Recent improvements in Cannabis measurement

January 2018

SRI has improved the cannabis measurement process by derivitizing the sample before injecting into the GC.

The process starts in the same way as before by adding 1 gram per gallon (264mg/liter) of Methyl Stearate (CAS# 112-61-8) to the Acetone extraction solvent.

Shown here is a liter bottle of Acetone which has had the Methyl Stearate added to it. For short, we call this the " dirty solvent " to distinguish it from clean acetone.

Here is the calibration mixture which is made by mixing the 3 common cannabinoids (CBD, d9THC and CBN) with the " dirty solvent.

The cannabinoid standards are typically delivered (from Restek, Lipomed, Cerilliant etc) in glass ampules at a concentration of 1mg/ml (1000ng/ul).

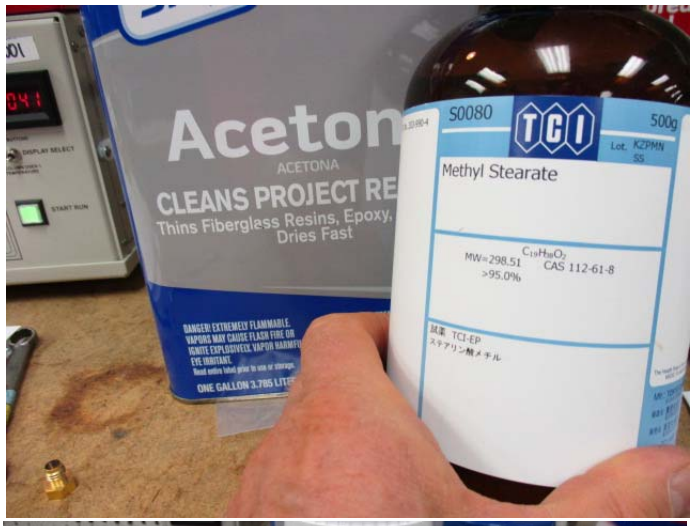
Use the 100ul syringe provided with the SRI GC to:

Add 100ul each to a 1.5ml vial. This changes the concentration from 1000ng/ul each to 333ng/ul each. There should be 300ul of liquid now in the vial.

Then add 300ul of " dirty solvent" to the vial.

There should now be 600ul of liquid in the vial.

The liquid is a mixture of methanol and acetone with the cannabinoids dissolved in it at 333ng/ul each, plus the methyl stearate. The calibration standard must use the same bottle of "dirty solvent" that you will use to extract the cannabis sample, so mark the calibration standard vial so you know which bottle of "dirty solvent" you used.



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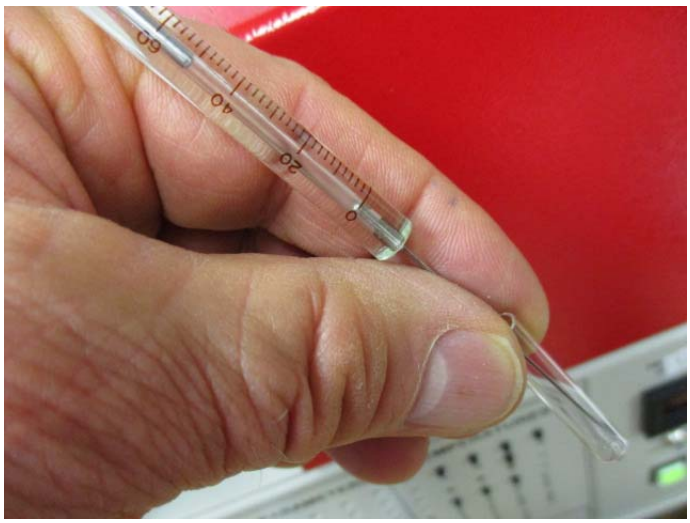
Obtain some smaller cheaper disposable glass vials.

These are the ones we use, but any small vial is OK.

Use the 100ul syringe to measure 50ul of the calibration standard into the vial.



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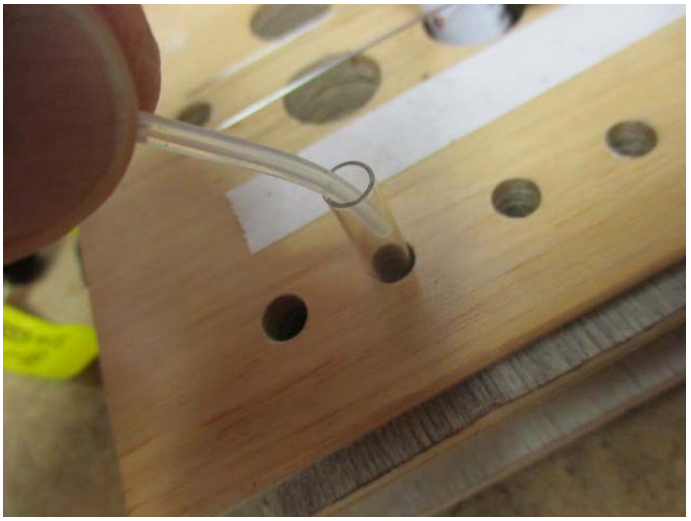
Obtain a small aquarium pump and some silicone tubing. You may need a couple sizes to reduce down to a size of tubing which will fit into the small vial.

Put the tubing into the vial about halfway down, but above the level of the 50ul of liquid. The air flow will quickly (<5minutes) evaporate the liquid leaving the cannabinoids and methyl stearate as a white film in the bottom of the vial.



You have to get rid of the methanol because it will prevent the derivatization from working. The acetone is OK, but the methanol is not OK. Unfortunately the standards from Restek etc are only available in methanol.

Use the 100ul syringe to add 50 ul of the MSTFA derivitzation reagent to the vial. There was originally 50ul of methanol/acetone in the vial. Now there is 50ul of the MSTFA so the concentration of the cannabinoids/methyl stearate is the same, just in the MSTFA instead of the methanol/acetone.

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Wash the 100ul syringe out with some kind of alcohol. It can be the denatured alcohol form the hardware store (a mix of methanol and ethanol).

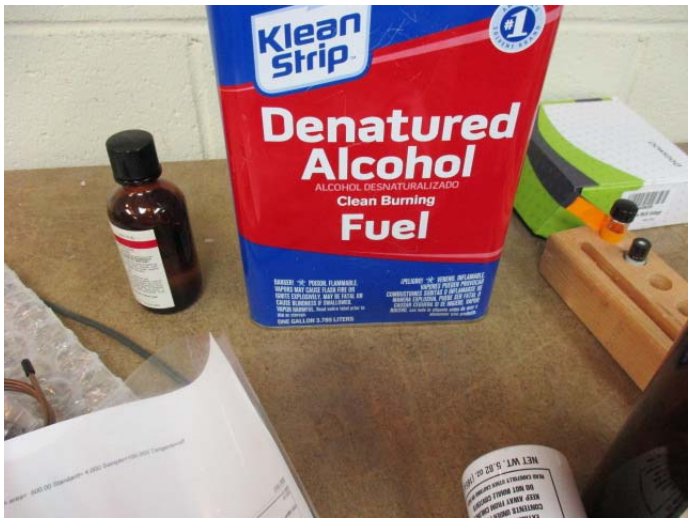
Or it can be methanol or ethanol.

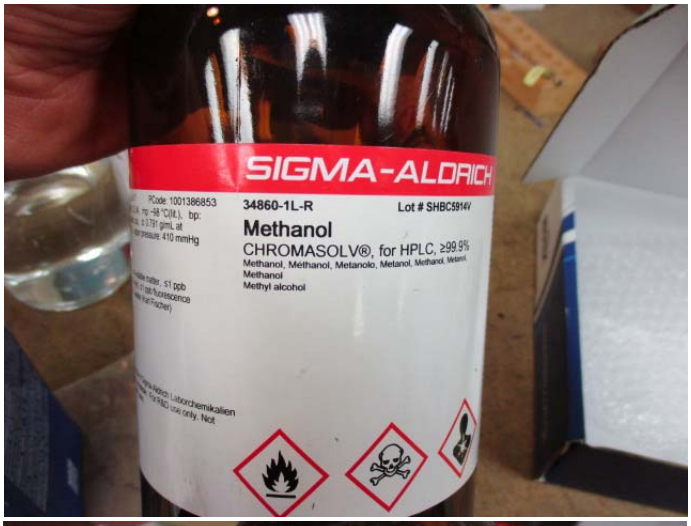
Any alcohol is OK.

The alcohol “kills” the MSTFA which is necessary because the MSTFA will polymerize on contact with water. If you don’t kill the MSTFA it will polymerize in the syringe and you won’t be able to move the plunger, ruining the syringe.

After you wash out the syringe with alcohol, wash the alcohol out with clean acetone. If you leave alcohol in the syringe you may contaminate the next batch of MSTFA, inadvertently “killing” the MSTFA,

We find it convenient to have one 40ml vial with alcohol and a second vial with clean acetone.





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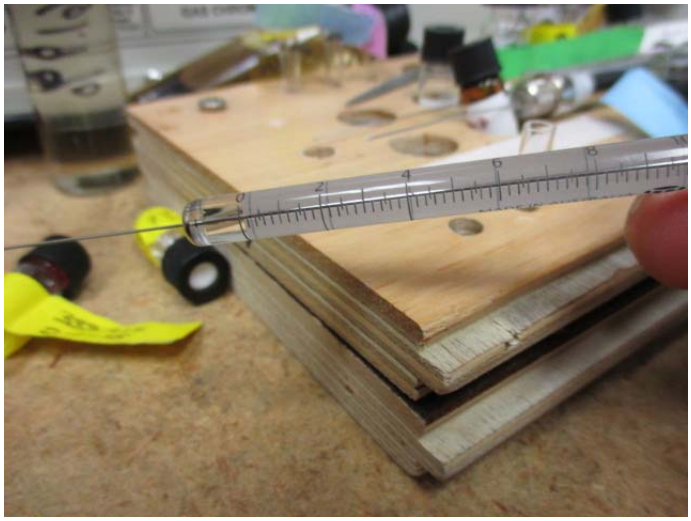
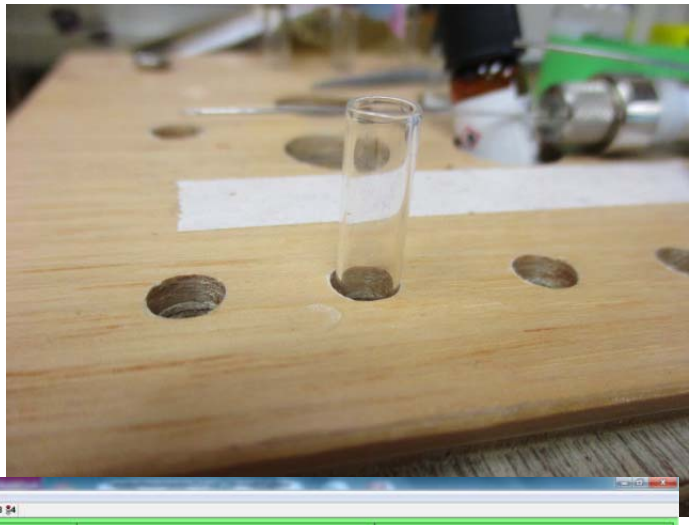
Use the 10ul syringe provided with the GC to inject 1ul of the derivitized 333 calibration standard into the GC.



Its handy if you have a block of wood to keep the tiny vial from tipping over.



Don't forget to wash the syringe with the alcohol followed by the acetone. The 50ul of MSTFA will polymerize after about 30 minutes which gives you time to make a couple replicate injections.

The chromatograms should look like the ones below.

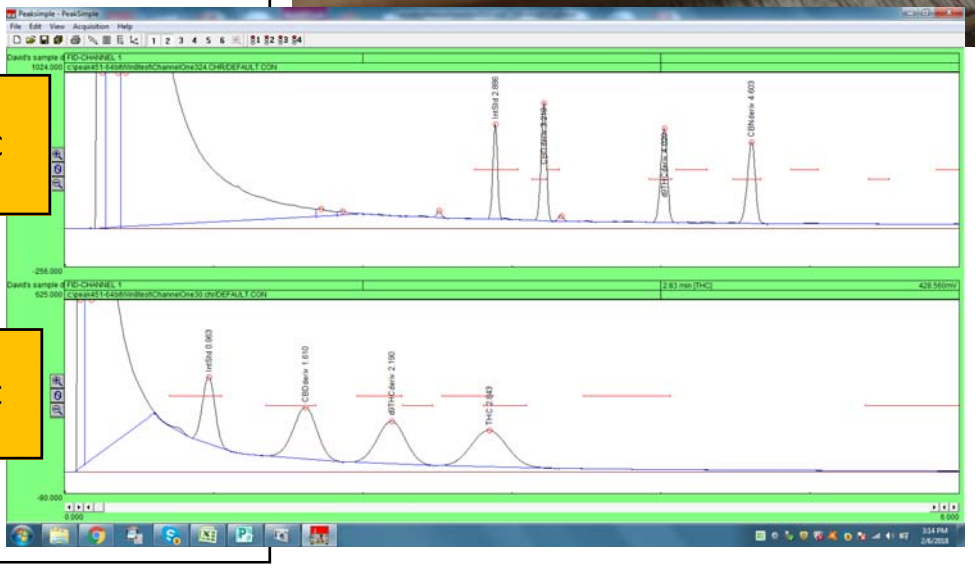
The chromatogram from the 310M&M GC are a little different from the Model 420, but you should see the methyl stearate peak followed by the 3 cannabinoid peaks. Calibrate these peaks in the normal way.

We like to name the CBD leak " CBDderiv", the THC peak d9THCderiv etc to make it clear that the derivitized peaks are different from the un-derivitized peaks. The derivitized peaks elute earlier than the un-derivitized peaks and have slightly larger peak areas.

The derivitized peaks should have their own calibration curves, also with distinct names,

301M&M GC

Model 420 GC



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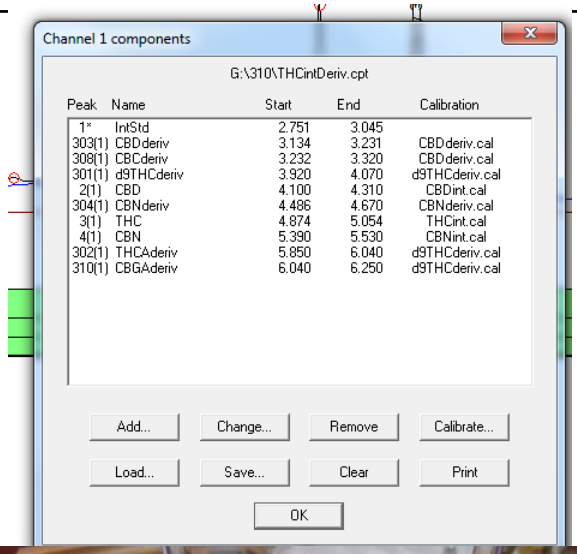
Here is a component table which has retention windows and calibration curves for both derivitized and un-derivitized peaks.



Prepare a cannabis sample in the normal way.

Weigh out 100mg of flower or 25 mg of concentrate.

Add the cannabis to the 40ml vial along with 40ml of the “ dirty solvent”.

Follow the exact same procedure as you did with the 333 calibration standard:

Use the 100ul syringe to measure 50ul of the cannabis extract into the small vial.

Evaporate until it is dry. This will be much quicker with acetone.

Add 50ul of the MSTFA then wash the syringe.

Use the 10ul syringe to inject 1ul of the derivitized extract into the GC.

Then wash the 1ul syringe.

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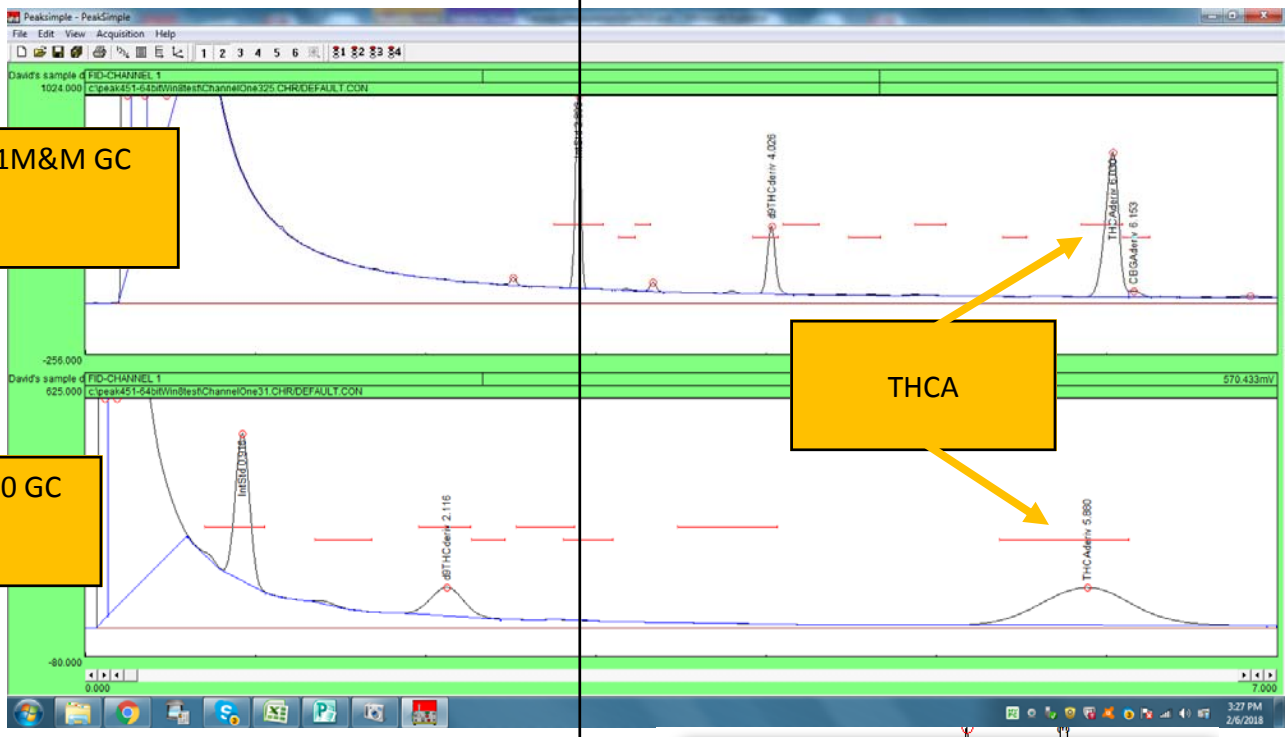
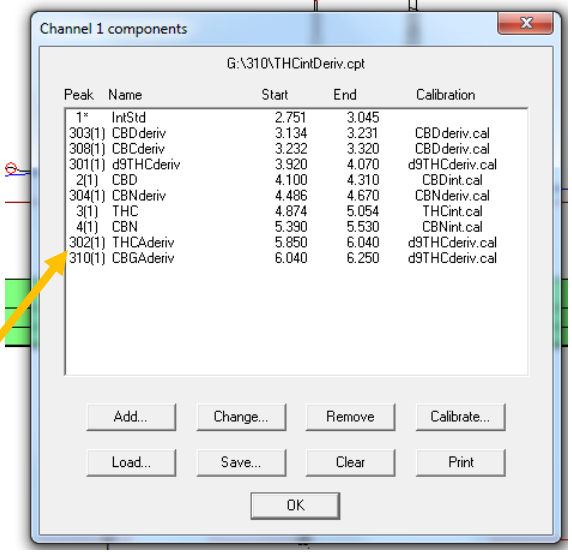


This sample was a fresh flower sample so it was mostly d9THC and THCA.

The chromatogram will show one peak which elutes in the d9THCderiv window. This window was already calibrated so you will see the calibrated answer in the Results screen. The other larger peak at the end of the chromatogram is the derivitized THCA.

You will have to create a new retention window for the THCA peak called THCAderiv.

This will show in the component table.



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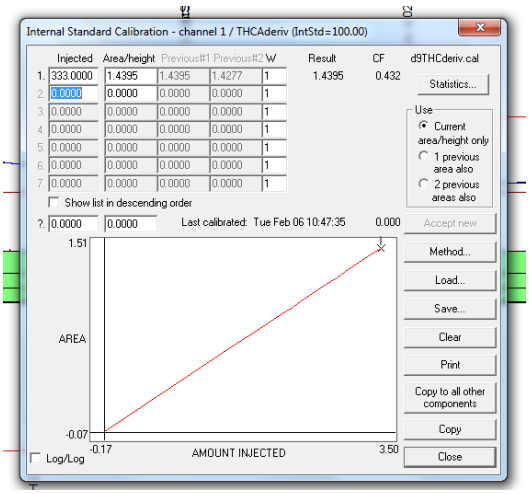
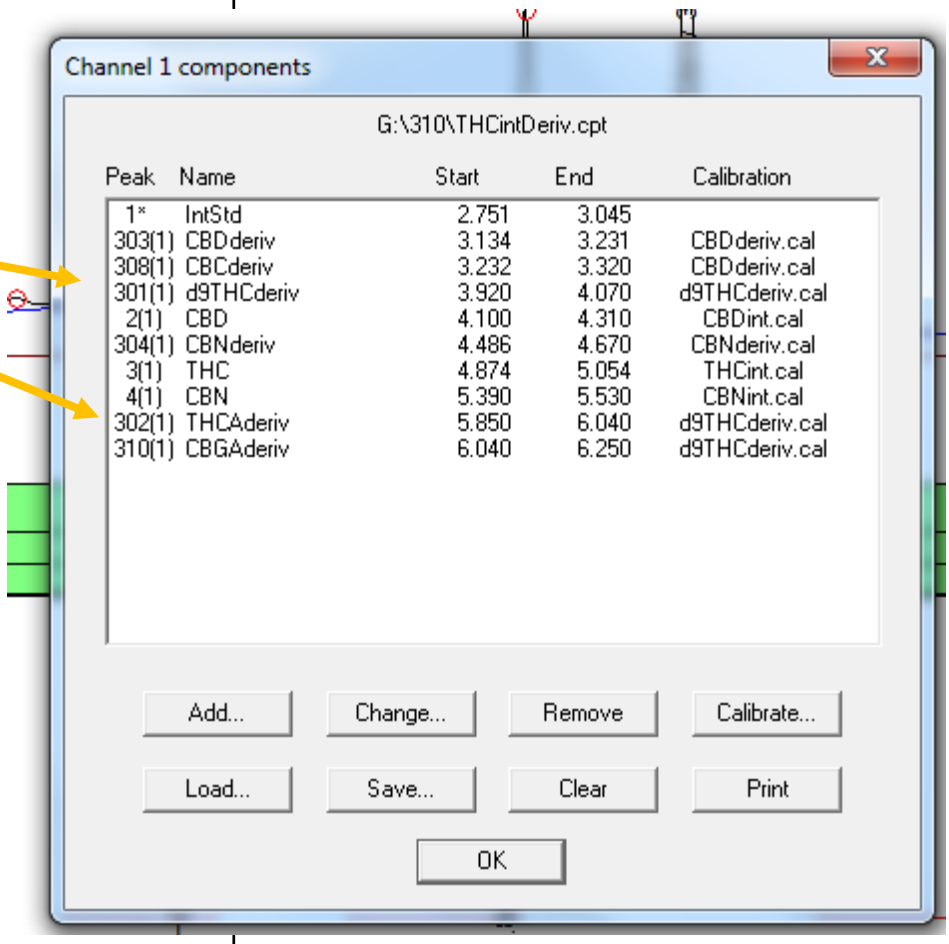
Apply the same calibration curve you created for the d9THCderiv to the THCAderiv.



In the component table notice that the d9THCderiv and the THCAderiv have the same calibration curve name.



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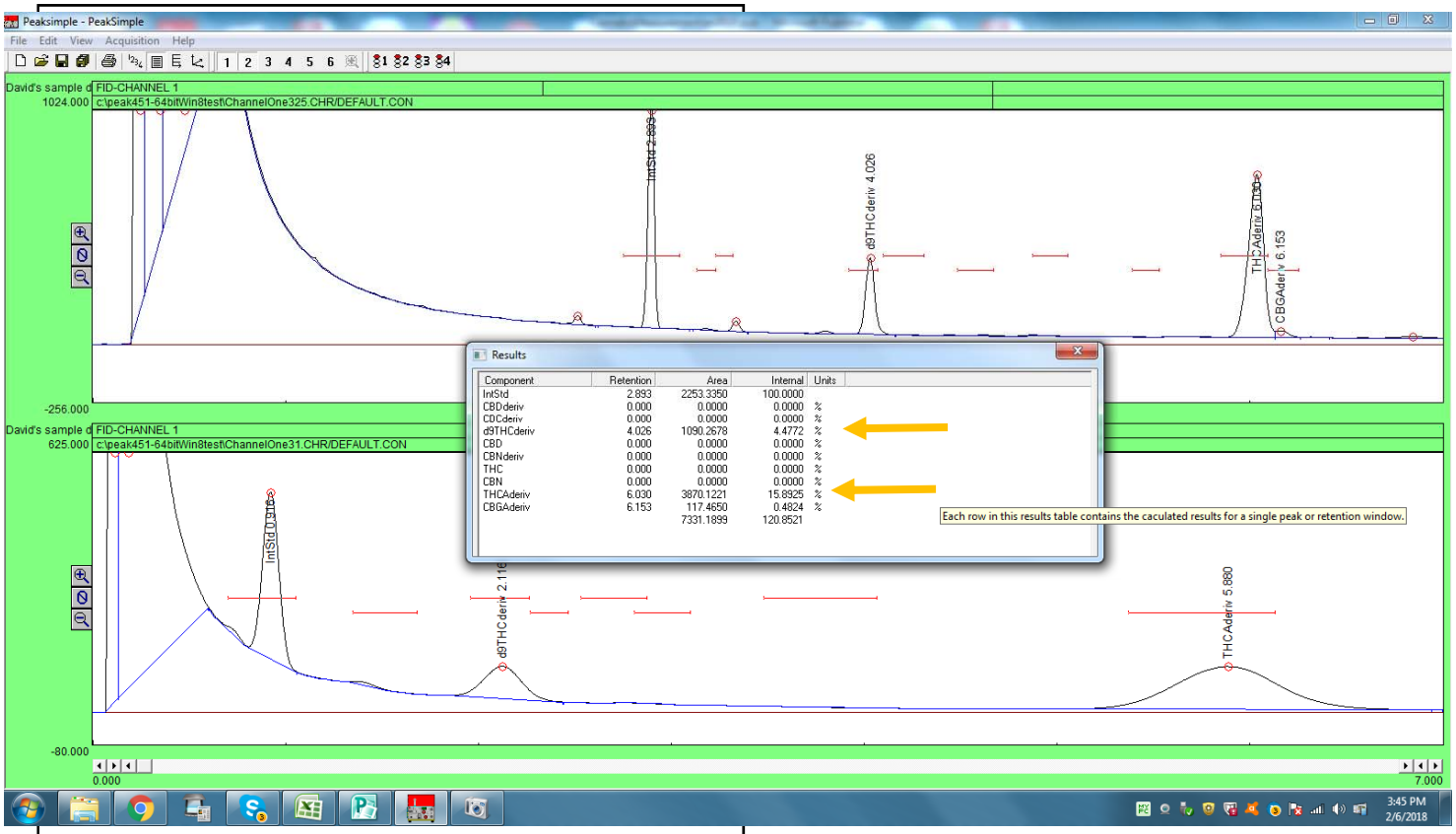

When you look at the Results screen you can see that for this sample the d9THCderiv calculated out at 4.47% and the THCAderiv calculated out at 15.89%.

To obtain the total potency you should multiply the THCA result by .877 (13.82%) then add that number to the d9THC (13.94+4.47=18.40%).

There is a way to do this automatically in the PeakSimple software, but we will leave that subject for a separate document.