THCA vs d9THC testing using the SRI 8610C GC

SRI gas chromatographs (GCs) that are configured for cannabis testing come in different sized chassis, with and without auto-samplers.

GC is the least expensive method (about 15 cents per sample) for testing cannabis potency and has long been the choice of many labs including the US Government's own lab.

HPLC systems can also be used to test cannabis although the equipment is more expensive to purchase and also more expensive to operate (about $3 per sample). HPLC has until now had one advantage over GC in that it can test for THCA as well as d9THC. THCA is the precursor molecule which the cannabis plant produces. With time and heat the THCA molecule loses one carbon and two oxygen molecules (de-carboxylates) to become d9THC. If the cannabis is smoked, the heat of the flame instantly decarboxylates the THCA into d9THC.

Edible forms of cannabis (medibles) are normally prepared with cannabis which has been deliberately decarboxylated prior to its addition to the flour or sugar "medible" ingredients. To verify that all the THCA in the cannabis leaves and flowers has been 100% decaboxylated (usually by simmering with butter, or otherwise heating above 100C) it is useful to be able to measure the THCA and also the d9THC in the same analysis.

Previously this was not possible with GC because the GC vaporizes the sample which is injected and due to the high heat, instantly decarboxylates any THCA in the sample, converting it into d9THC.

Recently we have learned how to stabilize the THCA molecule (derivitize) so it does not decarboxylate in the GC so we can measure THCA and d9THC separately in the same analysis just like using a HPLC but at much lower cost than buying and operating an HPLC system.

SRI 8610C GC

SRI 8610CV GC with Cobra auto-sampler

SRI Tech Support: 310-214-5092

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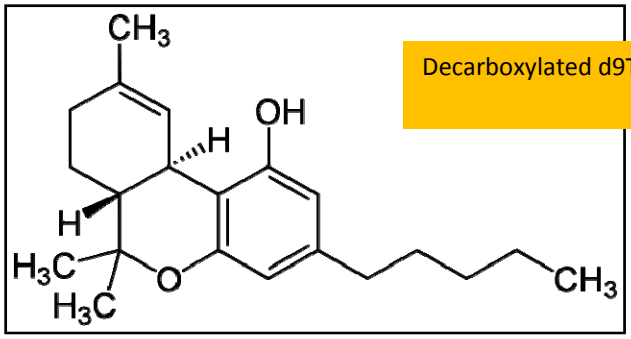
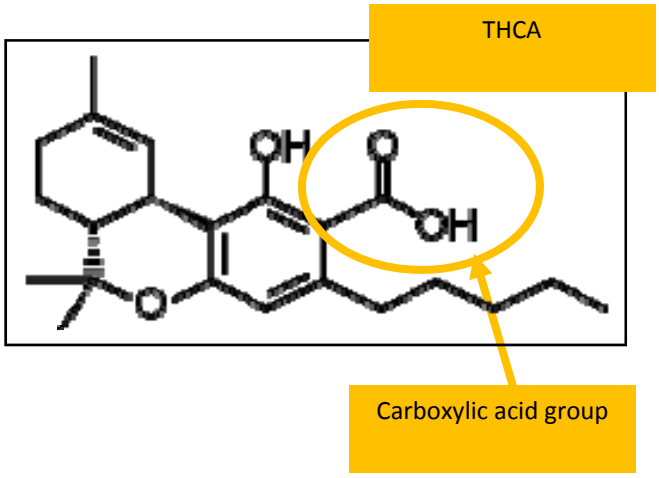
The decarboxylated d9THC molecule is shown at right.

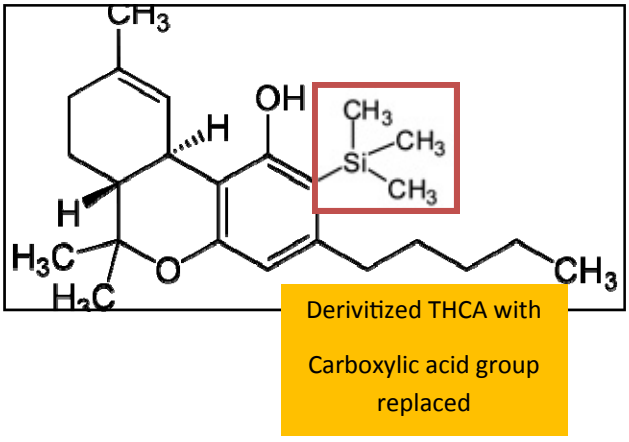


The precursor THCA molecule looks identical to d9THC except there is a carboxylic acid group attached.

When heated above 100C the carboxylic acid group converts to CO2 and is removed from the molecule leaving d9THC.



Derivitizing the THCA substitutes a more stable silicon based group for the carboxylic acid group.



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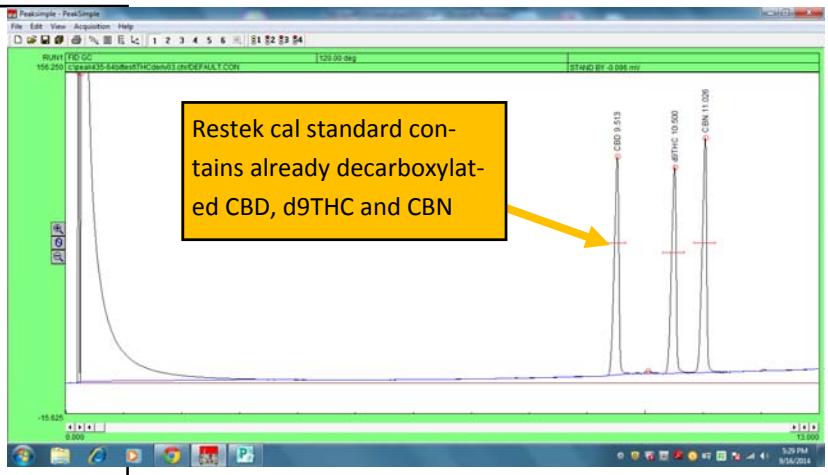
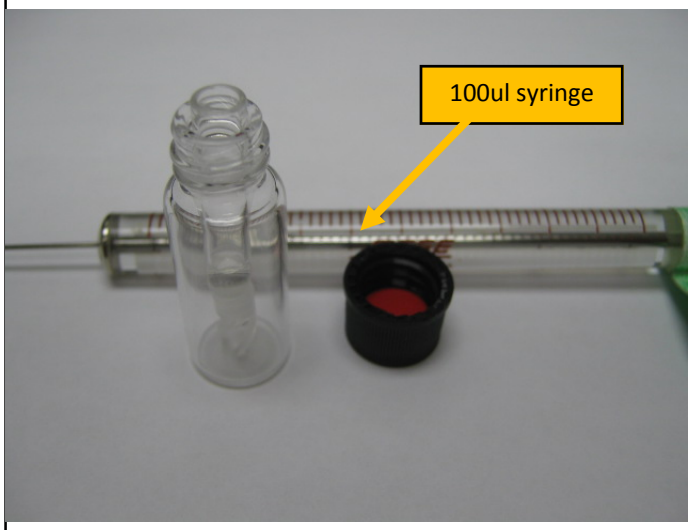
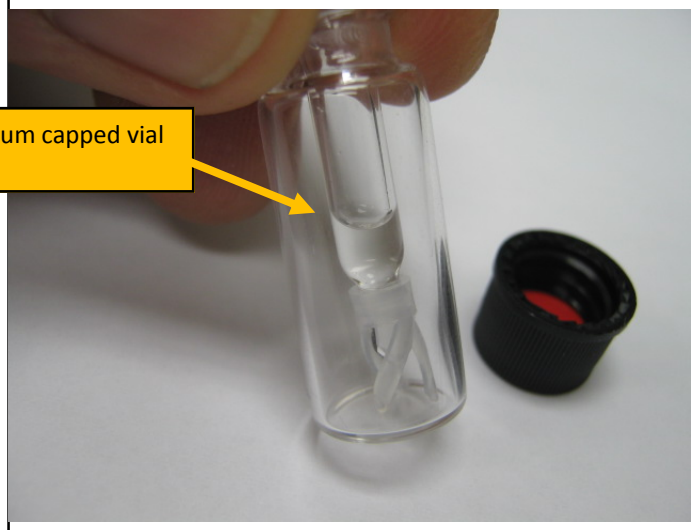
The chromatogram to the right shows the three peaks from Restek's #34014 calibration standard (www.restek.com). The CBD, d9THC and CBN are all completely decarboxylated in this mix.

1 ul of the Restek 34014 standard was injected into the GC with no sample prep at all.



50ul of the Restek 34014 mix was transferred into a 100ul vial using the 100ul syringe SRI provides with the GC. Vials and inserts like this are widely available.





A small, 50ul volume is all that is required. The Restek standard is in methanol.

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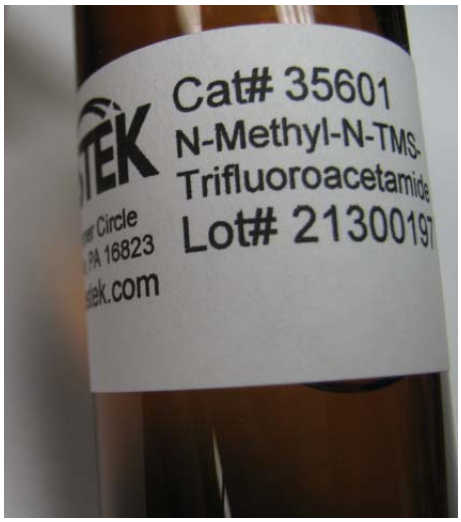
A small air compressor such as a fish aquarium pump is connected to a bent syringe needle (27gage 1.25” long) and placed in the vial containing the Restek mix to speed up the evaporation of the methanol solvent. It takes about 10 minutes to evaporate.

Its important that the end of the needle be above the liquid level so the liquid does not splash from the air bubbles.

Once the methanol solvent is completely dry, you will see some residue. This is the CBD, d9THC and CBN which have high boiling points and do not quickly evaporate.

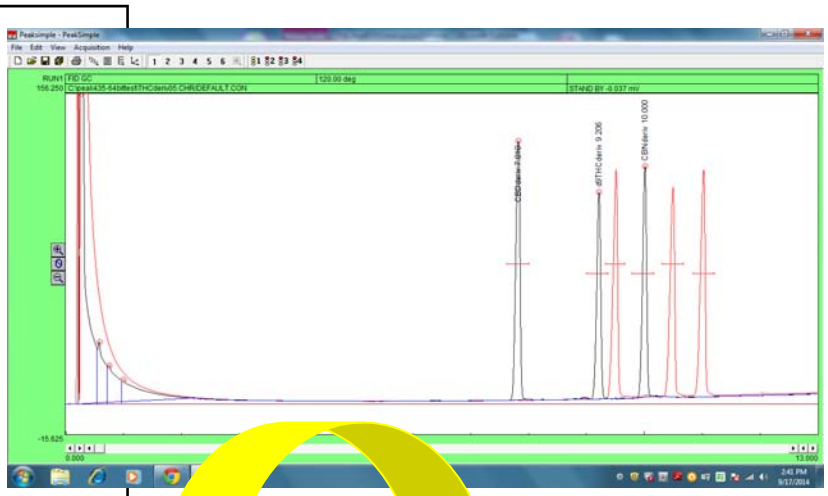
Add 50ul of the MSTFA derivitizing reagent. The CBD, d9THC and CBN will re-dissolve in the MSTFA. You may have to swirl the MSTFA a little to make sure the residues dissolve especially the ones at the very bottom of the 100ul insert which has a little blob at the bottom.

It’s important that the methanol is evaporated completely, as the derivitizing process will not work if methanol is present.



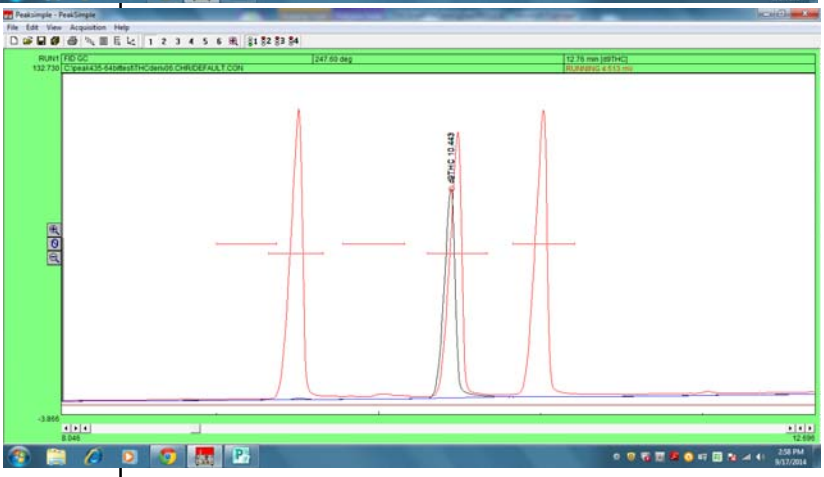
THCA vs d9THC testing using the SRI 8610C GC

The derivitized Restek standard looks like the chromatogram to the right. The peaks in red are the original un-derivitized, already decarboxylated CBD, d9THC and CBN peaks.



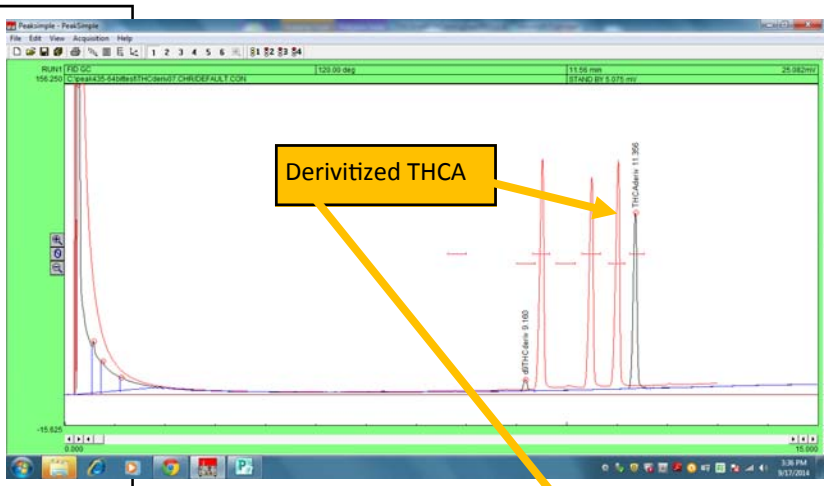
The peaks in black are the derivitized CBD, d9THC and CBN. You can see that the retention time of the derivitized peaks has shifted earlier.

Here is the chromatogram of un-derivitized THCA in black. Notice that the peak comes out at the same time as the d9THC. The un-derivitized THCA decarboxylates in the GC and becomes d9THC, so it makes sense that it elutes at the same time as d9THC.

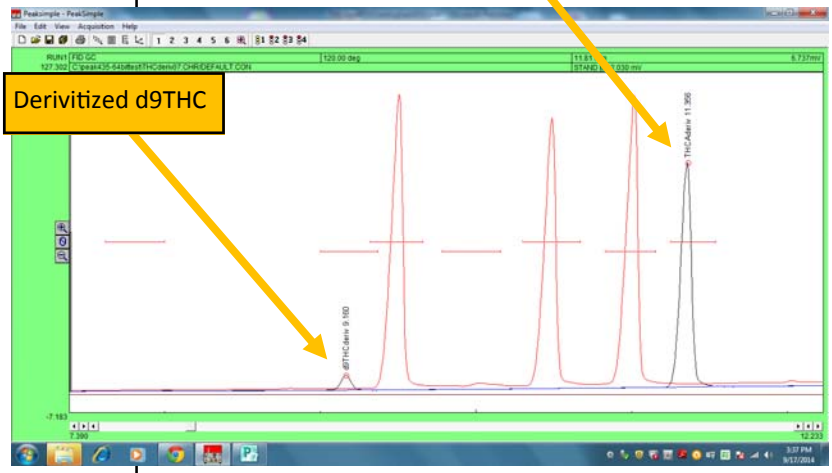


THCA vs d9THC testing using the SRI 8610C GC

Here is what the derivitized THCA looks like in black.



This is the same chromatogram zoomed in for more detail. Notice that a small THCderiv peak was also detected because some of the THCA standard had spontaneously decarboxylated in storage to d9THC. This may be why the standards are shipped in dry ice.



This is a typical cannabis flower extract where denatured alcohol is used as the extraction solvent

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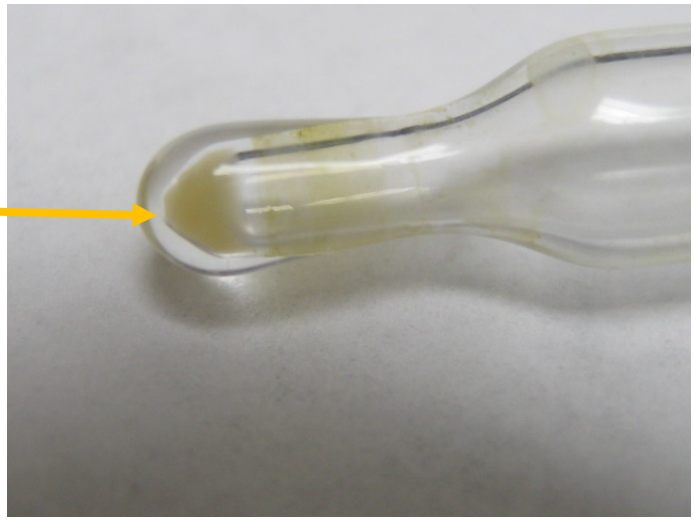
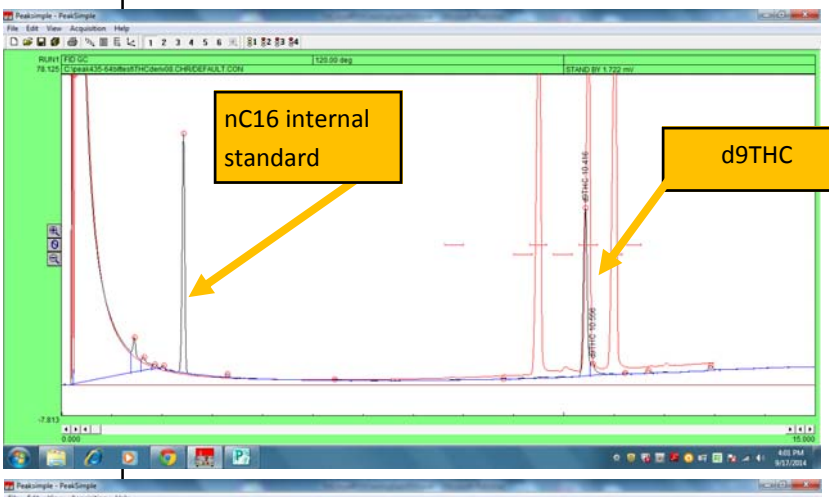
After evaporating the alcohol from 50ul of the cannabis extract you can see the residue including the green colored chlorophyll in the bottom of the 100ul insert.



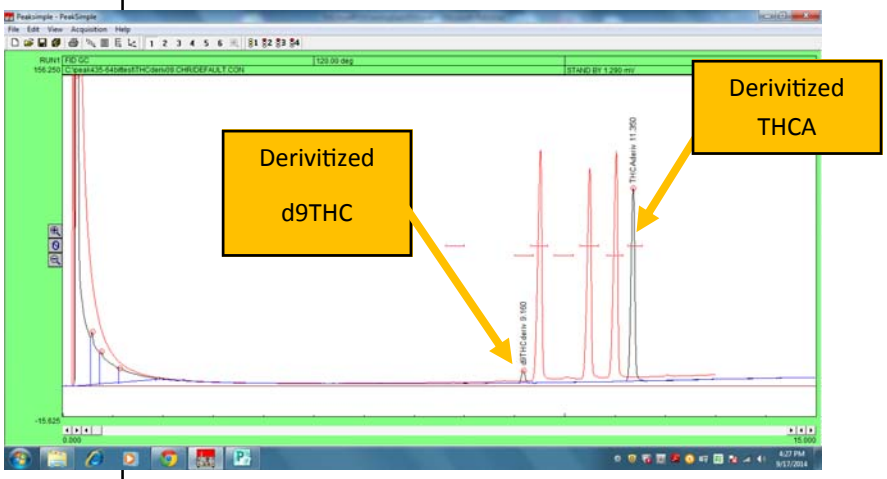
50 of MSTFA is added as before to derivitize the residue.

This is the chromatogram of the un-derivitized cannabis. The early peak is the nC16 internal standard peak which we typically add to the extraction solvent. The benefits of the internal standard are discussed in another publication.



This shows the same extract after derivitization. Apparently there was a ratio of about 10:90 of d9THC to THCA in the extract.

The cannabis used for this sample was very fresh and un-cured, so it might be expected that the extract would contain mostly THCA.



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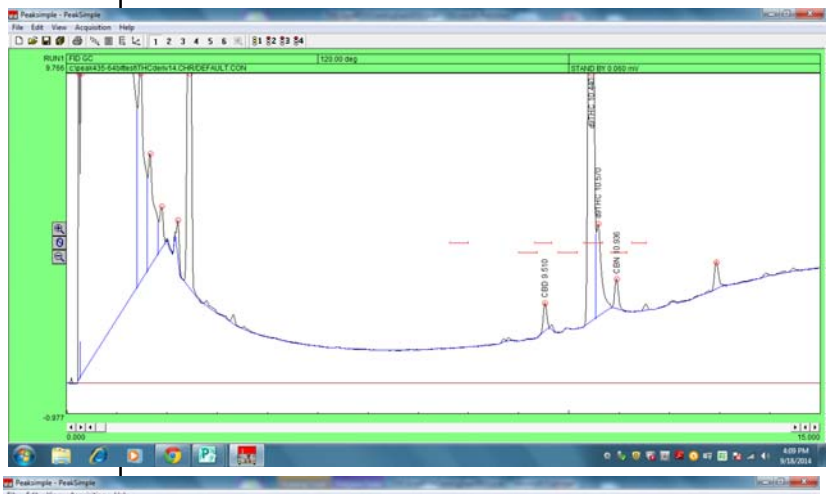
Here is another un-derivitized cannabis extract.



Here is the derivitized sample in black overlaid against the underivitized in red. In this case the ratio of d9THC to THCA is about 40/60 leading us to guess that this cannabis was not as fresh as the previous sample.