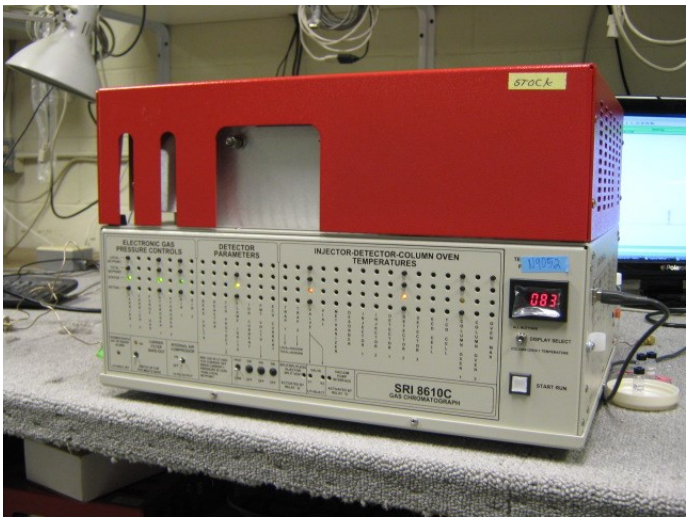
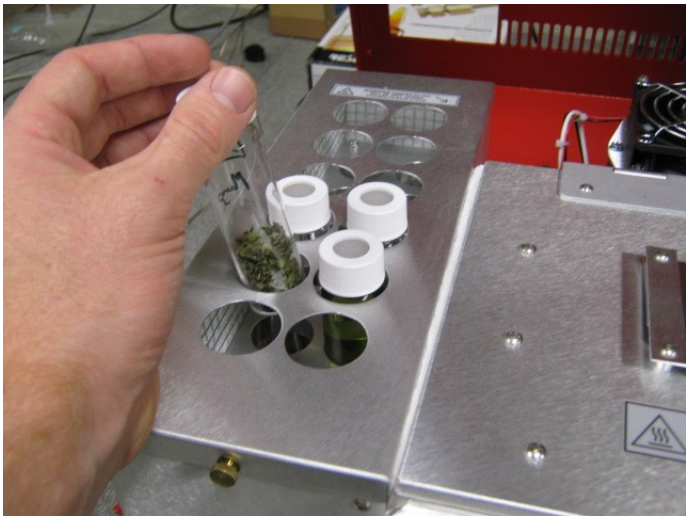
Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

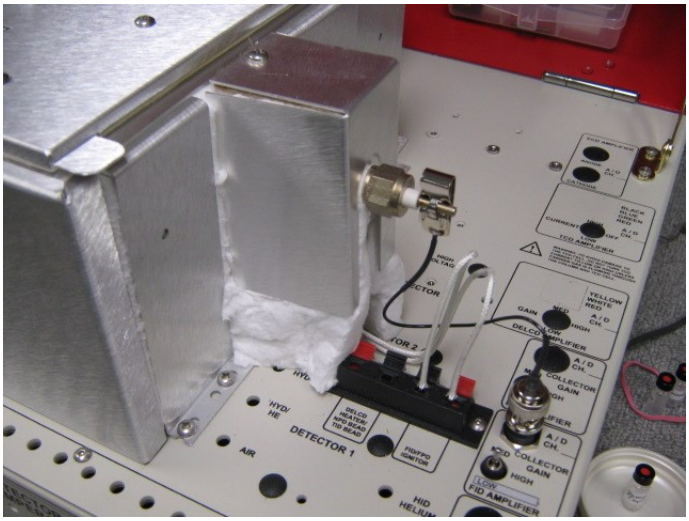
The SRI Medical Herb Potency 8610C GC is shown at right. This GC can also be used to test for residual solvents (i.e. butane, acetone, gasoline residue, etc.) in medical cannabis. These solvents are used in the extraction process to create medical cannabis hash oils and concentrates.



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).



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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.

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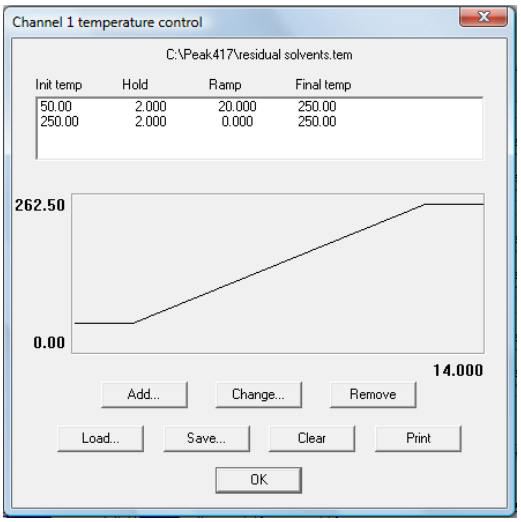
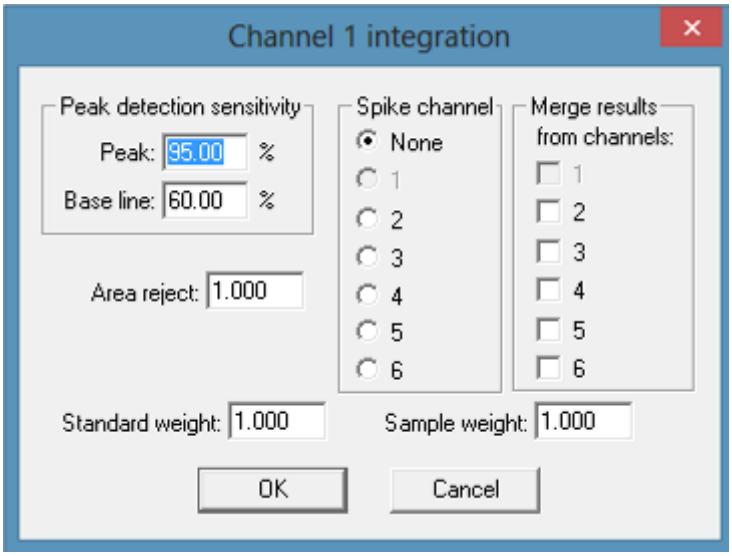
Set the column oven temperature as shown at right. Although we are only interested in the early eluting solvents and adulterants, the "heavier" terpene molecules are also injected onto the column, and these must be allowed time to come out. The light hydrocarbons come out during the two minute hold, 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.

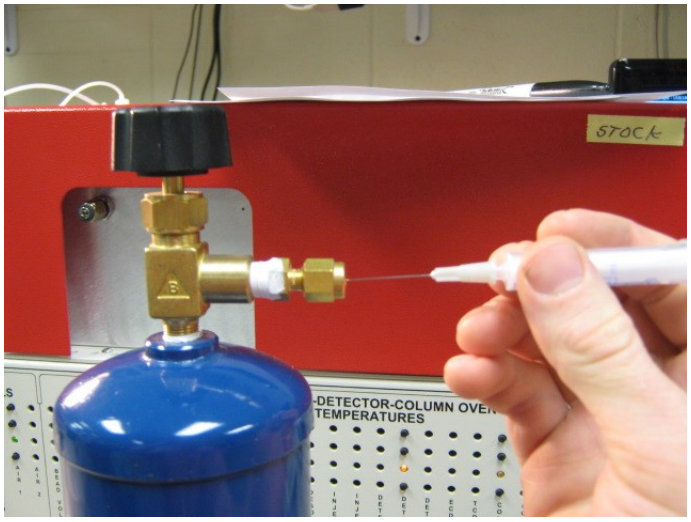
Set the Integration parameters as shown.



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).

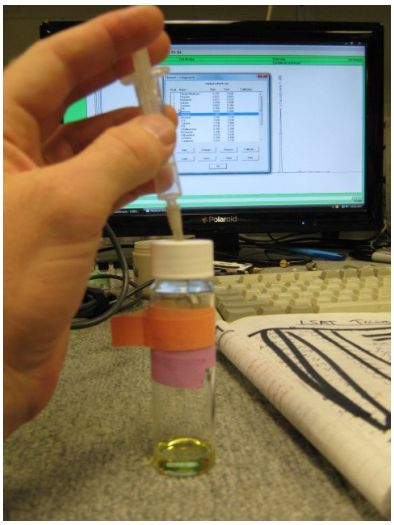
Pressurize the gas cylinder by turning the release valve slightly counterclockwise. Pierce the septum with a 3 mL gas syringe and withdraw 1 mL of gas. Remove the syringe from the gas sample bottle.



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.

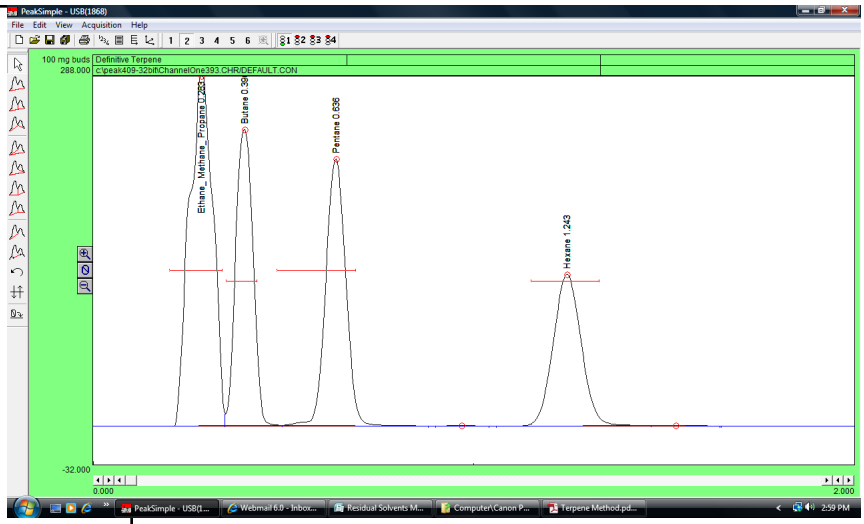


With the syringe plunger still at the 1mL mark, place the needle up against the septum of the injection port (but not poking through it yet). Press the Start Run button or press the spacebar on the keyboard.

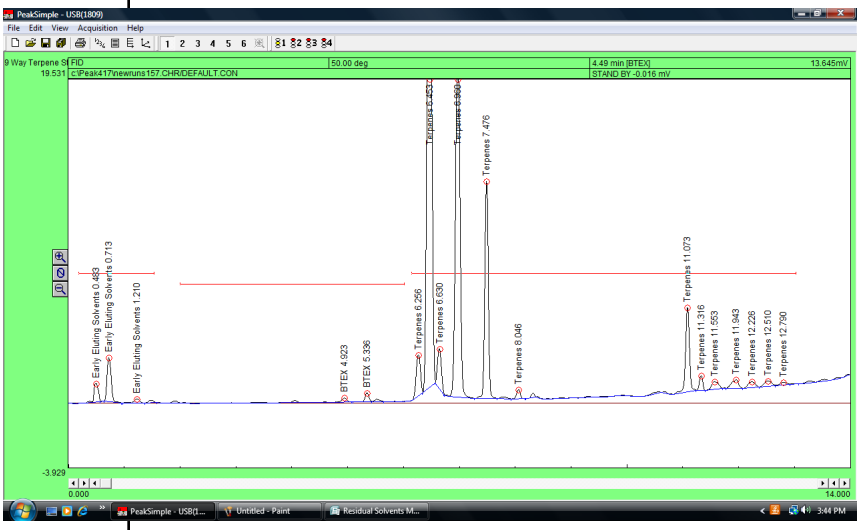
Insert the syringe all the way through the septum as far as it will go. Immediately depress the plunger and quickly withdraw the syringe.

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

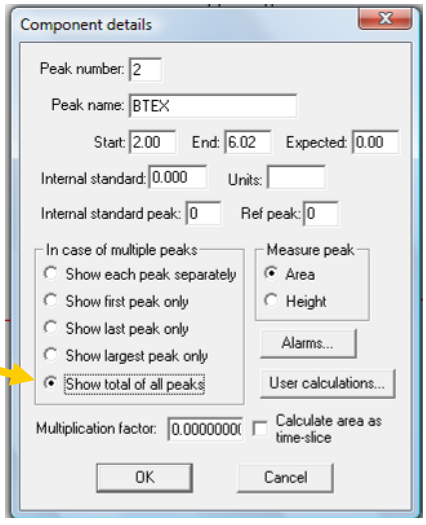
After injecting the C1—C6 standard we see four peaks: ethane, methane, and propane (which all elute together); butane; pentane; and hexane. Identify the peaks so that each peak is defined by a "retention window". See the PeakSimple tutorial describing the process of creating retention windows.



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.



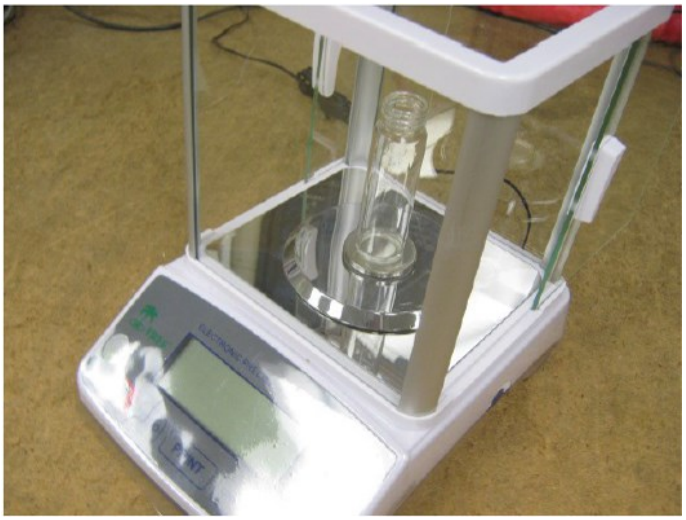
In this case, all the peaks under the retention window need to be quantified. In the Edit Component screen select "Show total of all peaks".



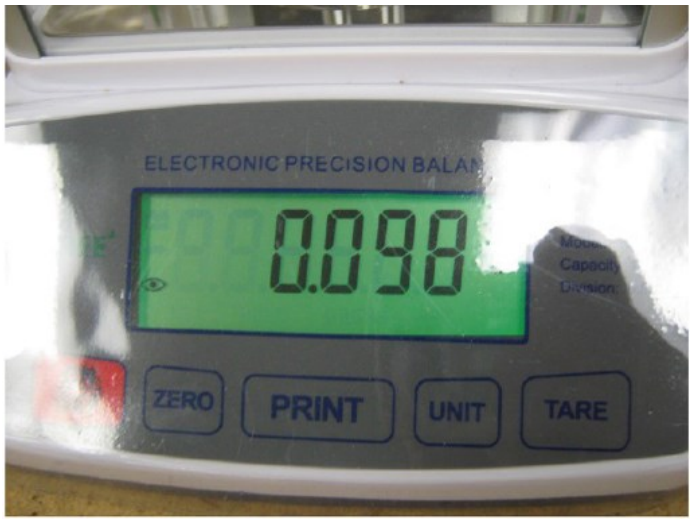
Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

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.

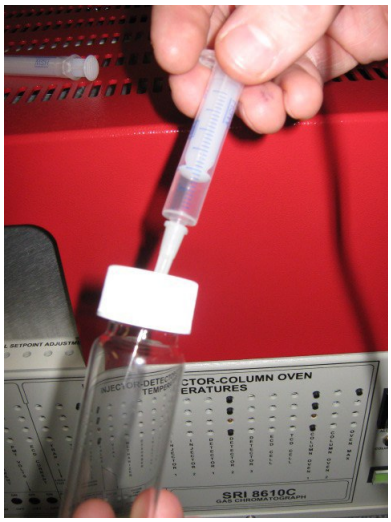


Don't worry if you are slightly under or above 100. In the photo at right, the reading is 98 milligrams which is close enough. Qualitative butane and residual solvent analysis does not depend on an exact measurement of sample, but the operator may find it advantageous to use the same sample for a subsequent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

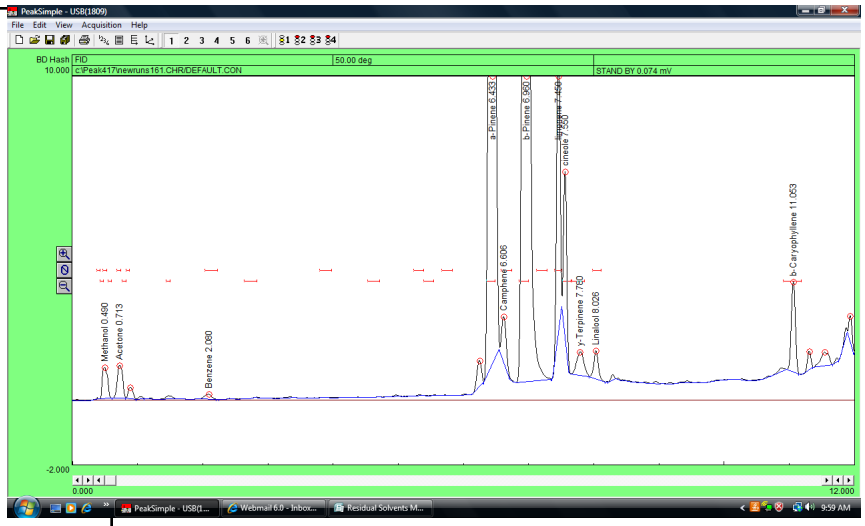


Inject the contents of the syringe into the injection port and start the run as shown previously.

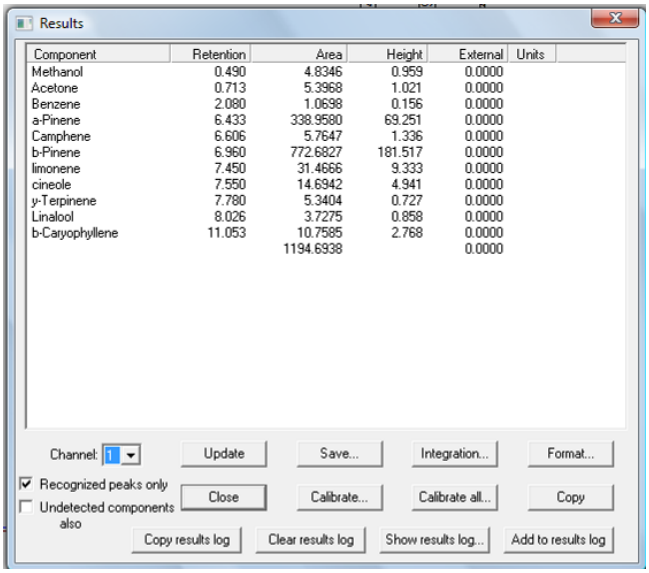
The picture at right shows a butane and residual solvent sample vial filled with 40 mL of extraction solvent and ready to be injected for cannabis potency analysis. See the PeakSimple tutorial describing the process for Medical Cannabis Potency testing.

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

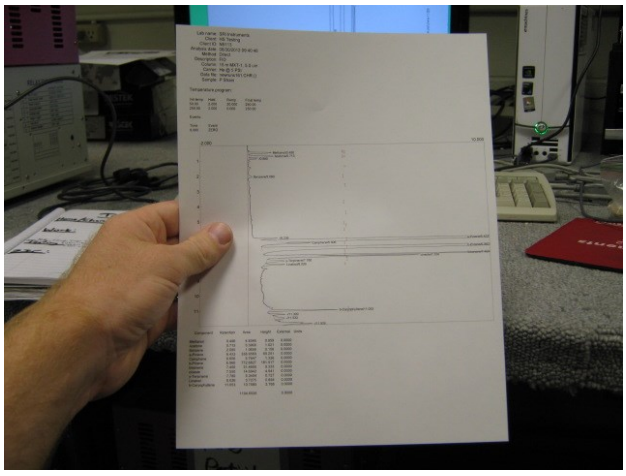
A real cannabis flower sample will look something like the chromatogram at right. This particular sample has standard levels of organic solvents (which are present in low levels naturally in plant matter) and multiple terpenes.



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

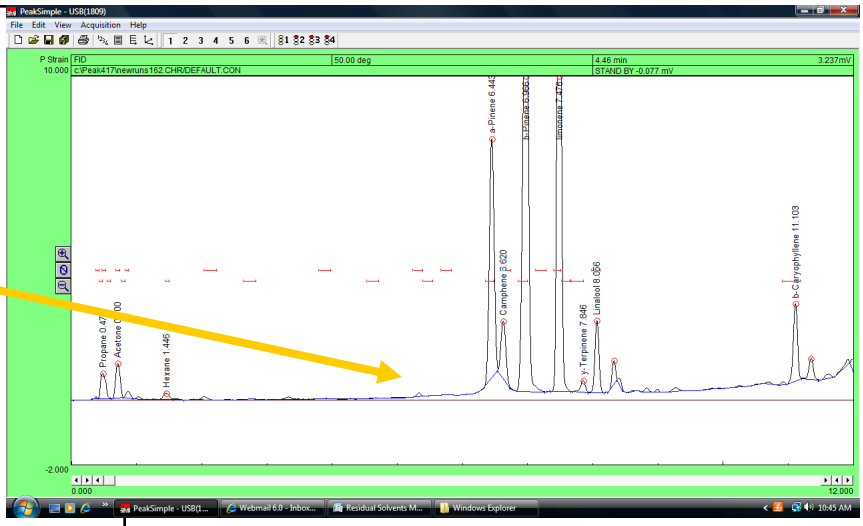


Print the chromatogram and results for a hardcopy record of the analysis.

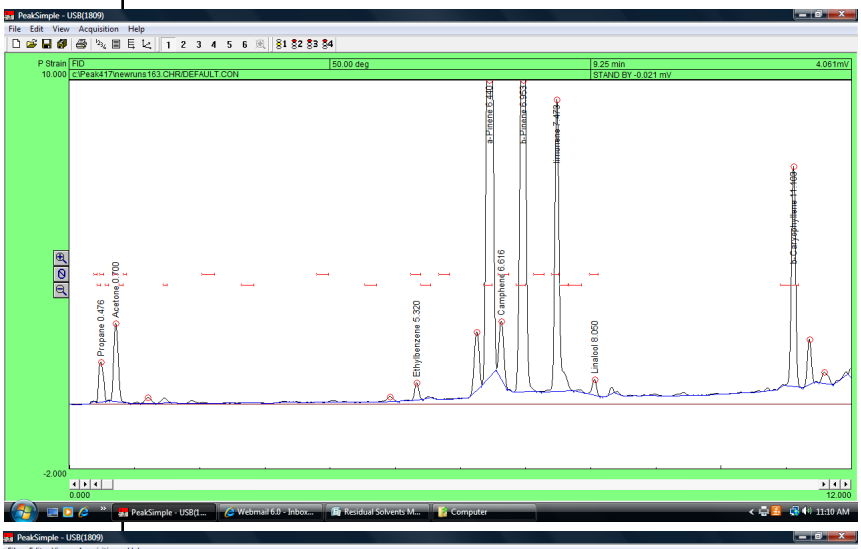


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

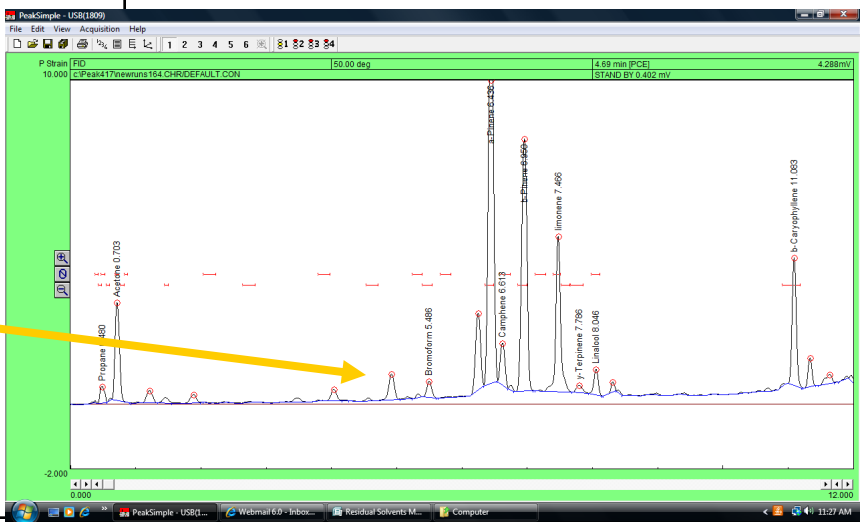
Super Sour Diesel flowers. Notice the lack of any BTEX-like adulterants.



Blue Dream concentrate.

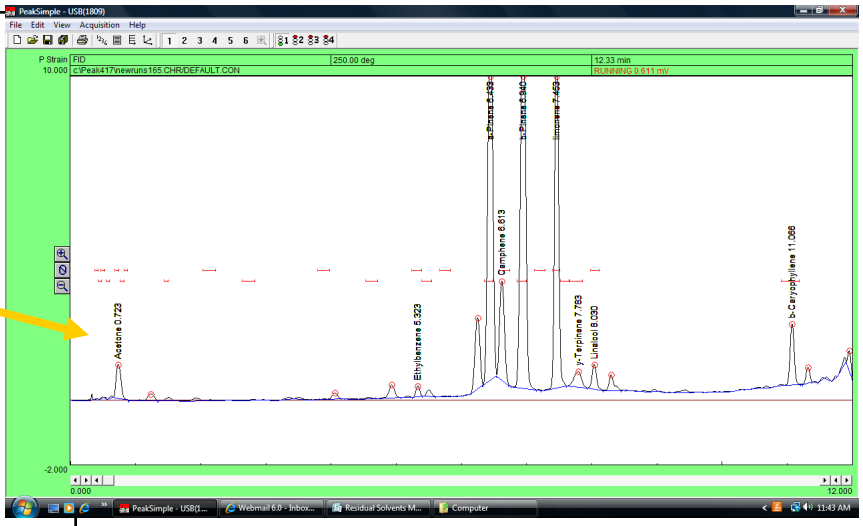


Mr. Nice concentrate. Notice the three small peaks in the BTEX area.

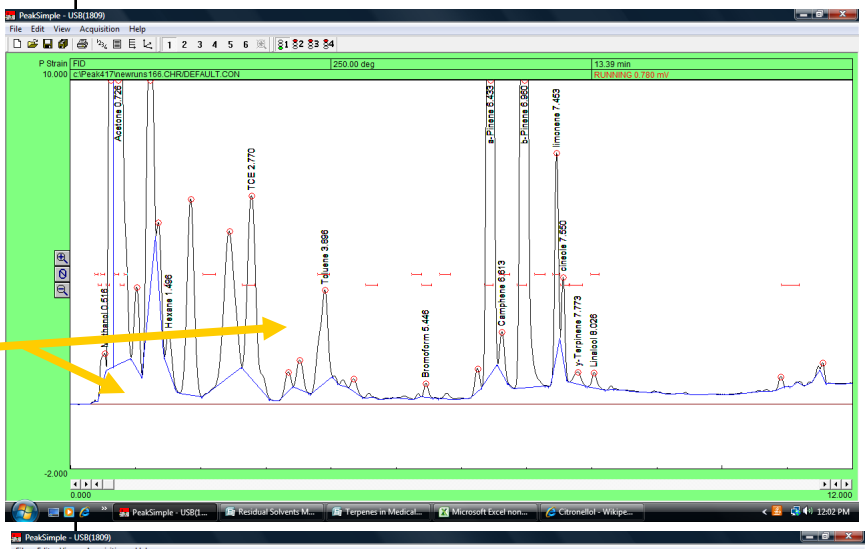


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

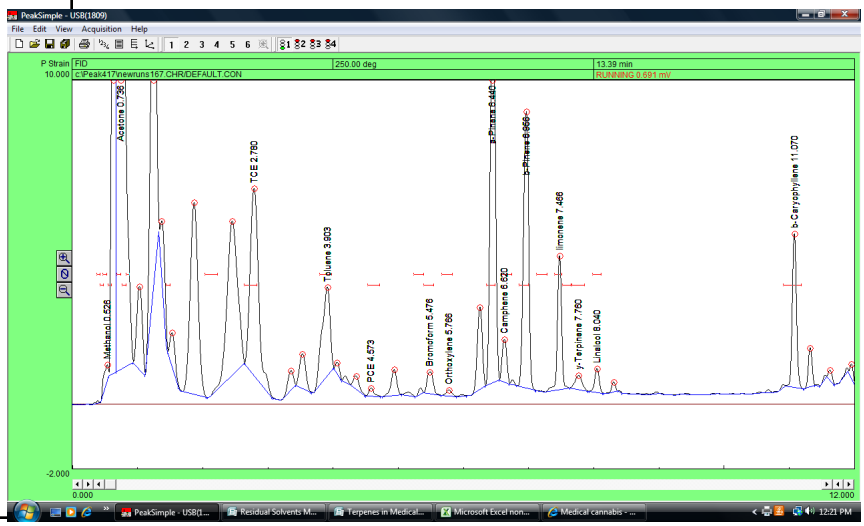
Sour OG Concentrate. Notice the low levels of organic solvents.



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

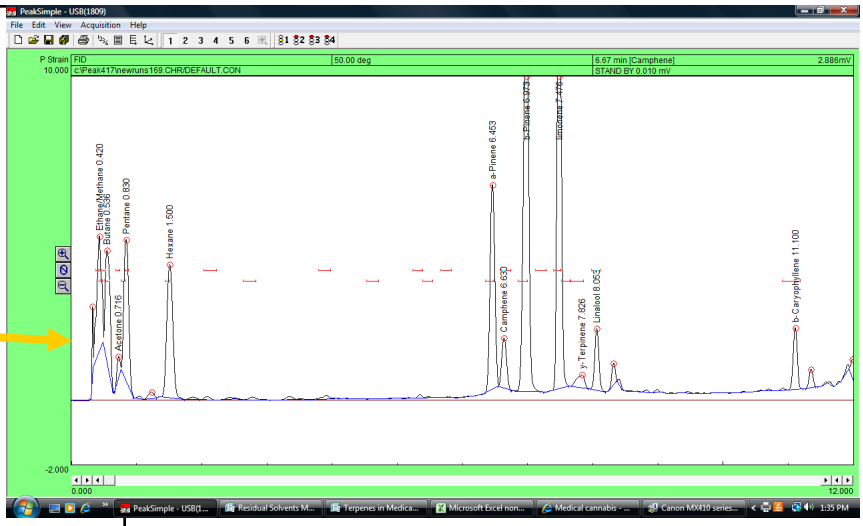


Mr. Nice Concentrate spiked with gasoline fumes.

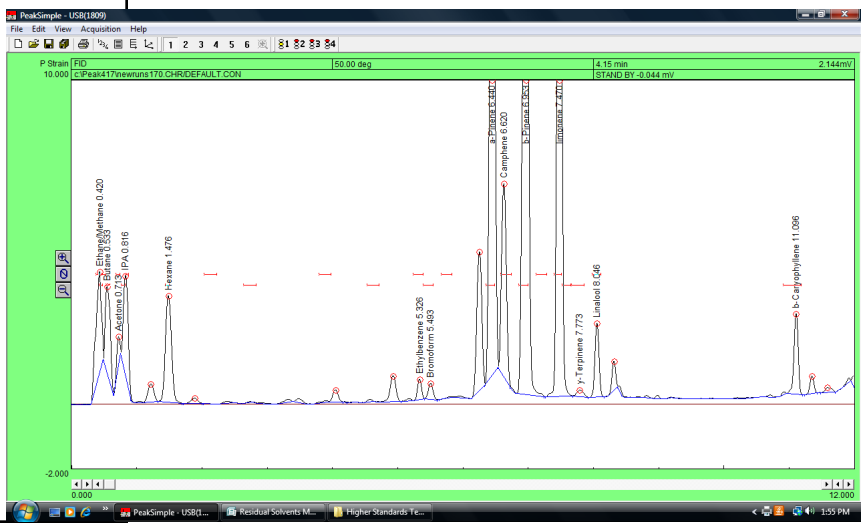


Butane and Residual Solvents in Medical Cannabis Flowers and Concentrates using the SRI 8610C FID GC

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.