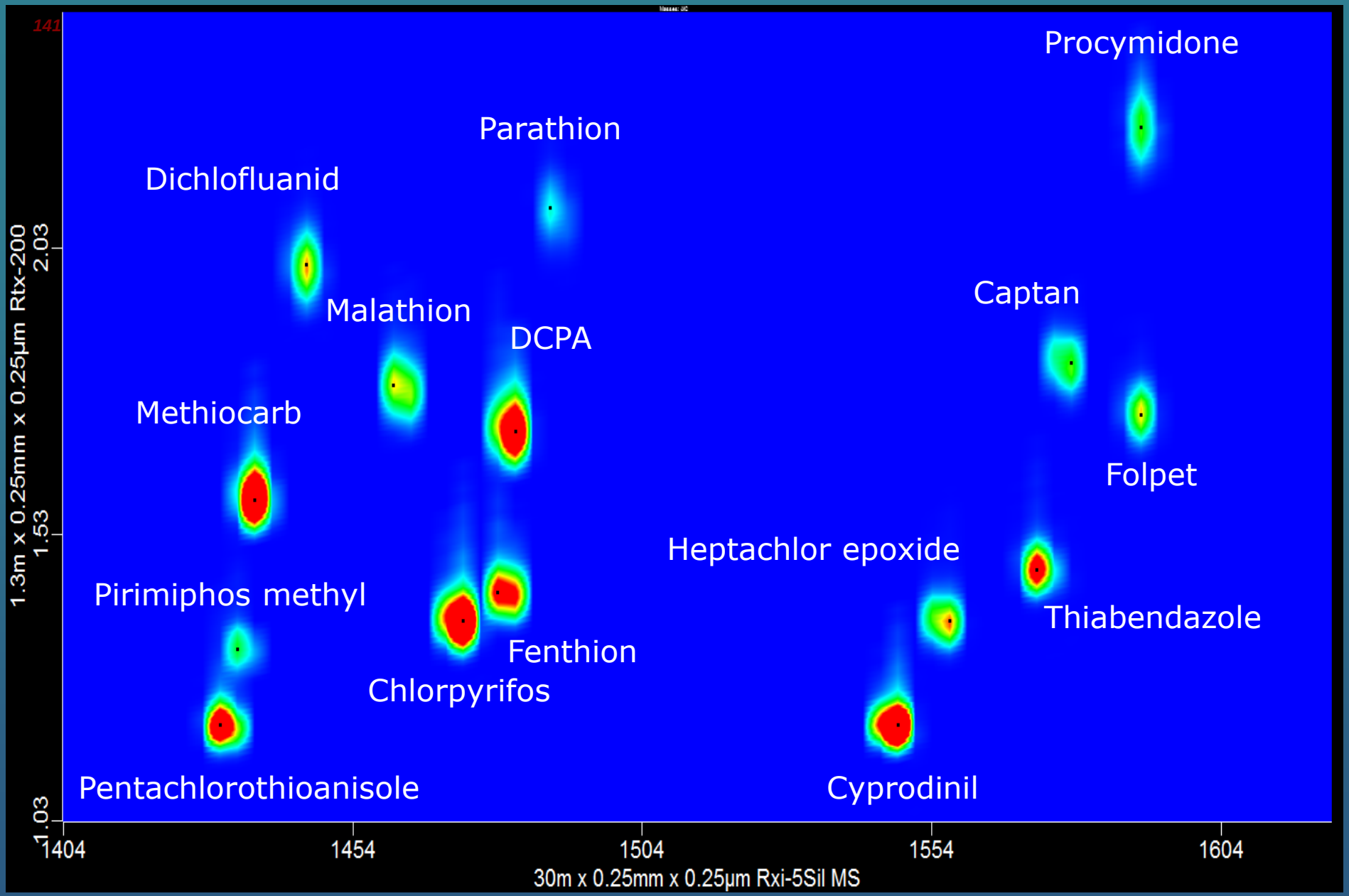


Pesticide Recoveries for Marijuana Spikes Later Eluting Compounds – Clean versus Unclean



Pesticide	Classification	SB3 cSPE	SB3 No cSPE
4,4'-DDD	Organochlorine	83	230
4,4'-DDT	Organochlorine	77	9
Bifenthrin	Pyrethroid	86	89
Dicofol	Organochlorine	84	ND
Azinphos methyl	Organophosphorus	79	53
trans-Permethrin	Organochlorine	68	17
Pyraclostrobin	Strobilurin	73	19
Fluvalinate	Pyrethroid	72	23
Difenoconazole	Triazole	67	21
Deltamethrin	Pyrethroid	68	20
Azoxystrobin	Strobilurin	72	27





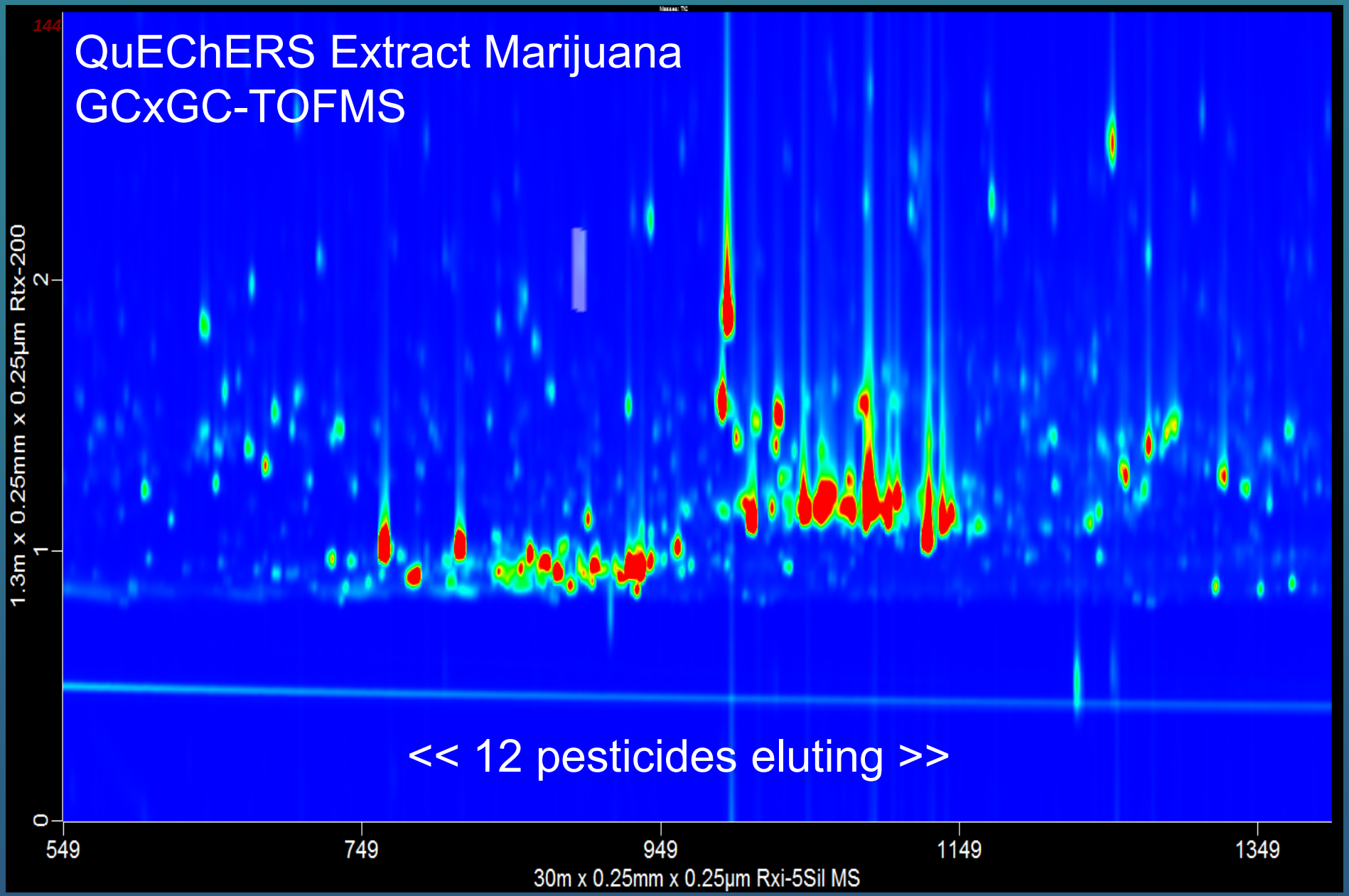
Pesticide Recoveries for Marijuana Spikes

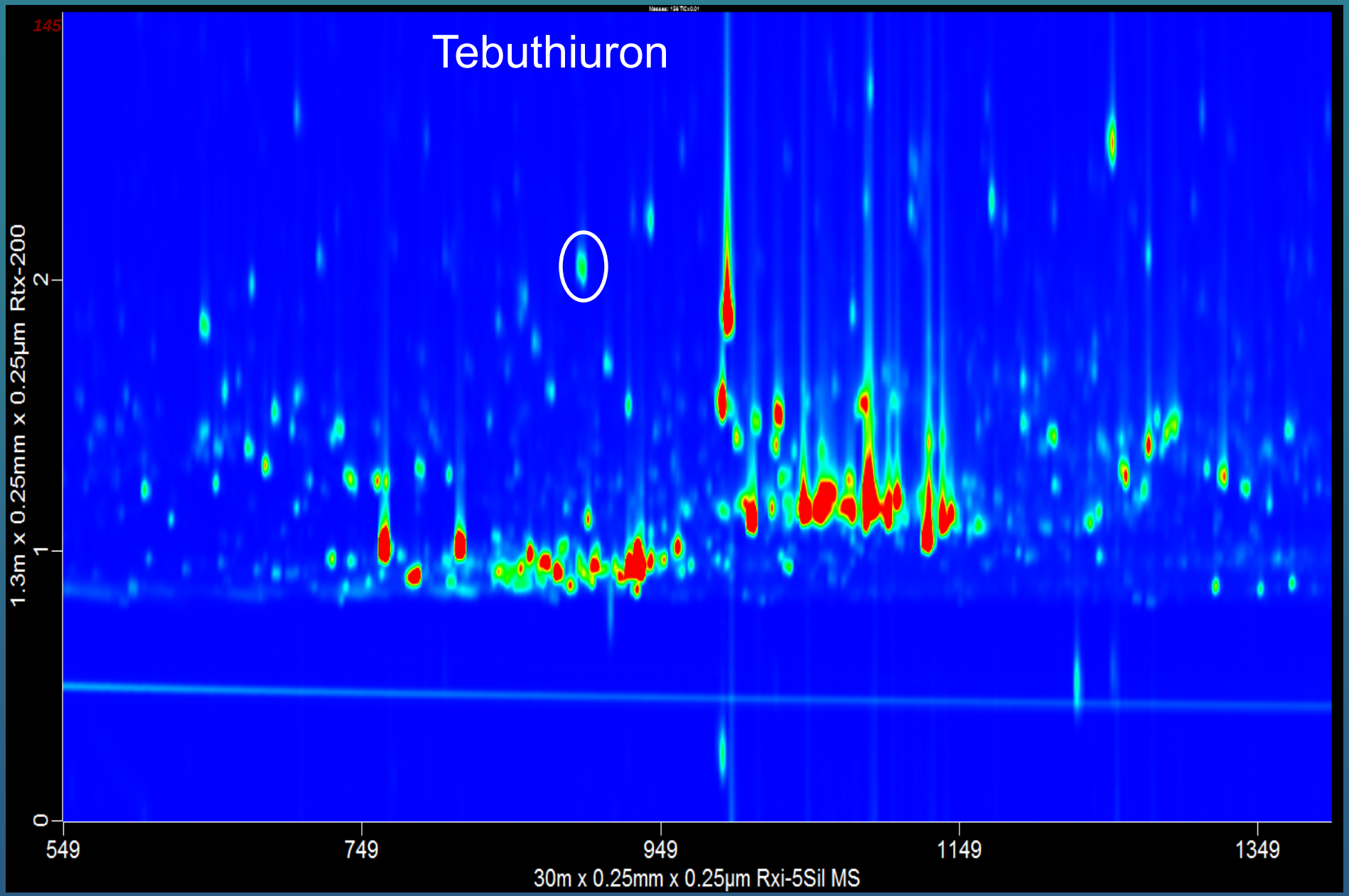
Pesticide	Classification	SB1 cSPE	SB3 Q + cSPE	S3 Q + cSPE
o-Phenylphenol	Unclassified	91	83	97
Tebuthiuron	Organonitrogen	100	104	94
Hexachlorobenzene	Organochlorine	73	44	71
Chlorothalonil	Organochlorine	77	80	81
Anthracene	QC STD	108	105	119
Diazinon	Organophosphorus	86	89	102
Carbaryl	Carbamate	91	103	100
Metalaxyl	Organonitrogen	93	96	90
Malathion	Organophosphorus	98	106	104
Chlorpyrifos	Organophosphorus	87	92	93
Captan	Organochlorine	71	80	91
Endosulfan I	Organochlorine	87	86	102

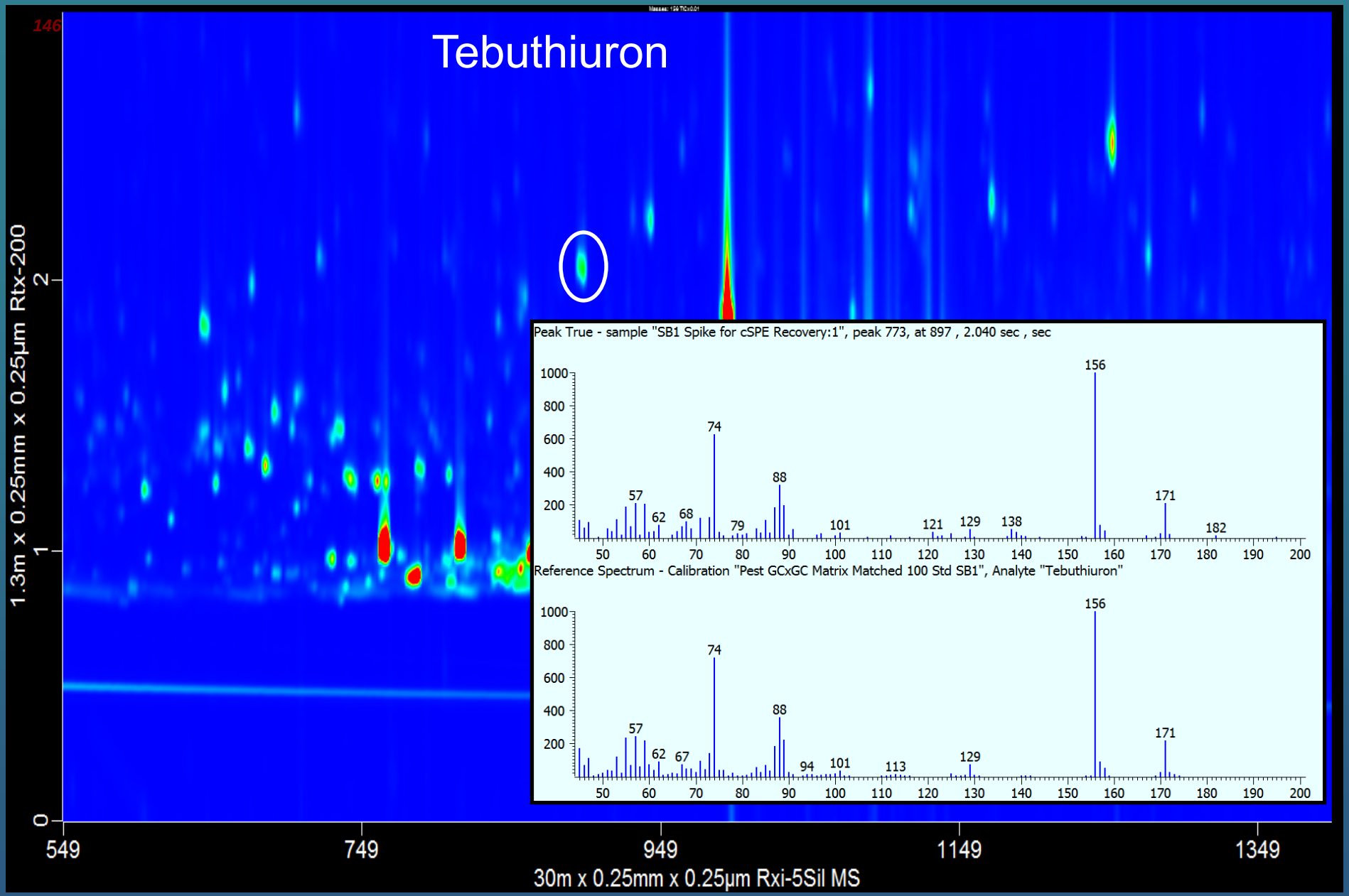
Pesticide Recoveries for Marijuana Spikes

Pesticide	Classification	SB1 cSPE	SB3 Q + cSPE	S3 Q + cSPE
Imazalil	Organonitrogen	83	77	91
Endosulfan II	Organochlorine	86	80	113
Endosulfan sulfate	Organochlorine	82	88	105
4,4'-DDT	Organochlorine	83	77	99
Bifenthrin	Pyrethroid	82	86	96
Dicofol	Organochlorine	40	84	73
Azinphos methyl	Organophosphorus	92	79	97
cis-Permethrin	Pyrethroid	72	64	91
trans-Permethrin	Pyrethroid	52	68	90
Cypermethrin	Pyrethroid	I	I	89
Deltamethrin	Pyrethroid	77	68	99

I = incurred pesticide

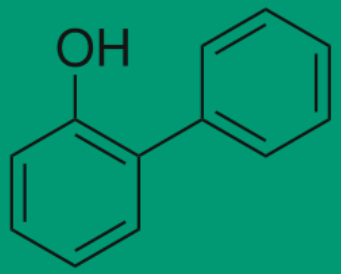


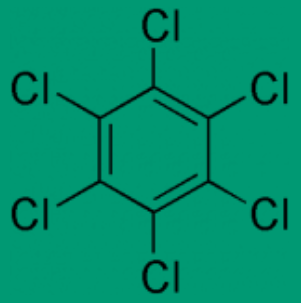




S1

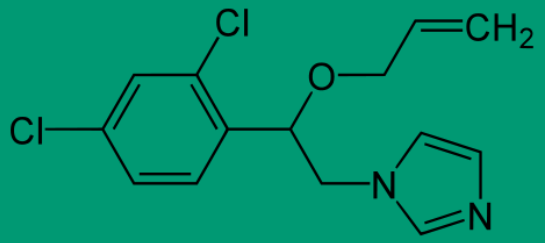
Pesticide	ppb
o-Phenylphenol	190
Hexachlorobenzene	23
Imazalil	1100

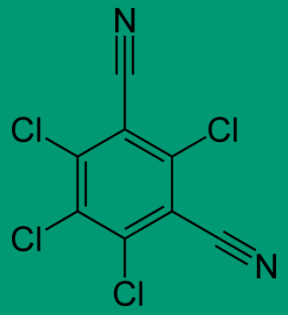




S2

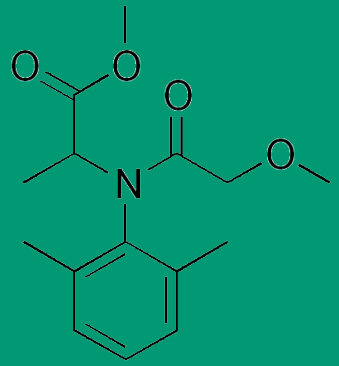
Pesticide	ppb
o-Phenylphenol	190
Chlorothalonil	330
Metalaxyl	400

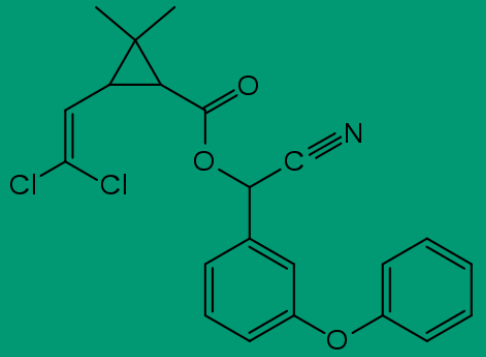




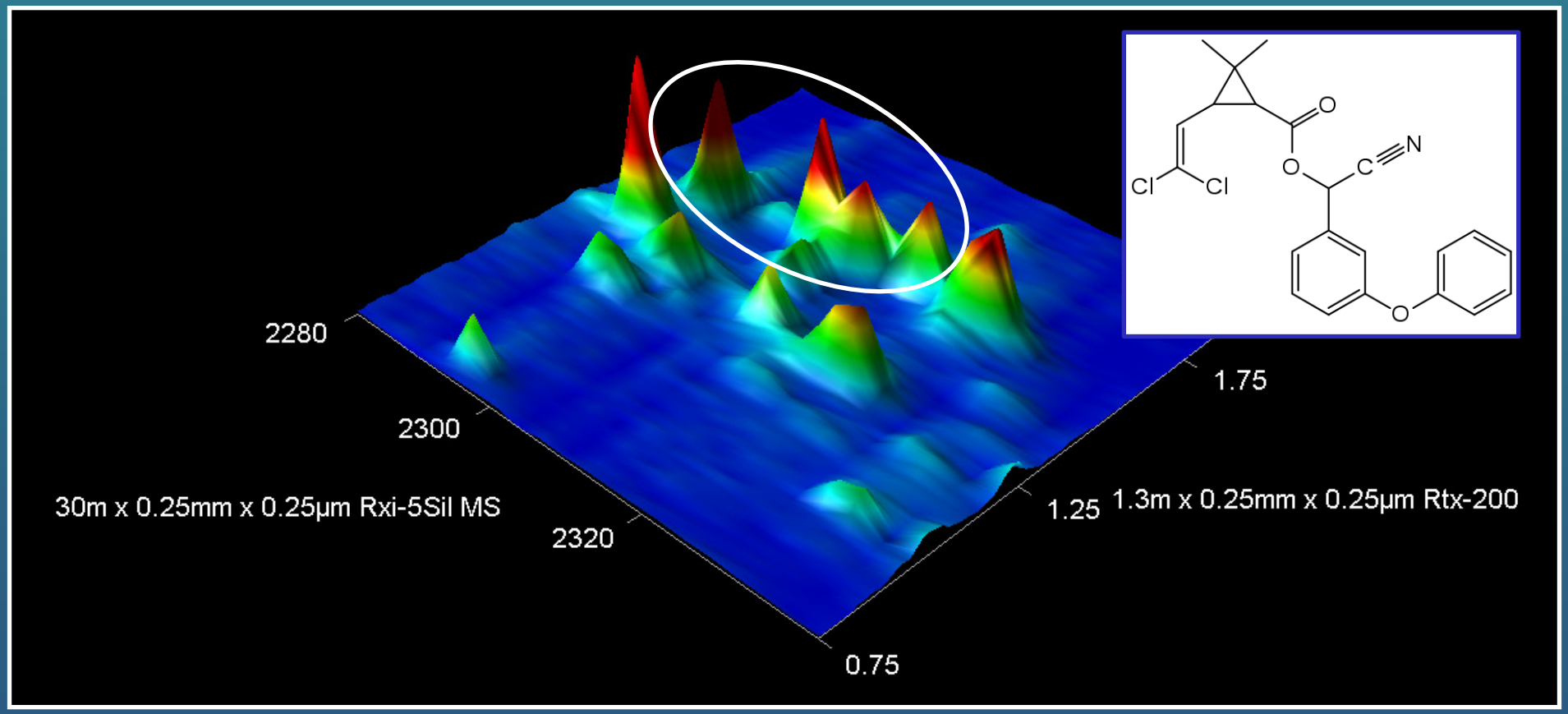
SB1

Pesticide	ppb
o-Phenylphenol	58
Chlorothalonil	29
Cypermethrin	2200



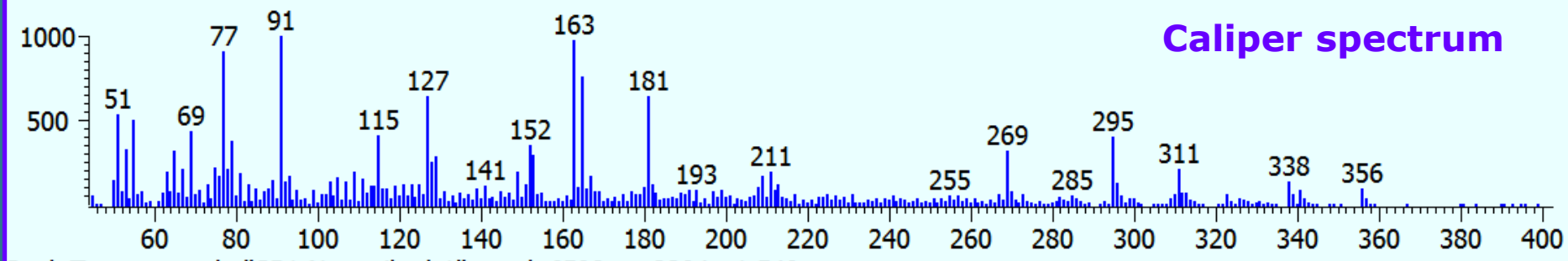


Incurred Cypermethrins in Marijuana
m/z 163 – Quantification Ion



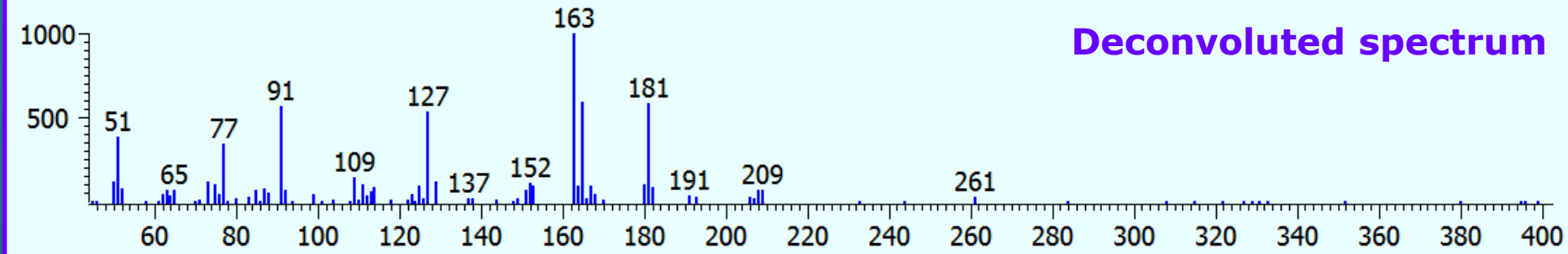
Incurred Cypermethrin in Illicit Marijuana – QuEChERS GCxGC-TOFMS

Caliper - sample "SB1 Not spiked:1", 2304 , 1.540 sec , sec to 2304 , 1.540 sec , sec



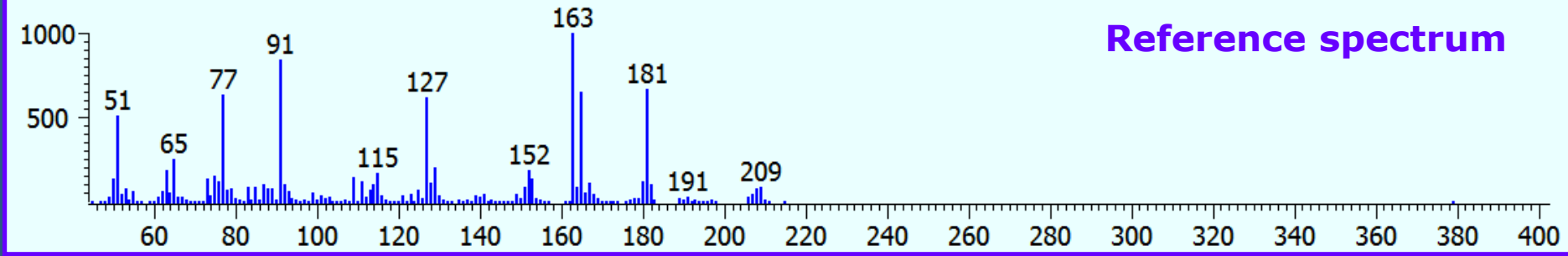
Caliper spectrum

Peak True - sample "SB1 Not spiked:1", peak 6598, at 2304 , 1.540 sec , sec



Deconvoluted spectrum

Reference Spectrum - Calibration "Pest GCxGC Matrix Matched 100 Std S2", Analyte "Cypermethrin 2"



Reference spectrum

Incurred Pesticides in Marijuana Sample 2Q2

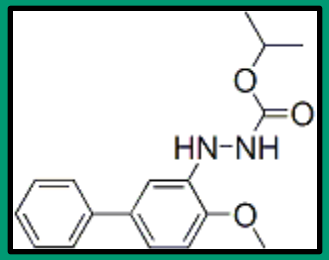
Pesticide	LC	GC
Imazalil	410	NA
Bifenazate	1100	2180
Piperonyl butoxide	37	41
trans-Permethrin	660	1100
cis-Permethrin	1200	690
o-Phenylphenol	NA	280
4,4'-DDE	NA	30

NA = not analyzed by this method

"Effervescent Health Formula"

Bifenazate

Chemtura AgroSolutions



Summary

- QuEChERS is a viable extraction approach for cannabis, but cartridge SPE cleanup necessary
- GCxGC-TOFMS was very helpful in pesticide determinations in cannabis
  - Sample extracts are VERY complex
  - Detectability boost through thermal modulation process

« Some answers on the Chromatography challenge posted before: Does a GC-capillary column produce different retention times when installed in the opposite direction?
LC/MS/MS Analysis of Synthetic Cannabinoid Metabolites in Urine – The Saga Begins »

First QuEChERS Extraction of Marijuana with GCxGC-TOFMS Analysis, dudes…

April 20th, 2011 by Jack Cochran

My Restek colleagues Julie Kowalski, Michelle Misselwitz, and Amanda Rigdon, along with Professor Frank Dorman from The Pennsylvania State University (PSU), report here what we believe is the first QuEChERS extraction of marijuana, with subsequent analysis using GCxGC-TOFMS. We were assisted in this task by Randy Hoffman, a Police Officer Specialist/Evidence Technician at PSU, who very kindly donated the samples confiscated from some students who probably should have had their minds on class, not grass.

Our interest in this topic is mainly about medicine, since at least 15 states (Pennsylvania is not one of them) and Washington DC have enacted laws to legalize medical marijuana. When you fill your prescription, how do you know your remedy is active (potency, or cannabinoid content), pesticide-free, and without bacteria or mold or fungus? Well, you probably don't, but eventually FDA might get involved and we'll need good, robust analytical methods, especially for pesticide analysis. We think that you might be able to do one extraction for both potency and pesticide determinations and we're high on QuEChERS, so we went for it.

First, the potency work, or cannabinoids determination. Although you don't need GCxGC for the BIG THREE (cannabidiol, Δ^9-THC, cannabinol; by the way, Restek has a reference material containing these compounds…), we used it to illustrate one of the benefits of that technique, the structured chromatogram. In the first figure below, the GCxGC contour plot (or chromatogram), you can see that compound classes position themselves in certain areas. This helps identification, and makes discovery of new compounds within classes a bit easier (e.g. perhaps there are undiscovered cannabinoids out there with medicinal benefits). Zooming in, we can see the terpenoid classes, which are thought to have therapeutic effects. Finally, you can see the cannabinoids, including cannabidiol, one of much interest given that "it has been shown to relieve convulsion, inflammation, anxiety, and nausea, as well as inhibit cancer cell growth" (http://en.wikipedia.org/wiki/Cannabidiol).

We quantified cannabidiol (CBD), Δ^9-THC (THC), and cannabinol (CBN) for 4 marijuana samples using QuEChERS and GCxGC-TOFMS and the results are presented in the table below. Since the samples had been stored in an evidence locker for over a year in some cases, the CBN content is relatively high versus fresh marijuana. CBN increases as THC degrades. The THC content is in line with what is typically reported for higher grade illicit marijuana.

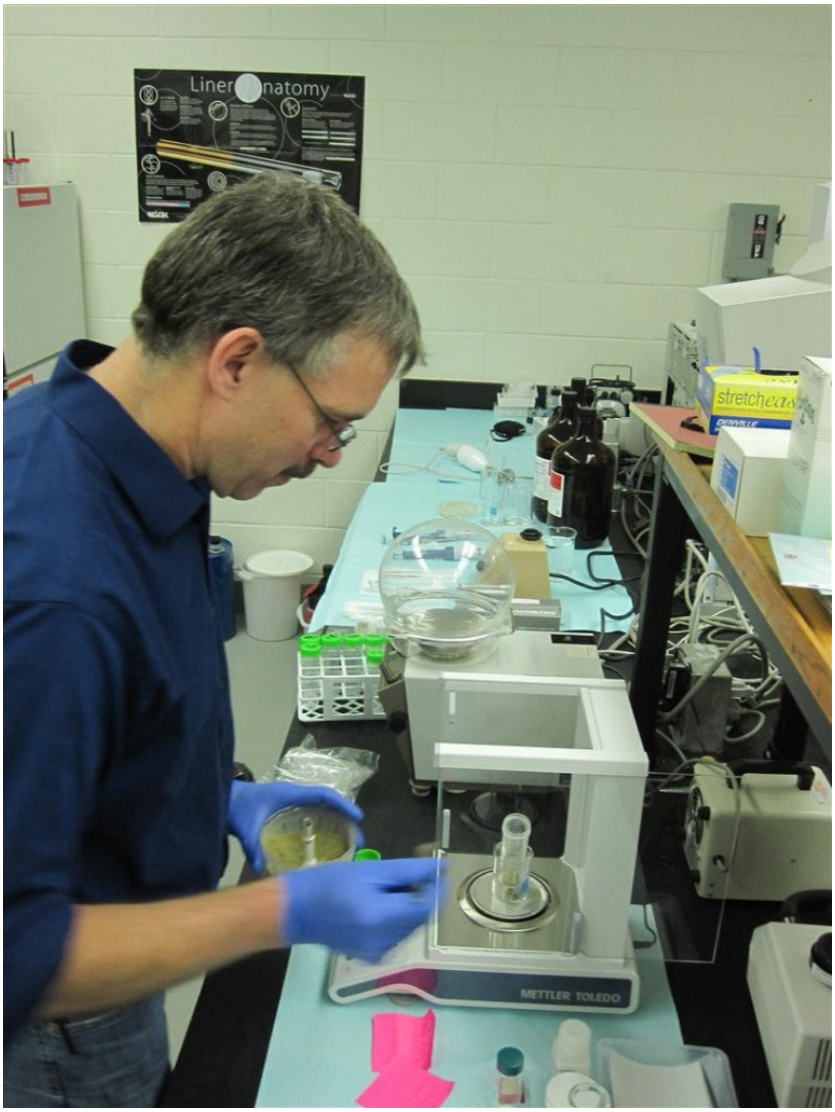
Stay tuned for a report on pesticide analysis of marijuana using QuEChERS and GCxGC-TOFMS. As you might imagine, the extracts are extremely complex, similar to what we saw in our dietary supplements work.

Siezed marijuana for QuEChERS extractions at PSU.

Marijuana for grinding prior to QuEChERS extractions.

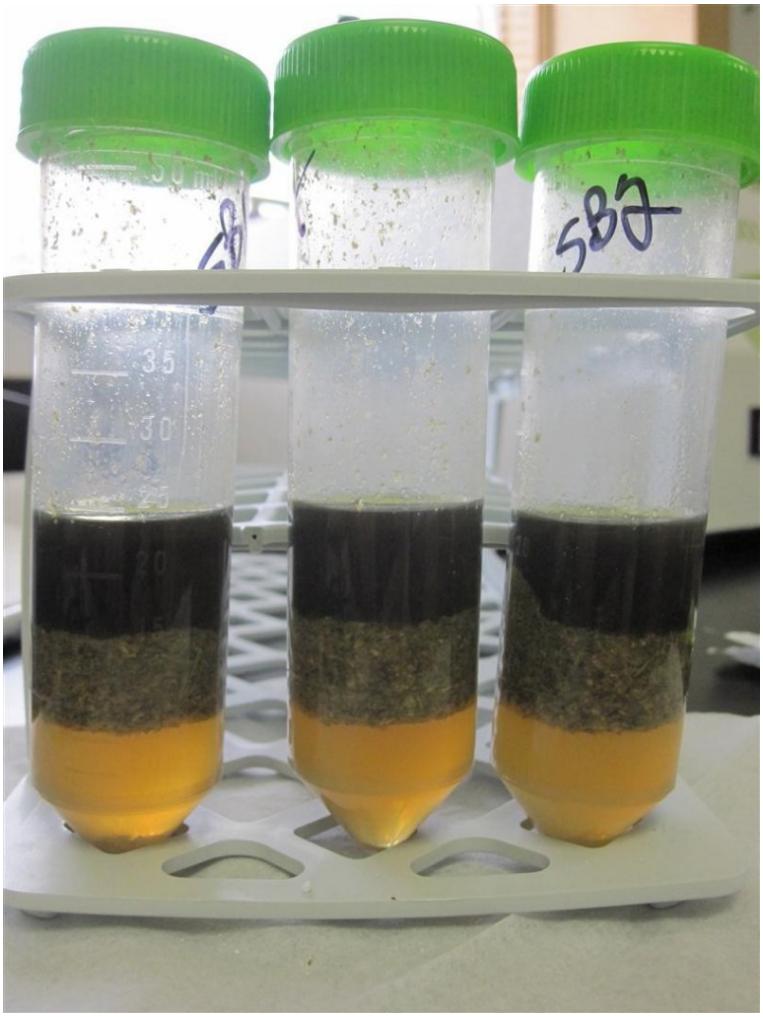
Professor Frank Dorman at Penn State University grinds the goods.

ht

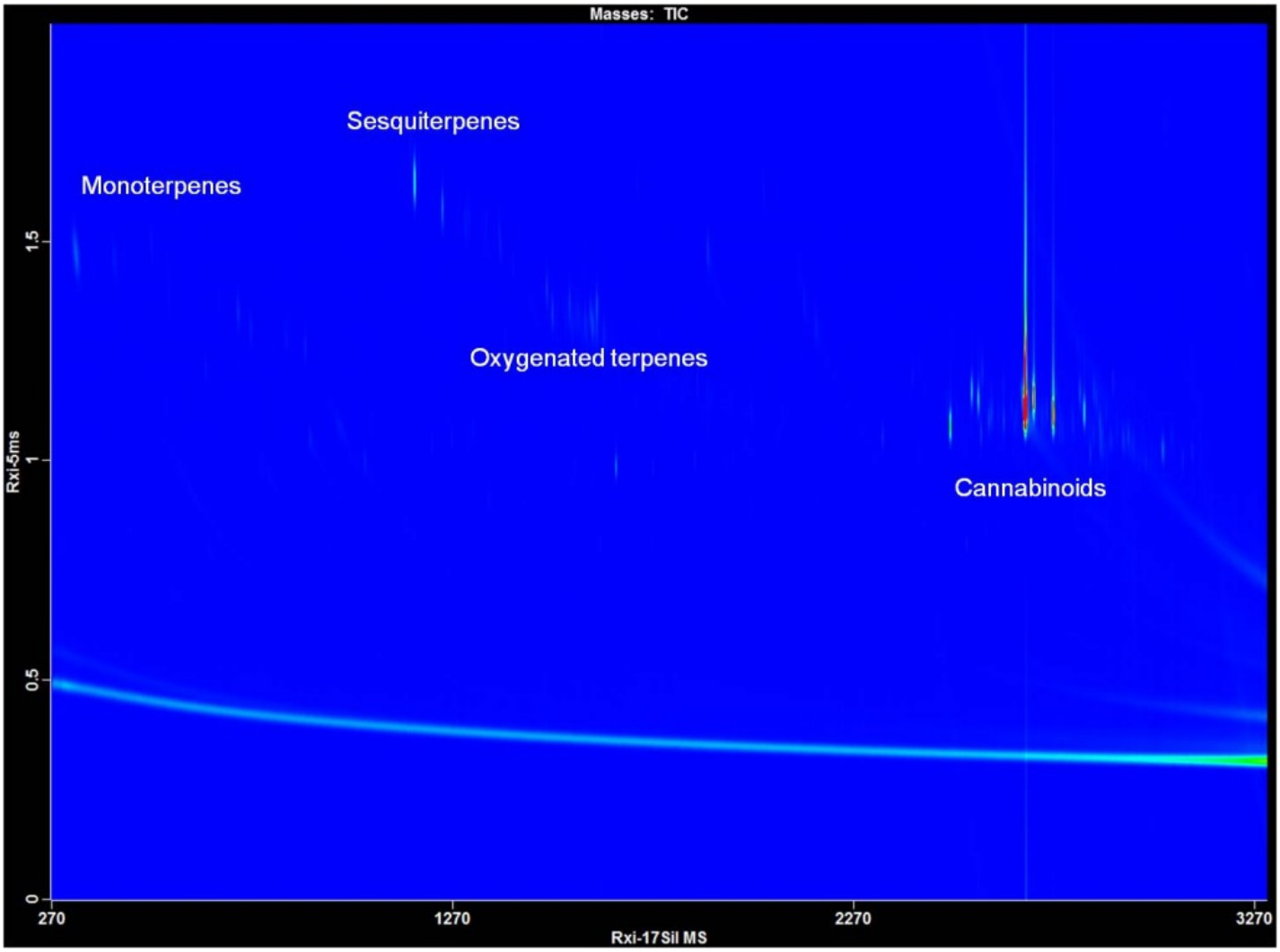


Weighing the marijuana into the QuEChERS extraction tubes. It is full of static!

h

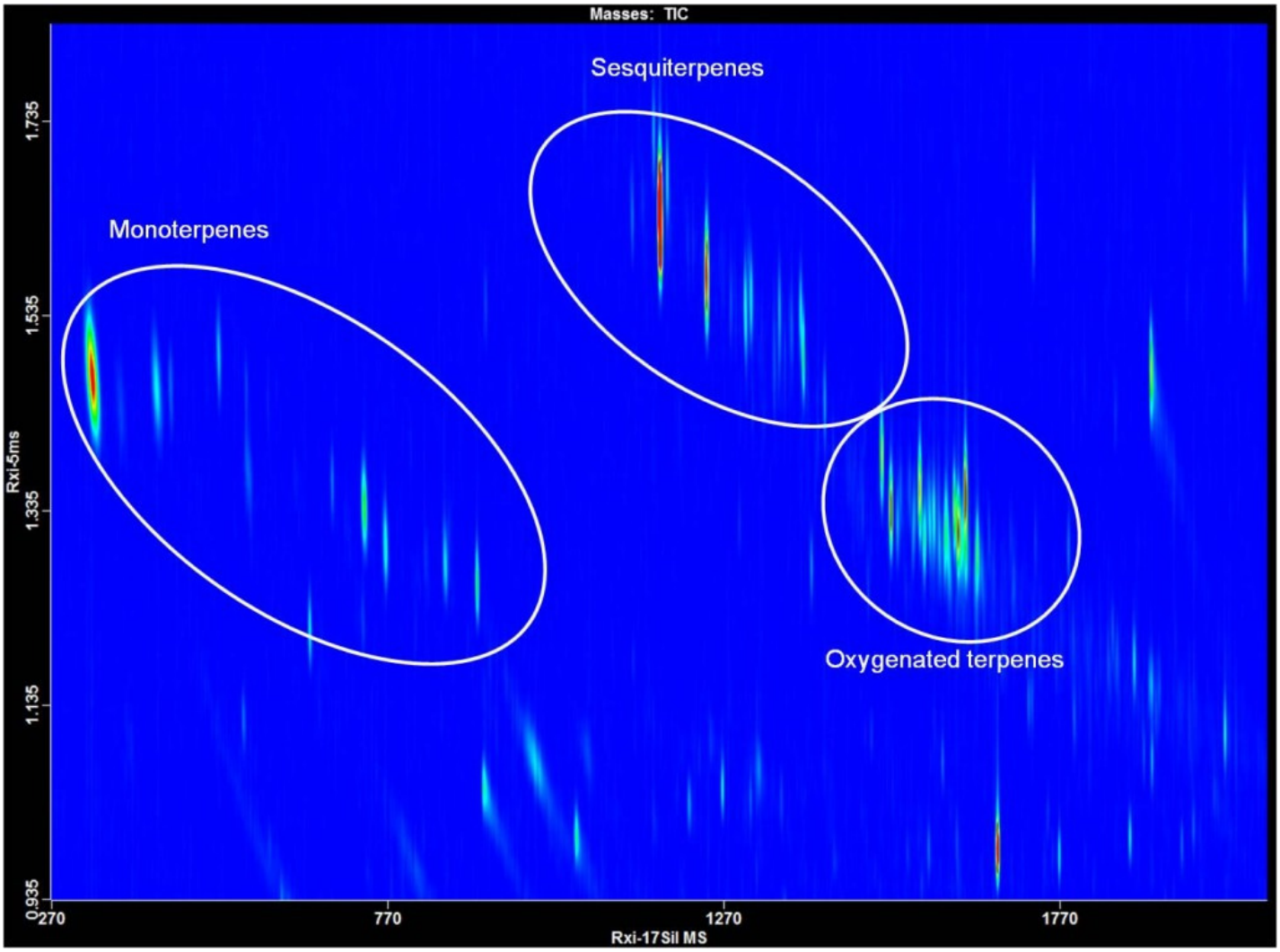


The first QuEChERS extracts of marijuana. They are almost black, and are very complex.

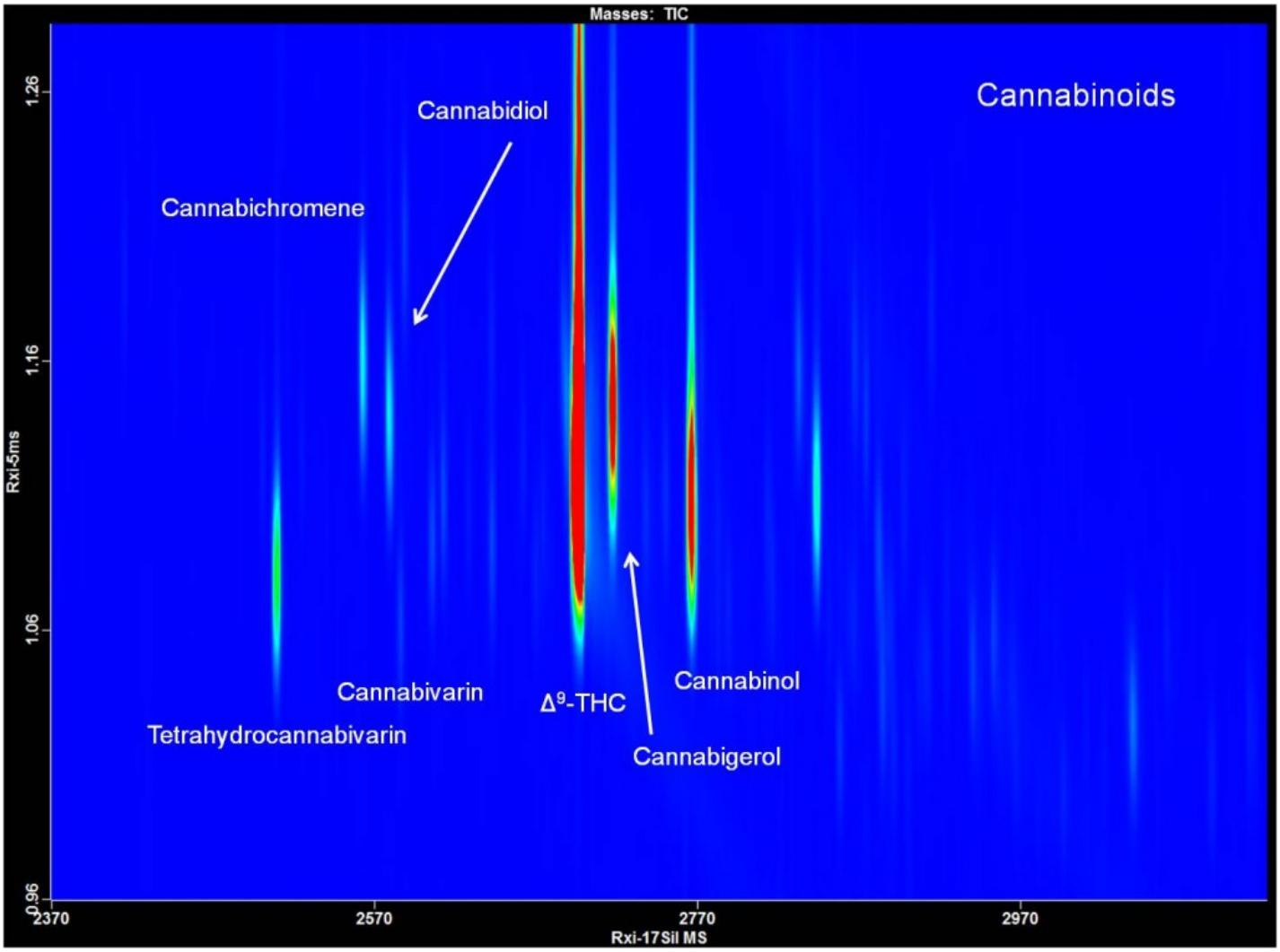


GCxGC-TOFMS contour plot of QuEChERS marijuana extract showing "structured chromatogram", where compound classes elute in certain regions. Rxi-17Sil MS x Rxi-5ms column combination.

h)



Zooming in on the GCxGC terpenoid region for QuEChERS extracts of marijuana.



The cannabinoid region of the GCxGC chromatogram of a QuEChERS extract of marijuana.

Sample	CBD	THC	CBN
S1	0.029	12	1.1
S2	0.016	4.3	1.3
S3	0.034	9.0	1.3
SB9	0.15	10	1.7

Cannibinoid results in percent for samples of marijuana analyzed by QuEChERS and GCxGC-TOFMS.

This entry was posted on Wednesday, April 20th, 2011 at 11:14 pm and is filed under GC/MS, QuEChERS, GCxGC, Medical Marijuana. You can follow any responses to this entry through the RSS 2.0 feed. You can leave a response, or trackback from your own site.

6 Responses to "First QuEChERS Extraction of Marijuana with GCxGC-TOFMS Analysis, dudes…"

1. Josh Wurzer says:
May 4, 2011 at 10:13 pm

Great work! I have been doing a Quechers extraction of cannabinoids for edible food products as well as a Quechers extraction of cannabis flowers for pesticide residue testing for over a year. Our laboratory tests medical cannabis samples in California. We would love to see restek offer standards for CBG, THCV, CBC, as well as THCA and CBDA (THCA and CBDA are the major cannabinoid constituents of raw cannabis flowers but are degraded into THC and CBD on a GC column).

I would love to share some data with your group if you are interested.

Thanks,
Josh Wurzer
Laboratory Director
SC Laboratories Inc.

2.  *Jack Cochran* says:
May 12, 2011 at 1:55 am

Greetings Josh!

Thanks so much for your kind comments and for letting me know of your use of QuEChERS. I'll forward your reference materials request to our standards group. I do know they are interested in expanding the line, but some of those neat compounds are SO expensive. I'm just getting ready to post on our pesticide results for the illicit marijuana we extracted. We found numerous pesticides, so the work turned out to be quite interesting. The samples are unbelievably complex, and needed a multidimensional technique, in this case, GCxGC, for the quantitative effort.

Regards,

Jack

3.  *Blake Meinert* says:
May 19, 2011 at 8:45 pm

Hello Jack,

I have had substantial experience analyzing for chlorinated pesticides in soil and water, but I had not considered their use in marijuana crops. What pesticides did you primarily see? Is there one primary pesticide of choice with growers that you know of? This is interesting.

Thanks,
Blake

4.  *Jack Cochran* says:
May 23, 2011 at 2:47 pm

Hi Blake:

The pesticides we saw on our small sample size were o-phenylphenol, hexachlorobenzene, metalaxyl, chlorothalonil, imazalil, and cypermethrin. Interestingly, all are fungicides except for the insecticide, cypermethrin. Mold/fungus apparently is a big problem for marijuana that is being dried/stored, so maybe this finding isn't surprising.

With the small sample size we had, and the fact that all of our samples were illicit marijuana, I'm not sure if there is a "primary pesticide of choice". At least with the medical marijuana, it may be that bifenazate (Floramite) and abamectin (Avid) are the "pesticides of choice" to control spider mites in indoor grow operations. But since this doesn't seem to be a well regulated area yet, I'm not sure if we know what to expect as regards pesticide use.

Jack

5. *High Quality Analysis of Pesticides in Marijuana using QuEChERS, cartridge SPE cleanup, and GCxGC-TOFMS « ChromaBLOGraphy* says:
May 22, 2011 at 3:28 am

[…] we reported on what we believe is the first application of QuEChERS for marijuana, using it for potency analysis with GCxGC-TOFMS. Ultimately, the plan was to determine pesticides […]

6. *The Bard Hits the Bong? « ChromaBLOGraphy* says:
July 2, 2011 at 9:47 pm

[…] cannabis to (1) develop methods for possibly fingerprinting marijuana types, (2) characterize marijuana potency, and (3) analyze for pesticides in marijuana with GCxGC-TOFMS and […]

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« Difficult QuEChERS extracts: Removing fatty acid interferences
Even More Technical Service "Red Flags" – GC »

Accurate Quantification of Cannabinoid Acids and Neutrals by GC – Derivatives without Calculus

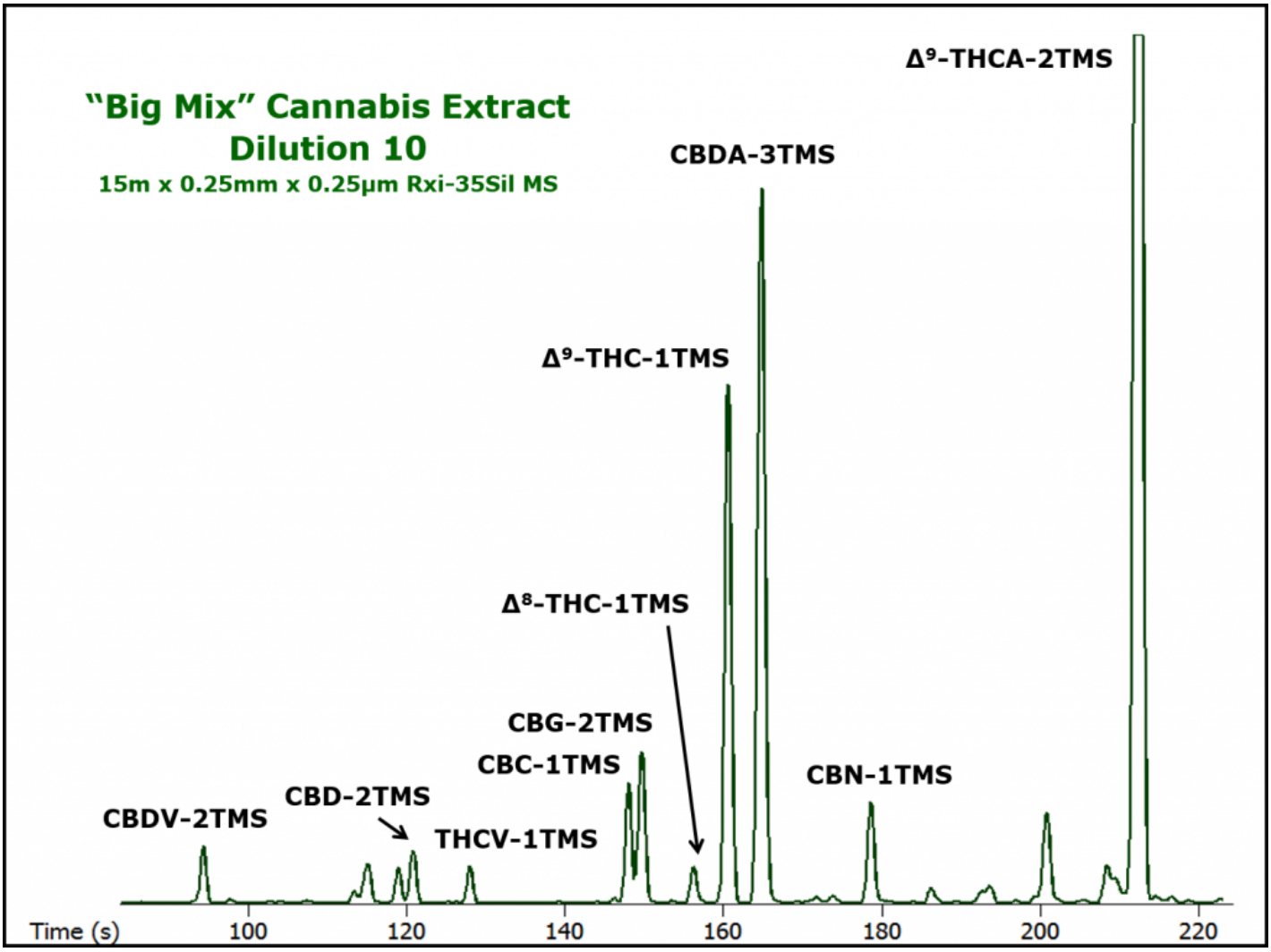
September 9th, 2015 by Amanda Rigdon

Derivatization is a widely-used technique for GC sample preparation across many industries and in widely varied matrices from soil to plastics to blood that is used to make polar and active compounds more amenable to good GC analysis. If you're careful about testing your derivatization procedure during method development, you can be confident that you'll have a reproducible method that can vastly improve the quality of your GC results. While derivatization does require some extra sample handling, the procedure I developed for cannabis plant matrix is very straightforward and easy to perform:

Derivatization Procedure for Cannabis and Hemp Plant Matrices:

- Place 100µL of plant extract into a 1mL Micro-Vial
- Evaporate to dryness at 50°C under a gentle stream of nitrogen
- Add 50µL ethyl acetate and 50µL BSTFA + 1% TMCS
- Incubate at 70°C for 30 minutes
- Cool and dilute with ethyl acetate if desired

In my last blog, I introduced the concept of derivatization for use in cannabis or hemp cannabinoid testing. Once acidic cannabinoids are derivatized, they no longer break down in the GC inlet and can be quantified separately from the neutral cannabinoids. I demonstrated this through derivatization of high-level solvent standards, but work with solvent standards is a far cry from matrix work, which means the procedure needed to be tested in matrix. To kick off the matrix test, I spiked an extract with the most common cannabinoids of interest, derivatized it using the procedure listed above, and my colleague, Jack Cochran, analyzed it via GC-FID with our Rxi-35Sil MS GC column. We can see that we have a beautiful chromatogram with all of the derivatized cannabinoids separated, and very little matrix interference.



In addition to confirming that all derivatization sites are indeed derivatized by analyzing the standards with GC-MS (this is shown in my last blog), we also tested derivatization efficiency using a cannabis extract previously generated at Penn State University with the help of Professor Frank Dorman and a Police Officer Specialist. Because derivatization is a chemical reaction, the derivatization reagent gets used up during the derivatization reaction. Because plant matrix contains many other derivatizable compounds like sugars and sterols, these other compounds may compete for the derivatizing reagent, possibly resulting in the reagent getting used up before all of our analytes of interest can be derivatized.

So how can we be sure our derivatization is going to completion in the presence of matrix? There are a couple things we can do, the first of which is really simple. We can see in our procedure that we use a hefty 50µL of derivatizing reagent per 100µL of cannabis extract. We know that our extract contains a lot less than 50mg of plant matrix, not all of which is derivatizable. This means that by adding 50mg of BSTFA per 100µL of sample, we can be confident that we have a significant excess of derivatizing reagent as compared to derivatizable groups in our sample. Excess derivatizing reagent means that it will never be completely used up, ensuring the reaction will go to completion no matter what.

A more quantitative way to test derivatization efficiency in a matrix where you can't get blanks is to evaluate analyte linearity with differing amounts of matrix. For example, if you derivatize four THCA-containing samples prepared using 10, 20, 50, and 100µL of cannabis extract and plot the area of THCA versus sample amount, you should end up with a straight line if your derivatization is going to completion. If it's not, then you'll likely see THCA area fall off for the samples containing more matrix since the derivatization reagent is being used up before all the analyte in the higher matrix level sample is derivatized. To test our procedure, we did just that. We can see that our linearity looks beautiful for all of the cannabinoids, indicating the derivatization does indeed go to completion.

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Terpenes in Impinger Extracts of Kryptonite and Blueberry Strains of Medical Cannabis

March 17th, 2014 by Jack Cochran

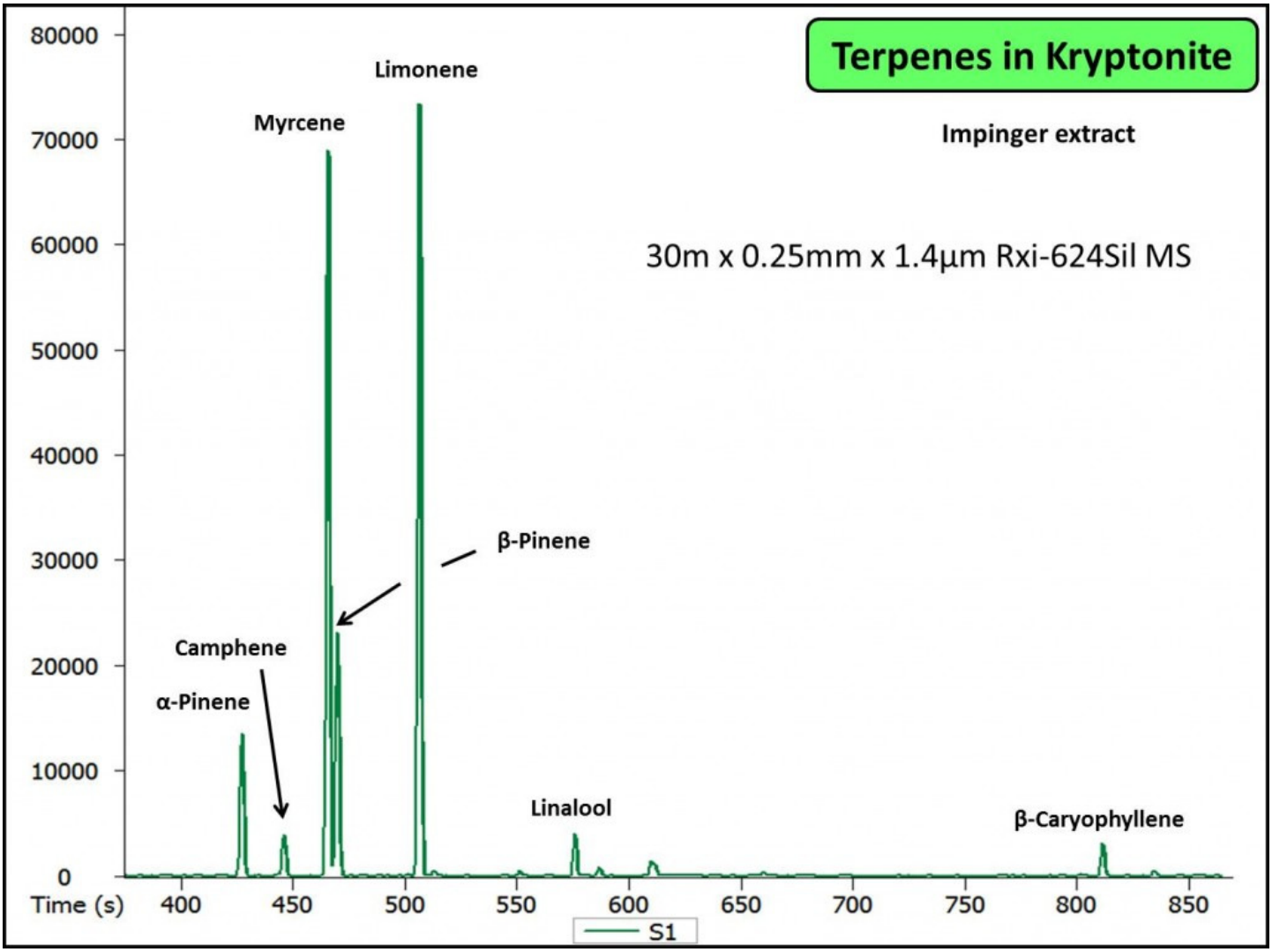
As noted in my earlier post, Terpenes in Medical Cannabis, terpenes are an important class of aroma compounds that may contribute to the medicinal benefits of cannabis, via the so-called "entourage effect". I profiled some of the terpenes listed as important for medical cannabis using our 30m x 0.25mm x 1.40µm Rxi-624Sil MS, achieving a promising separation on a standard I put together. Shown below are some impinger extracts provided by SRI Instruments for Kryptonite and Blueberry strains of medical cannabis. Importantly, these extracts do NOT contain any cannabinoids, which would elute late, if at all, from the thick-film 624Sil MS column, nor do they contain chlorophyll, another compound that plays havoc with GC inlet liners and stationary phases. Part of the beauty of headspace extraction techniques for terpenes is leaving the involatile material behind, and in this case, compressed air was used to sweep the terpenes from the cannabis to a vial containing methanol for trapping the terpenes.

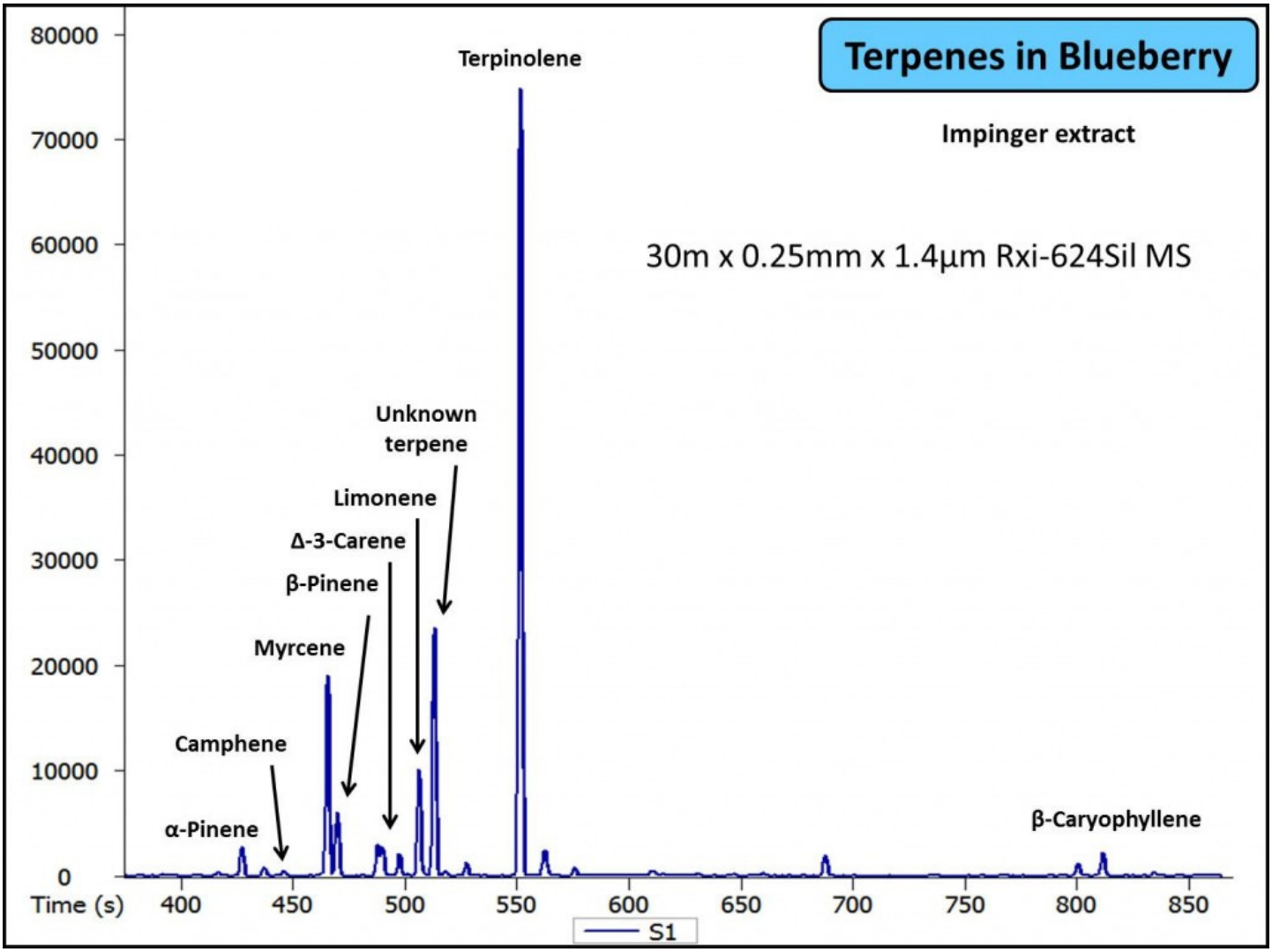
As you look at the chromatograms below, it is important to note that this is ONLY qualitative work at this point and that different headspace methods (e.g. purge-and-trap, static headspace, SPME, etc.) could yield much different chromatograms. Solvent extraction or steam distillation, would likely be even more different, including resulting in more intense peaks for later eluting (less volatile) terpenes. The point of this work is to show initial efforts to characterize chromatographic elution order for some medical marijuana terpenes and analyze the first "real world" samples to show how terpene profiles for different medicines can be dissimilar.

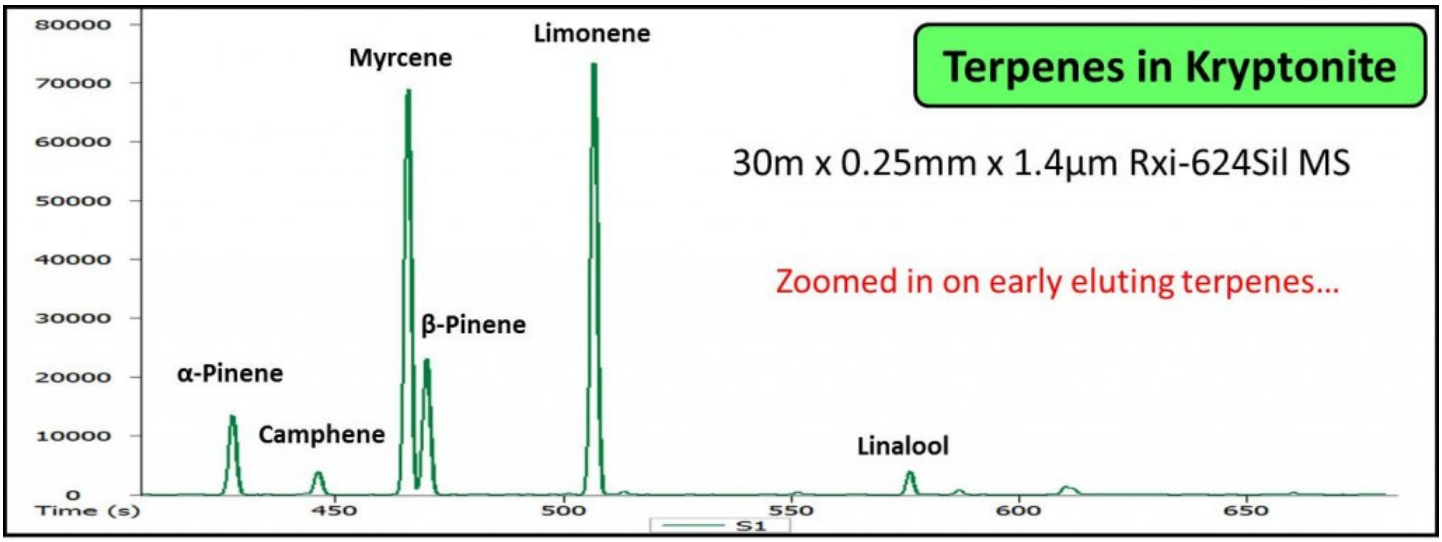
Take a look back on the GCxGC-TOFMS work that shows very nice multidimensional separations of terpenes, sesquiterpenes, and oxygenated terpenes in a solvent extract for cannabis.

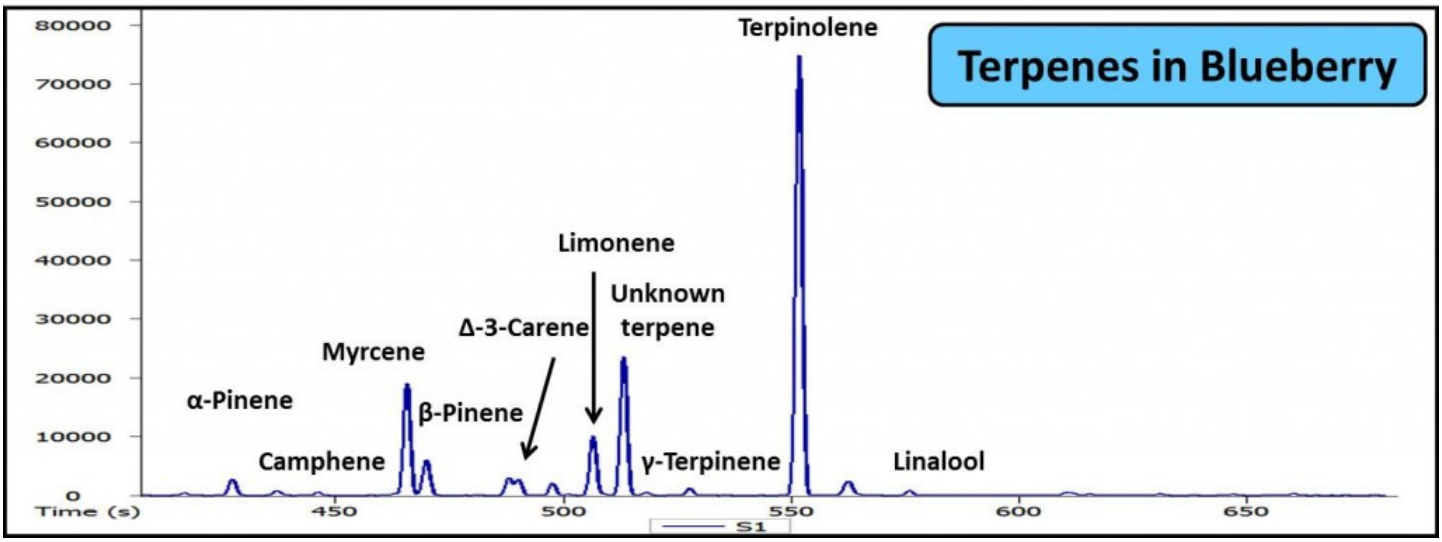
Restek continues to support the medical cannabis analysis community with GC and LC columns, accessories, and reference materials. Check out our Medical Marijuana web page.

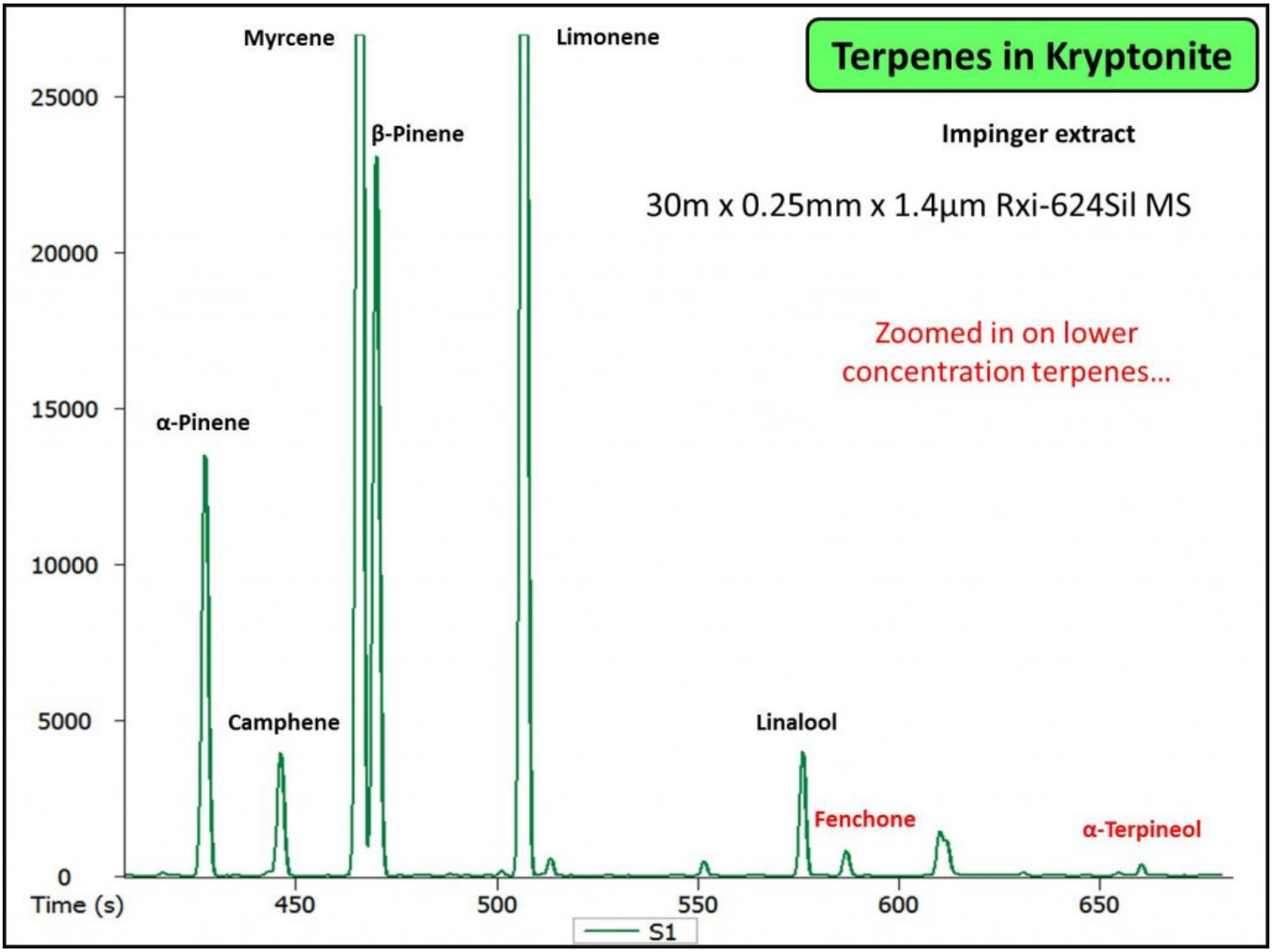
ht

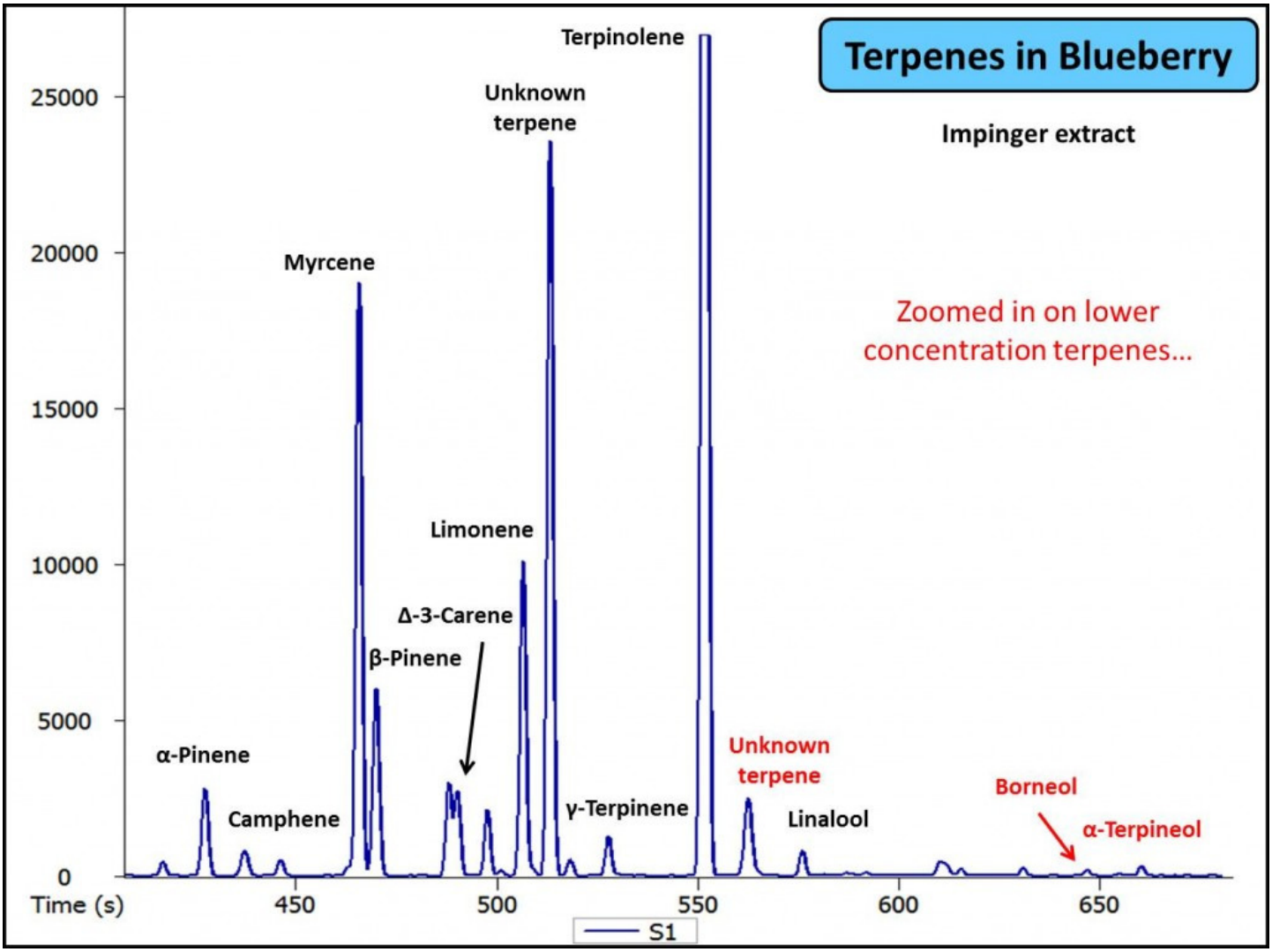












This entry was posted on Monday, March 17th, 2014 at 6:32 pm and is filed under QuEChERS, GCxGC, Medical Marijuana. You can follow any responses to this entry through the RSS 2.0 feed. You can leave a response, or trackback from your own site.

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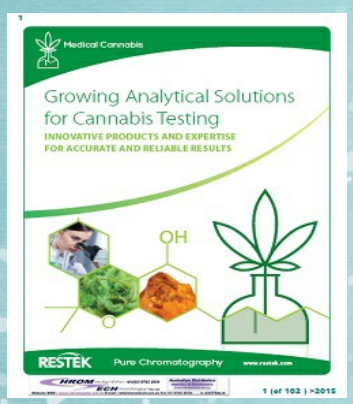

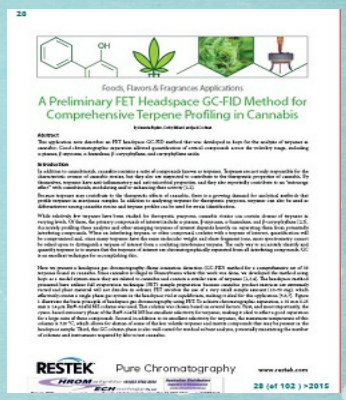

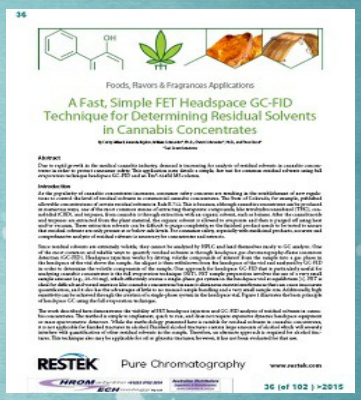

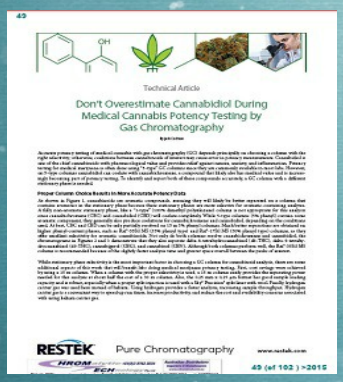

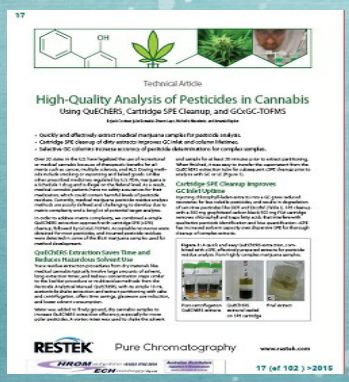

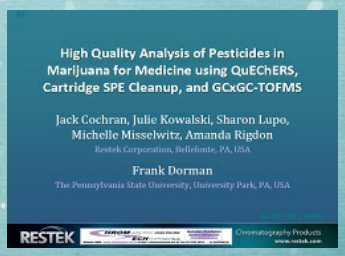

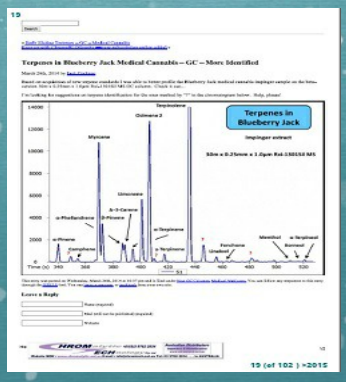

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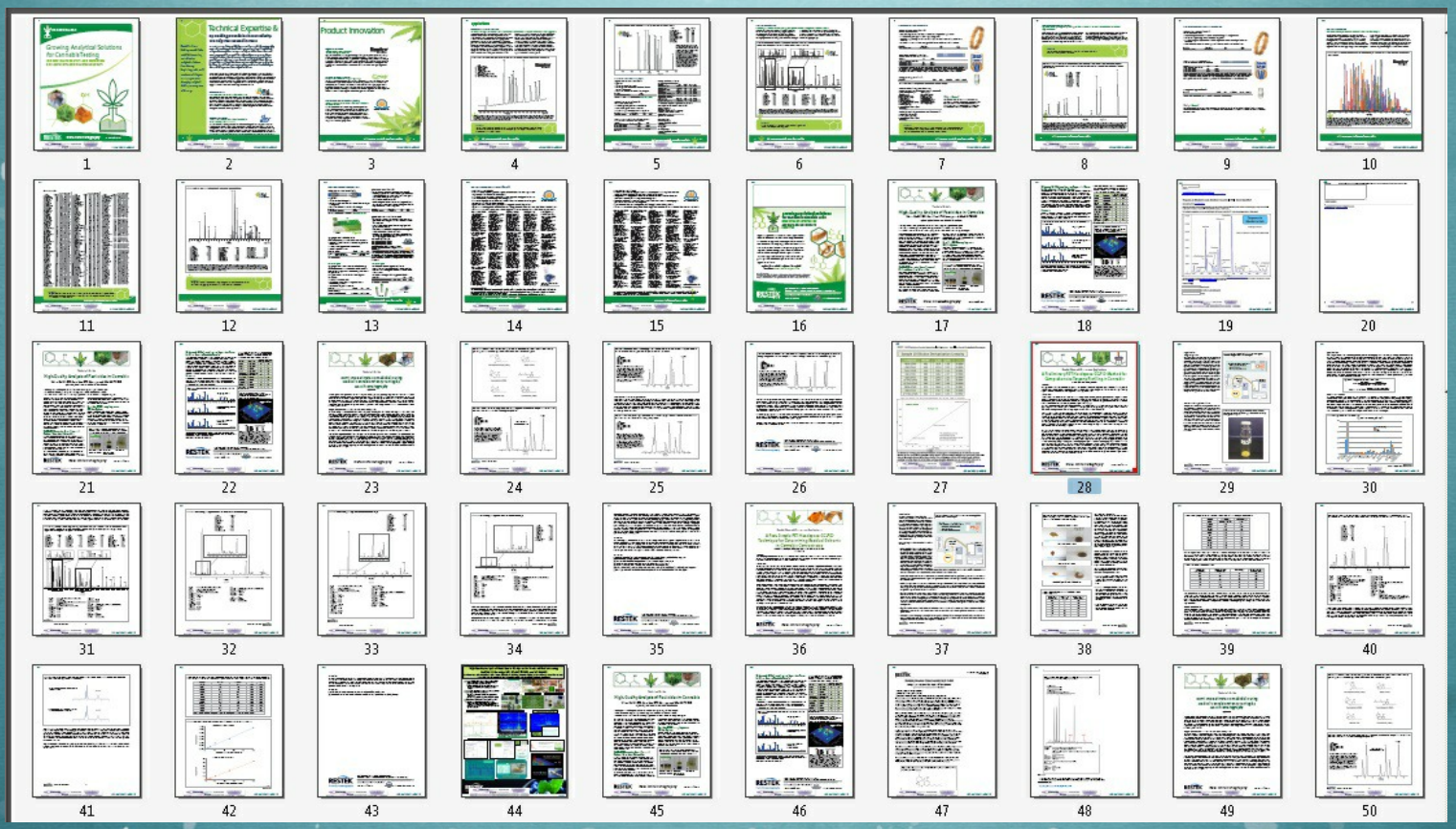

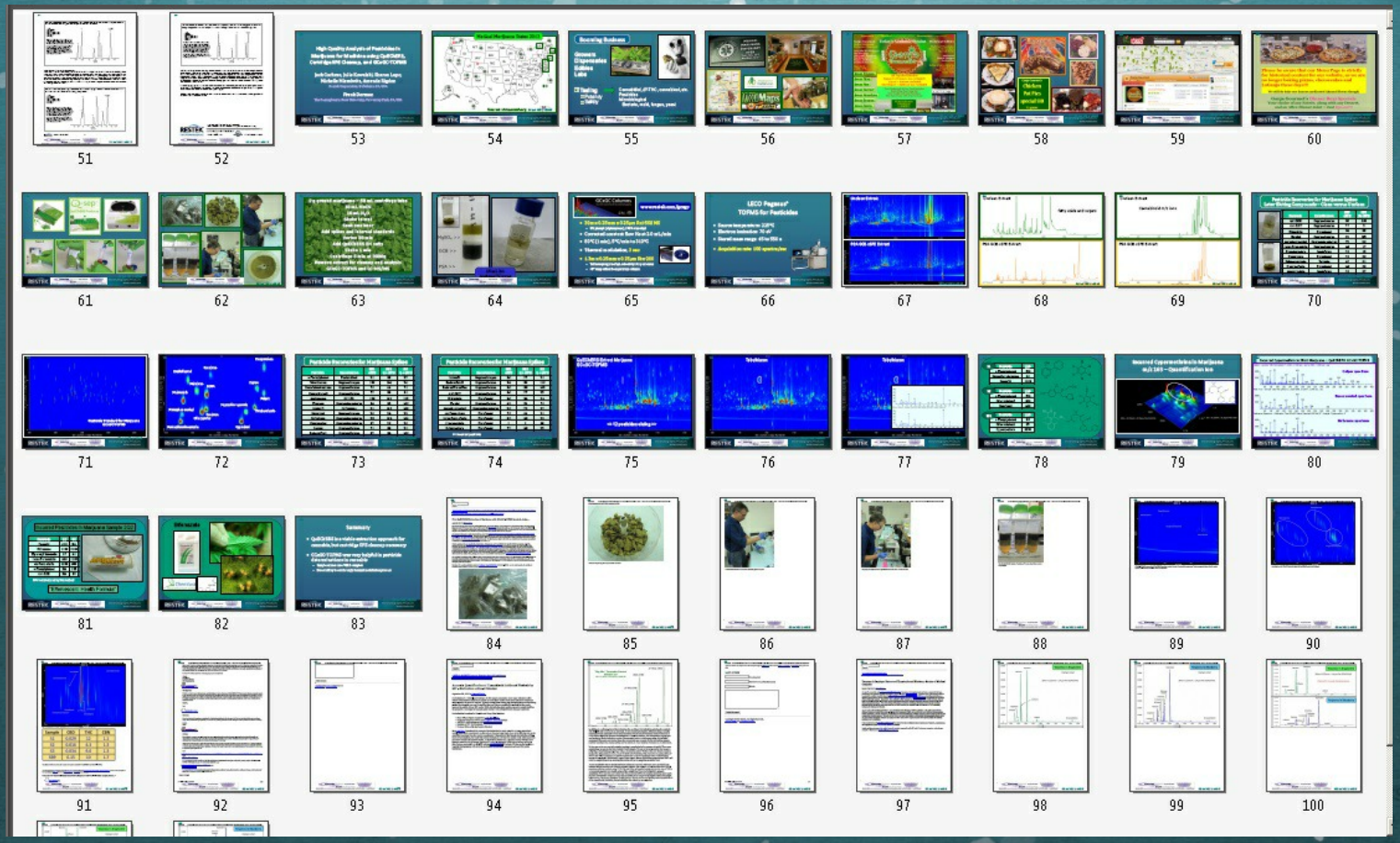

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Restek APPLICATIONS : MEDICAL CANNABIS . . . 102p CT-republished >2015

Hints - DISCLAIMER

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2 A Preliminary FET Headspace GC-FID Method for Comprehensive Terpene Profiling in Cannabis_1-8_FFAN2045-UNV 8p p28

3 Solvents in Cannabis Concentrates_1-8_FFAN2009A-UNV p36

4 High Quality Analysis of Pesticides in Marijuana for Food and Medicine using Quechers, Cartridge SPE, GCxGC-TOFMS and LC-MS-MS_MJrafa2011_med_mj_1-1

5 Don't Overestimate Cannabidiol During Medical Cannabis Potency Testing by GC_FFAR1954-UNV_1-4

6 High-Quality Analysis of Pesticides in Cannabis Using Quechers Cartridge SPE Cleanup and GCcxGC-TOFMS_FFAR1950-UNV_1-2 p49 2p

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7 High Quality Analysis of Pesticides in Marijuana for Medicine using Quechers, Cartridge SPE Cleanup and GCxGC-TOFMS_Cannabis_Pittcon2013_1-31

BLOGS . . . *& Ongoing !*

8 Accurate Quantification of Cannabinoid Acids and Neutrals by GC - Derivatices without Calculus - Blog

9 Terpenes in Impinger Extracts of Kryptonite and Blueberry Strains of Medical Cannabis.

10 Terpenes in Blueberry Jack Medical Cannabis - GC - More Identified

See SRI GCs-Cannabis - for some h'ware related Custom GCs and accessories

Restek prolific & on-going effort (& societies in general) " a work in progress" - and a potential "drug of least harm" and "potent"ial beneficial . . . even when / and for Australia when we wake up to reality R&D sure! but QC is the issue & Restek has (at least some of) THE answers ! Hints Disclaimer see flip.chromalytic.net.au/books/gydm/

Resources

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High Quality Analysis of Pesticides in Marijuana for Medicine Using QuEChERS, Cartridge SPE Cleanup, and GCxGC - TOFMS (PDF)

.....

A Preliminary FET Headspace GC-FID Method for Comprehensive Terpene Profiling in Cannabis (PDF)

... for Comprehensive Terpene Profiling in **Cannabis** Abstract This application note describes an FET headspace GC -FID method that was developed in hops for the analysis of terpenes in **cannabis**. Good chromatographic...

High Quality Analysis of Pesticides in Marijuana Using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS (PDF)

...High-Quality Analysis of Pesticides in **Cannabis** Using QuEChERS, Cartridge SPE Cleanup, and GCx in the U.S. have legalized the use of recreational or medical **cannabis** because of therapeutic benefits...

A Fast, Simple FET Headspace GC-FID Technique for Determining Residual Solvents in Cannabis Concentrates (PDF)

... for Determining Residual Solvents in **Cannabis** Concentrates By Corby Hilliard; Amanda Rigdon; William Schroeder **cannabis** industry, demand is increasing for analysis of residual solvents in **cannabis** concentrates in order...

Medical Marijuana

We have the industry's most comprehensive selection of cannabinoid-related certified reference materials (CRMs), manufactured and QC tested in our ...

Don't Overestimate Cannabidiol During Medical Cannabis Potency Testing by Gas Chromatography (PDF)

...Don't Overestimate Cannabidiol During Medical **Cannabis** Potency Testing by Gas Chromatography By Jack Cochran Accurate potency testing of medical **cannabis** with gas chromatography (GC) depends...

ChromaBLOGraphy: Terpenes in Medical Cannabis

Thanks to a colleague at SRI Instruments who sent me some terpenes, and with the use of other terpenes I had in house, I was able to collect numerous chromatograms of these compounds that may contribute to the "entourage effect" of medical cannabis. This means they may have therapeutic effects in their own right, or as synergists with cannabinoids. […]

ChromaBLOGraphy: Terpenes in Cannabis – to MS or not to MS?

In my last blog, I showed how FID is a more suitable detection method for cannabis residual solvent analysis than MS. But what about terpene analysis? Can our old friend the FID hold its ground against the mighty mass spectrometer for this application? Actually, it can! Terpenes are much larger molecules than residual solvents, so […]

ChromaBLOGraphy: Do I smell Cannabis in the Lab?

Last week I was traveling in Europe to present seminars on practical topics like trace analysis, faster analysis and troubleshooting. During such seminars you visit companies and you always learn something. One of the companies we visited was a company that did forensic analysis and were specialized in cannabis measurement. My colleague already explained to […]

Growing Analytical Solutions for Cannabis Testing (PDF)

...Medical **Cannabis** Growing Analytical Solutions for **Cannabis** Testing INNOVATIVE PRODUCTS of performance, all Rxi® capillary columns for the **cannabis** industry are manufactured and individually tested...

A Fast, Simple FET Headspace GC-FID Technique for Determining Residual Solvents in Cannabis Concentrates

... **cannabis** industry, demand is increasing for analysis of residual solvents in **cannabis** concentrates in order . **Introduction** As the popularity of **cannabis** concentrates increases, consumer safety concerns ... As the **cannabis** industry expands, demand is increasing for analysis of residual solvents in **cannabis** concentrates in order to protect consumer safety. This application note details a simple, fast...

News: Cannabis Testing Opens Up a Whole New Market

Author(s): Michelle Taylor, Editor-in-Chief Chromatography Techniques / Laboratory Equipment Published By: Chromatography Techniques / Laboratory Equipment Year of Publication: 2015 Links: http://www.chromatographytechniques.com/articles/2015/06/cannabis-testing-opens-whole-new-market http://www.laboratoryequipment.com/articles/2015/06/cannabis-testing-opens-whole-new-market Abstract: Given recent law and attitude changes in the United States, the cannabis industry is on the … Continue

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ChromaBLOGraphy: Residual Solvents in Cannabis – to MS or not to MS?

Over the past few months, I've gotten numerous questions about the best detection method for terpenes and residual solvents in cannabis. It seems that a lot of people are purchasing GC-MS instruments for both of these analyses. While GC-MS is indeed a powerful tool, it's not really necessary for either analysis. In fact, the use […]

ChromaBLOGraphy: Optimization of Cannabis Analyses from the Emerald Conference

Good news, everyone! I've added some comprehensive speaker notes to the talk I delivered at the Emerald Conference about the optimization of cannabis analyses. In this talk, I outlined some easy ways to improve your potency, terpenes, and residual solvents analyses for cannabis. I added the notes in the hope that the talk can be […]

ChromaBLOGraphy: Early Eluting Terpenes – GC – Medical Cannabis

I've already had a request to zoom in on the early eluting part of the previously posted gas chromatogram of medical cannabis terpenes on the Rxi-1301Sil MS so the separations can be better viewed. So here it is… More later…

Pittcon 2016

ChromaBLOGraphy: Analyzing Residual Solvents in Cannabis Concentrates: A Sticky Situation

Along with the increasing demand for various forms of cannabis concentrates comes increased concern regarding residual solvents in these products. In many cases, cannabis concentrates are prepared by extracting either the acidic or decarboxylated forms of cannabinoids from plant material using organic solvents. Some of the solvents used for extraction can have detrimental health effects, […]

News: Restek to Offer Free Cannabis Chromatography Seminar After ACS

Chromatography is a necessary tool for the cannabis business, and cannabis labs can take advantage of Restek's chromatography expertise to make themselves more successful. We have designed a seminar specifically for cannabis labs. Attendees will learn about LC, GC, and … Continue reading →

News: Terpenes Standards for Medical Cannabis Analysis Just Released by Restek

Restek is pleased to announce the release of new multicomponent terpenes standards for medical cannabis analysis. These new blends contain the most important terpenes for cannabis labs and are formulated for maximum stability. High-concentration (2,500 µg/mL) solutions provide value and … Continue reading →

A Preliminary FET Headspace GC-FID Method for Comprehensive Terpene Profiling in Cannabis

... that was developed in hops for the analysis of terpenes in **cannabis**. Good chromatographic separation allowed -caryophyllene, and caryophyllene oxide. **Introduction** In addition to cannabinoids, **cannabis** contains ... of terpenes in **cannabis**. Good chromatographic separation allowed quantification of critical compounds across...

ChromaBLOGraphy: Terpenes in Impinger Extracts of Kryptonite and Blueberry Strains of Medical Cannabis

As noted in my earlier post, Terpenes in Medical Cannabis, terpenes are an important class of aroma compounds that may contribute to the medicinal benefits of cannabis, via the so-called "entourage effect". I profiled some of the terpenes listed as important for medical cannabis using our 30m x 0.25mm x 1.40µm Rxi-624Sil MS, achieving a […]

ChromaBLOGraphy: Cannabis Analysis – We've Come a Long Way, Baby!

A little less than a year ago at Pittcon in Chicago, my colleague Frank Dorman from the Pennsylvania State University and I sat down over beers with Ken Snoke and Wes Burk from Emerald Scientific, a small startup distribution business for cannabis labs. We were joined by Bill and Christi Schroeder and Ted Flood from […]

Don't Overestimate Cannabidiol During Medical Cannabis Potency Testing by Gas Chromatography

...Accurate potency testing of medical **cannabis** with gas chromatography (GC) depends principally and the fastest analysis time, making it the ideal GC column choice for medical **cannabis** potency testing ... Proper GC column choice is essential for accurate and robust medical **cannabis** potency testing...

ChromaBLOGraphy: 1st Annual Medical Cannabis Summit at Pittcon 2014

Happy post-Pittcon, everyone! It was a very productive week, and as always, I had a wonderful time. This year was special for me in that amid the talks, posters, and general bustle of the show, I had the pleasant opportunity to finally meet some folks from the medical cannabis industry face-to-face. Even though Restek has […]

ChromaBLOGraphy: Terpenes in Blueberry Jack Medical Cannabis – GC – More Identified

Based on acquisition of new terpene standards I was able to better profile the Blueberry Jack medical cannabis impinger sample on the beta-version 30m x 0.25mm x 1.0µm Rxi-1301Sil MS GC column. Check it out… I'm looking for suggestions on terpene identification for the ones marked by "?" in the chromatogram below. Help, please!

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Resources

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News: **The Practical Chemist: Calibration Part II – Evaluating Your Curves**

Author: Amanda Rigdon Restek Corporation Published By: Cannabis Industry Journal Year of Publication: 2016 Link: https://www.cannabisindustryjournal.com/column/calibration-part-ii-evaluating-your-curves/ Abstract: Despite the title, this article is not about weight loss – it is about generating valid analytical data for quantitative analyses. In the … Continue reading →

ChromaBLOGraphy: **Important Medical Marijuana Cannabinoids Analyzed by GC-FID on Rxi-35Sil MS and Rtx-35**

In a previous post, "Don't overestimate cannabidiol during medical cannabis potency determinations with gas chromatography. Use stationary phase selectivity for accuracy and hydrogen for fast analysis.", I recommended a 15m x 0.25mm x 0.25µm Rxi-35Sil MS GC column for fast separations of CBC, CBD, delta-8-THC, delta-9-THC, CBG, and CBN with hydrogen carrier gas. This same […]

ChromaBLOGraphy: **Accurate Quantification of Cannabinoid Acids and Neutrals by GC – Derivatives without Calculus**

Derivatization is a widely-used technique for GC sample preparation across many industries and in widely varied matrices from soil to plastics to blood that is used to make polar and active compounds more amenable to good GC analysis. If you're careful about testing your derivatization procedure during method development, you can be confident that you'll […]

NACRW 2016

Here's a sneak peek at what we'll be up to during this year's show.

ChromaBLOGraphy: **Screening for Bifenazate (Floramite) in Medical Marijuana Using QuEChERS and GC-FID – Is it Possible?**

Bifenazate (CAS# 149877-41-8) is an acaricide made by Uniroyal Chemical and sold under the trade name Floramite. Floramite is registered in the US for control of mites on a wide variety of plants, and is widely employed in greenhouses and other indoor growing environments. Its effective control of spider mites leads to its application by [...]

ChromaBLOGraphy: **US Drug Enforcement Administration Exerts Federal Control Over Synthetic Marijuana Compounds (JWH-type; CP-47,497; and Cannabicyclohexanol)**

I heard on the news the other day that the DEA is taking action to control synthetic marijuana compounds such as JWH-018, JWH-073, and JWH-200, in addition to a couple others. It hasn't been that long ago that herbal "incenses" that provide a pot-like high when smoked popped up for sale on the internet, and [...]

Restek Advantage, 2011.2 (PDF)

.... However, LC is also a viable technique for medical **cannabis** potency testing. As shown in this article , the same straightforward sample preparation technique can be used for **cannabis** potency testing by either...

Restek GCxGC Columns: Your One Source for 2D Gas Chromatography (PDF)

.... While many **cannabis** labs do purport to check for pesticides in marijuana, it is unlikely...

News: **Restek at Pittcon 2015: Free Starbucks and 30 Years of Pure Chromatography**

What better place than The Big Easy to throw a party? And what better excuse than our 30th anniversary? We have your invitation to innovation, and there's no RSVP needed. Just stop by Booth #2600 to join in the fun! … Continue reading →

ChromaBLOGraphy: **The Bard Hits the Bong?**

I was listening to "Wait Wait… Don't Tell Me!" (WWDTM) today on the local NPR station and heard an interesting story. WWDTM is a humorous quiz show on topical news items, and features celebrities and comedians bantering with each other and the host, Peter Sagal, while chosen listeners are called and asked questions. The prize [...]

ChromaBLOGraphy: **MXT-35 GC-FID: Medical Marijuana Cannabinoids**

I'm now adding a 15m x 0.53mm x 0.50µm MXT-35 to the GC column data collection effort for cannabinoids, thanks to Ron Stricek's help with getting that column manufactured for me. This column is made out of metal and has the features/benefits listed in this brochure. In addition to the ruggedness of the metal column and the […]

U.S. Customer Application Form

ChromaBLOGraphy: **High Quality Analysis of Pesticides in Marijuana using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS**

Recently we reported on what we believe is the first application of QuEChERS for marijuana, using it for potency analysis with GCxGC-TOFMS. Ultimately, the plan was to determine pesticides via the QuEChERS approach, combining it with cartridge SPE cleanup as we did for dietary supplements, since sample complexity would defeat the typical dispersive SPE cleanup [...]

ChromaBLOGraphy: **Restek's EZGC Online Suite that includes the Method Translator and Flow Calculator, and a Chromatogram Modeler, wins a TASIA Award – Chris Nelson, One of the Suite Builders**

The Analytical Scientist is a very smartly produced scientific magazine full of interesting articles, including many on chromatography. I've had the pleasure of working with two of the minds behind this publication, Rich Whitworth and Frank van Geel, on a TAS GCxGC contribution, and have been impressed with the volume of quality work they've put […]

ChromaBLOGraphy: **Choosing an Internal Standard**

Adding an internal standard (IS) to an assay can be an excellent way to often improve method precision and accuracy. An IS can account for signal suppression (or enhancement), that may be caused by the sample matrix. When using an IS, the response of your target compound(s) is compared to the response of the IS. […]

News: **Join Restek at EAS 2015**

Going to EAS this year? If so, be sure to visit the Restek team at Booth 512. We would love to hear about your work, help you optimize your analyses, and show you why Restek is your first and best … Continue reading →

ASMS 2016

Learn more about what we'll be doing at the 64th American Society for Mass Spectrometry Conference on Mass Spectrometry and Allied Topics

Resources

Showing 26 to 50 of 67

ChromaBLOGraphy: **Residual Solvents in Cannabis…and Terpenes with Simon & Garfunkel**

In my last blog post, I wrote about our ongoing method development for residual solvents in cannabis. We've been really busy since then, and now we have a complete application note published on this subject. Because we can't legally get our hands on real cannabis concentrates here in Pennsylvania, the application note proves our concept […]

ChromaBLOGraphy: **Possible Internal Standards for Medical Cannabis Potency Testing by GC**

I am often asked about internal standards for use in medical cannabis potency testing with gas chromatography. I finally got some time in the lab to check this out and came up with a couple of possibilities after testing numerous compounds for favorable retention times versus typically analyzed cannabinoids. Internal standards are mostly used by adding to […]

ChromaBLOGraphy: **Medical Cannabis Terpenes Standards now available from Restek!**

As many of you dedicated ChromaBLOGraphy readers know, Restek has supported the medical marijuana market for years with reference materials, and GC and LC consumables. We find the field fascinating, so much so that we try to anticipate the upcoming needs of medical cannabis analysts through method development for compounds in addition to cannabinoids that […]

High-Quality Analysis of Pesticides in Cannabis Using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS

... legalized the use of recreational or medical **cannabis** because of therapeutic benefits for ailments on the federal level. As a result, medical **cannabis** patients have no safety assurances for their medication...

ChromaBLOGraphy: **Low-Pressure System: Gas Chromatography, Not Weather… Synthetic Cannabis**

We have a very bright guy named Jaap de Zeeuw who works for Restek. Years ago he invented a system for low-pressure gas chromatography by where he used a 0.53mm GC column attached to a mass spectrometer, and a restrictor column (e.g. 0.50m x 0.10mm) press-fitted to that column and installed in a split/splitless GC [...]

ChromaBLOGraphy: **Internal Standard versus External Standard Quantification in Medical Cannabis Potency Analysis with GC-FID**

The ChromaBLOGraphy series continues for the use of internal standards with medical cannabis potency testing by GC-FID (I've listed the first two parts in the series immediately below as web-links). This third part demonstrates the positive impact an internal standard can have on quantitative accuracy. Possible Internal Standards for Medical Cannabis Potency Testing by GC […]

News: **Don't Overestimate Cannabidiol During Medical Cannabis Potency Testing by Gas Chromatography**

Author(s): Jack Cochran Restek Corporation Published By: Restek Corporation Year of Publication: 2014 Link: http://www.restek.com/Technical-Resources/Technical-Library/Pharmaceutical/fff_FFAR1954-UNV Abstract: Proper GC column choice is essential for accurate and robust medical cannabis potency testing. Using an Rxi®-35Sil MS column under the instrument conditions shown here … Continue reading →

News: **A Fast, Simple FET Headspace GC-FID Technique for Determining Residual Solvents in Cannabis Concentrates**

Author(s): Corby Hilliard1; Amanda Rigdon1; William Schroeder, Ph.D.2; Christi Schroeder, Ph.D.2; Theo Flood2 1. Restek Corporation, 2. Cal-Green Solutions Published By: Restek Corporation Year of Publication: 2014 Link: http://www.restek.com/Technical-Resources/Technical-Library/Foods-Flavors-Fragrances/fff_FFAN2009-UNV Abstract: Due to rapid growth in the medical cannabis industry, demand is … Continue reading →

News: **A Preliminary FET Headspace GC-FID Method for Comprehensive Terpene Profiling in Cannabis**

Author(s): Amanda Rigdon, Corby Hilliard, and Jack Cochran Restek Corporation Published By: Restek Corporation Year of Publication: 2014 Link: http://www.restek.com/Technical-Resources/Technical-Library/Foods-Flavors-Fragrances/fff_FFAN2045-UNV Abstract: This application note describes an FET headspace GC-FID method that was developed in hops for the analysis of terpenes in … Continue reading →

ChromaBLOGraphy: **Pennsylvania joins other states in banning "bath salts" and synthetic cannabis (aka "Spice")**

Pennsylvania is set to outlaw sales of the currently legal drugs known colloquially as "bath salts" and "Spice". The active compounds in these drugs are apparently being made by enterprising chemists and sold as products not for human consumption, which allows them to skirt current drug laws in the US. Often the "bath salts" contain [...]

ChromaBLOGraphy: **Faster GC Analysis of Medical Cannabis Terpenes with Same 624Sil MS Selectivity**

The chromatograms below show what happens when you translate a GC method (previously used for

medical cannabis terpenes here and here) from a 30m x 0.25mm x 1.40µm Rxi-624Sil MS GC column to a 30m x 0.25mm x 1.00µm Rxi-1301Sil MS column. Both of these columns have arylene-modified cyanopropylphenyl dimethyl polysiloxane-type stationary phases. As should be […]

ChromaBLOGraphy: **Comparison of Phencylidine and Prazepam as Internal Standards in Medical Cannabis Potency Analysis with GC-FID**

Recently in the ChromaBLOGraphy posts below I proposed the use of Phencylidine (PCP) as an internal standard (ISTD) for cannabinoids analysis with GC-FID when using the Rxi-35Sil MS GC column. I demonstrated that the RSD% of Average Response Factors (Avg RFs) and Correlation Coefficients (CCs) for the calibration curves generated using the ISTD technique were […]

ChromaBLOGraphy: **The separation problem with CBC and CBD in GC analysis of medical cannabis with 5% phenyl-type columns.**

In my post, "Don't overestimate cannabidiol during medical cannabis potency determinations with gas chromatography. Use stationary phase selectivity for accuracy and hydrogen for fast analysis.", I showed how an Rxi-35Sil MS GC column provides excellent separation for cannabichromene (CBC), cannabidiol (CBD), delta-8-THC, delta-9-THC, cannabigerol (CBG), and cannabinol (CBN). I focused on the separation of cannabichromene [...]

ChromaBLOGraphy: **Calibration Curves for Cannabinoids Based on PCP Internal Standard – Medical Cannabis GC-FID**

Yesterday's ChromaBLOGraphy post concerned the use of internal standards (ISTDs) for GC-FID potency testing of medical cannabis. In that post I defined desirable characteristics for an ISTD and said that one of the benefits of ISTD use is better quantitative accuracy. Good quantitative accuracy starts with good calibration, which I demonstrate in this post by showing a […]

ChromaBLOGraphy: **Using delta-9-THCA and delta-8-THC as Standards to Determine Medical Cannabis Potency with GC-FID**

January 2, 2014 Update: We now have 34014 (Cannabinoids Standard with CBD, d9-THC, CBN) and 34067 (Delta-9-Tetrahydrocannabinol (THC) Standard) back in stock and ready for sale. http://www.restek.com/catalog/view/11258/34014 http://www.restek.com/catalog/view/10385/34067 As some of you in the medical cannabis analysis community already know, Restek is currently unable to provide our cannabinoids standard (34014 – CBD, d9-THC, CBN) for potency testing due to raw […]

News: **High-Quality Analysis of Pesticides in Cannabis Using QuEChERS, Cartridge SPE Cleanup, and GCxGC-TOFMS**

Author(s): Jack Cochran, Julie Kowalski, Sharon Lupo, Michelle Misselwitz, and Amanda Rigdon Restek Corporation Published By: Restek Corporation Year of Publication: 2014
Link: http://www.restek.com/Technical-Resources/Technical-Library/Pharmaceutical/fff_FFAR1950-UNV
Abstract: As medical cannabis is more frequently prescribed, patient safety must be ensured. Pesticide residue testing is … Continue reading →

News: **The Practical Chemist: Calibration – The Foundation of Quality Data**

Author: Amanda Rigdon Restek Corporation Published By: Cannabis Industry Journal Year of Publication: 2016 Link: https://www.cannabisindustryjournal.com/column/calibration-the-foundation-of-quality-data/
Abstract: This column is devoted to helping cannabis analytical labs generate valid data right now with a relatively small amount of additional work. The … Continue reading →

ChromaBLOGraphy: **Don't overestimate cannabidiol during medical cannabis potency determinations with gas chromatography. Use stationary phase selectivity for accuracy and hydrogen for fast analysis.**

It's important to properly quantify cannabidiol in medical marijuana samples, as it is one of the chief cannabinoid compounds with pharmacological value, including relief against nausea, anxiety, and inflammation. However, on typically used "5 type" GC columns, it can coelute with cannabichromene, a compound that likely also has medical value and is more and more [...]

ChromaBLOGraphy: **Accurate Quantification of Cannabinoid Acids by GC – Is it Possible?**

I think by now we've all heard that GC potency testing for cannabis or hemp has some drawbacks. That being said, GC is a popular, rugged, and cost-effective laboratory workhorse and is still employed in many cannabis laboratories. The major drawback of GC versus HPLC cannabinoid testing is the fact that the acidic cannabinoids convert […]

Global Advantage, 2012.1 (PDF)

... inexpensive instrumentation. However, LC is also a viable technique for medical **cannabis** potency testing . As shown in this article, the same straightforward sample preparation technique can be used for **cannabis**...

Restek Reference Standards (PDF)

... • Fatty acid methyl esters (FAMEs) • QuEChERS • Derivatization reagents • **Cannabis** PETROCHEMICAL...

ChromaBLOGraphy: **Analysis of Nicotine and Related Compounds in Urine Using Raptor™ Biphenyl**

As Applications Chemists in the LC lab, one of the most exciting parts of our jobs is the variety of analyses we are exposed to. One day you are developing a method for potency analysis in cannabis samples, the next you are looking at anti-epileptic drugs in urine. We're regularly challenged to think outside the […]

News: **The Practical Chemist: Easy Ways to Generate Scientifically Sound Data**

Author: Amanda Rigdon Restek Corporation Published By: Cannabis Industry Journal Year of Publication: 2016 Link: https://www.cannabisindustryjournal.com/column/easy-ways-to-generate-scientifically-sound-data/ Abstract: The inaugural installment of a new column that will provide simple ways for laboratories to improve the quality of their chromatographic data, including … Continue reading →

ChromaBLOGraphy: **CBDV and THCV on the Rxi-35Sil MS GC Column with Other Cannabinoids**

We continue to use the 15m x 0.25mm x 0.25µm Rxi-35Sil MS for medical cannabis potency testing by GC-FID. Even though we don't currently offer CBDV and THCV as reference materials, I wanted to find out where they eluted on the 35Sil MS because of their potential for being medically significant. CBDV

apparently has demonstrated […]

ChromaBLOGraphy: Medical Marijuana Musings

At Restek, we're into medical marijuana, from an analytical standpoint, as cannabis is one of the most complex (and controversial) natural products. That complexity can make it very challenging to analyze, especially as the suite of analytes expands beyond the usual cannabinoids (e.g. delta-9-THC, cannabidiol, cannabinol) to include other cannabinoids (e.g. cannabigerol, cannabichromene, delta-8-THC, cannabivarin, etc.) and [...]

RESTEK Pure Chromatography

Restek Corporation, U.S., 110 Benner Circle, Bellefonte, PA 16823

ESOP A Company of Owners

Step by step Cannabis Potency Testing using the SRI odel GC une

The SRI Model 420 Gas Chromatograph (GC) is an ultra low cost and easy to operate GC which measures CBD and THC in cannabis and concentrate samples with the same accuracy as vastly more expensive and complicated laboratory instruments The Model 420 is equipped with a built-in hydrogen generator so only water and electricity are required for operation.

Why send samples to a lab when you can measure CBD and THC yourself in minutes at a cost of less than cents per analysis.

verything you need to begin is included in the kit except for

A Windows computer with USB connection (laptop OK)

Distilled water from the grocery store (about $1)

Denatured alcohol from the hardware store (about $15)

You get

An electronic balance to weigh the sample

Six extraction bottles

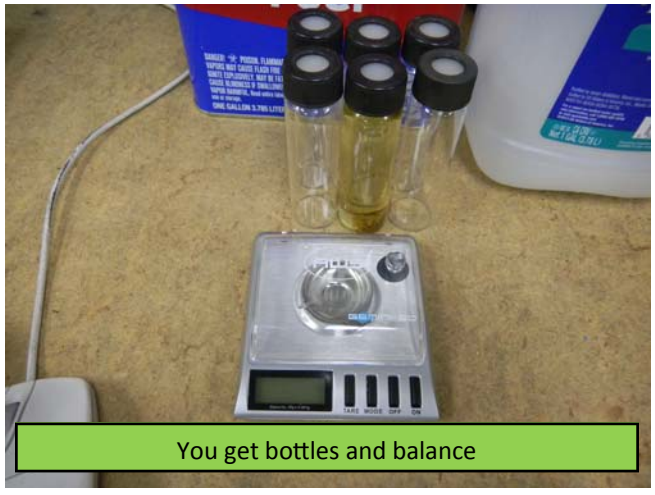
Calibration standard-enough for 400 analyses

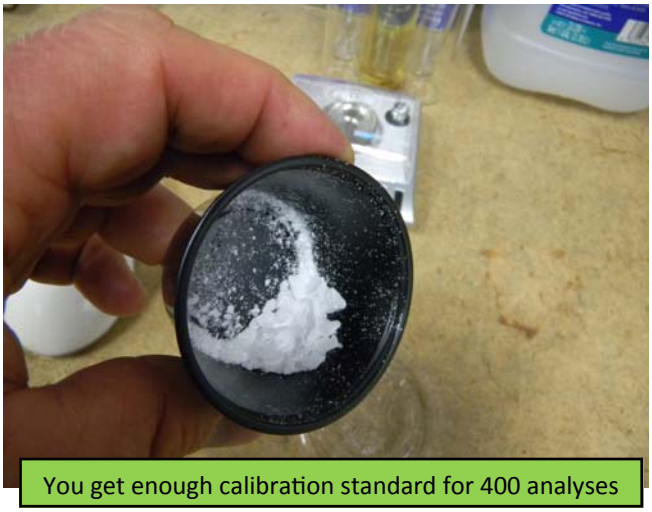
Two injection syringes

To Order:

8610-0420 Model 420 GC kit for cannabis potency testing $4995.00

Just needs distilled water and electric power


You get bottles and balance


You get enough calibration standard for 400 analyses

Step by step Cannabis Potency Testing using the SRI odel GC une

SRI also manufactures more capable gas chromatographs for testing cannabis.

http //www.srigc.com/home/product detail/medical-cannabis-cannabinoid-gc

These GCs can distinguish between CBD and CBC, and between THC and CBG which the simpler odel can not do. The more capable GCs allow for more sophisticated analyses demanded by professional labs.

The SRI C is the perfect si e GC (gas chromatograph) for measuring CBD, CBDA, d THC, d THC, THCA, CBC, CBG and CBN levels in medical cannabis.

It can also be used to test for synthetic cannabinoids like SPIC , butane residuals, terpenes, aromas and edibles.

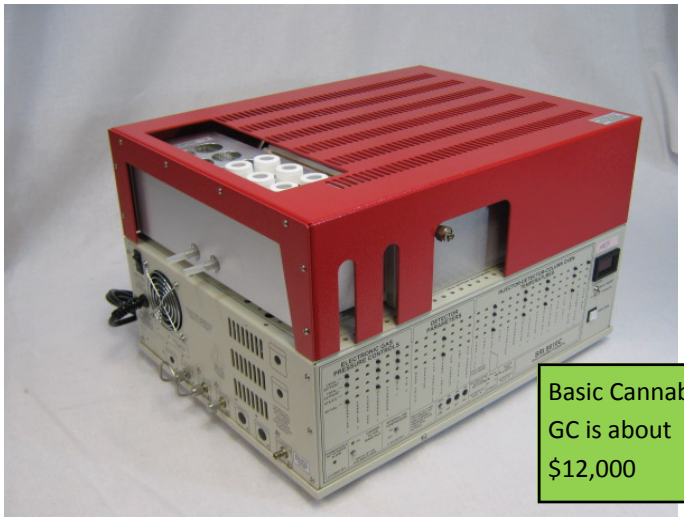
The basic cannabis testing GC is , (une prices) with a single FID detector and column. A simple minute column change converts from cannabinoid analysis to residual solvents or terpene analysis.

With or FID detectors and columns, cannabinoids, residual solvents and terpene profiles can all be performed simultaneously on one GC with no hardware changes, completely avoiding downtime from column change-overs.The included built-in °C incubator speeds up the extraction process and is especially helpful in getting concentrates, medibles and/or butters to dissolve.

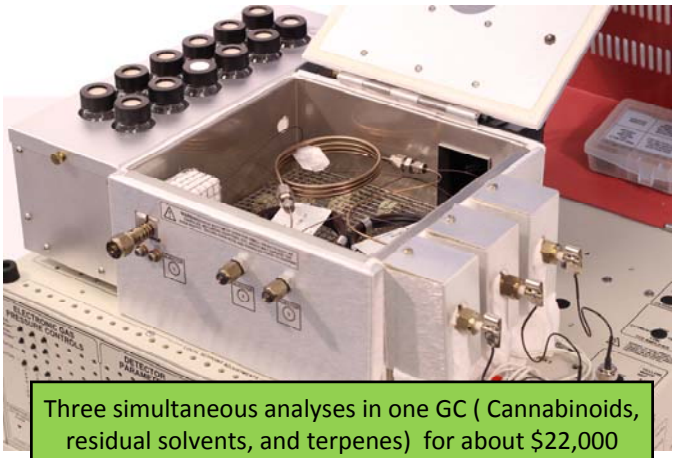
8610-0091 Basic Cannabis GC $12,170.

8610-0291 Basic Cannabis GC plus 2nd channel for residual solvents or terpenes $18,500.

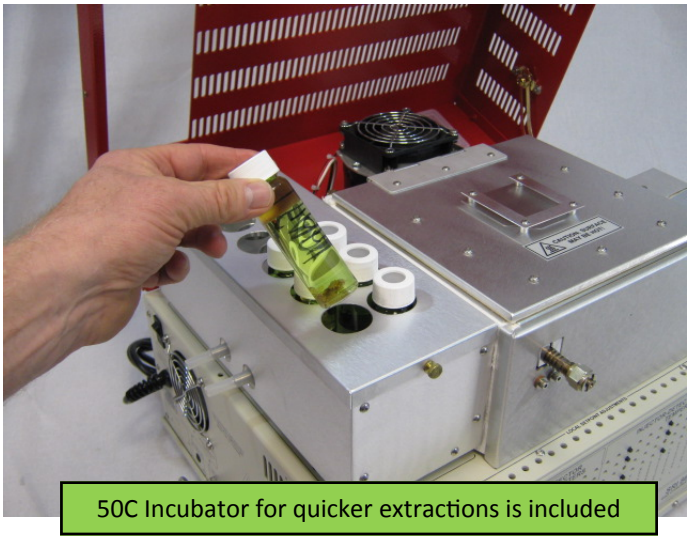
8610-0391 Basic Cannabis GC plus 2nd and 3rd channels for residual solvents and terpenes simultaneously $22,500.



Basic Cannabis GC is about $12,000



Three simultaneous analyses in one GC (Cannabinoids, residual solvents, and terpenes) for about $22,000

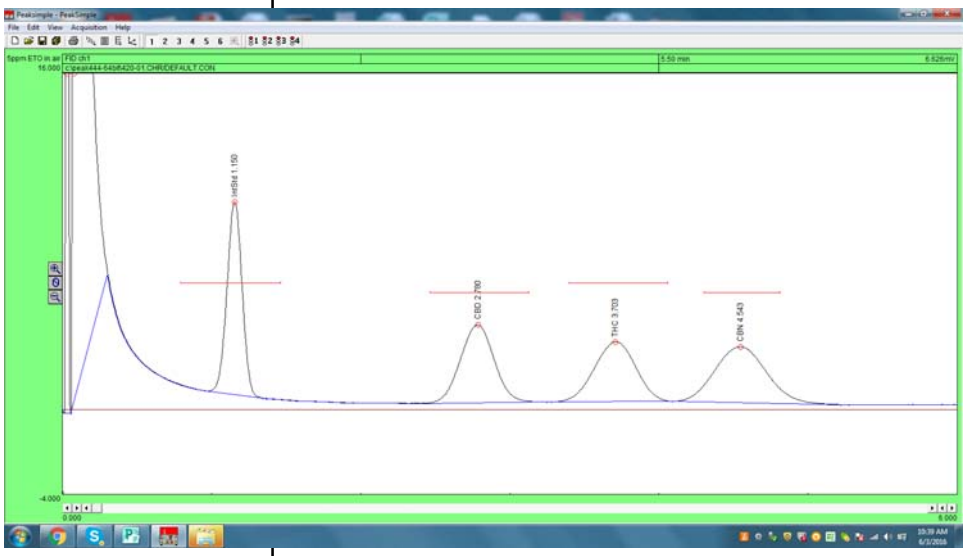


50C Incubator for quicker extractions is included

SRI

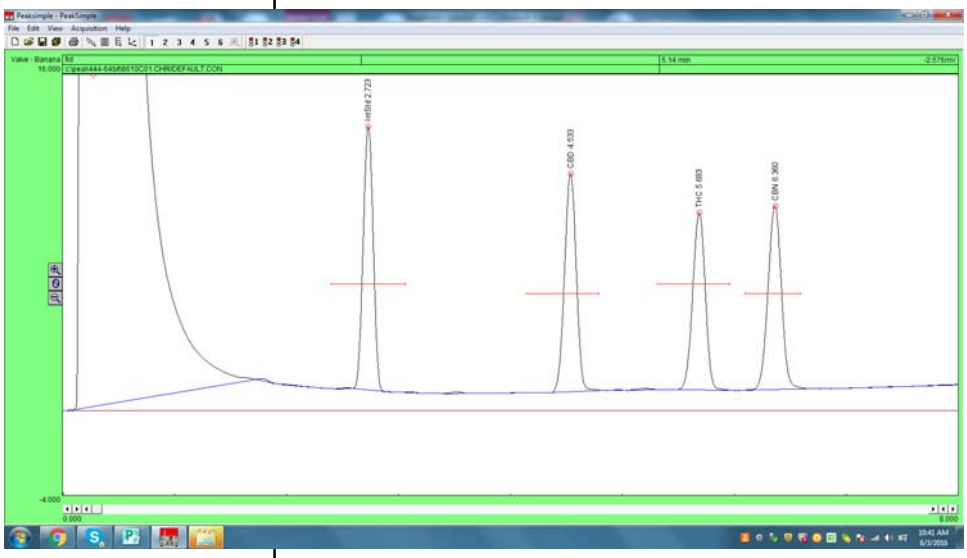
Step by step Cannabis Potency Testing using the SRI odel GC une

This chromatogram shows the injection of a calibration chromatogram with CBD, THC and CBN on the . odel GC.



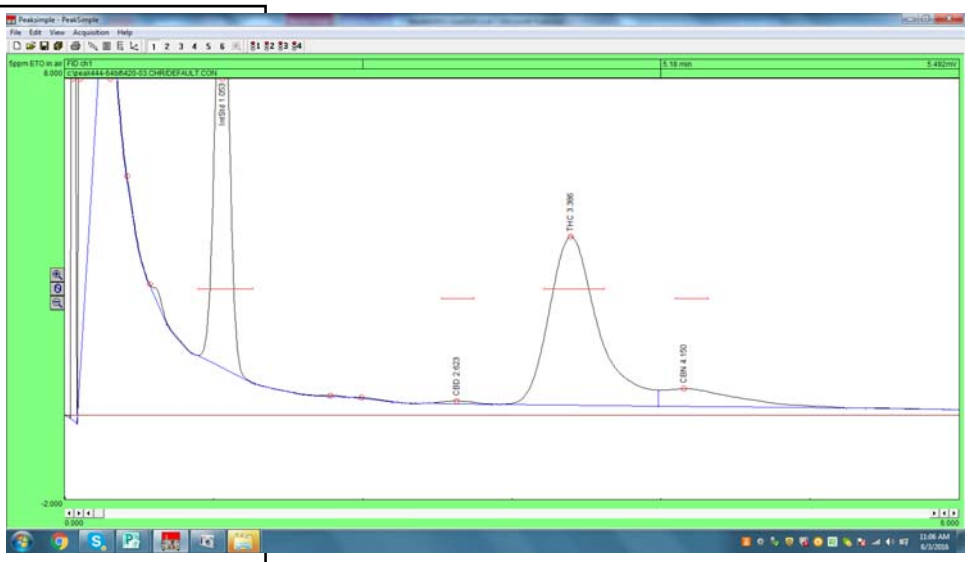
This shows the same calibration sample on the twelve thousand dollar odel C configured for cannabis testing. This is the GC we suggest for professional labs.

The peaks are a little sharper but aside from that, there is no major difference.



Step by step Cannabis Potency Testing using the SRI odel GC une

This chromatogram shows a real cannabis sample on the odel GC.

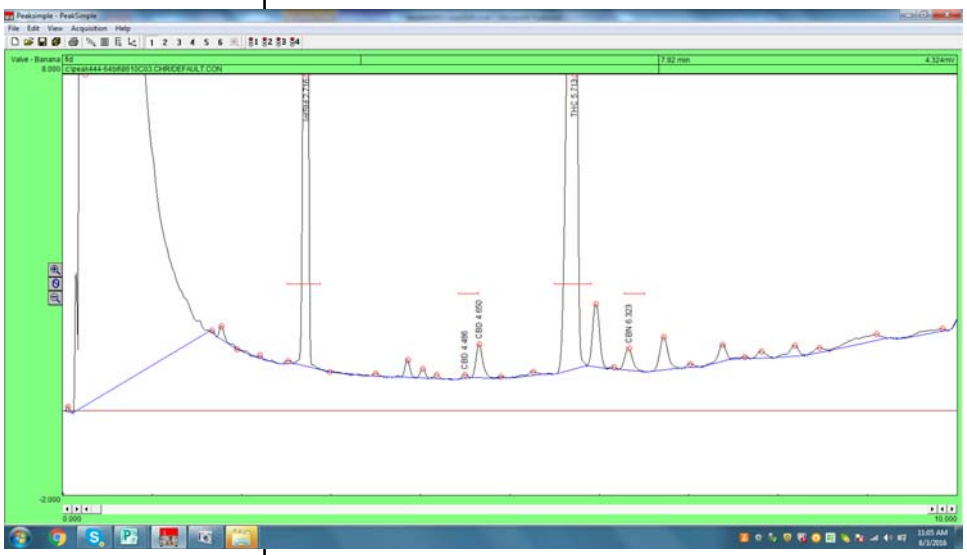


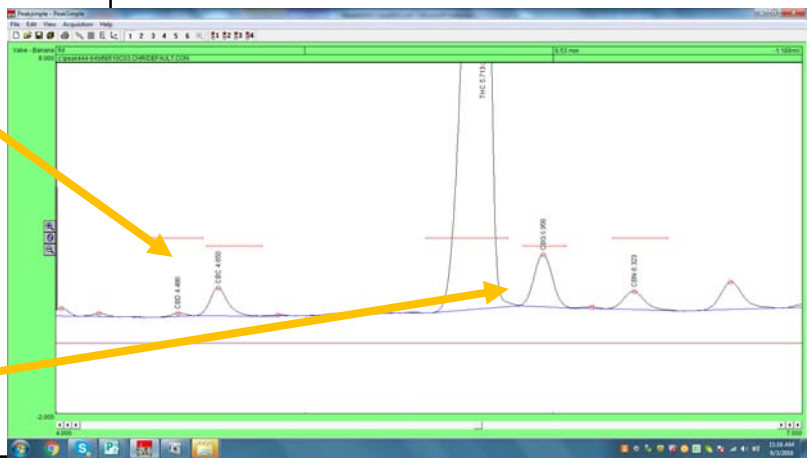
This is the same sample on the C GC.

Notice that there are many more peaks which are separated.

These are all real cannabinoid peaks which the more expensive GC can resolve but which the od-el can not.

specially note that the CBD peak is immediately next to the CBC peak.





And the CBG peak is well resolved from the THC.

Step by step Cannabis Potency Testing using the SRI odel GC une

Step

Buy a gallon of denatured alcohol at the hardware store (Home Depot etc). The usual cost is about for the gallon. Denatured alcohol is used for stove fuel in boat stoves and is a mix of / methanol and ethanol. Its poisonous to drink and flammable so use it in a well ventilated area away from flames and don't smoke around it.

Step

Find the white internal standard powder. There will be about gram of methyl stearate in a plastic cup supplied with the GC. ethyl Stearate is made from palm oil and is commonly found in cosmetics. Don't eat it either.

Step

Put the entire contents into the gallon of denatured alcohol. Don't spill any. Use a popsicle stick or Q-tip to sweep all of it into the gallon container. It takes a while to dissolve if the denatured alcohol is cold, so put the denatured alcohol in the sun to warm up and shake it one or twice once it is warm. Remember its flammable so don't put it in the oven or on the stove.

Step by step Cannabis Potency Testing using the SRI odel GC une

Step

Set up the balance (scale) which comes with the odel GC. You have to put in the batteries and check the calibration with the little gram weight which comes with it.

If you have a more expensive balance then you can use that instead. The import thing is that the balance can read the weight down to milligram (. gram).

Step

Weigh approximately milligrams of cannabis into the little weighing dish. It does not have to be exactly milligrams as long as you record the actual weight. In the photo, it reads milligrams

For concentrates, weigh milligrams of concentrate instead of milligrams. An easy way to do this is to put a little strip of paper on the balance, tare the balance to read and then dab about milligrams of concentrate on the paper.

Step by step Cannabis Potency Testing using the SRI odel GC une

Step

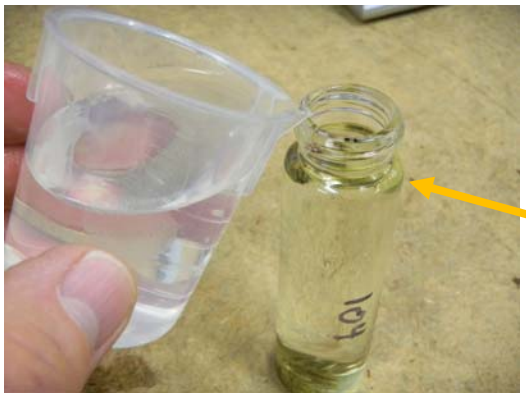
Put the milligrams of cannabis (or milligrams of concentrate) into the milliliter bottle. Be careful not to spill any as the weight of the cannabis is important to getting an accurate answer.

Write the name of the sample and the weight on the bottle with a magic marker

Step

Pour some of the alcohol into the beaker which comes with the odel . The beaker makes it less likely you will spill and makes it easier to fill the ml bottle (the gallon is heavy).

Put the cap on the ml vial, give it a shake, and let it sit on the table for at least minutes. This gives the alcohol time to dissolve the THC and CBD etc.



Fill the 40ml bottle to the neck where the glass narrows.

Step by step Cannabis Potency Testing using the SRI odel GC une

Step

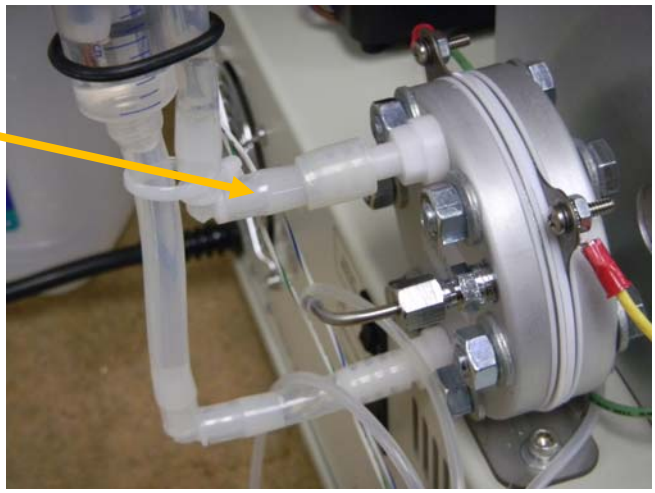
Buy a gallon of distilled water at the grocery store (about). ake sure it says " Distilled Water" , not "purified" water or "de-ioni ed" water. Do not use household tap water.

Fill the water reservoir with the distilled water. The water reservoir holds milliliters which is enough for about hours of operation.

Make sure the water reservoir is full before turning on the Model 420 power.

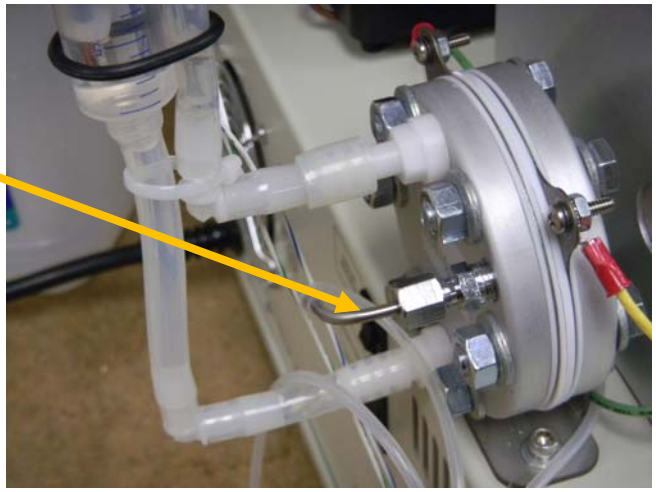
The hydrogen generator (which is built-in to the od-el) produces hydrogen gas and oxygen gas.. The oxygen gas and extra water bubbles up through the return tube and back into the water reservoir.

Oxygen bubbles up through this tube along with extra water back into the reservoir



Step by step Cannabis Potency Testing using the SRI odel GC une

Hydrogen exits the hydrogen generator along with extra water from the metal tube.



The hydrogen flows into a water separator mounted on the left side of the odel .

Water gradually accumulates in the water separator.

very time the reservoir is filled, the accumulated water in the water separator must be drained by turning the red stopcock.

The water will slowly flow out of the separator and out this tube.

Put the tube in the beaker to avoid getting the tabletop wet. Do not re-use the water, just pour it down the sink.

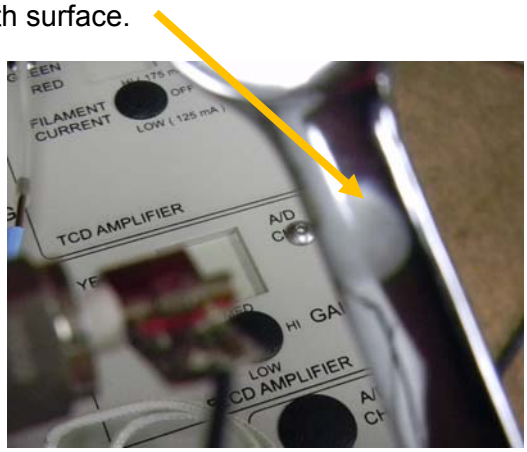
Step by step Cannabis Potency Testing using the SRI odel GC une

Step

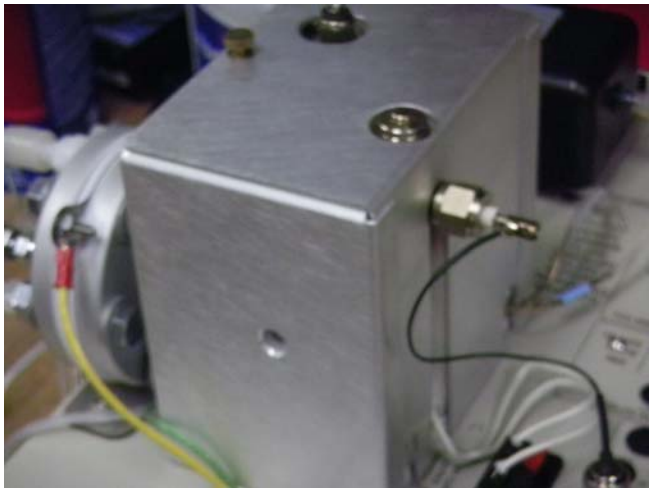
Turn on the main power switch located on the left side of the odel . The displays on the front will illuminate. The left side display controls the GC's column operating temperature. This is normally set to degrees Centigrade and fluctuates about de- gree up or down after it heats up. The green digits on the bottom is the setpoint and the red digits at the top is the actual temperature. The red digits will change a little, but not more than about degree.

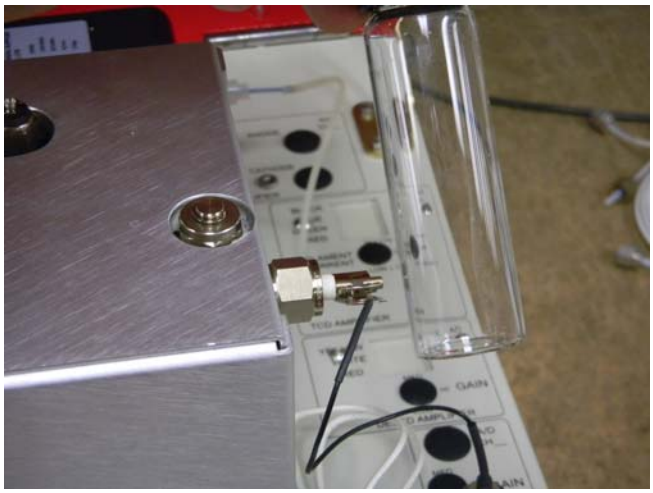
The right side display shows the hydrogen generator voltage (the red digits at the top) and the current (amps) (blue digits at bottom). When the hydrogen generator is operating correctly the values will be as shown in the photo.

Under the odel 's red lid is the GC oven, injector and FID (flame ioni ation)detector. The FID detector has a tiny hydrogen flame which burns inside the stainless steel body. When hydrogen burns it makes water which shows up as water vapor on the side of the ml bottle or even better on a shiny wrench or other smooth surface.



The flame lights itself as long as the hydrogen is flowing .

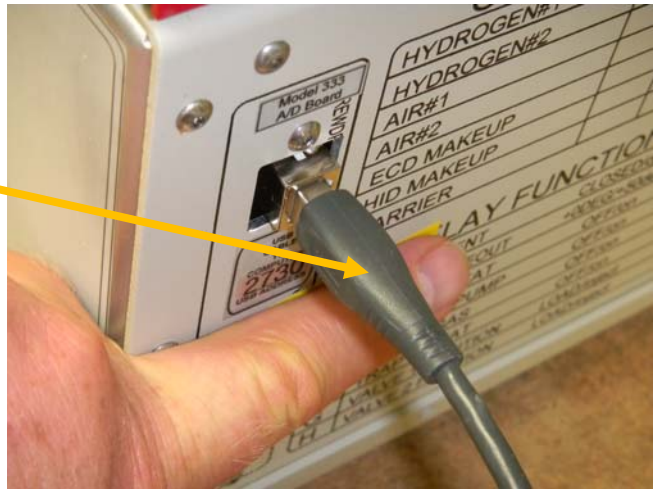




Step by step Cannabis Potency Testing using the SRI odel GC une

Step

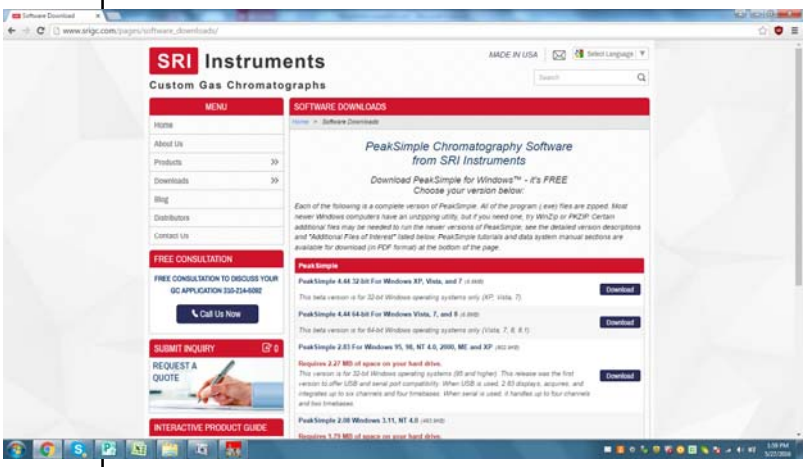
Plug the odel into your Windows P or later computer using the provided USB cable.



Download the PeakSimple software from SRI's website.

Click here to download PeakSimple

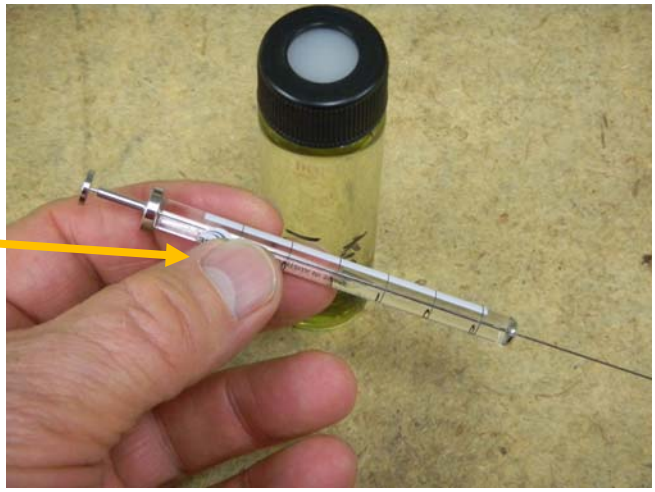
There will be a special version of the software which has everything already set up for the CBD and THC analysis.



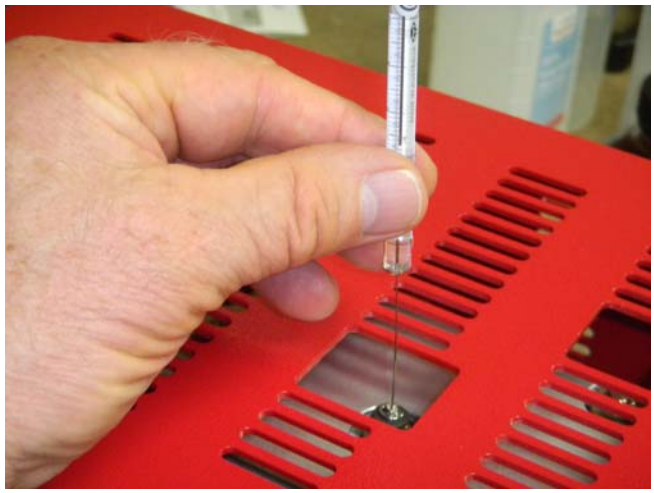
Step by step Cannabis Potency Testing using the SRI odel GC une

Step

Use the provided ul (microliter) syringe to suck up ul (microliter) of the cannabis extract you previously prepared. This may have a greenish color by now.

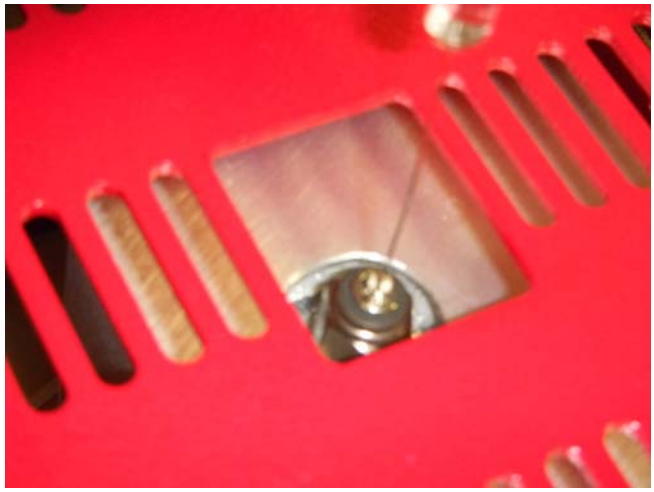


Its not critical to measure exactly ul, but try to be somewhat close to ul. Pull the syringe plunger back after you fill the ul so there is some air in the syringe needle. This makes it less likely to lose some sample if you accidentally touch the plunger while making the injection.



Position the syringe in the injector but do not push it down yet. You will feel the rubber septum when the tip of the syringe touches it.

When you are ready, press the computer's spacebar to start the analysis and within a few seconds push the syringe down all the way and depress the plunger.

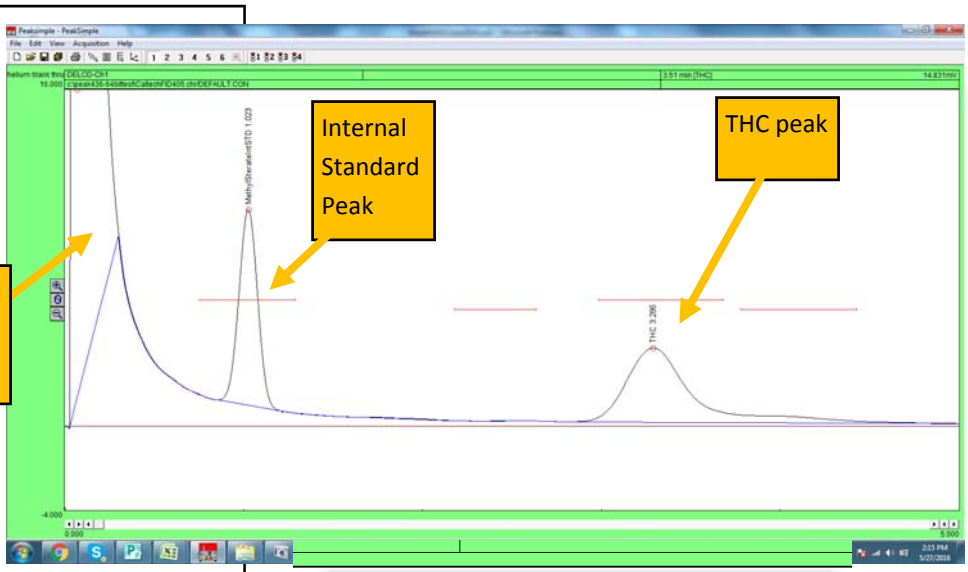


This injects the ul of cannabis extract into the GC.

Step by step Cannabis Potency Testing using the SRI odel GC une

Step

A chromatogram will appear on the computer screen which looks something like this. It takes about minutes altogether.

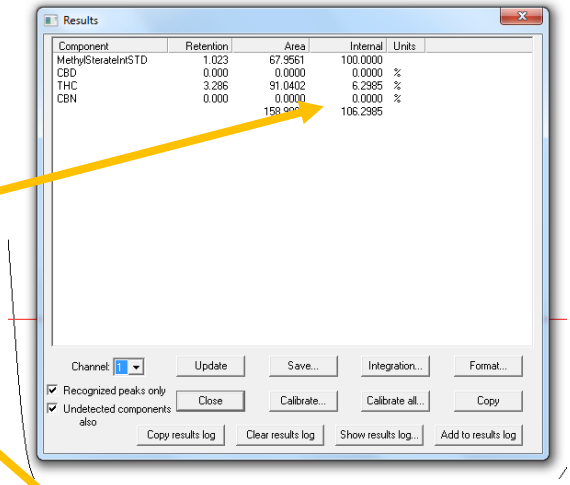


The first peak is very large and appears almost immediately. This is the denatured alcohol peak.

The second peak is the methyl stearate internal standard peak.

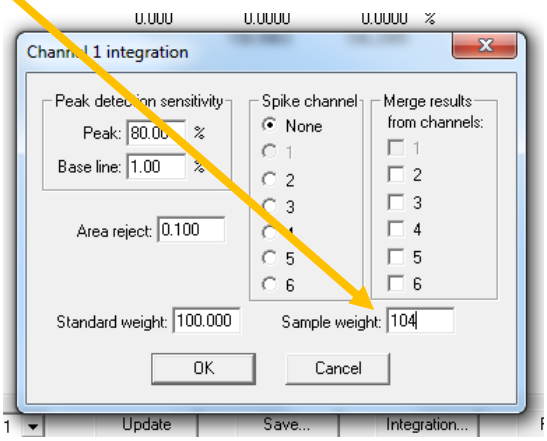
The rd peak is the THC peak.

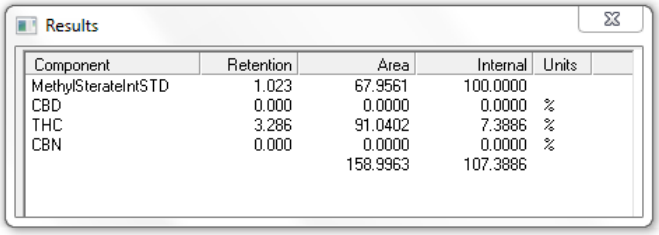
The PeakSimple software calculates the si e of the THC peak (the area under the curve, not the height) and compares it to the si e of the Internal Standard peak. This gives you the answer which shows up in the software s Results screen.



Another screen (just one mouse click away) lets you enter the actual weight of the cannabis we put in the ml bottle (milligrams).

So you enter the number in this box and that corrects the answer.





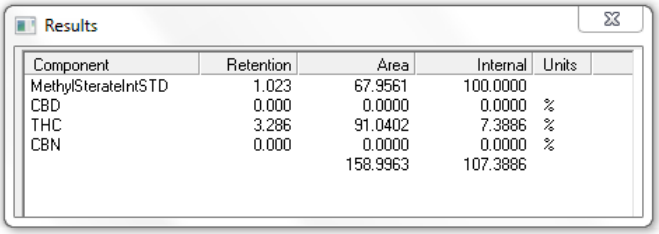
Results

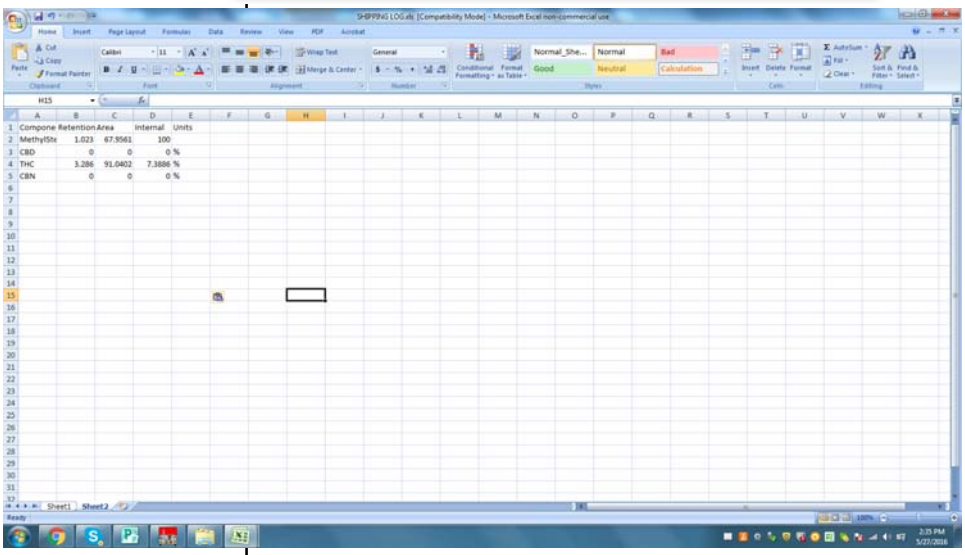
Component	Retention	Area	Internal	Units
MethylSterateIntSTD	1.023	67.9561	100.0000	
CBD	0.000	0.0000	0.0000	%
THC	3.286	91.0402	7.3886	%
CBN	0.000	0.0000	0.0000	%
		158.9963	107.3886	

In this case the answer comes out to be . THC.

Step by step Cannabis Potency Testing using the SRI odel GC une

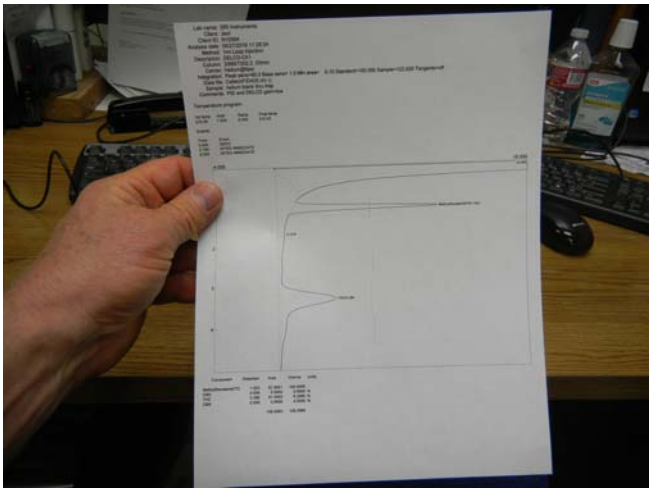
The Results are easily pasted into xcel, Word or other program with just a couple mouse clicks.





You can also print to a pdf or to paper.

The odel GC is now ready to measure the next sample.



Step by step Cannabis Potency Testing using the SRI odel GC une

The odel GC comes with a one year warranty.