APPLICATIONS

Update . . . >Jan 2016

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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

SRI Gas Chromatographs

: *Custom configurations specific to Cannabis*

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.
- Medical Cannabis Terpene Measurement using the SRI 8610C FID GC.
- Measuring THC in Butter using the SRI 8610C GC.

Disclaimer / preamble . . . "draft Only"

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 moreso potential loss of revenue through difficult to control naturally grown and their replacement of controlled substances like tobacco and alcohol under more and more severe taxation tariifs and which are "protected" substances despite arguably but proven more damaging heatlh and social problems.
Vested interest are the prime interest including the medical/pharmaceutical system as well as the attracted criminal element both out to exploit the publics naivety or gullibility.
Chromalytic Tech does NOT condone the use of such illicit materials *if it at all ameliorates 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 required.
There is in fact a valid argument for the legalisation of all drug use.

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

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

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

Gas Chromatotography (and perhaps to a lesser extents HPLC being less affordable and more complex) is recognised as a relatively simple low cost QC technique. HPTLC is another promising complementary technique.
Under suitable Laboratory control, licensing etc. with proper technical supervision.

For "Cannabis" the GC has been well researched and documented to the point where effective QC is now possible.
Chromalytic offers such GC equipment to qualified Labs and researchers but by definition GC is such a Universal techniquie NO GC system can be defined as being applicatble to marijuna (or any other drug testing) ONLY!
There is NO legal restriction on the use of GC for ANY purpose including potential use for drug testing.

DISCLAIMER :
"Catch-ALL legalities" Buyer BEWARE To prevent diversion of such or other equipment that might be somehow related to potential drug manufacture the 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 eqiuipment to unauthorised end-users.*
- equires setting up an account with Chromalytic Technology with full traceability; ID etc
- Declaration as to the intended use

We accept NO Responsibility whatsover for the use or misuse of such equipment either legal or illegal.
Any results and interpretation of such and resulting consequneces 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.

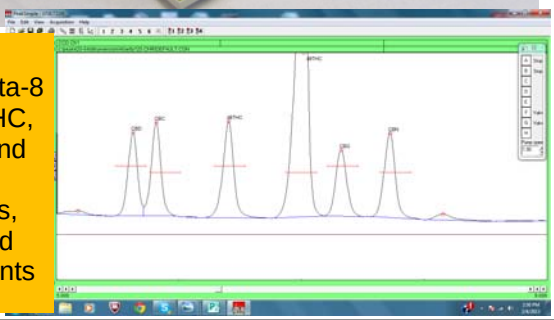
Four common configurations have become popular for measuring medical cannabis.

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)

8610C Gas Chromatograph for Medical Cannabis Analysis

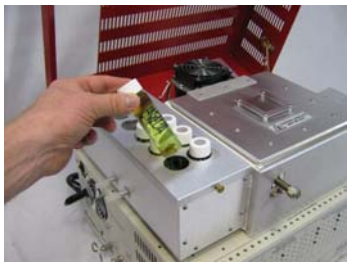


Measure CBD,CBC, delta-8 and delta-9THC, CBG, CBN and other cannabinoids, terpenes and residual solvents

Flame Ionization Detector (FID)
Heated Flash Injector
400°C column oven
Built-in Incubator for heated extractions
15 meter Capillary column
PeakSimple Data System built-in
Hydrogen regulator/tubing kit
Field portable system
Heavy Duty shipping container
Low power consumption (< 800 watts)
Ships via FedEx/UPS or airline baggage
Small footprint for crowded lab benches
Friendly, easy to reach US tech support
Free training
Two Year warranty
Made in USA

Complete system
US$ 12,015.00 plus shipping

The SRI 8610C is the perfect size GC (gas chromatograph) for measuring CBD, THC and CBN levels in medical cannabis. It can also be used to test for synthetic cannabinoids like SPICE, butane residuals, terpenes, aromas and most edibles. The SRI 8610C is rugged enough for mobile applications and light enough to carry around. Simple operation makes training new operators easy. The built-in 50°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 8 minutes so up to 7 samples an hour can be analyzed. The included PeakSimple software (Windows XP/Vista/Win7/8) controls the GC as well as acquiring and calibrating the data. Simple one click export of the data to Excel or Word makes your final report look professional. Get half a day of free training with your GC at our tech support center near LAX (Los Angeles) airport.



System consists of two part numbers:

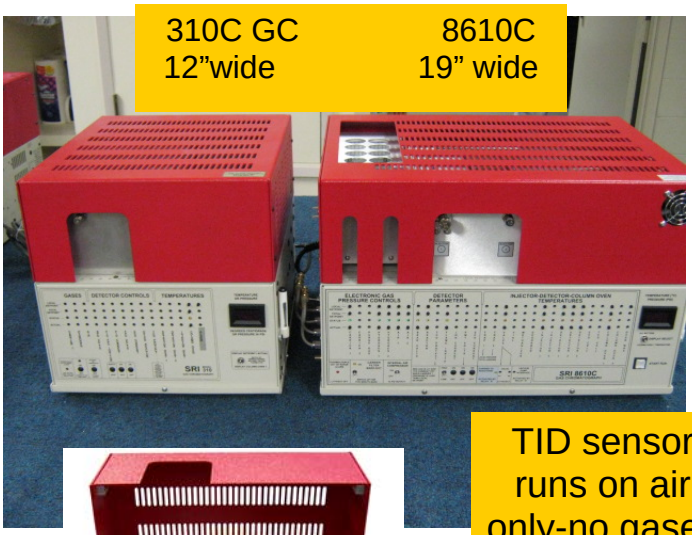
8610-0091	**Cannabis Potency Testing GC complete**	**$11,585.00**
8600-C350	**Hydrogen Gas line kit**	**430.00**
Total	**USD ADD Import Frt&GST In Australia**	**USD$12,015.00**

Medical Cannabis Gas Chromatograph (GC)
Gasless and Simple Configuration

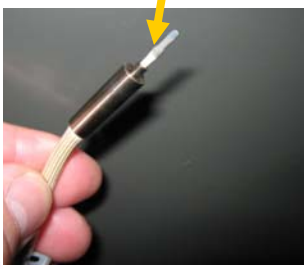
Configuration #1
"Gasless" TID Detector based Potency Configuration
Part# 8610-0094 $9999.00

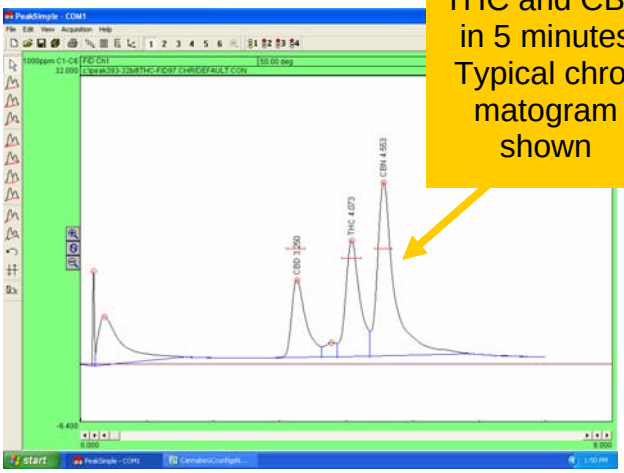
This GC is configured on the ultra-compact 310C chassis (only 12 inches wide) and includes an TID (thermionic ionization detector) which requires no gas cylinders to operate. All required gas is provided by the built-in "whisper quiet" air compressor and dryer. This GC configuration is appropriate for 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





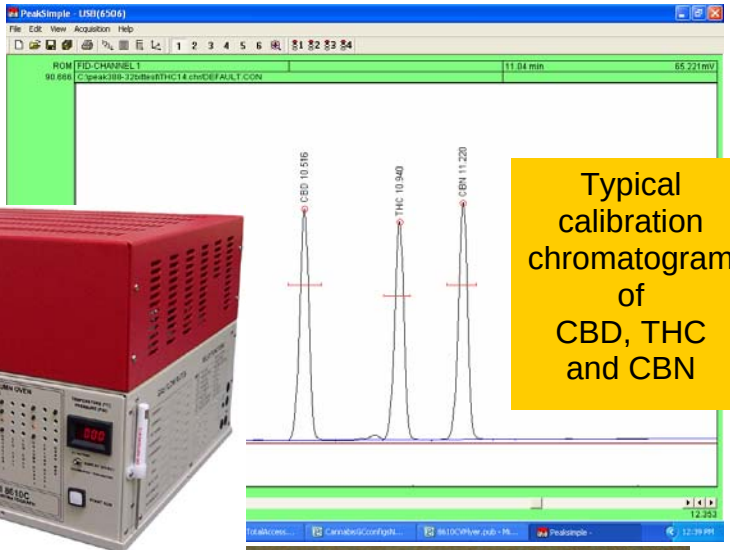
Measure CBD, THC and CBN in 5 minutes Typical chromatogram shown

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.

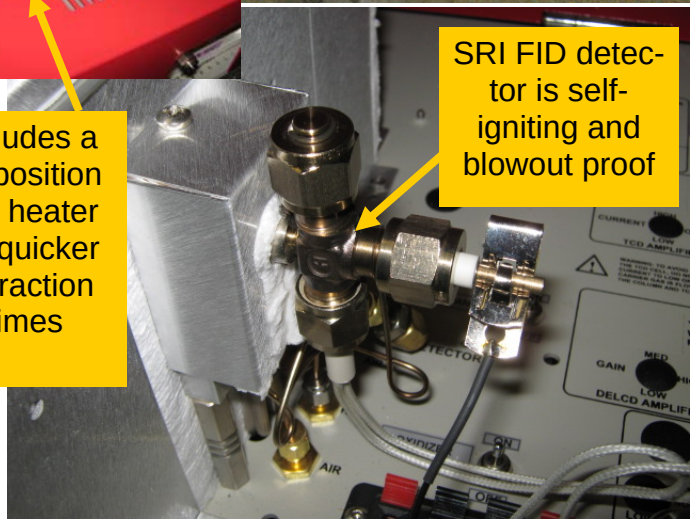
User's will need a hydrogen cylinder, Windows computer and AC power. Syringes and a starter pack of vials is included.



Typical calibration chromatogram of CBD, THC and CBN

Hydrogen Gas cylinder

Includes a 12 position vial heater for quicker extraction times



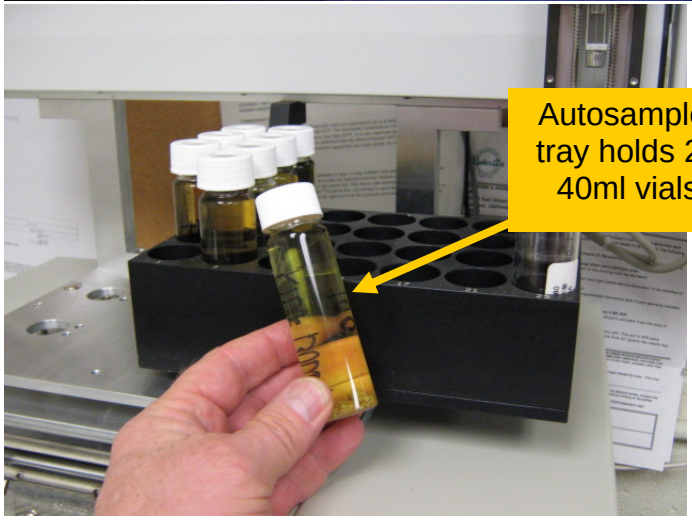
SRI FID detector is self-igniting and blowout proof

Medical Cannabis Gas Chromatograph
Automated Hi-volume Configuration

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

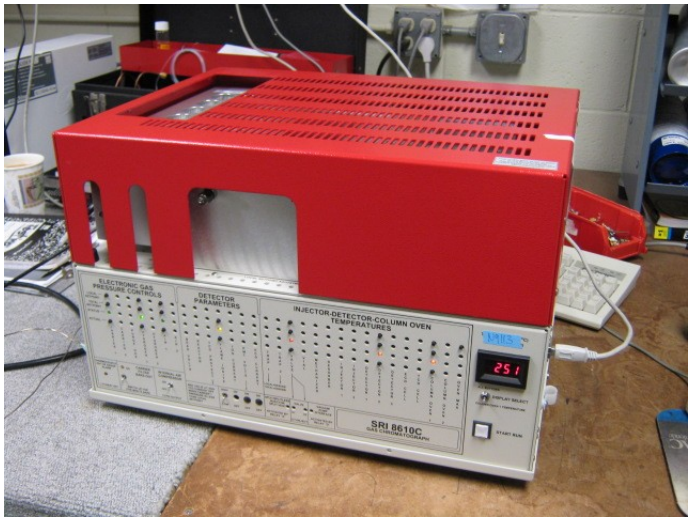
This GC configuration is appropriate for user's who have higher numbers of samples per day to analyze for CBD, THC and CBN. The autosampler accommodates 28 of the 40milliliter extraction vials so users do not have to transfer the THC extract from the extraction vial to a smaller autosampler vial thus 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.



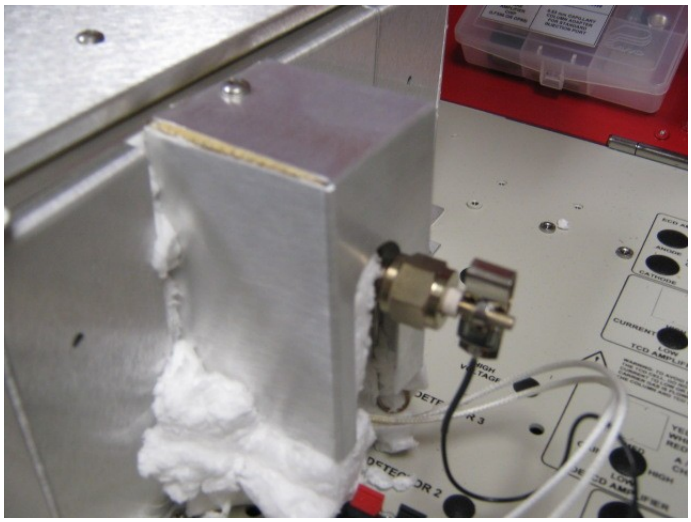
Medical Cannabis Potency Testing using the SRI 8610C FID GC

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).



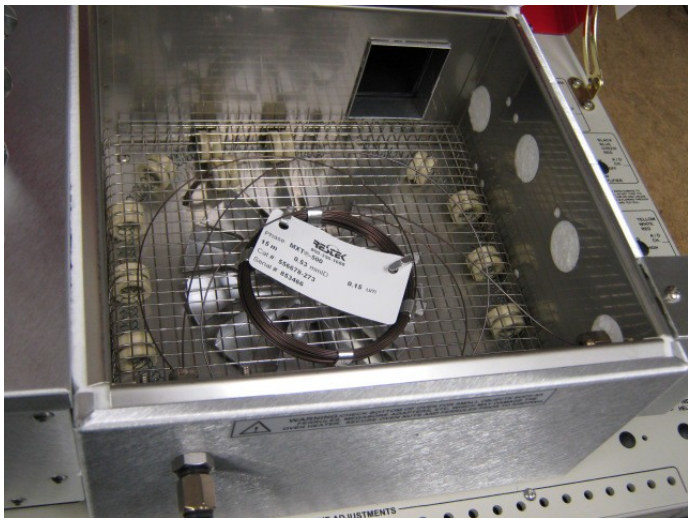
The 12-vial sample heater aids in a quicker extraction of the cannabinoids in solvent and maintains the extracted samples at 50° C for better reproducibility.

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.

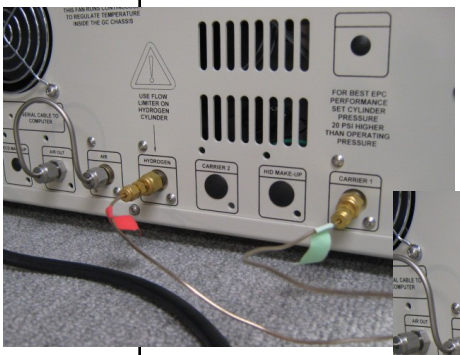


Medical Cannabis Potency Testing using the SRI 8610C FID GC

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.



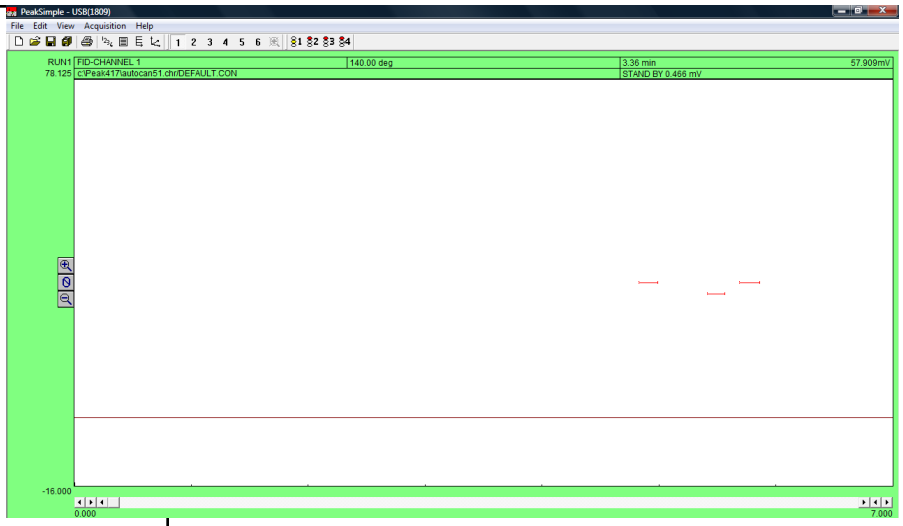
Hook up the gas lines to the left side of the GC. The GC can be operated with hydrogen or helium as a carrier gas. When using hydrogen as a carrier gas, cap off the hydrogen gas inlet and connect the hydrogen to the carrier 1 inlet.



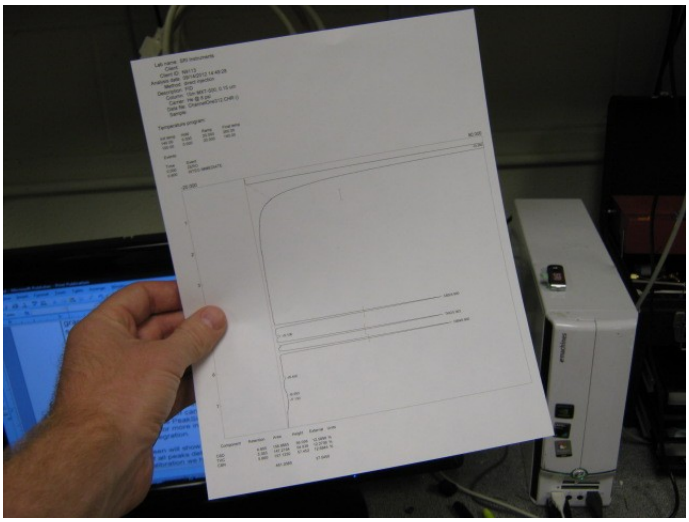
The entire GC plugs into any Windows computer using a USB cable.

Medical Cannabis Potency Testing using the SRI 8610C FID GC

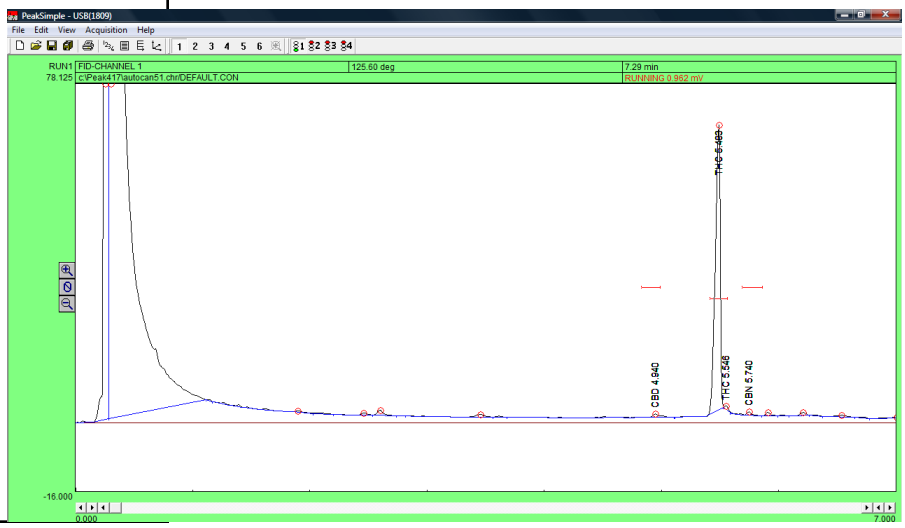
SRI's PeakSimple software is included with the GC. PeakSimple software collects the GC data and generates a calibrated result which can be printed or transferred to other programs such as Excel or Word.



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

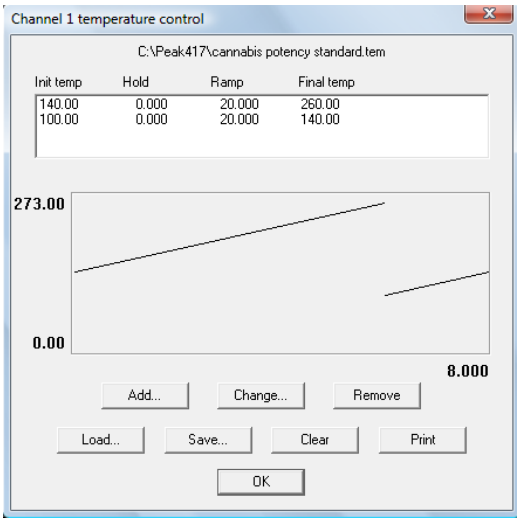


An actual cannabis sample is shown at right.

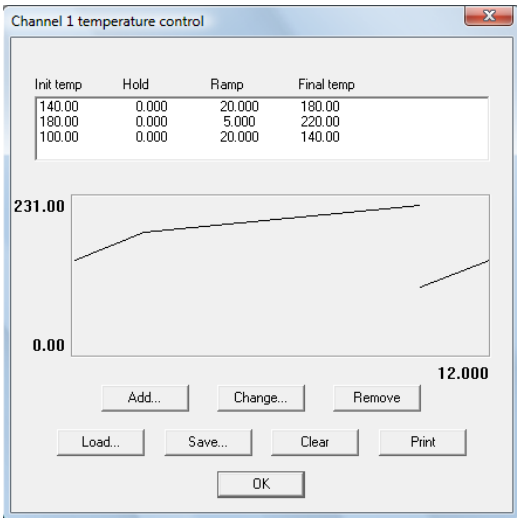


Medical Cannabis Potency Testing using the SRI 8610C FID GC

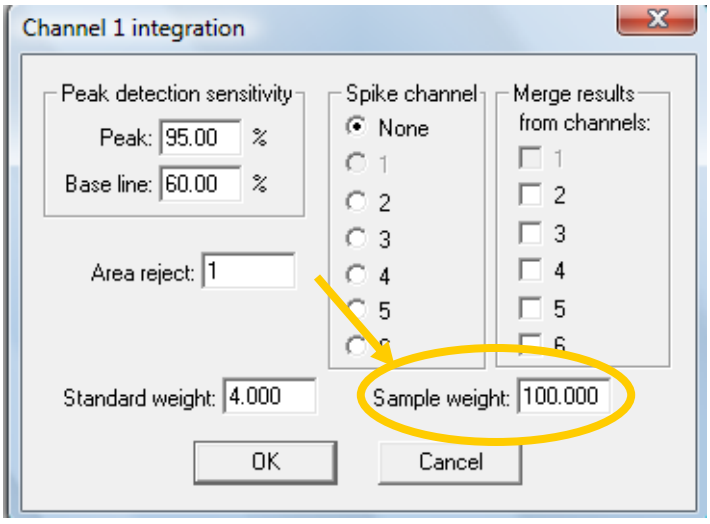
For a quick 8-minute analysis that optimizes speed *and* peak separation, set the column oven temperature as shown to the right.



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.

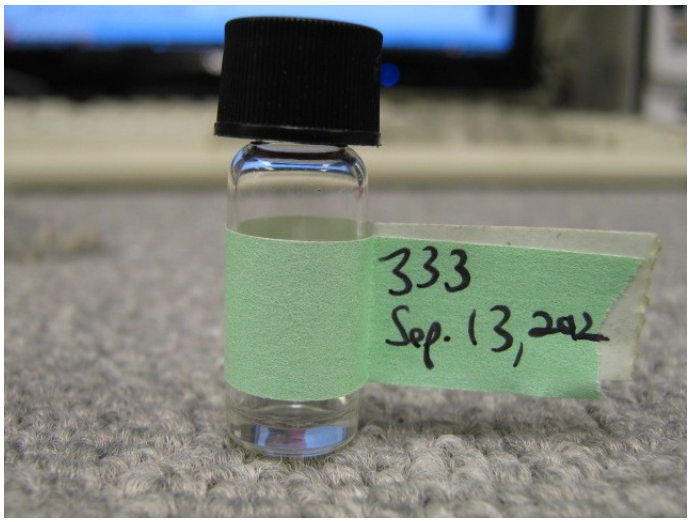
Whether you have three vials (individual standards of THC, CBD, or CBN) or one vial of 3-way standard, they will each be at a concentration of 1000ng/ul. We will refer to these standards as primary standards. Ideally, when not in use they should be kept in a refrigerator with an unpierced septum so that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

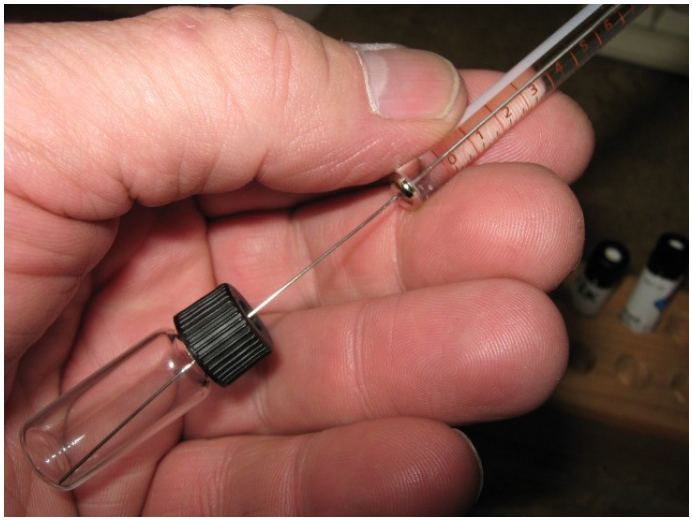
If you have separate cannabinoid standards, use the 100uL syringe, which is included with the SRI GC, (Restek#24863) to transfer 100uL of each 1000ng/uL (primary) standard into another 2mL vial. If you have the 3-way standard, use the 100uL syringe to transfer 100uL of the standard into another 2mL vial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

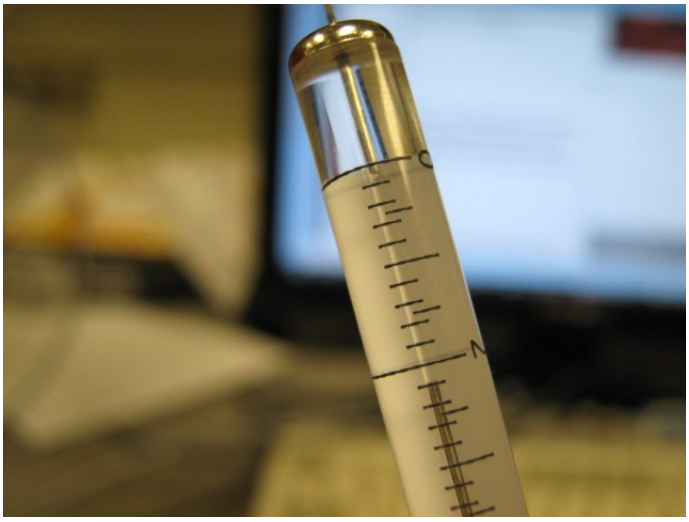


Medical Cannabis Potency Testing using the SRI 8610C FID GC

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.

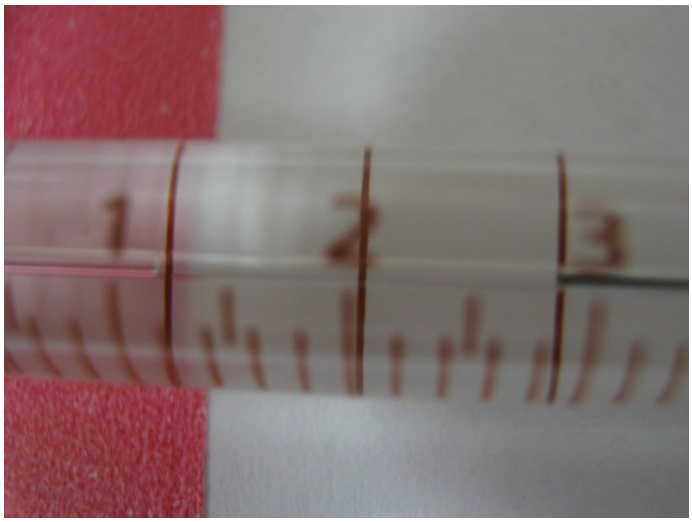


With air bubbles removed, push the plunger to the 1ul mark. It is important to be as precise as possible. Wipe the needle with your fingers or a tissue to remove any liquid from the outside of the needle.

Medical Cannabis Potency Testing using the SRI 8610C FID GC

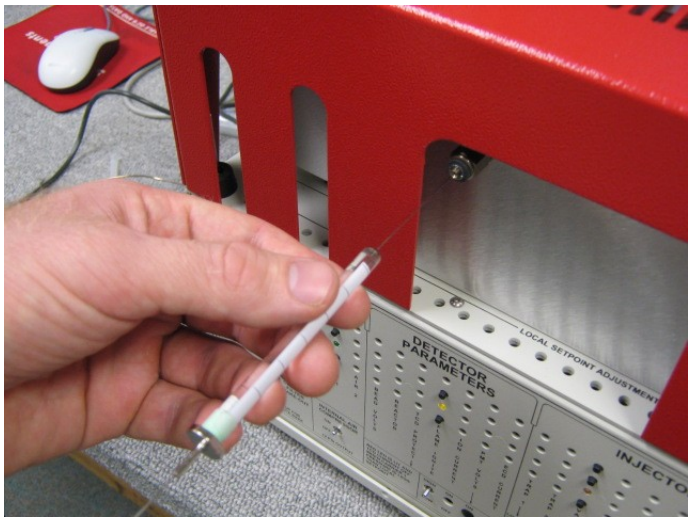
Pull the plunger back to the 3ul mark and note the amount of liquid. It should be 1.6-1.8ul because the needle also contains .6-8ul and this adds to the 1ul you measured with the plunger.

Leave the plunger at the 3ul mark.

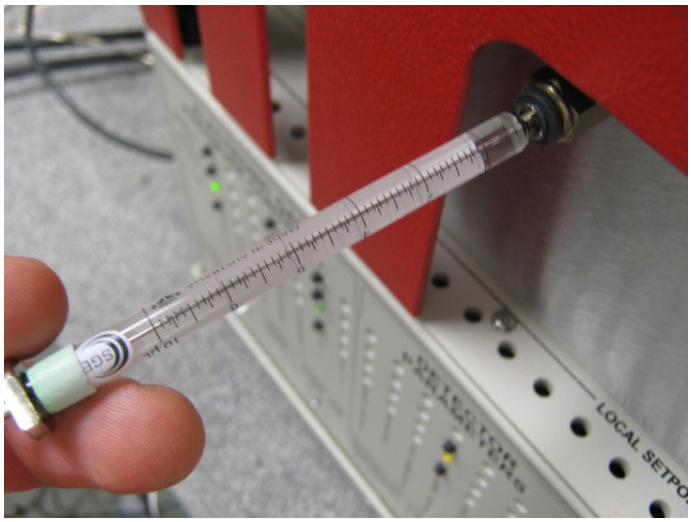


With the plunger still at the 3ul mark, place the needle up against the septum (but not poking through it yet).

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

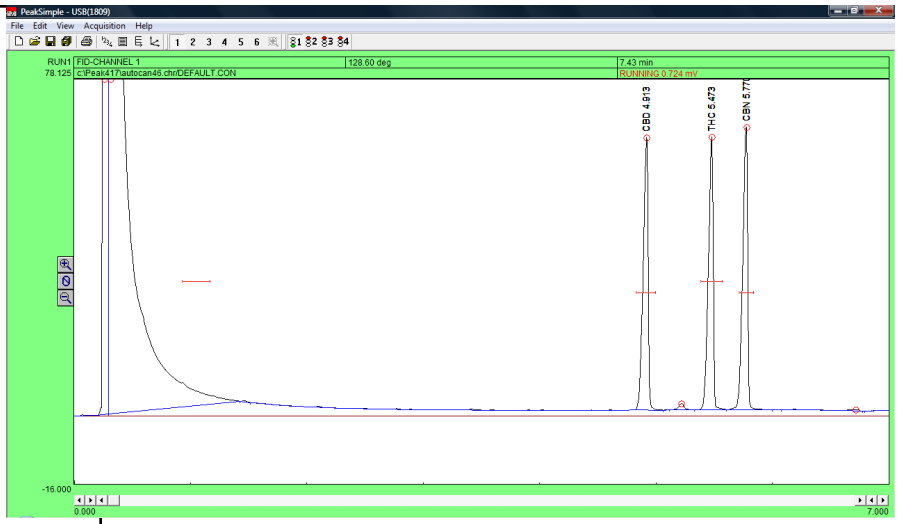


Insert the syringe all the way through the septum as far as it will go. Immediately depress the plunger. Twist the syringe one half turn (to wipe off any liquid on the tip of the needle) and then withdraw the syringe.

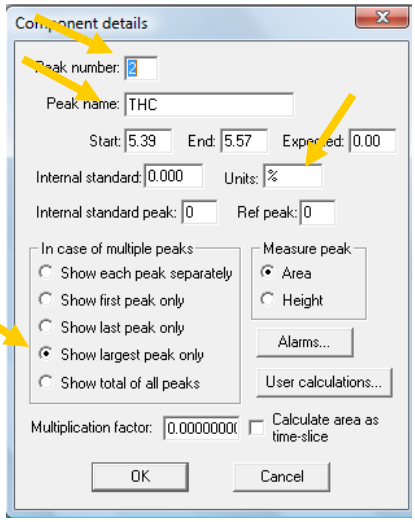


Medical Cannabis Potency Testing using the SRI 8610C FID GC

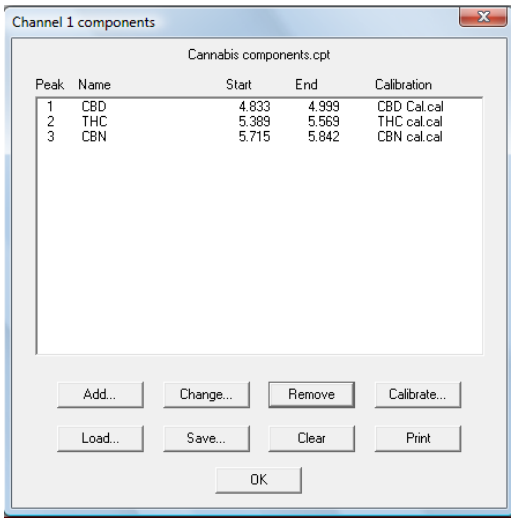
Once the run is completed you should see a large solvent peak near the beginning, then closer to the end, three peaks of roughly equal size (there will also probably be a small Delta-8 THC peak between the 1st and 2nd peak). Add retention windows to the three peaks by right clicking on the peak and selecting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

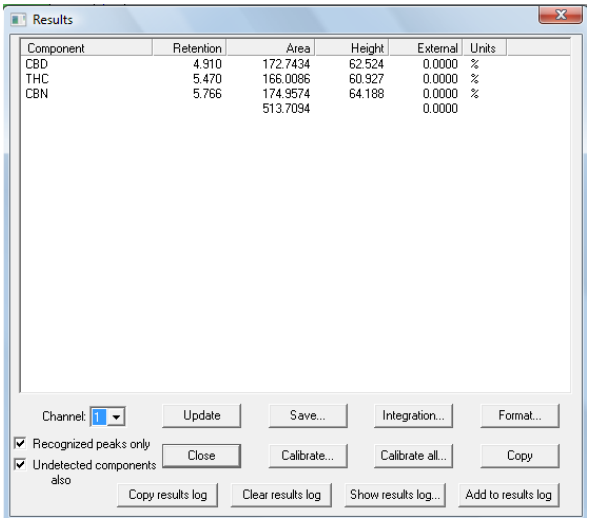


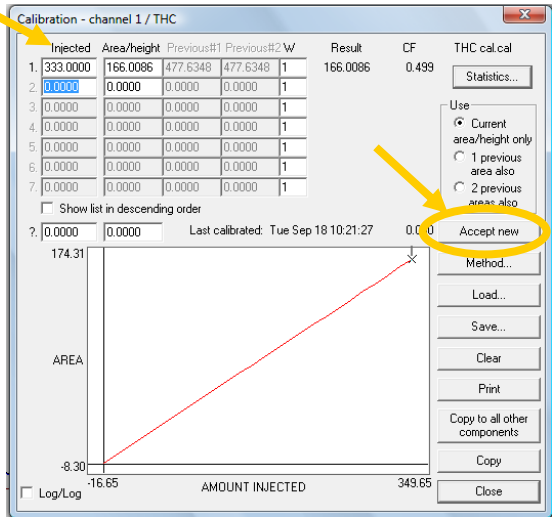
Medical Cannabis Potency Testing using the SRI 8610C FID GC

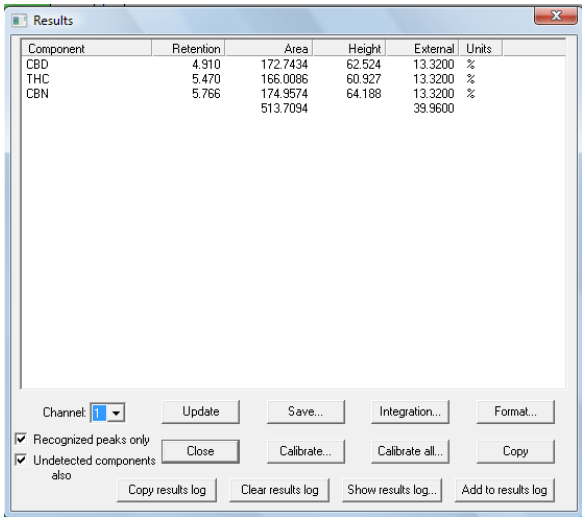
Check the Results screen. If you injected a primary standard the area counts for the cannabinoids should be between 420 and 540 and roughly equal to each other (+/- 30). If you injected the working standard the area counts should be between 140 and 180 and roughly equal (+/- 10).

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.



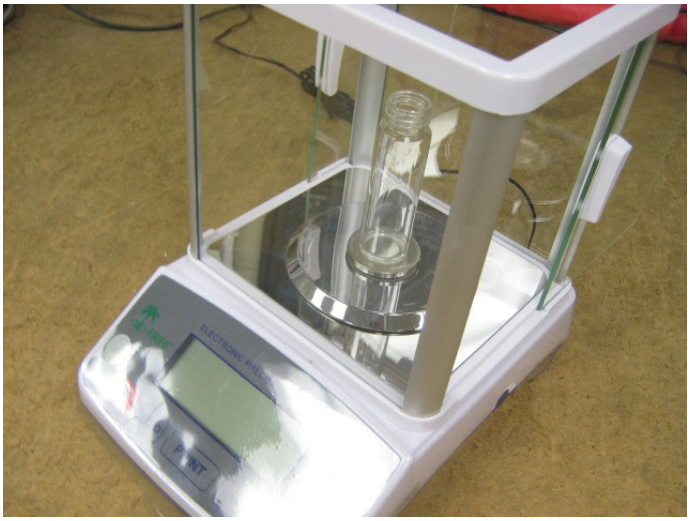




Medical Cannabis Potency Testing using the SRI 8610C FID GC

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.

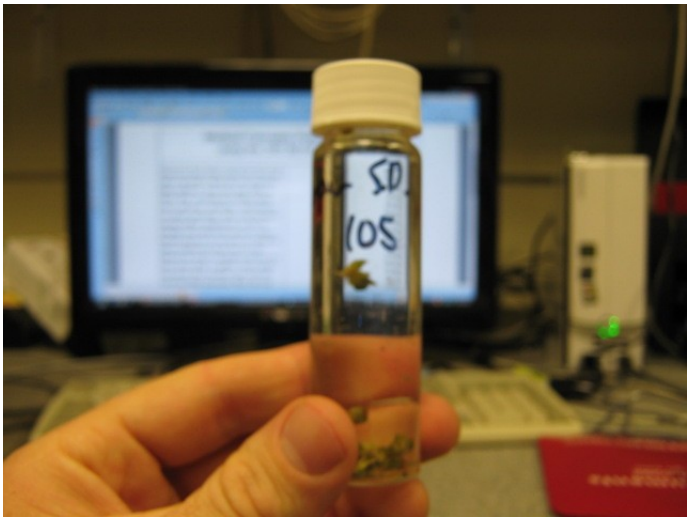


Don't worry if you are slightly under or above 100. In the photo at right, the reading is 98 milligrams which is close enough. You will enter the reading in the sample weight field in Peak-Simple software which will mathematically correct the calculated answer to compensate for weights slightly above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

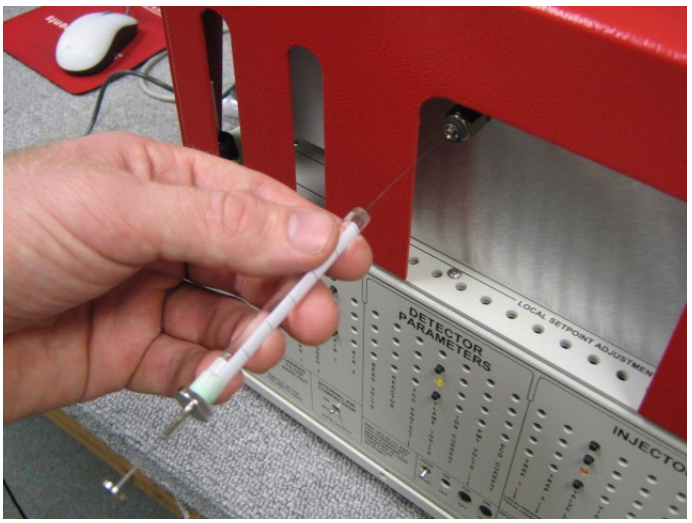
Medical Cannabis Potency Testing using the SRI 8610C FID GC

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).

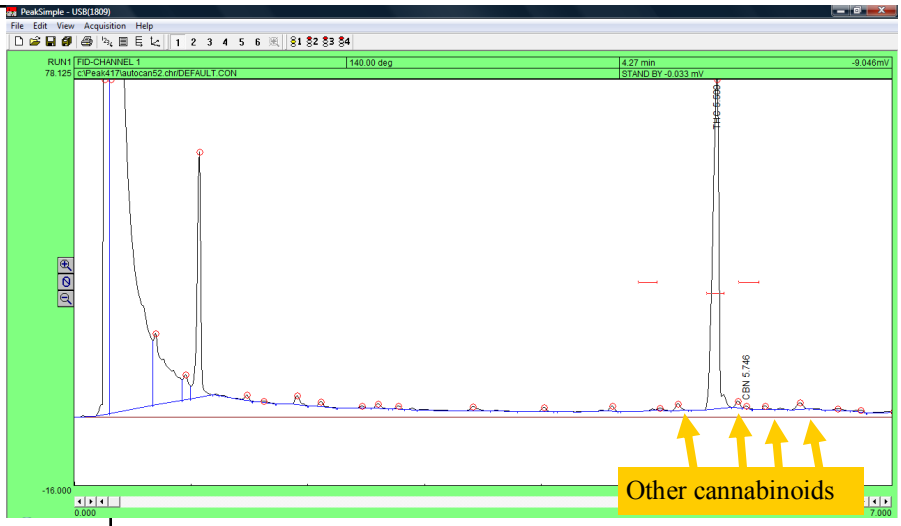


Use the 10ul syringe which comes with the GC to inject 1ul of the extract as shown previously with the calibration standard. It is important to be very precise with the syringe since the overall accuracy of the test depends on this. Don't forget to enter the exact Sample weight in the proper field on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



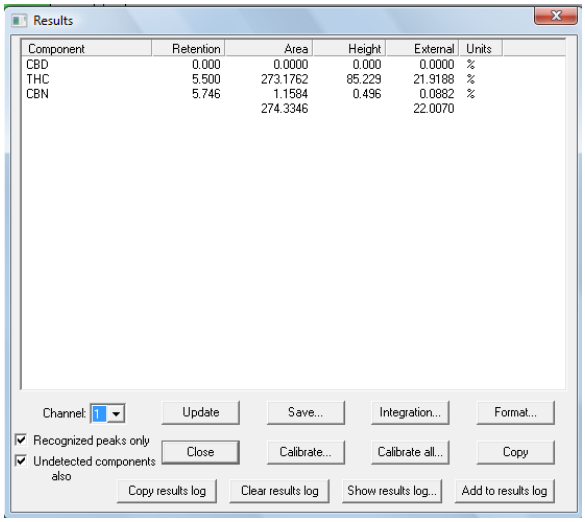
CBN may or may not be detected or it may blend into the much larger THC peak. When this happens you can use the slower temperature program and/or lower the carrier pressure to get better separation of the peaks.

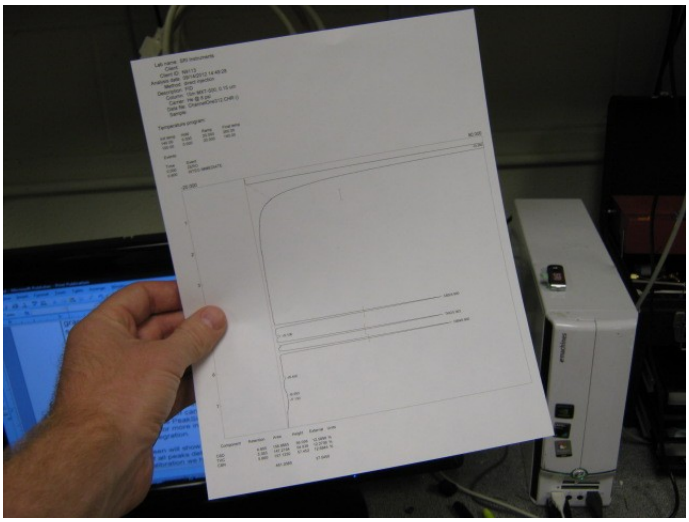
There may be other peaks which are not CBD, THC or CBN. These other peaks are cannabinoids (CBC, Delta-8 THC, CBG, and others) for which there may or may not be calibration standards available at this time.

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.

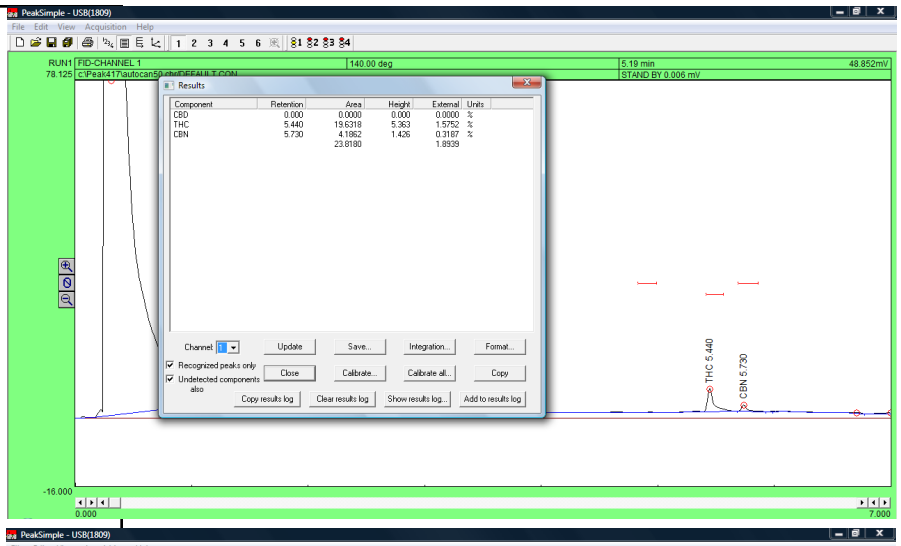
Print the chromatogram and results for a hardcopy record of the analysis.



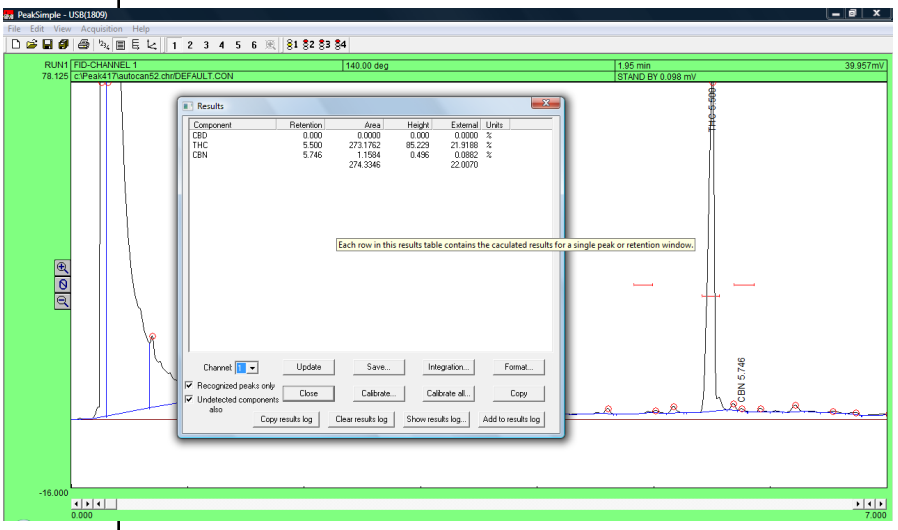


Medical Cannabis Potency Testing using the SRI 8610C FID GC

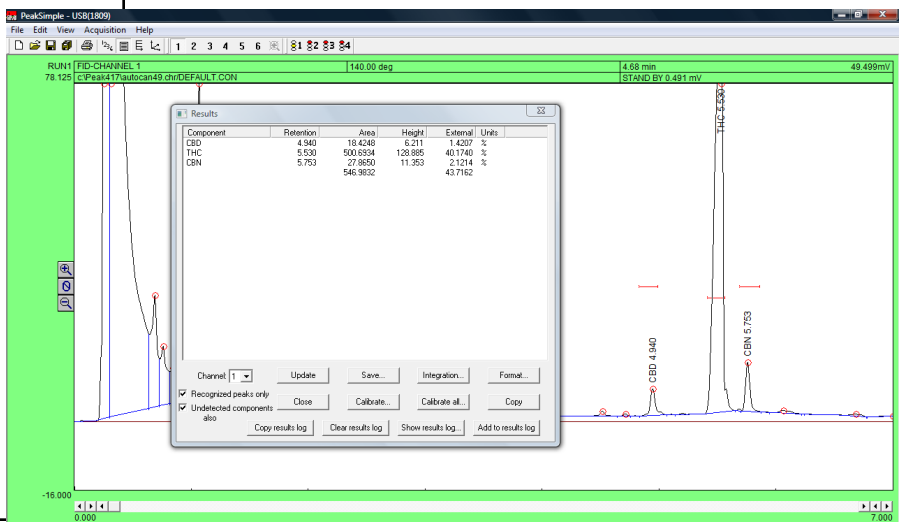
Chromatogram of a low-potency cannabis flower sample with 1.6 % THC.



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



Chromatogram of a typical cannabis concentrate with 40.2% THC.



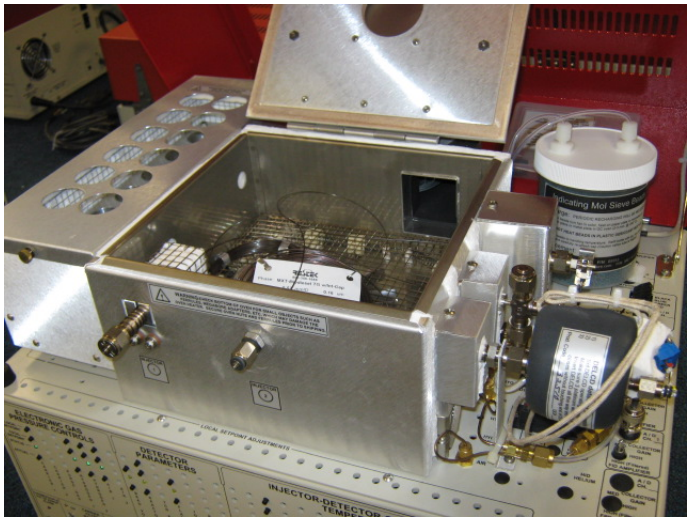
Medical Cannabis Gas Chromatograph Pesticides and Potency Configuration

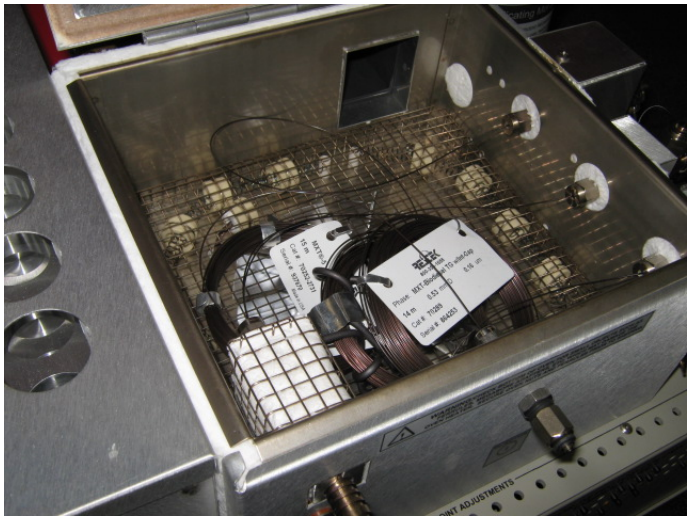
Configuration #4
Potency plus Pesticides
GC configuration
Part# 8610-0092 $21,889.00

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.

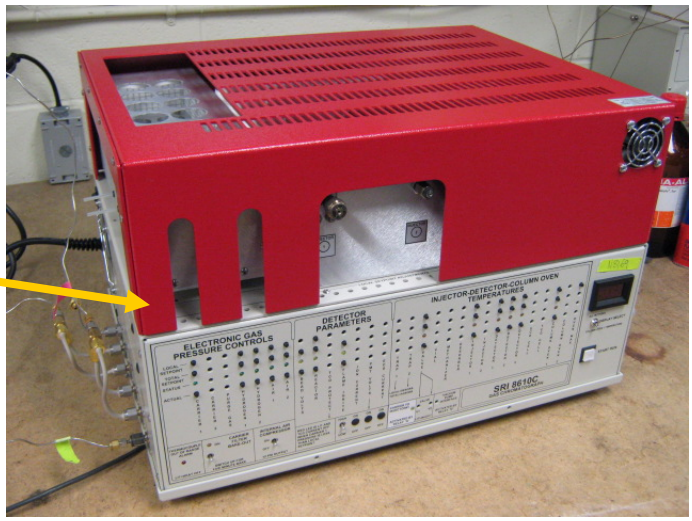
This GC configuration is appropriate for users with prior GC experience since the pesticide screen is more complex than the potency test. It should be understood that while 90% of all pesticides can be detected with this GC configuration, it is not possible to measure every possible pesticide since there 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

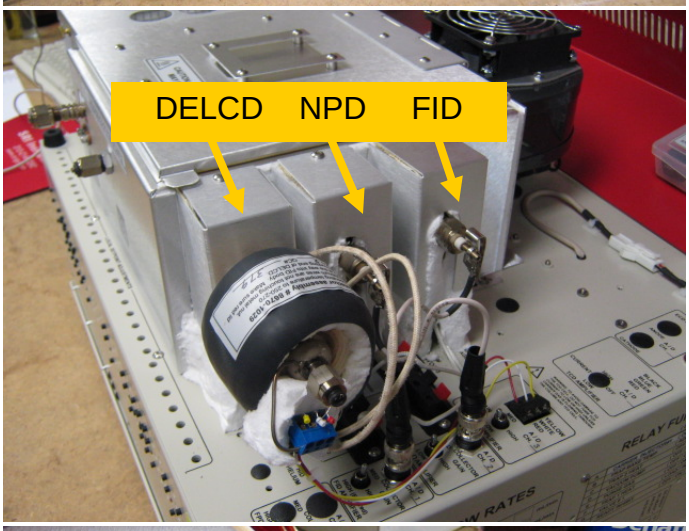
The SRI 8610C Gas Chromato-graph (GC) configured for Medical Cannabis Potency and Pesticide testing is shown at right.



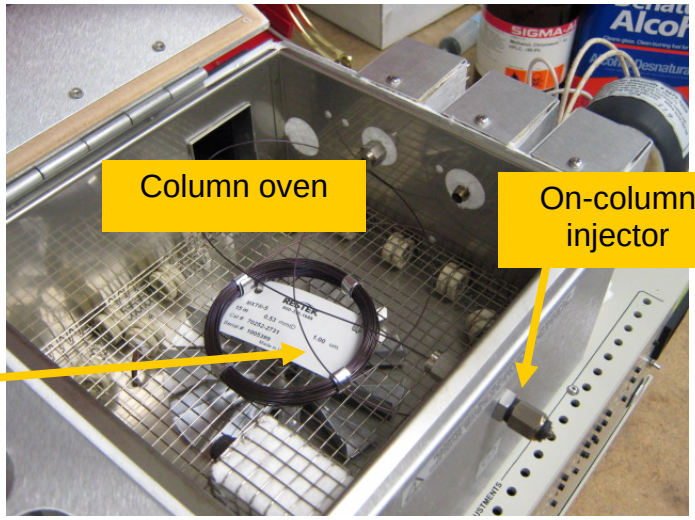
The GC is equipped with three detectors:

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.

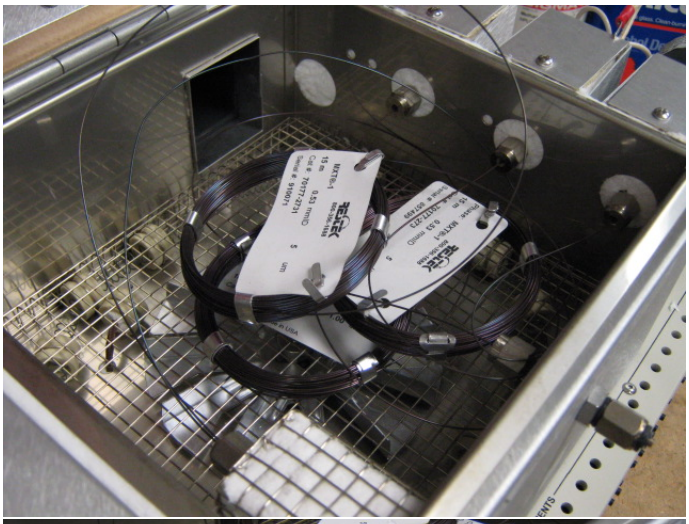


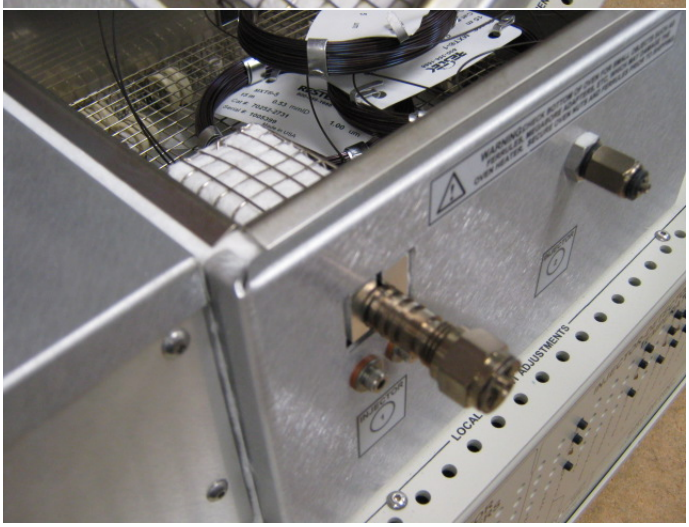
Medical Cannabis Pesticide Screening using the SRI 8610C GC

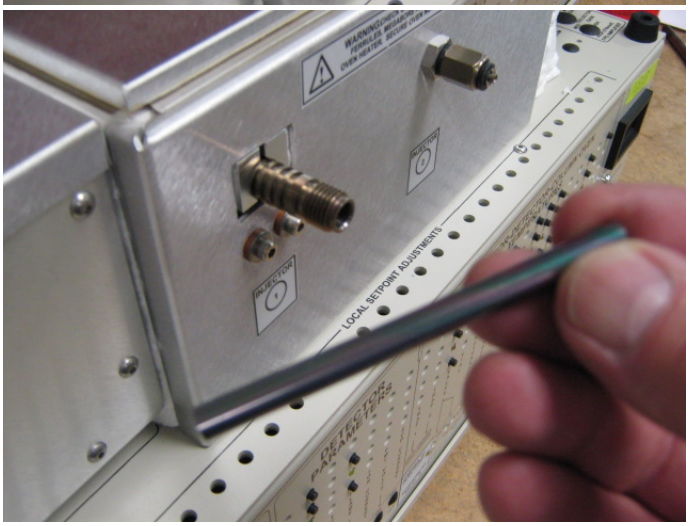
Two additional columns can be connected to the Heated Injector. One of these columns goes to the NPD detector and the other to the DELCD detector. The Heated Injector splits the sample onto the two columns using a two hole ferrule Restek part# 20246

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.



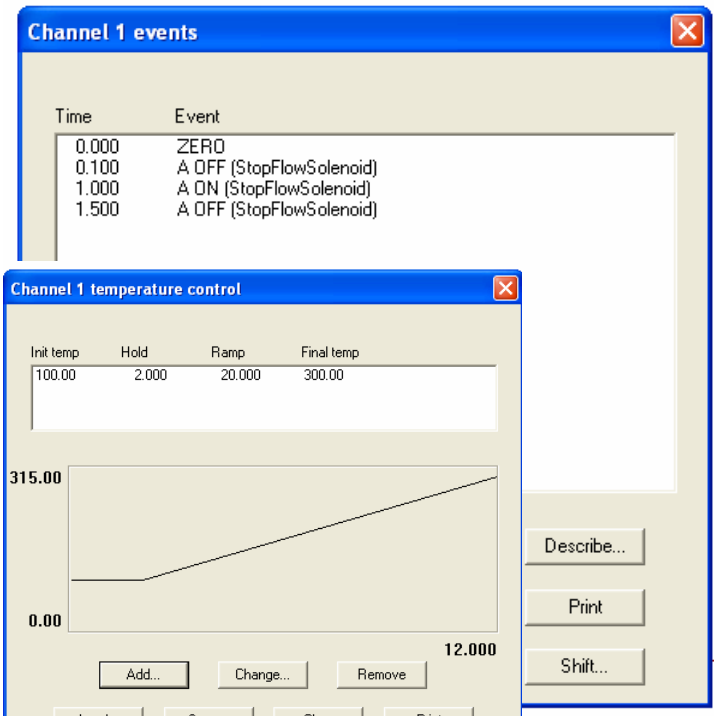


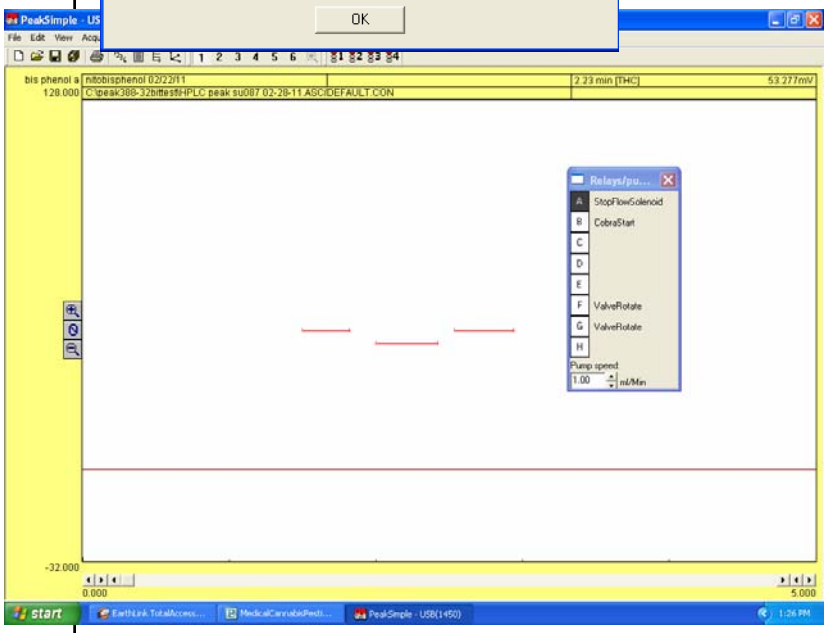


Medical Cannabis Pesticide Screening using the SRI 8610C GC

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.

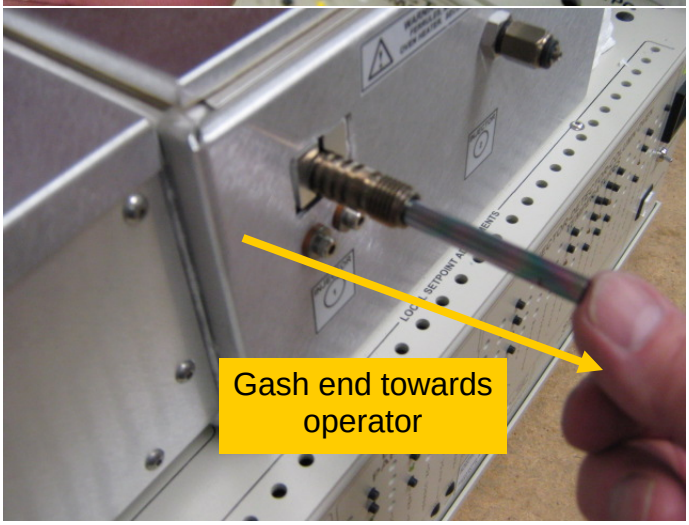
Weigh the tube until you get approximately 100 milligrams. You do not have to get exactly 100 so long as you are close (95-105 mg). The photo are right shows the weight at 99 milligrams. You can correct for the actual sample weight in the PeakSimple software after the analysis.

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.

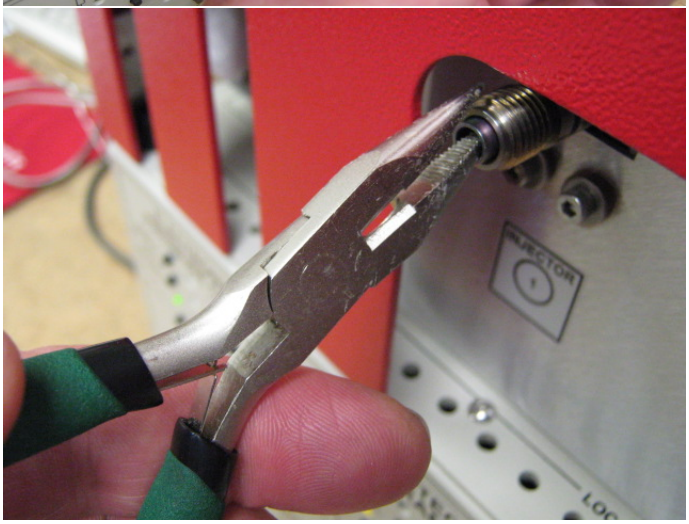
<u>The gash end MUST be towards the operator.</u>

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.

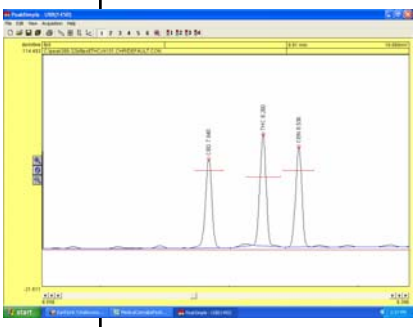
If your GC is equipped with a second injector and FID detector for potency measurement, you can inject the potency extract in the other injector anytime in the first 1 minute of the analysis.

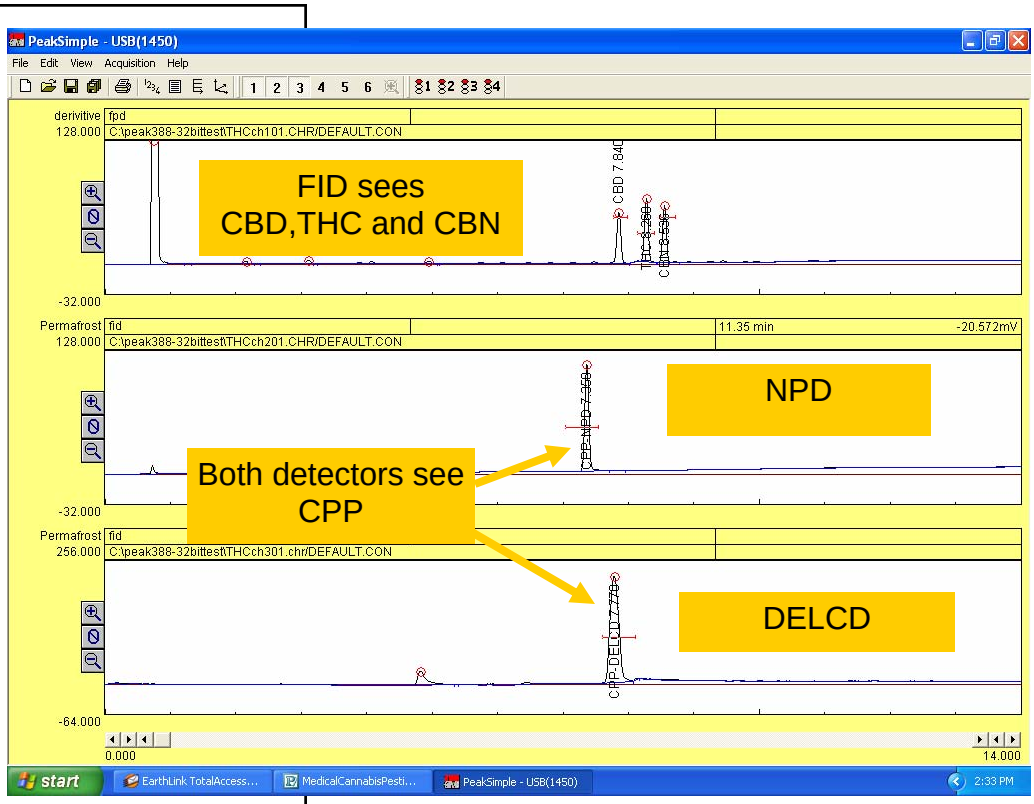
At 1 minute into the analysis, the carrier gas is turned off for 30 seconds. During that 30 second period remove the tube from the HOT injector using a tool to avoid burning your fingers. Place the HOT tube in a beaker to cool off. You must replace the septum nut within the 30 second window.



Medical Cannabis Pesticide Screening using the SRI 8610C GC

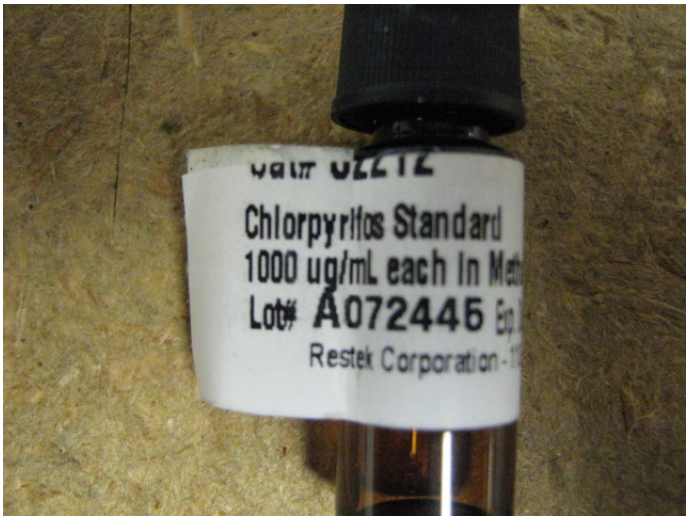
To calibrate the Potency channel (channel 1), inject 1ul of the 333ng/ul calibration mixture into the on-column injector. You should see three equal size peaks.





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

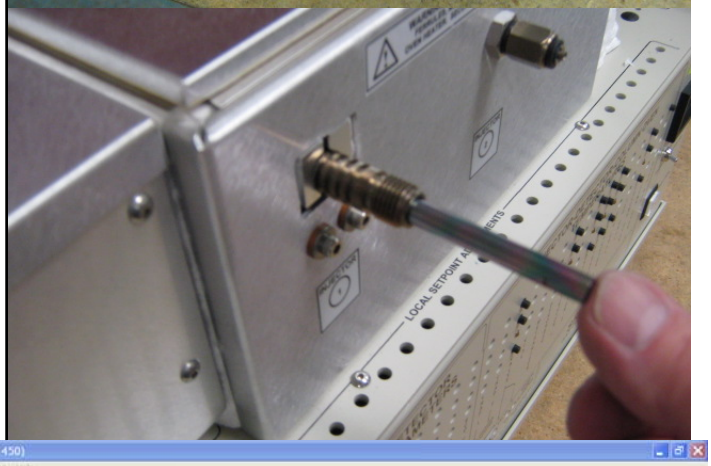
The two pesticide detectors (NPD and DELCD) are calibrated with a pesticide standard such as Chlorpyrifos. Restek part# 32212 is 1000ug/ml (1000ppm) of chlorpyrifos (CPP) in methanol. CPP was chosen as the calibration pesticide because it has both phosphorus (which the NPD detects) and chlorine (which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

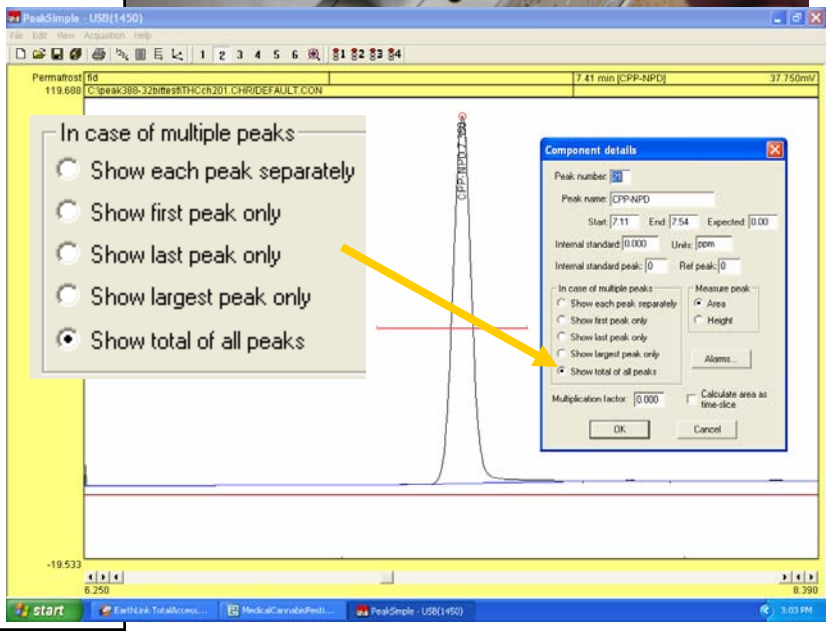
Then desorb using the standard program and events.



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

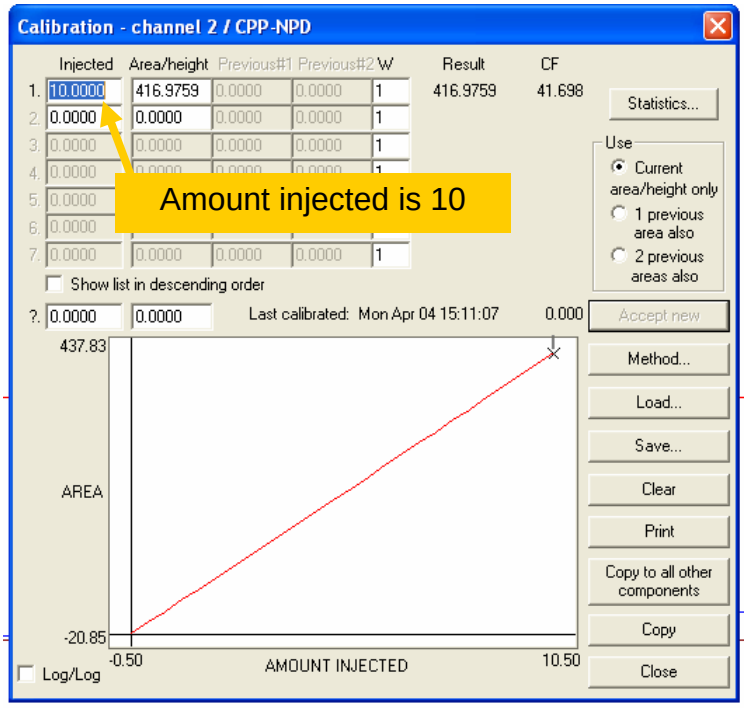
Create a calibration curve for the CPP in both NPD and DELCD channels. Note that the amount injected is set to 10.

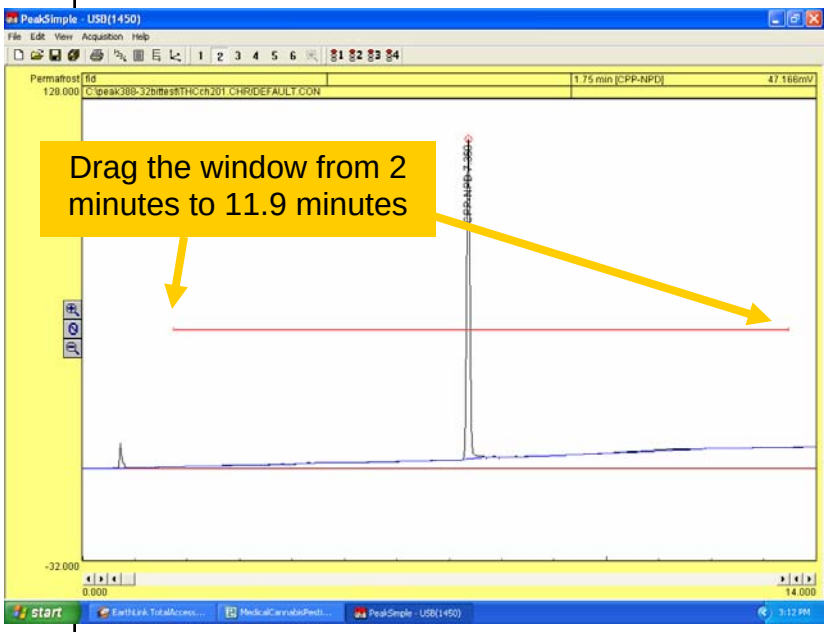
We injected 1ul of CPP standard which contains 1000 nanograms of CPP. Since we will be desorbing 100milligrams of cannabis, 1000 nanograms is 10ppm, hence the number 10 in the amount injected column.

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%

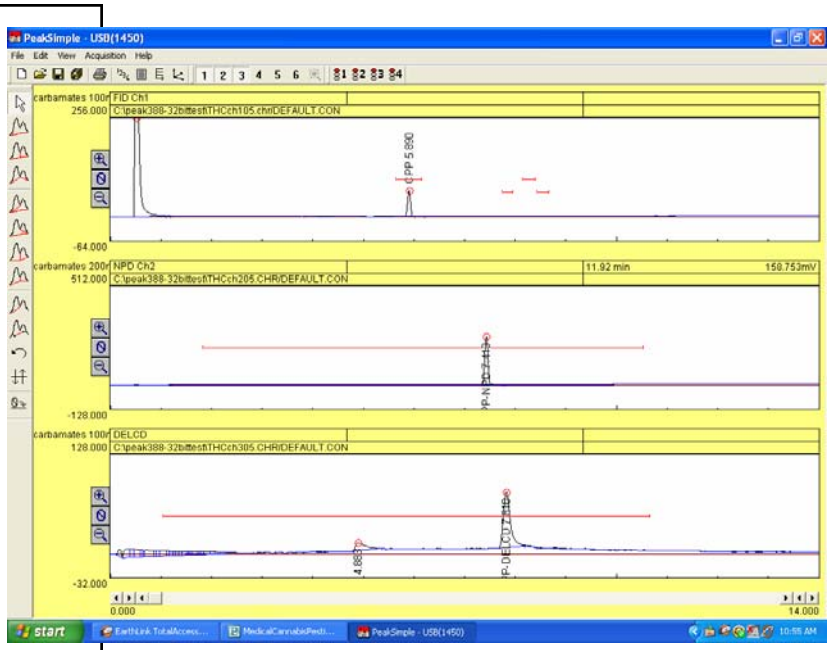




Medical Cannabis Pesticide Screening using the SRI 8610C GC

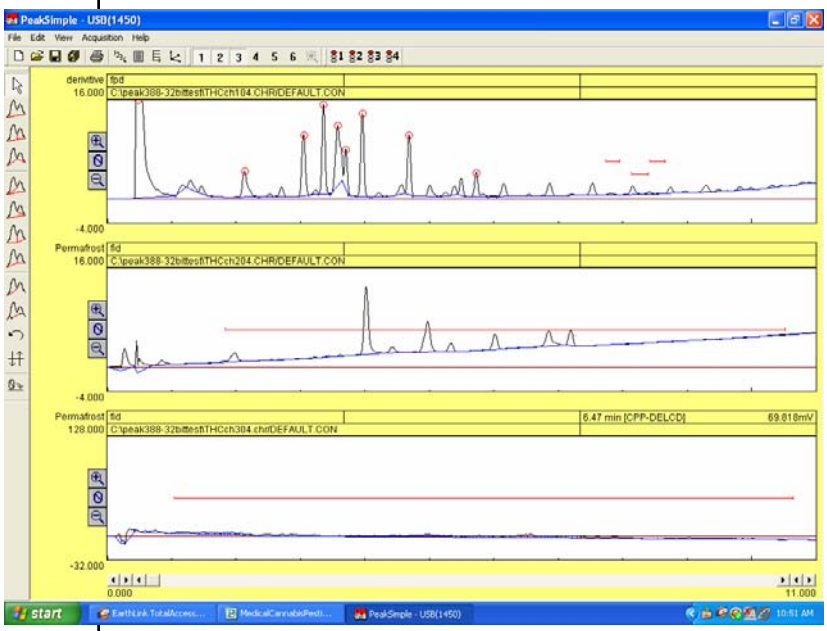
The chromatograms at right show the CPP peak on all three channels. The top channel (FID) was injected with the CPP standard just for comparison. Normally the FID channel is used for potency (CBD, THC, CBN).

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



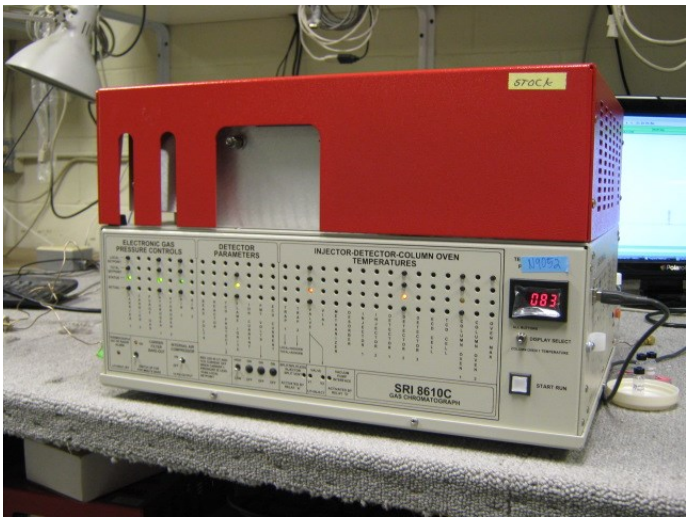
The chromatograms to the right show carbamate pesticides.

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

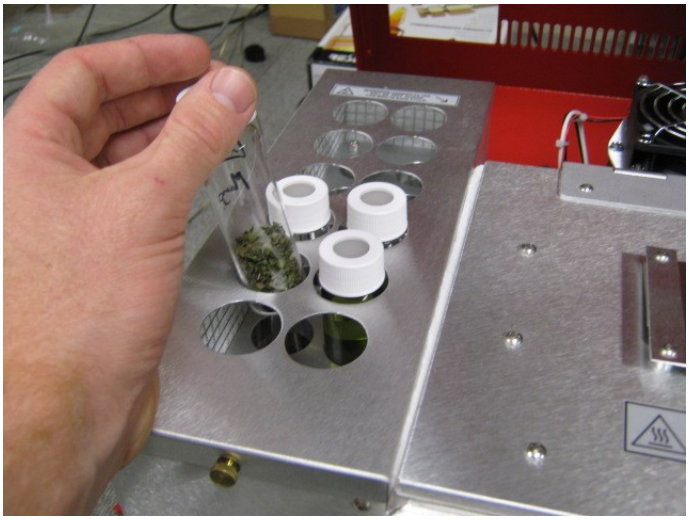


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

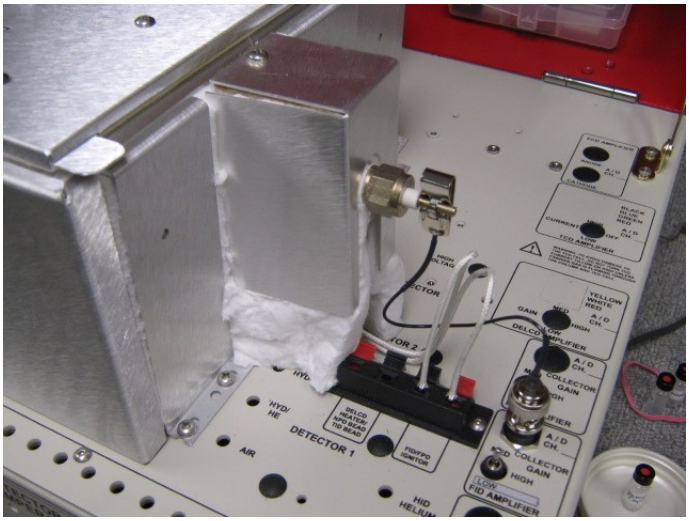
The SRI Medical Herb Potency 8610C GC is shown at right. This GC can also be used to test for residual solvents (i.e. butane, acetone, gasoline residue, etc.) in medical cannabis. These solvents are used in the extraction process to create medical cannabis hash oils and concentrates.



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).

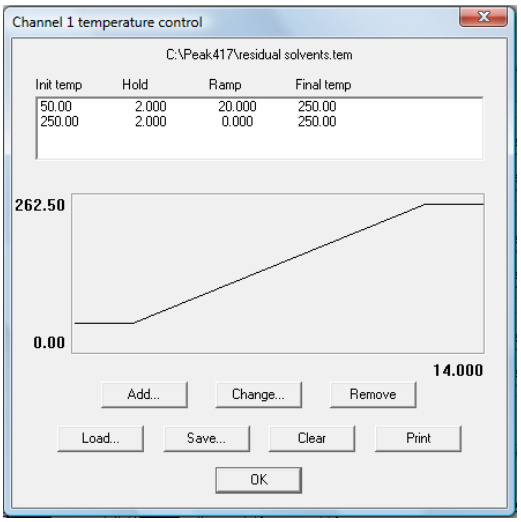
Many types of columns could be used to separate these molecules, but SRI suggests a 15 meter MXT-1 with a 5 micron film thickness and .53mm id. This column can distinguish between solvents like pentane and hexane and does a good job of separating terpene molecules.

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.

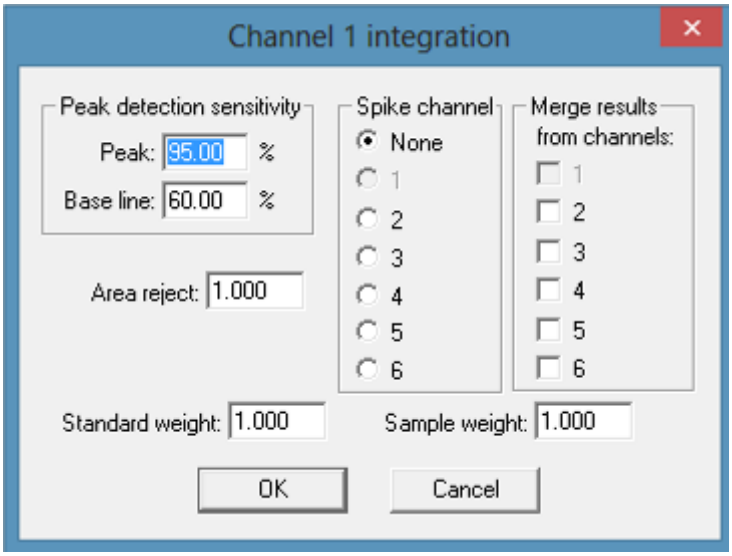
Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

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.



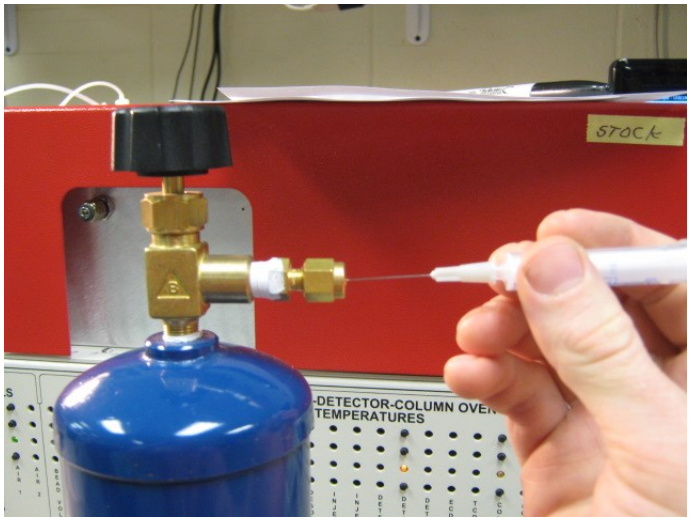
Set the Integration parameters as shown.



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).

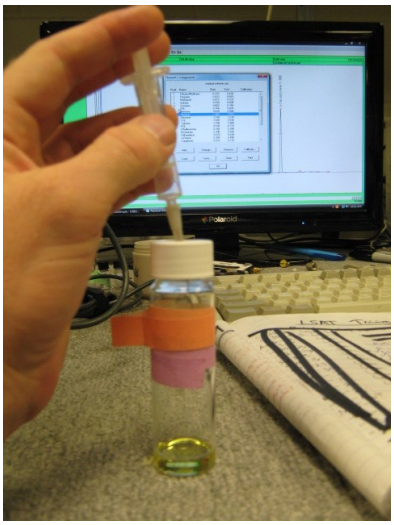
Pressurize the gas cylinder by turning the release valve slightly counterclockwise. Pierce the septum with a 3 mL gas syringe and withdraw 1 mL of gas. Remove the syringe from the gas sample bottle.



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.

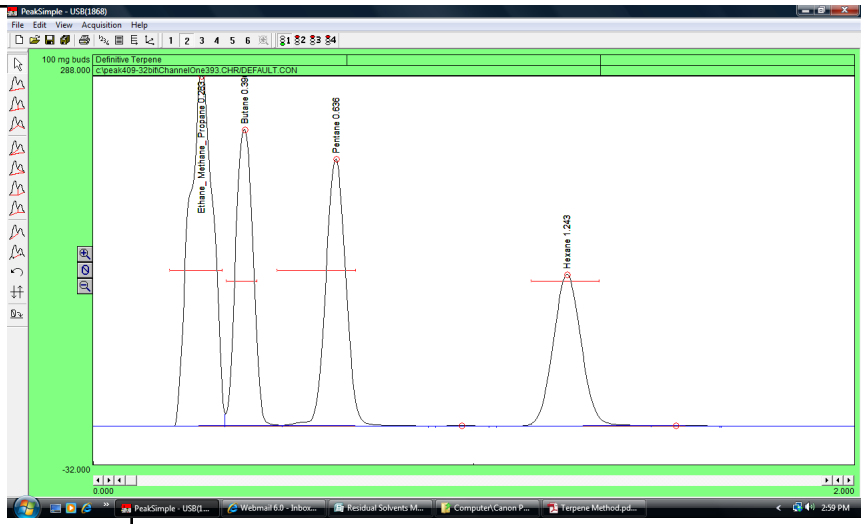


With the syringe plunger still at the 1mL mark, place the needle up against the septum of the injection port (but not poking through it yet). Press the Start Run button or press the spacebar on the keyboard.

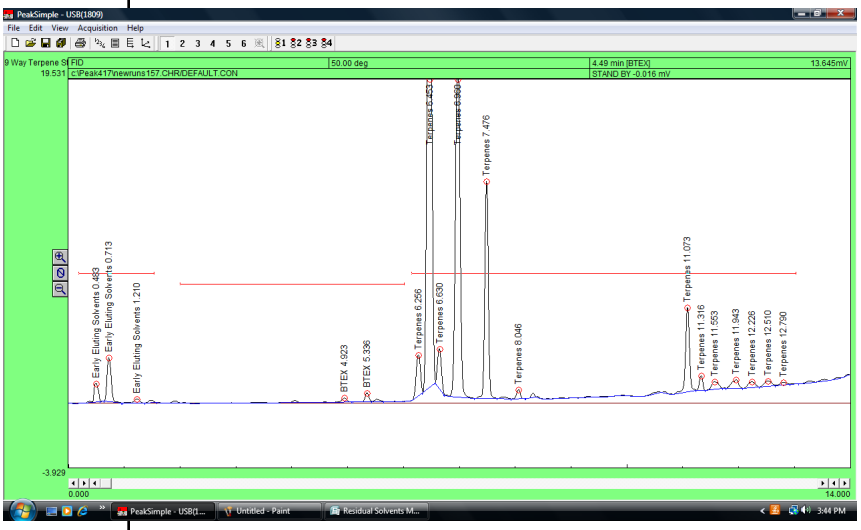
Insert the syringe all the way through the septum as far as it will go. Immediately depress the plunger and quickly withdraw the syringe.

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

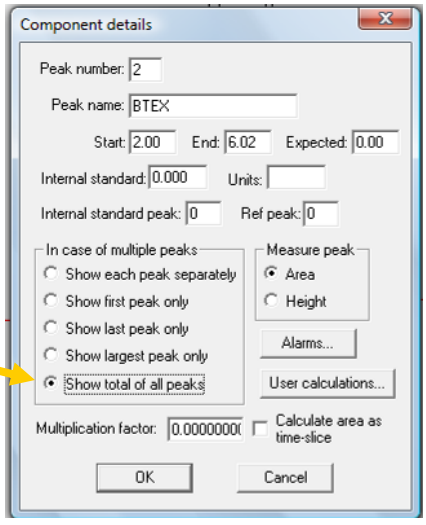
After injecting the C1—C6 standard we see four peaks: ethane, methane, and propane (which all elute together); butane; pentane; and hexane. Identify the peaks so that each peak is defined by a "retention window". See the PeakSimple tutorial describing the process of creating retention windows.



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.



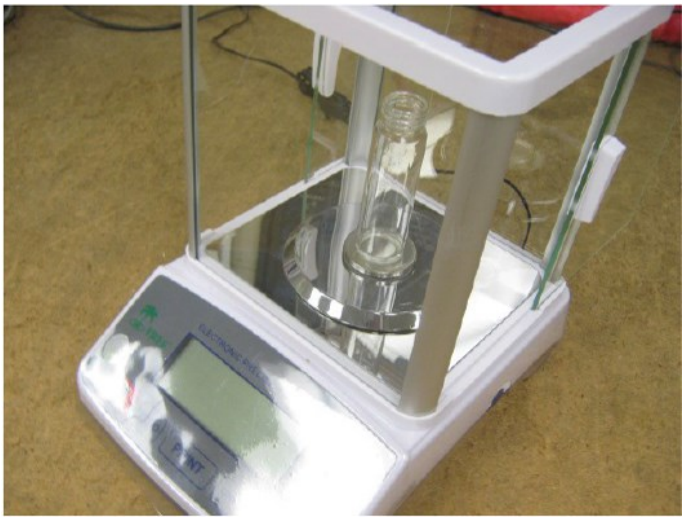
In this case, all the peaks under the retention window need to be quantified. In the Edit Component screen select "Show total of all peaks".



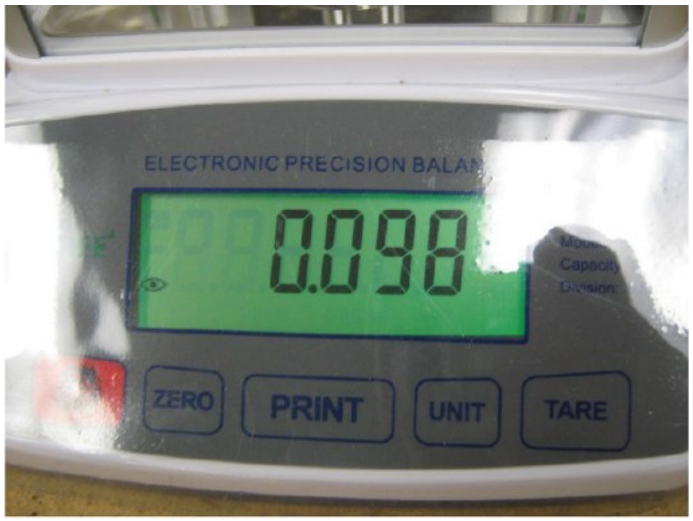
Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

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.

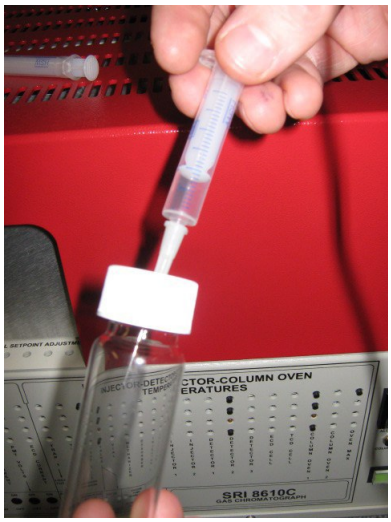


Don't worry if you are slightly under or above 100. In the photo at right, the reading is 98 milligrams which is close enough. Qualitative butane and residual solvent analysis does not depend on an exact measurement of sample, but the operator may find it advantageous to use the same sample for a subsequent 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.

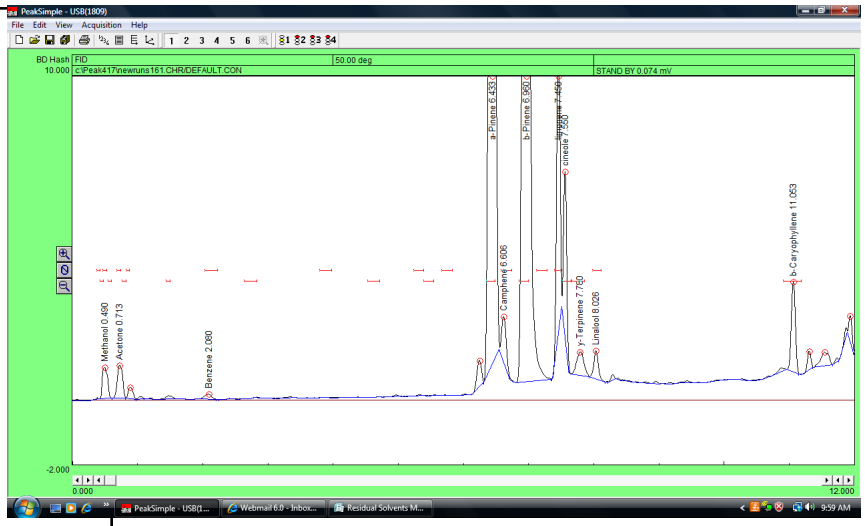


Inject the contents of the syringe into the injection port and start the run as shown previously.

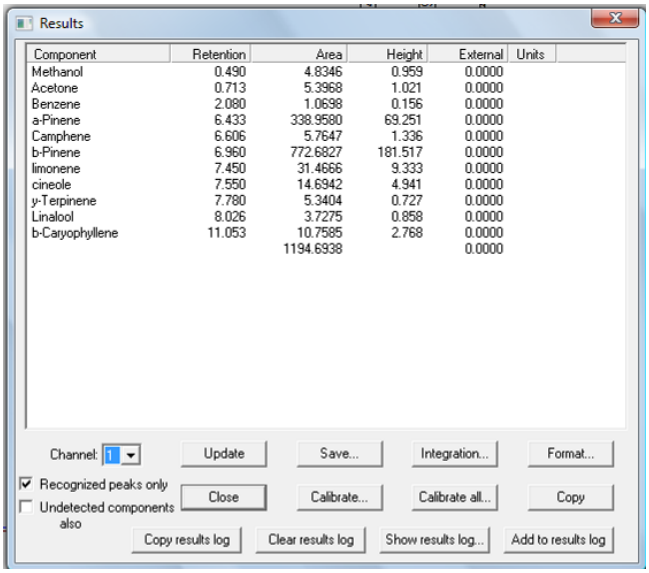
The picture at right shows a butane and residual solvent sample vial filled with 40 mL of extraction solvent and ready to be injected for cannabis potency analysis. See the PeakSimple tutorial describing the process for Medical Cannabis Potency testing.

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

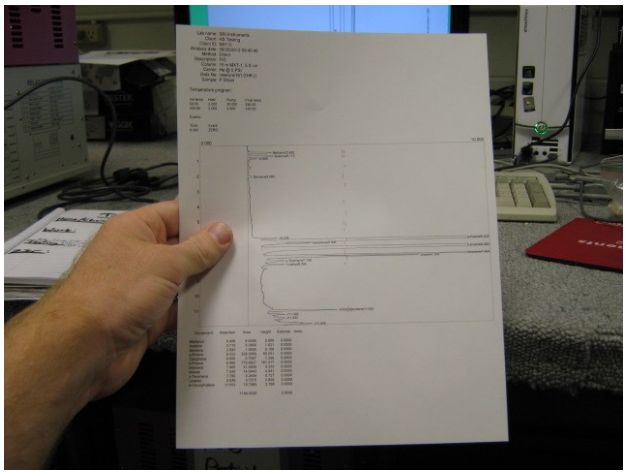
A real cannabis flower sample will look something like the chromatogram at right. This particular sample has standard levels of organic solvents (which are present in low levels naturally in plant matter) and multiple terpenes.



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

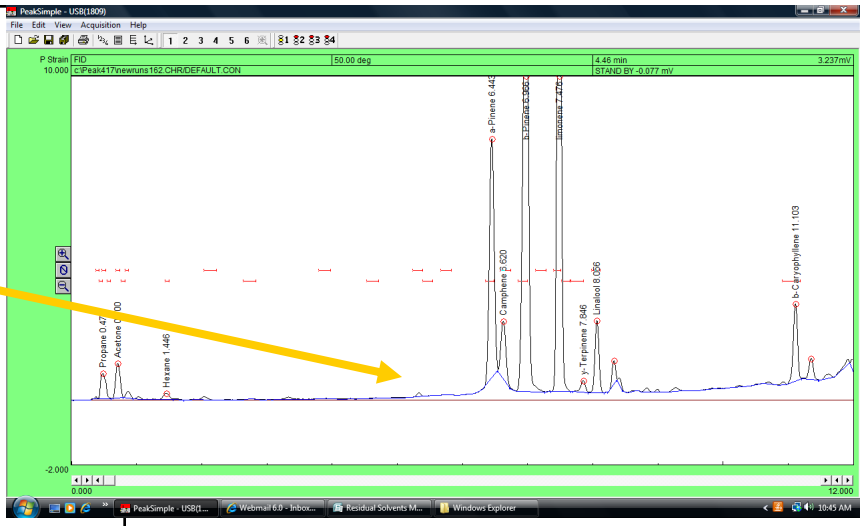


Print the chromatogram and results for a hardcopy record of the analysis.

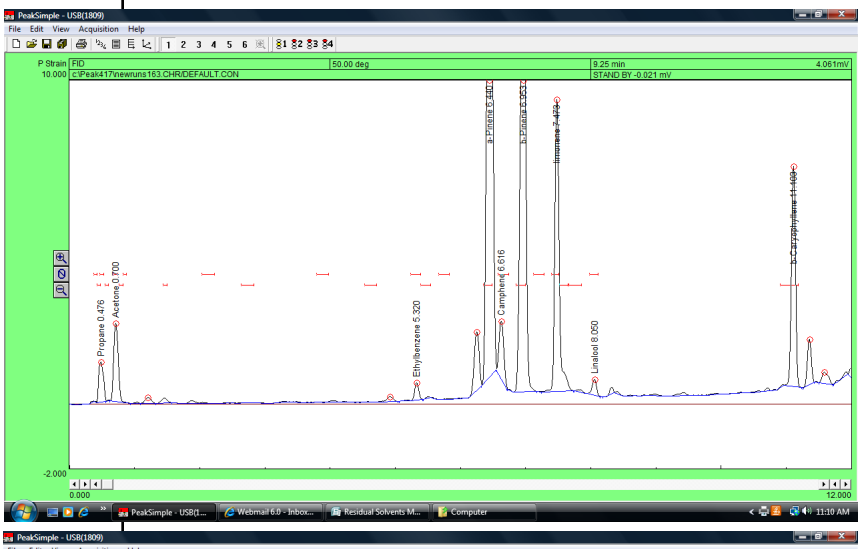


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

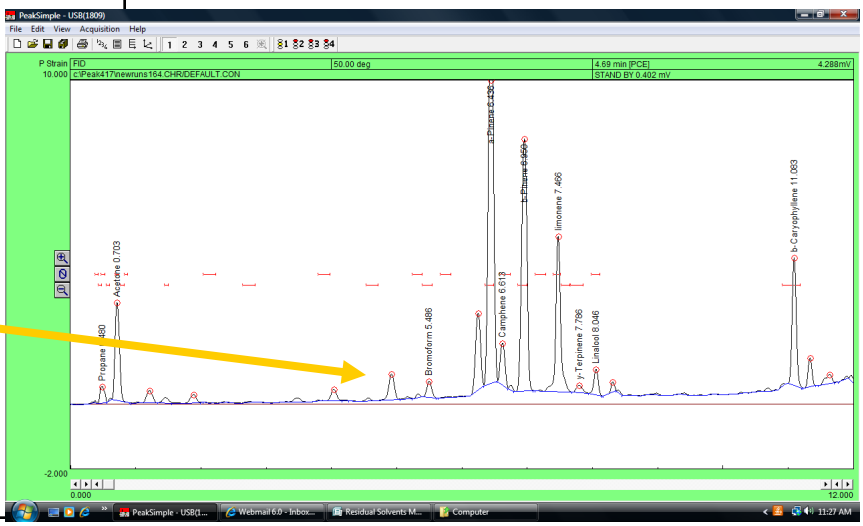
Super Sour Diesel flowers. Notice the lack of any BTEX-like adulterants.



Blue Dream concentrate.

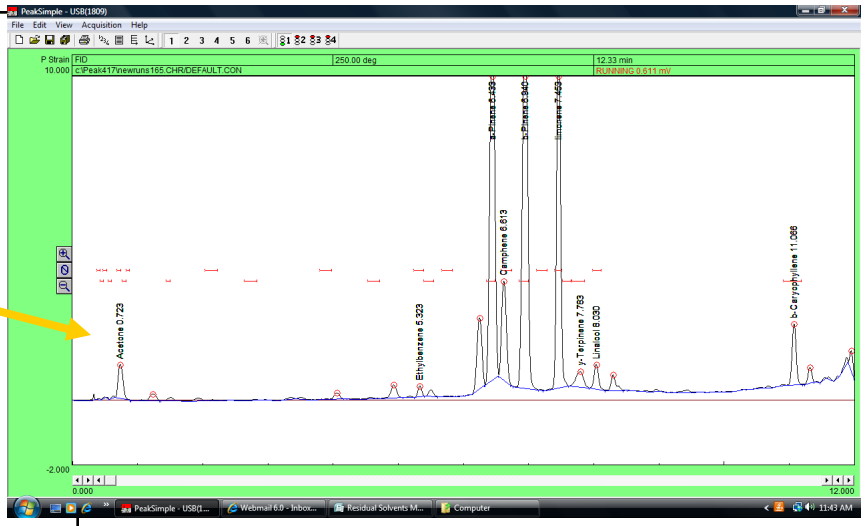


Mr. Nice concentrate. Notice the three small peaks in the BTEX area.

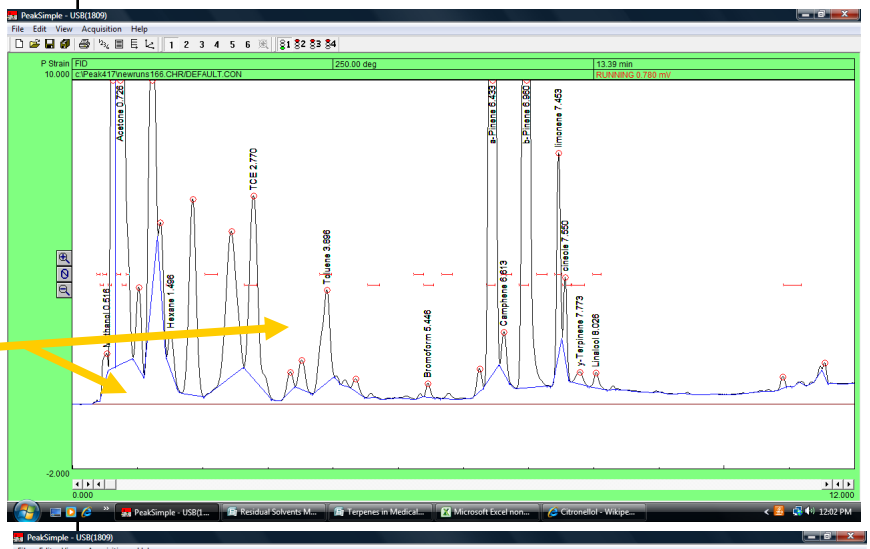


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

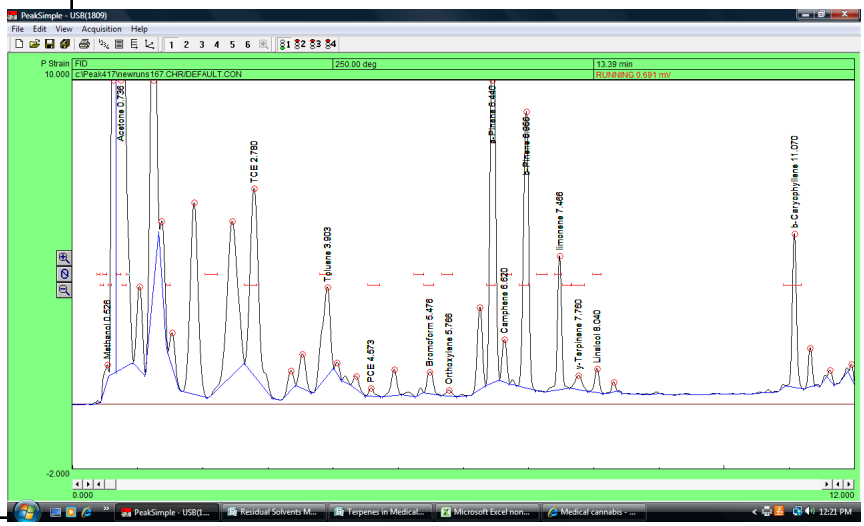
Sour OG Concentrate. Notice the low levels of organic solvents.



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

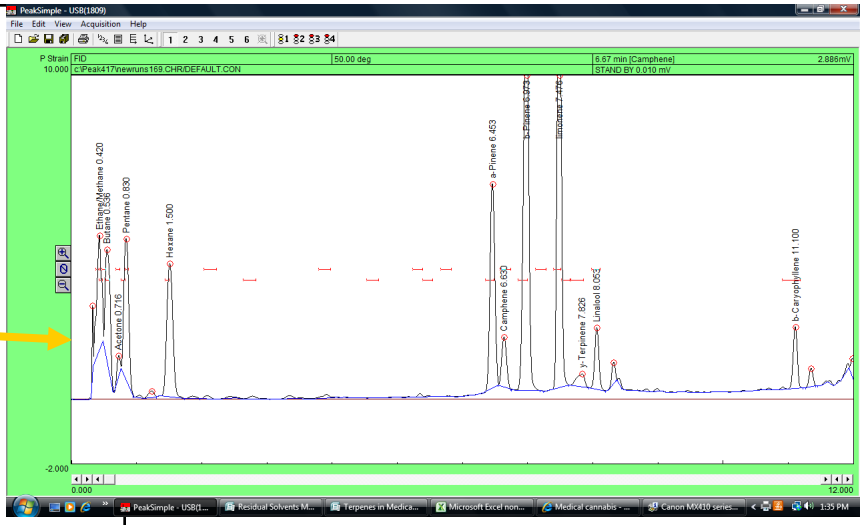


Mr. Nice Concentrate spiked with gasoline fumes.

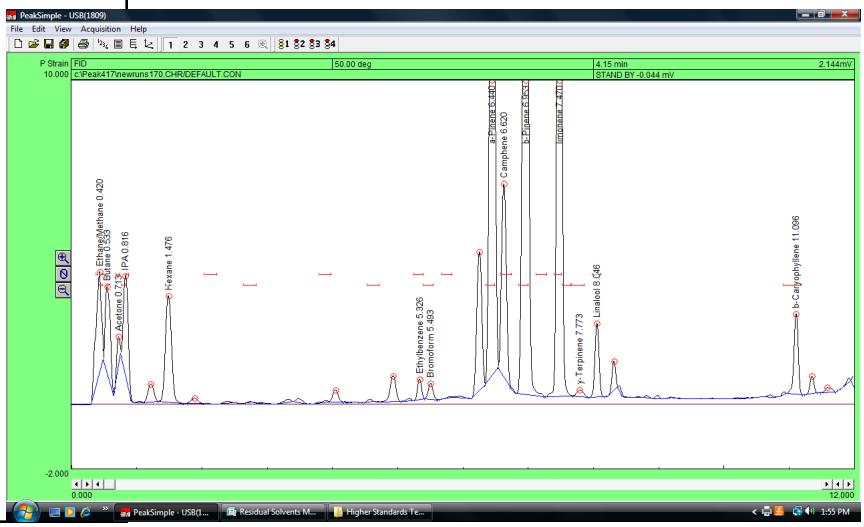


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

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



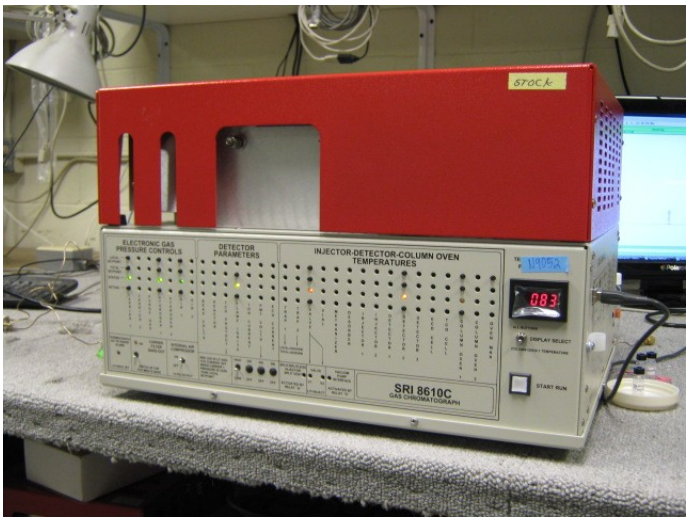
Sour OG concentrate spiked with organic solvents (C_1-C_6).



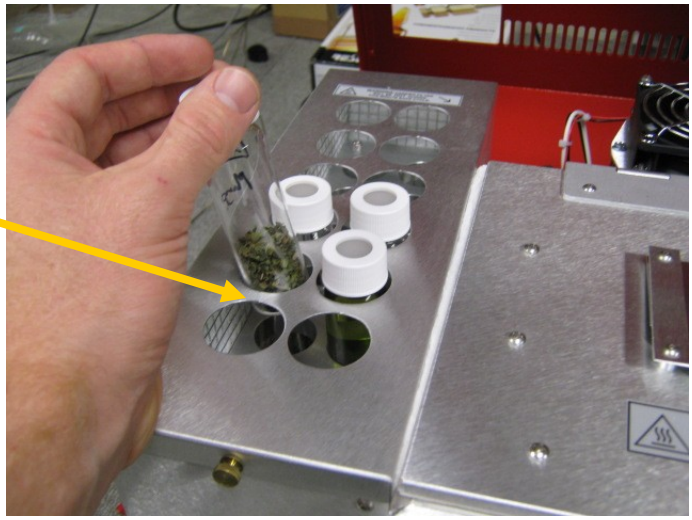
Real world medical cannabis samples will always contain some concentration of organic solvents (plant matter gives off trace amounts of ethane, methane and other gases as it slowly decays), so the presence of minute quantities of these gases should not be alarming. As the operator gains experience running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

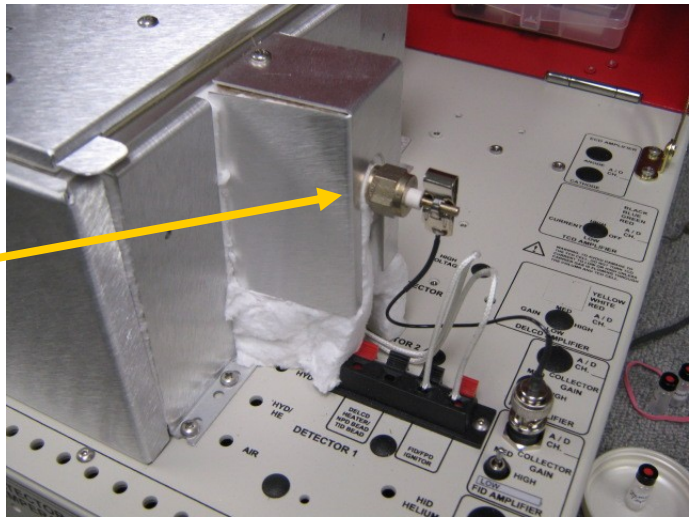
The SRI Medical Herb Potency 8610C GC is shown at right. This GC can also be used to test for the presence of terpenes in cannabis. The word terpene is usually taken to mean the non-psychoactive volatile molecules which make up the characteristic odor of cannabis even though delta-9-THC, CBD and other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

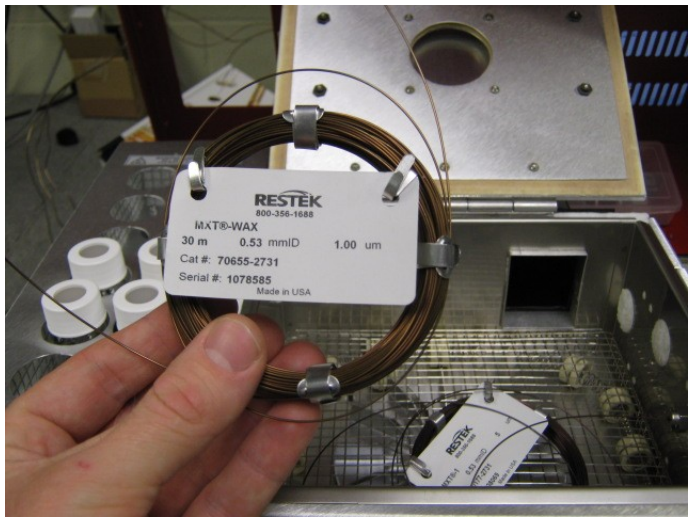
The terpene molecules commonly found in cannabis include:

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

But there are many more.

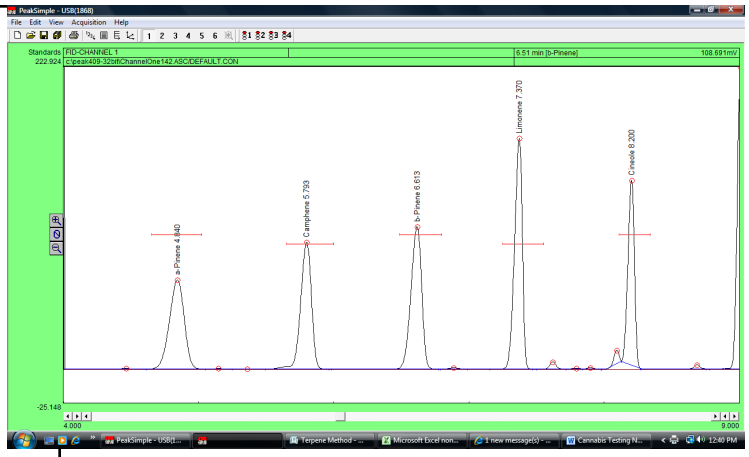
Many types of columns could be used to separate these molecules, but SRI currently suggests a 30meter MXT-WAX with 1 micron film thickness and .53mm id. The terpene analysis can be performed on other columns but the MXT-WAX provides the best separation.

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

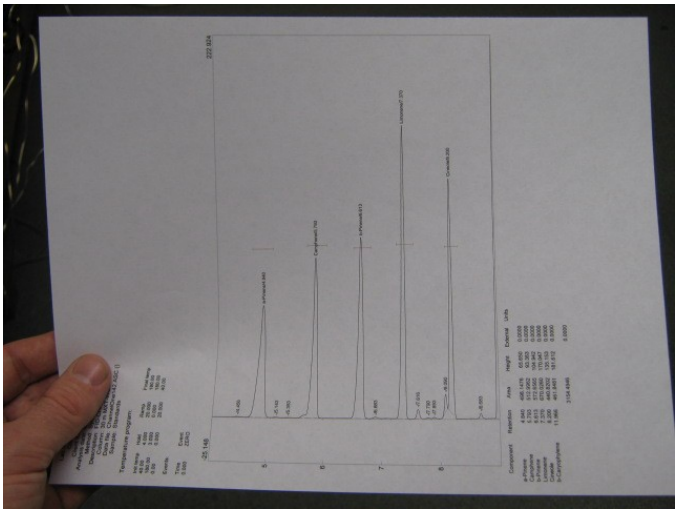


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

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.

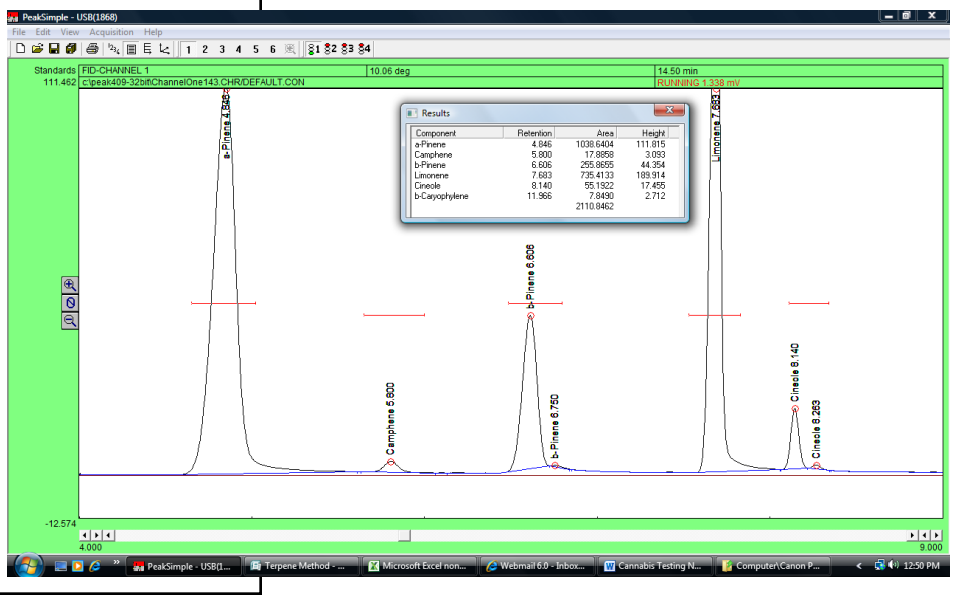


The chromatogram hardcopy printout at right shows a five terpene standard which was injected to identify these volatile odor compounds.



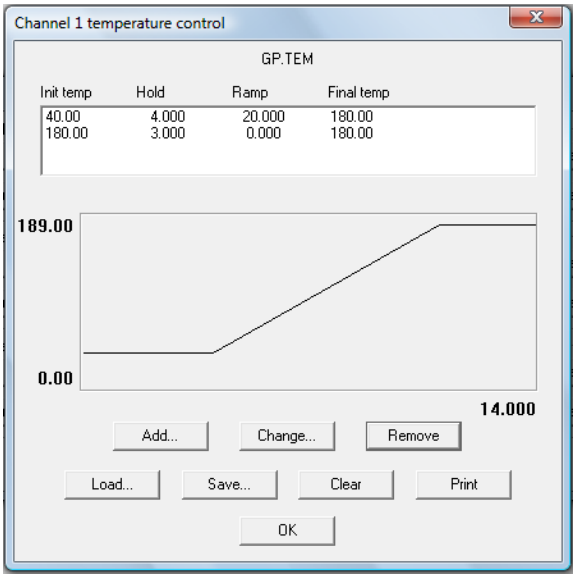
An actual cannabis sample run on the MXT-Wax column is shown at right.

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

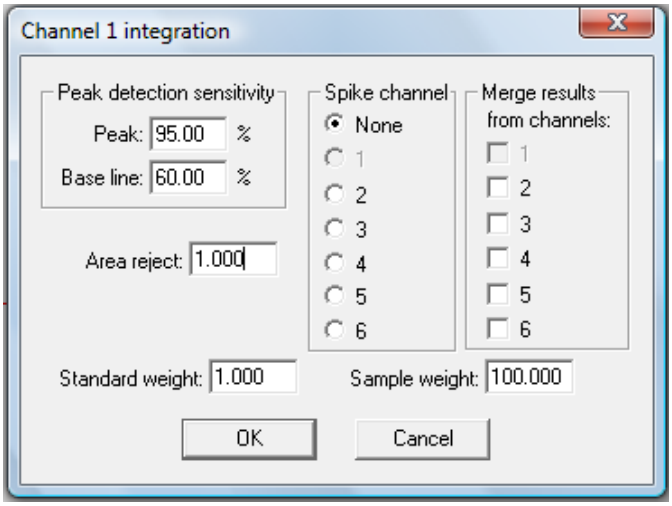


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

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

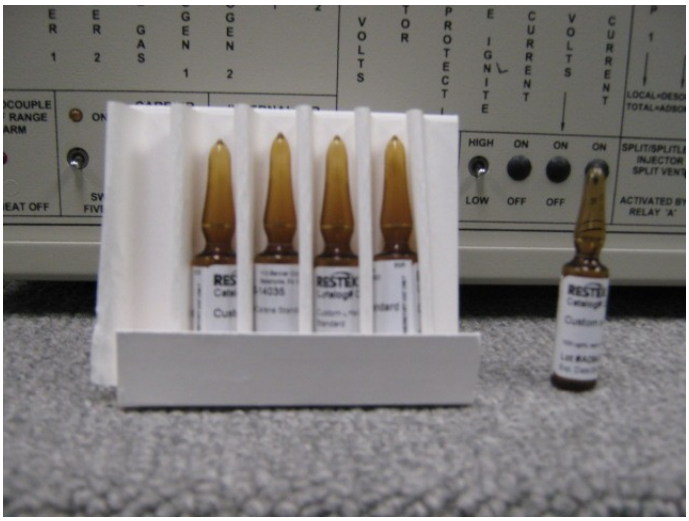


Set the Integration parameters as shown.

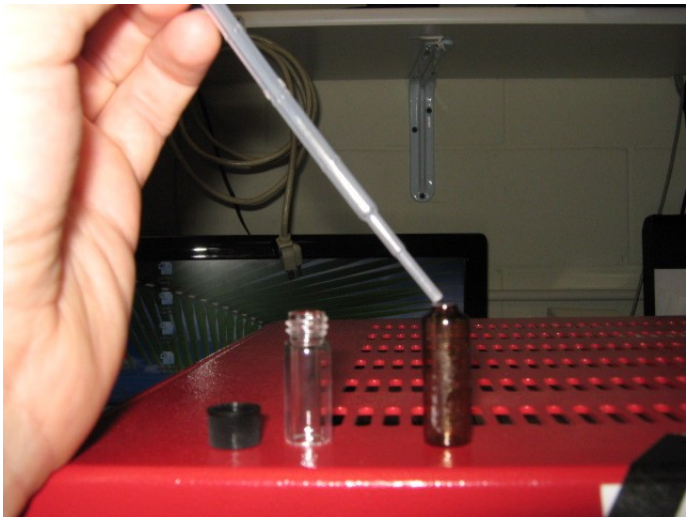


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

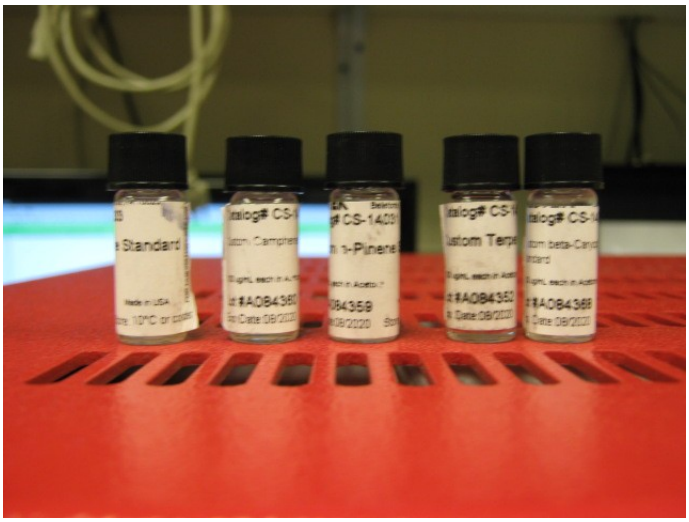
In order to identify terpenes in cannabis obtain the standards from a chromatography supplier like Restek (restek.com) (800) 356-1688.



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.

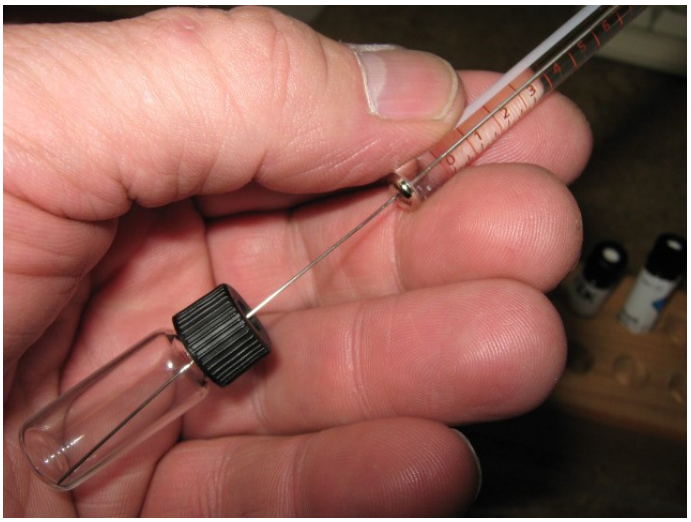


You will end up with one vial per terpene standard. There are 5-10 main terpenes in cannabis.

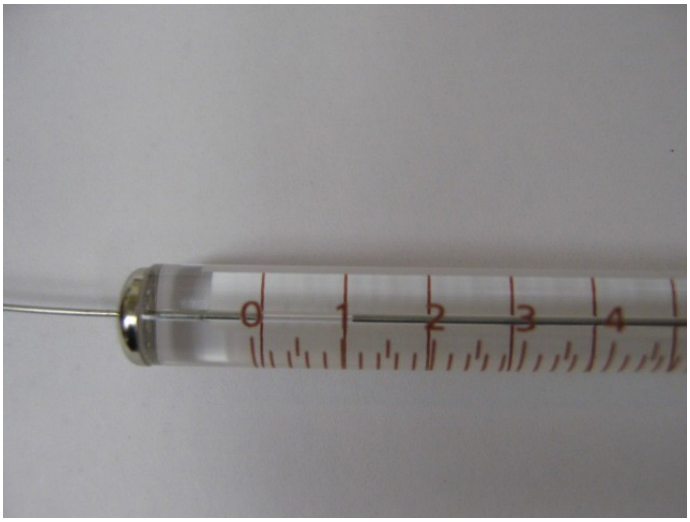


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

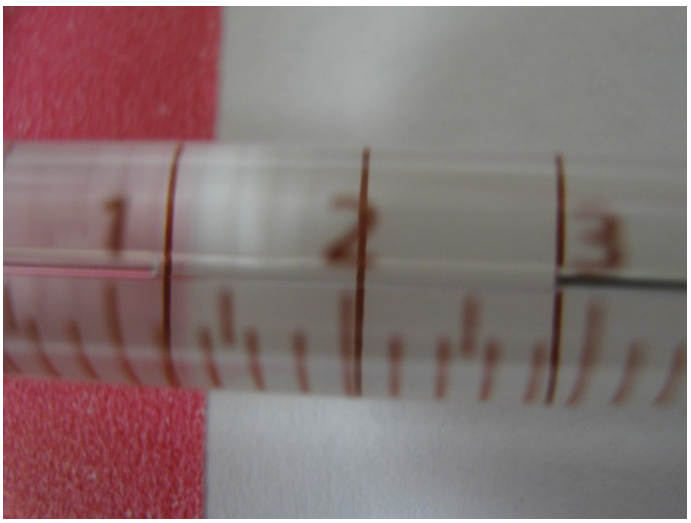
To qualitatively identify each terpene, the standard must be injected into the GC. Rinse the syringe first, then: use the 10uL syringe delivered with the GC (SRI #8670-9550) to withdraw 3-4uL of the standard. Puncture the septum rather than open the vial to avoid letting the methanol solvent evaporate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

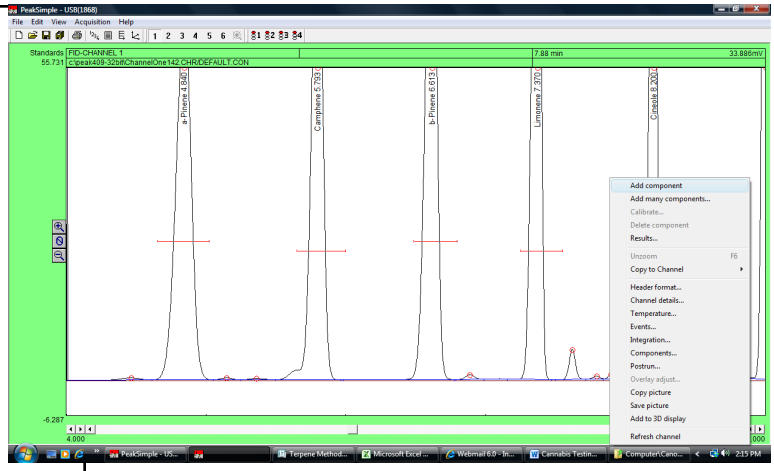
With the plunger still at the 3uL mark, place the needle up against the septum of the injection port (but not poking through it yet).

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

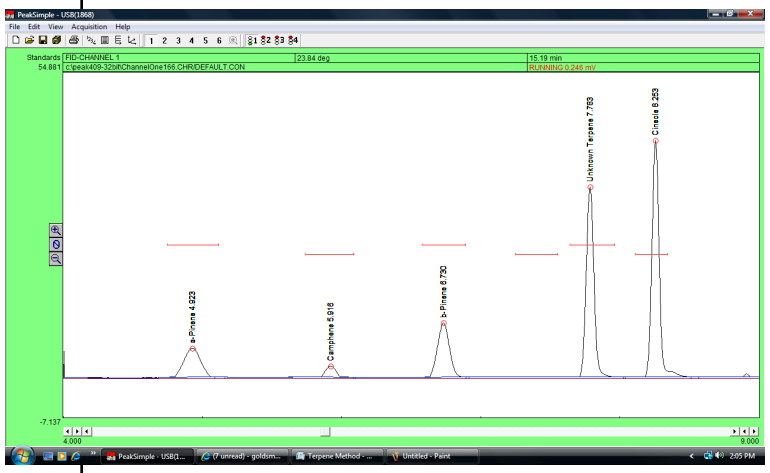
Insert the syringe all the way through the septum as far as it will go. Immediately depress the plunger. Twist the syringe one half turn (to wipe off any liquid on the tip of the needle) and then with-draw the syringe.

Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

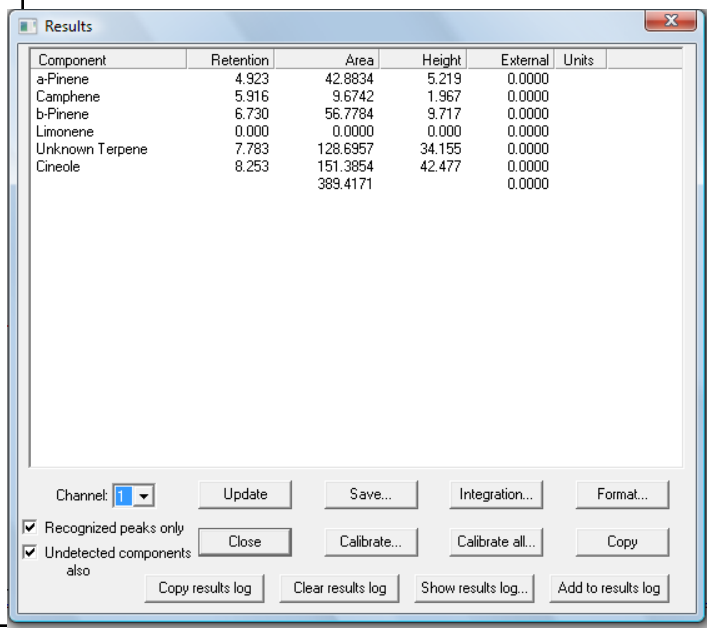
For this terpene standard we have five peaks. Identify the peaks so that each peak is defined by a “retention window”. See the PeakSimple tutorial describing the process of creating retention windows.



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



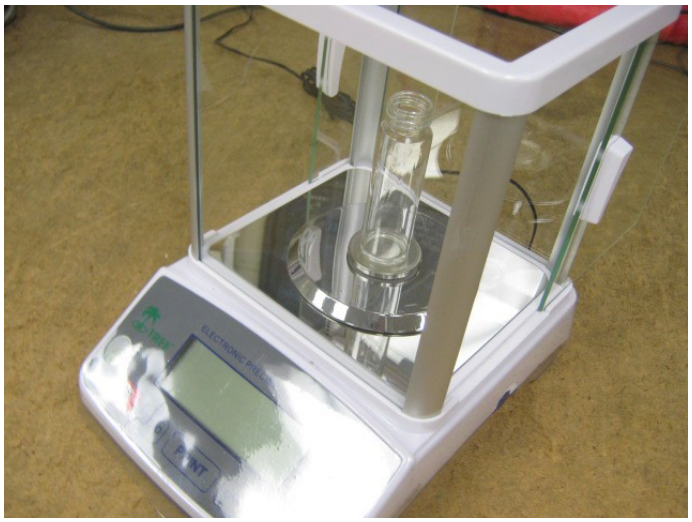
Navigate to the View/Results screen to see the report.



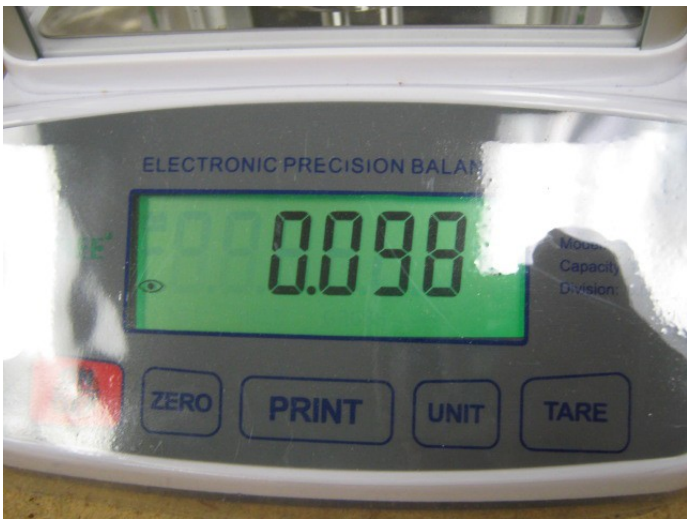
Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

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.



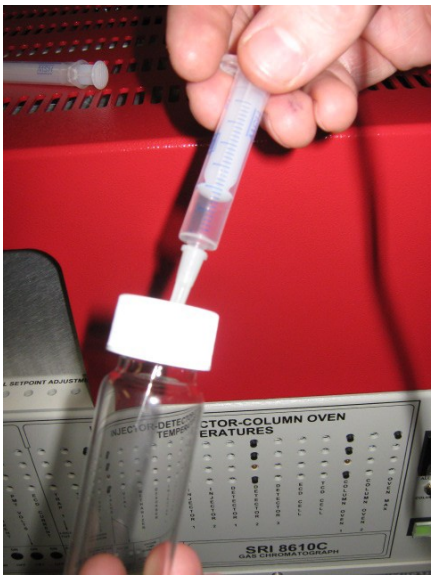
Don't worry if you are slightly under or above 100. In the photo at right, the reading is 98 milligrams which is close enough. Qualitative terpene analysis does not depend on an exact measurement of sample, but the operator may find it advantageous to use the same sample for a subsequent potency analysis. 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.

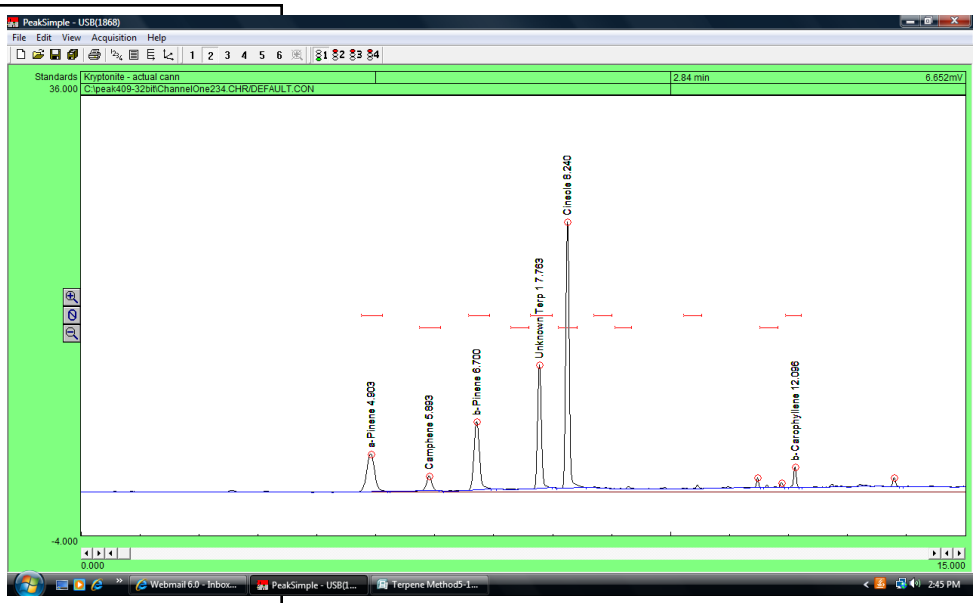


Inject the contents of the syringe into the injection port and start the run as shown previously.

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.

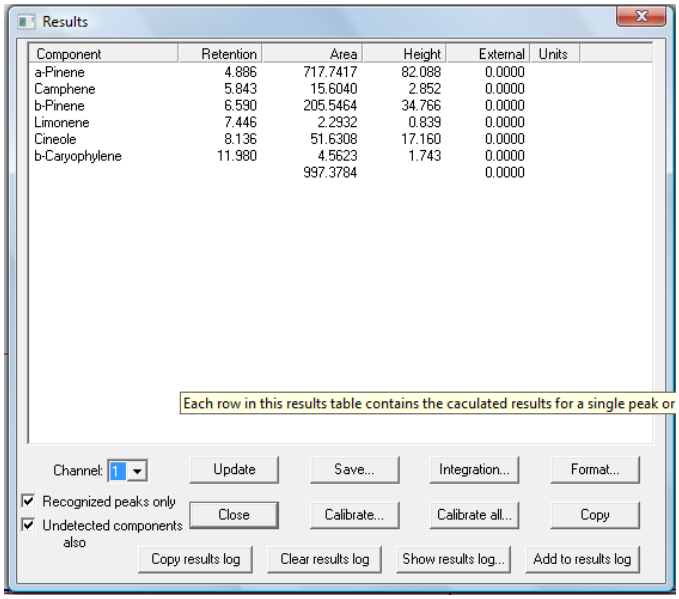
Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

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.

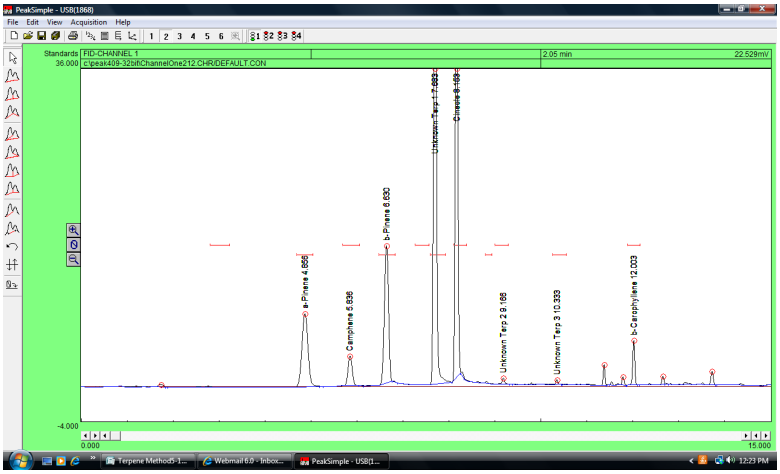
The Results screen will display the area counts of all peaks detected and identified with retention windows.



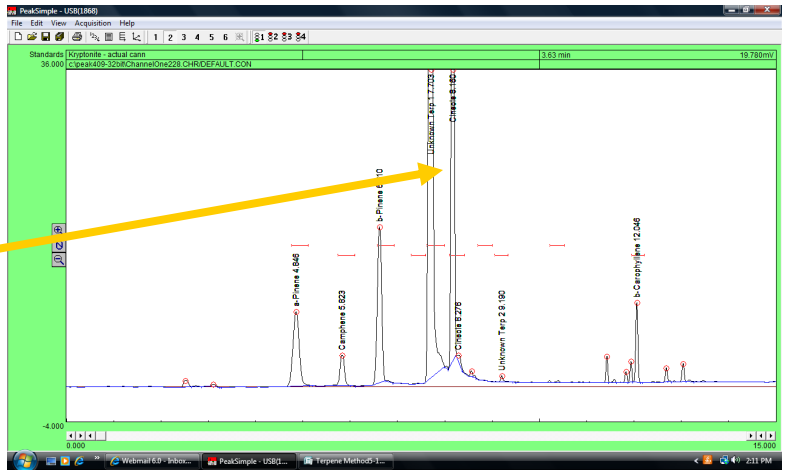
Print the chromatogram and results for a hardcopy record of the analysis.

Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

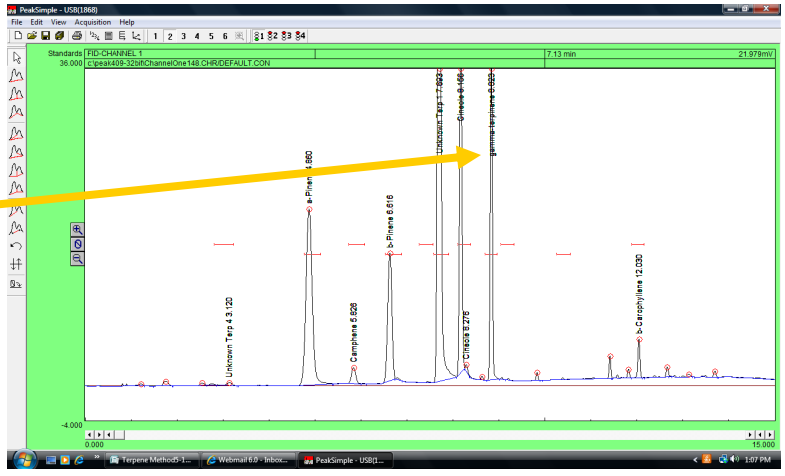
Here is what terpene analysis on the strain King Louie 13 OG looks like. Notice the presence of at least six terpenes: α– and β-Pinene, Camphene, Myrcene, Cineole, and β-Carophyllene.



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.

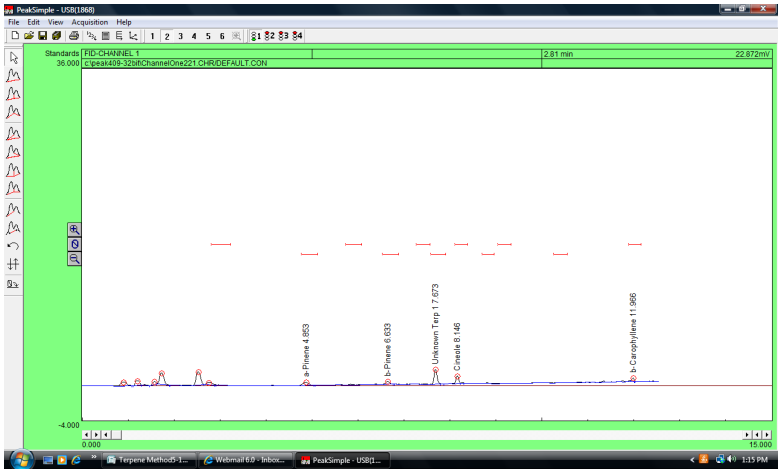


This sample of Green Crack has high concentrations of γ-terpinene. This terpene has a characteristic low-intensity lemon smell and is commonly used as an aromatic in foods, soaps, perfumes, and flavors.

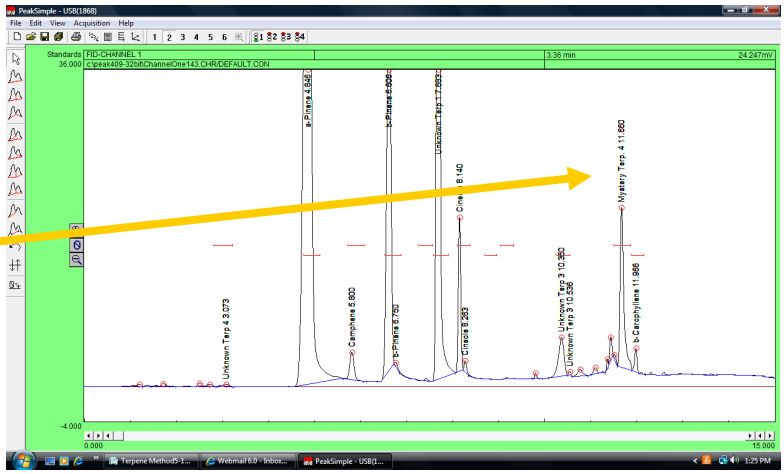


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

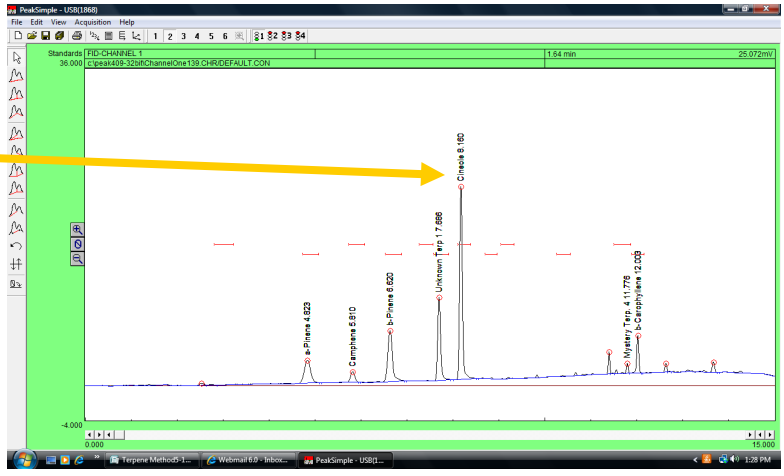
This sample was from visibly low-quality medical cannabis called Mango. Notice its overall low terpene concentrations.



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

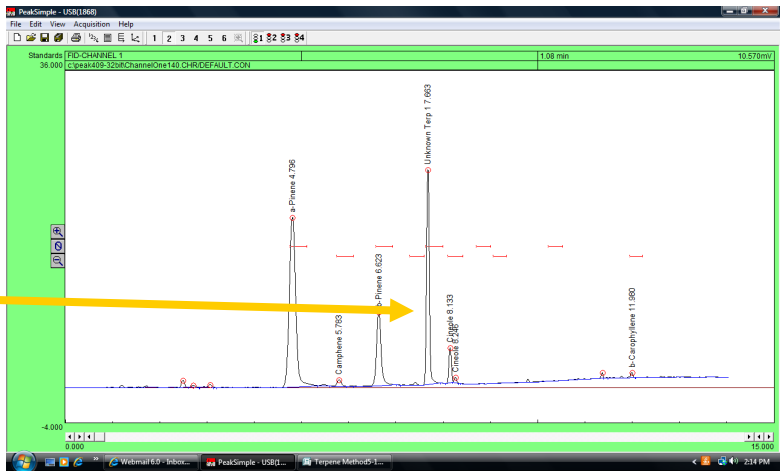


This is a strain called Super Sour Diesel. This chromatogram shows that it has the highest concentrations of the terpene cineole.

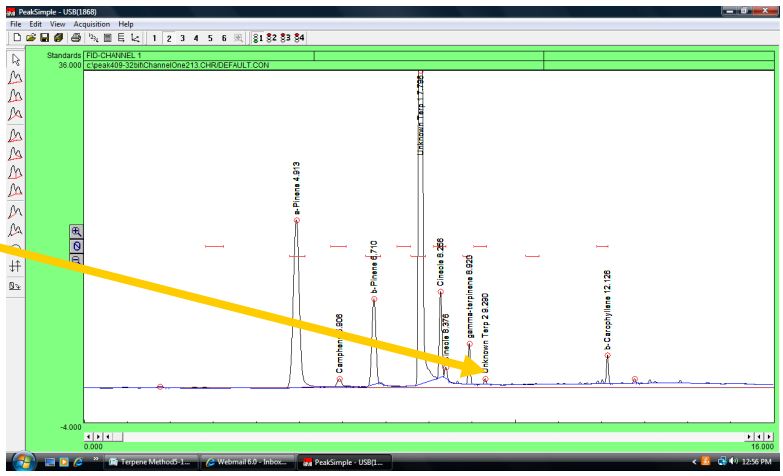


Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

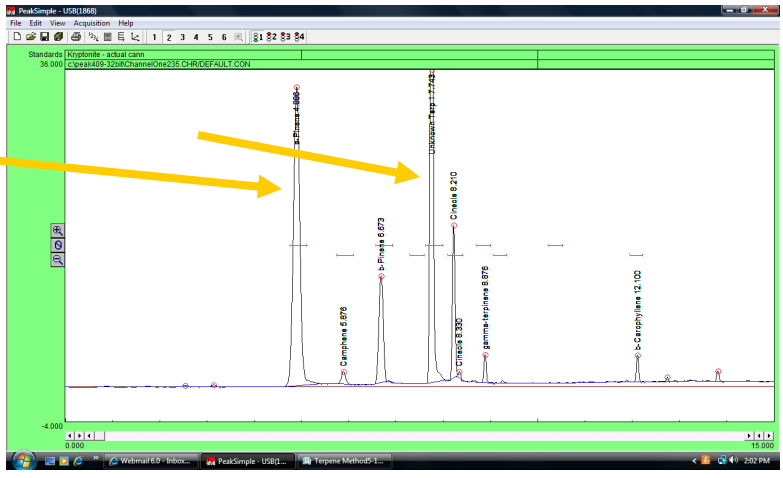
This is from a sample named Allen Wrench. Notice the high concentration of myrcene. This is typical of most strains as myrcene is the most common terpene in cannabis. Myrcene has a clove-like, earthy, vegetative, citrusy-mango smell.



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

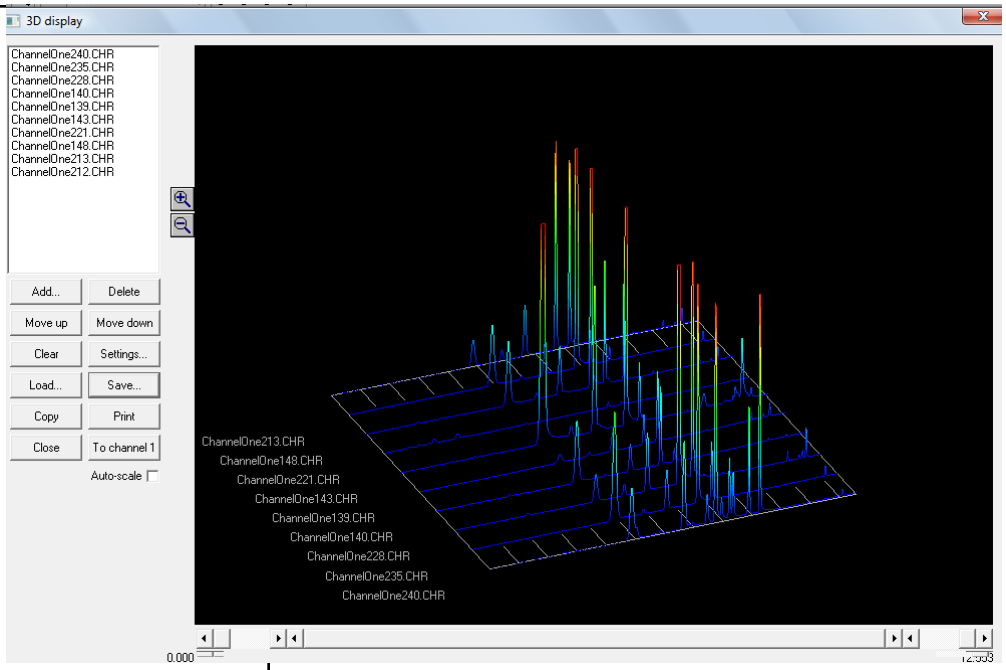


This is an outdoor variety of the strain Strawberry. Notice the nearly similar levels of α-pinene and myrcene. The terpene α-pinene has the characteristic odor of pine trees and is used in cleaning products like Pine-sol.



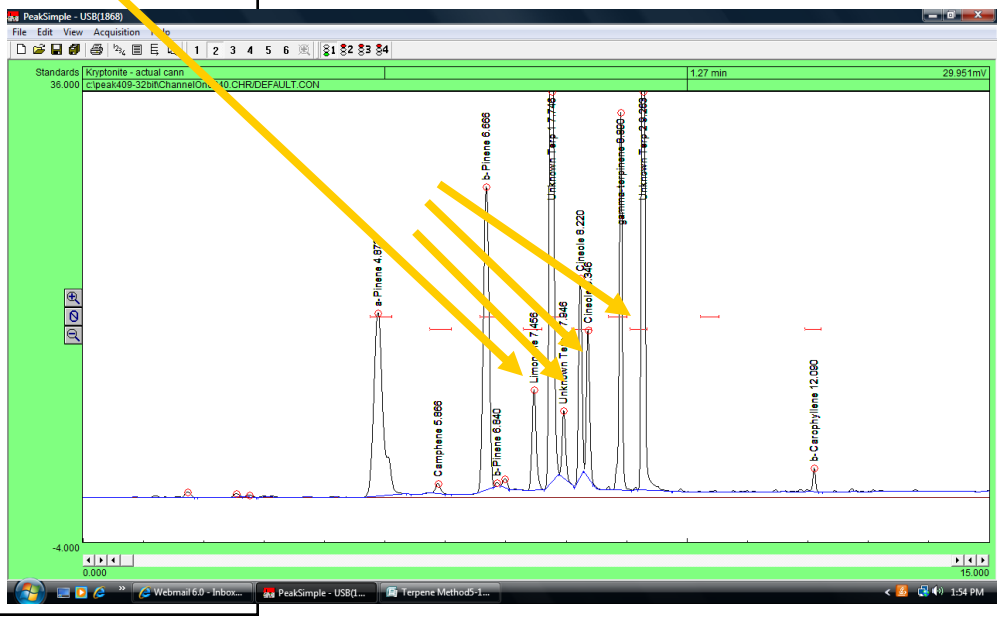
Medical Cannabis Terpene Measurement using the SRI 8610C FID GC

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.

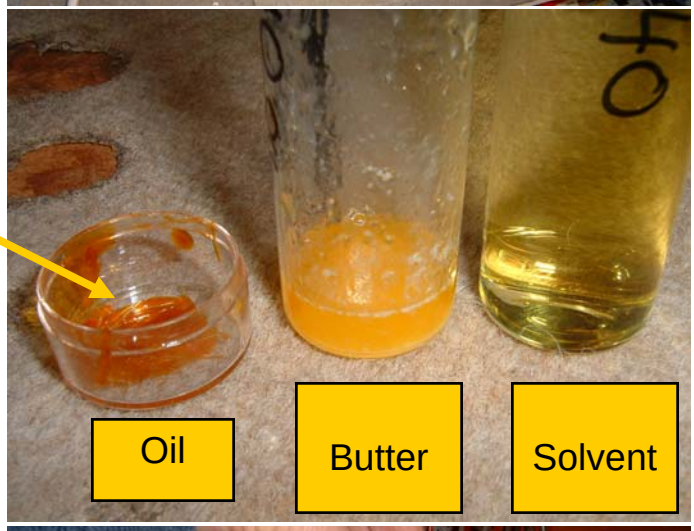
SRI Instruments welcomes your feedback, knowledge and experience with terpene analysis. Please contact us if you have any questions or information to provide.



Measuring THC in Butter using the SRI 8610C GC

The THC in butter analyses were performed using an SRI 8610C GC configured for cannabinoid analysis.

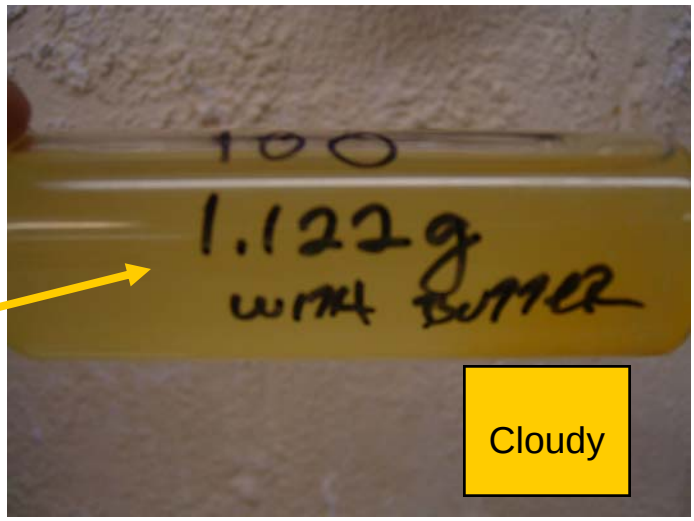
100 milligrams of a cannabis oil was weighed into two identical 40ml vials. The oil was a CO2 extract with an orange color. We used the oil for this test because it was very uniform in consistency.



The first vial was filled with methanol and placed in the built-in sample incubator which is part of this GC configuration. To the second vial was added 1 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 50C. A third vial with no oil was loaded with 1 gram of butter for comparison.

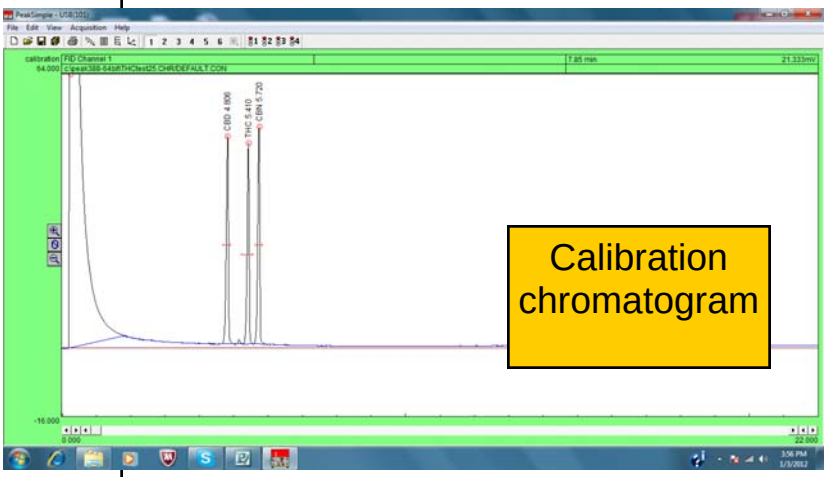
Measuring THC in Butter using the SRI 8610C GC

After 30 minutes in the incubator the two butter vials were filled with methanol and placed back into the incubator. Once the methanol warmed to 50C the butter vials were shaken for 30 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 30 minutes.



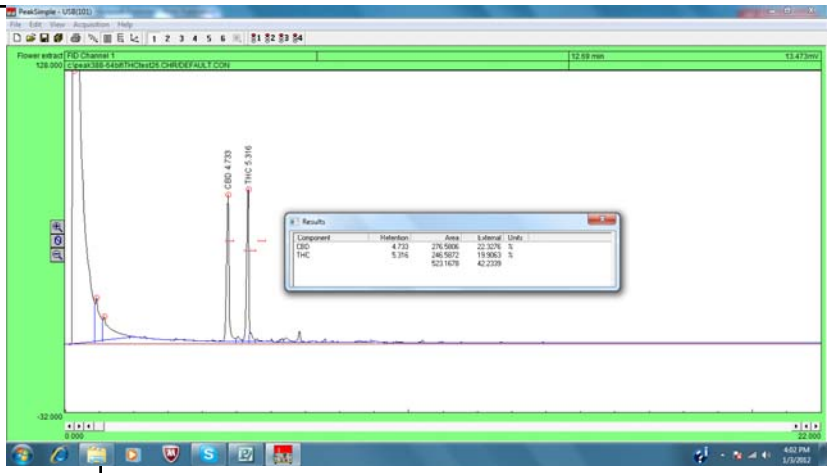
After another 30 minutes the butter solids dropped to the bottom of the vial leaving clear liquid in the top of the vial. Interestingly, the suspension did not clear at room temperature, only when heated in the incubator.

Meanwhile the GC was calibrated with a mixture of CBD, delta9THC and CBN each at a concentration of 333ng/ul. 1ul was injected on-column into a 15 meter MXT500 capillary column with .53mm id and a film thickness of .15 micron. The temperature program was set to start at 140C hold for 0.00 minutes , then ramp at 20 degrees per minute to 380 C then hold. The FID was set to 380C. Hydrogen carrier was used at 5psi or 10ml/min.

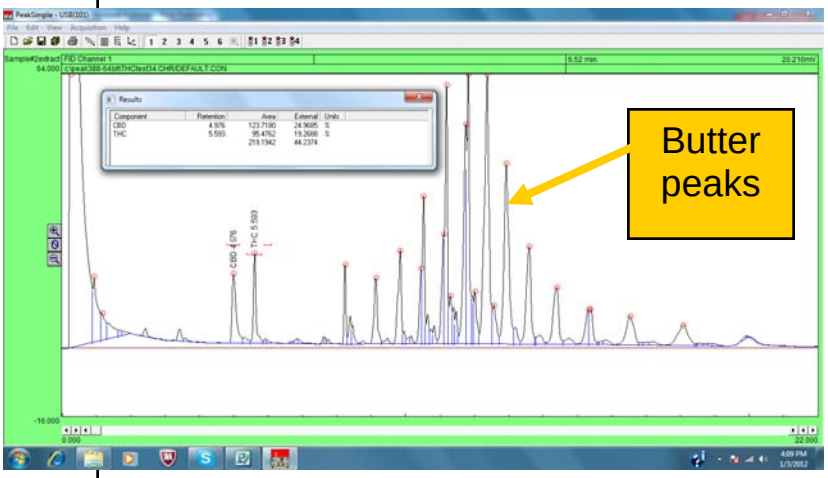


Measuring THC in Butter using the SRI 8610C GC

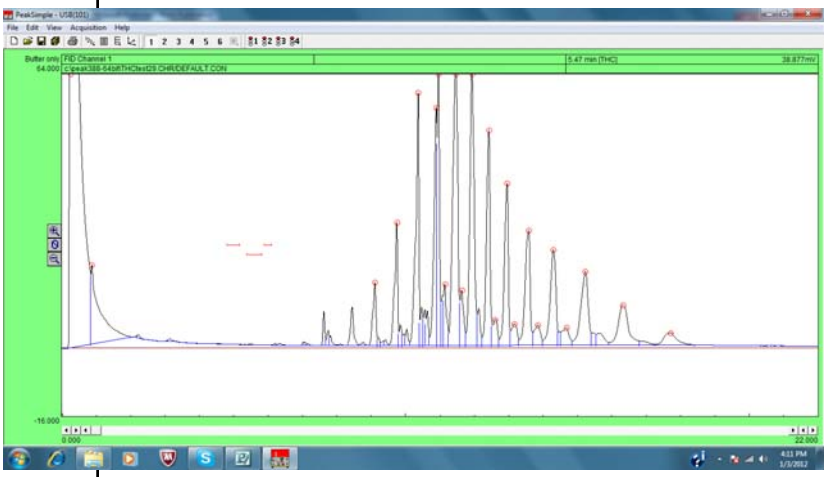
The oil only extract was injected and the results showed 22.3% for CBD and 19.9% for d9THC. Presumably this particular oil was prepared from industrial hemp since the CBD was so high.



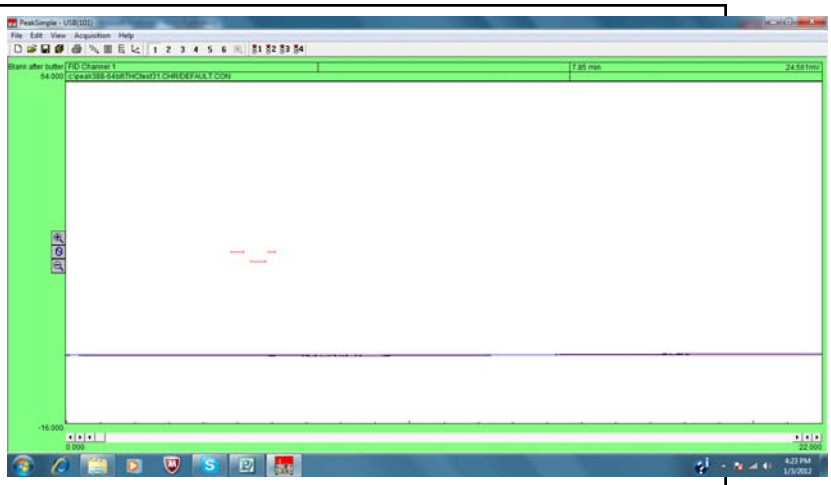
The vial with butter and oil was injected and the results showed 24.9% for CBD and 19.3% d9THC. Some thickening of the CBD is apparent while the THC peak looks much the same as the non-butter vial.



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.



Measuring THC in Butter using the SRI 8610C 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 3% earlier with the 1 gram butter injections, but returned to the normal time in subsequent injections of non-butter samples.

We made a more concentrated butter extract (3 grams butter in 40 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 MXT500 column which was used is rated to over 400C which allows this high temperature operation. In addition the thin film promotes fast elution of the high boiling molecules. Even so, the analysis took 22minutes.

The peculiar shape of the CBD peak and the evidence that the butter increases the CBD number but not the d9THC is not explained and requires further investigation.

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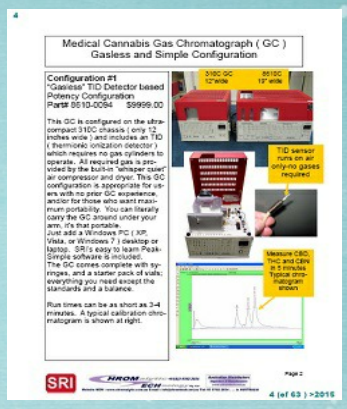

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