

Chromat Calibration

Updated October 27th, 2017

Calibrating the Chromatograph

Performing the Chromat Calibration is highly recommended when starting a new well. The MLogger already has a default calibration on it but that calibration might not work due to various reasons such as changes in air pressure due to altitude or weather systems, chromat column packing, air temperature, sensitivity of the GC filament, and/or the Carrier Air Pump. This is why it is best to do a field calibration at the beginning of the well so that the chromatograph can be as accurate as possible. Calibration checks can also be performed during the course of the well to verify accuracy.

Go through the following steps to calibrate chromatograph.



Make sure that the Chromat Runs are stopped by looking at the Status Bar on the main TControl Window. If there is a run currently in it's cycle, then go to the Setup menu \rightarrow Chromat tab \rightarrow select Stop Continuous Runs. This will allow the current run to finish without starting another run. Once the Status Bar displays "Chromat Stopped", then proceed to the next step.

Chromat Running

Chromat Run 2:21

CCD: 0.034 GC: 0.259 TCD: 0.184 **Aux 1: N/A**

Chromat Stopped

CCD: 0.045 TCD: 0.185 GC: 0.045

Aux 2: N/A

Aux 1: N/A Aux 2: N/A **Chromat Stopped**



2. Turn the Zero Air knob clockwise so that it is pointed to Zero Air. This pulls a vacuum of fresh air from the Zero Air port that is on the rear panel of the MLogger.







- 3. Zero the GC filament.
 - a. Set the GC Flowmeter to 0.5scfh.
 - b. Turn the GC potentiometer knob on the front panel of the MLogger counterclockwise to decrease the voltage or clockwise to increase the voltage to 0.05v. Note: The voltage will not rest directly on 0.05v, so just get it as close as possible.

GC Potentiometer



GC Voltage (high)

CCD: 0.059 TCD: 0.049 GC: 0.097

Aux 1: N/A Aux 2: N/A Chromat Stopped

In this example, the GC voltage is a little bit higher than the target 0.05v, so the GC knob needs to be turned counterclockwise, slightly, to attain the desired voltage.

GC Voltage (zeroed)

CCD: 0.048 TCD: 0.054 GC: 0.056

Aux 1: N/A Aux 2: N/A Chromat Stopped



4. Attach the gas regulator to the bottle of 1% Blend (both are provided by Terra SLS). Make sure that the regulator is screwed on tight. Then, attach a short piece of ¼" polyflow from the regulator to the Test Gas port on the front panel.

1% Blend w/ Regulator



Attached to Test Gas port





5. Turn the Test Gas knob clockwise so that it is pointed to Test Gas. This will change the flow through the GC flowmeter to come directly from the Test Gas port.

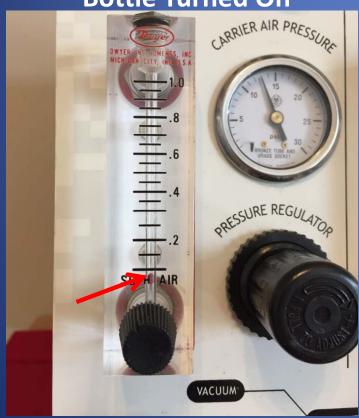
Pointed to Test Gas





- 6. Turn on the bottle of Blend by turning its knob counterclockwise for 2 or 3 turns. Ensure the bottle has gas in it by checking the PSI on its regulator gauge. Note: If the pressure gauge shows 0, tighten the Valve Fitting on the regulator.
- 7. Turn the GC flowmeter knob counterclockwise to raise the flow up to 0.5scfh or higher.
- 8. Wait 30-45 seconds to ensure that the sample loop is completely full.



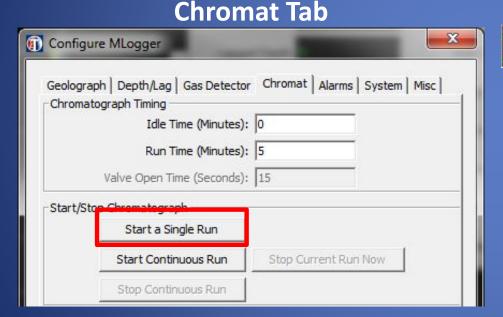


GC Flowmeter Turned Up





- Go to the Setup menu → Chromat tab → verify Run Time is set to 5 minutes → click Start a Single Run.
- 10. As soon as the Chromat Run time in the Status Bar starts counting, then turn the bottle of Blend off by turning its knob clockwise until it is tight. The BB in the GC flowmeter should drop all the way to the bottom.
- 11. Wait 5 minutes for the run to finish.



Chromat Run Started

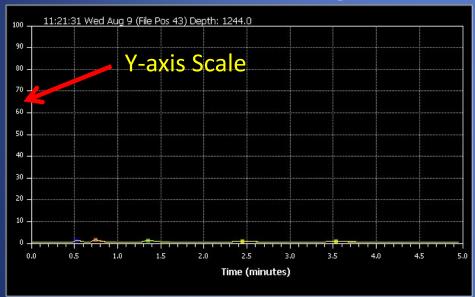
CCD: 0.033 TCD: 0.049 GC: 0.241

Aux 1: N/A Aux 2: N/A Chromat Run 0:01

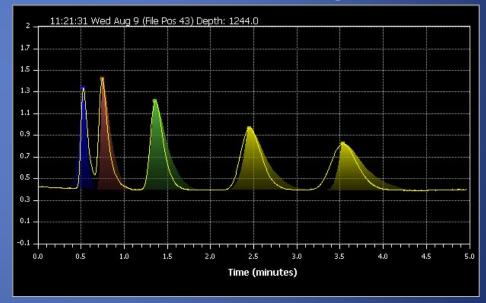


12. Once the run has finished, the peaks may be hard to see if the scale in the graph isn't set low enough; by default it is set to 0-100. Right-click anywhere on the y-axis scale (vertical scale) to get a scale change window and set the Minimum to 0 and the Maximum to 2 and then click OK. Or, left-click anywhere in the graph to change to a "Curve Zoom" scale, which will change the y-axis scale to a scale that best fits the curve.

Before Scale Change



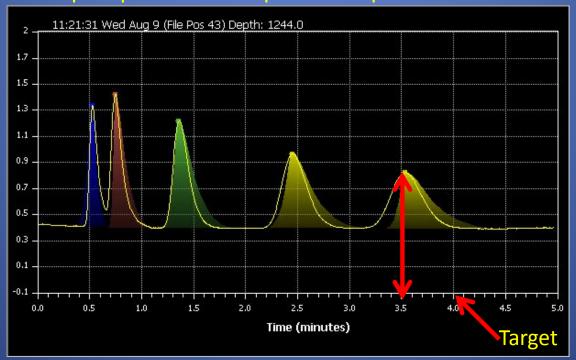
After Scale Change





13. Set the Carrier Air Pressure.

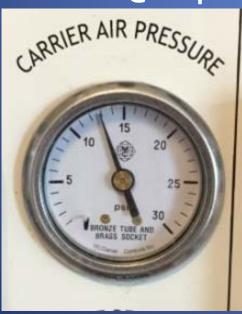
In the example below, the nC4 peak is centered around the 3.5 minute mark on the x-axis scale. This is OK but, for best results, try to center the nC4 peak around the 4.0 minute mark. This spreads the peaks out farther and gives a little bit better separation between the C1 and C2 peaks. Since the nC4 peak came in too fast, the Carrier Air Pressure needs to be decreased. If nothing needs to be changed, skip to step 16. Note: The required Carrier Air Pressure will be different from MLogger to MLogger due to the breakdown of the Chromat Columns over time and other variables such as the pumps and atmospheric air pressure.





- 13. Set the Carrier Air Pressure (cont'd)
 - a. Adjust the Carrier Air Pressure by using the Pressure Regulator knob just below the gauge. To use the knob, pull outward on it until it clicks. Turning the knob clockwise and counterclockwise will increase or decrease the pressure on the gauge. As a rough rule of thumb, adjusting the pressure by 1 psi will shift the center of the nC4 peak by about 15 seconds on the x-axis scale. In this example, the peak is centered at around 3.5 minutes. So, decreasing the pressure by 2 psi should shift the peak roughly 30 seconds to the 4.0 minute mark. Note: Anytime the Carrier Air Pressure is adjusted, a new calibration run should be done.

Pressure @ 13 psi



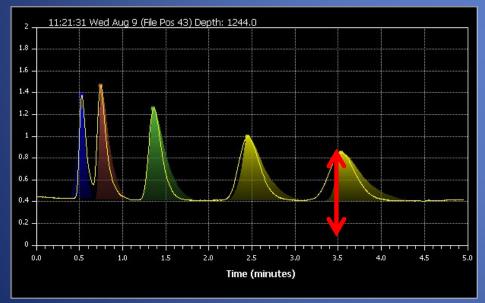
Pressure @ 11 psi



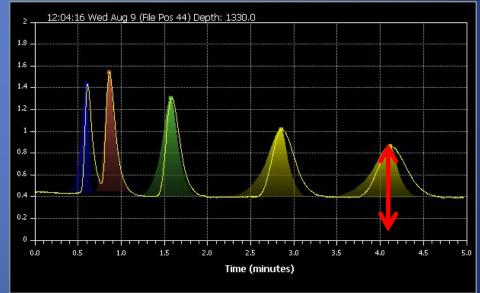


- 13. Set the Carrier Air Pressure (cont'd)
 - b. Once the Carrier Air Pressure has been adjusted, do another calibration run by repeating steps 1-11. In this example, decreasing the Carrier Air Pressure from 13 psi to 11 psi got the nC4 peak pretty close to being centered around the 4.0 minute mark (it's just slightly over). This looks pretty good. If needed, adjust the Carrier Air Pressure again and repeat steps 1-11 again. If the adjustment is fine, then push the Pressure Regulator knob back in so that it is locked in place.

Before Adjustment



After Adjustment





- 14. Now that the Carrier Air Pressure is set and the bottle is turned off and the BB in the GC flowmeter has dropped, turn the Test Gas knob counterclockwise so that it is pointed back to Rig.
- 15. Adjust the GC flowmeter back to 0.5scfh.

Point back to Rig



Set GC Flowmeter back to 0.5





16. Right-click anywhere in the chromatogram graph on the main TControl window to open the Chromat Records and Chromat Charting windows.

Chromat Records Chromat Records Calculated PPM Scale Factor Methane (C1): 10697 C1: 7834 Ethane (C2): 9405 C2: 10422 Propane (C3): 12269 C3: 10927 isoButane (iC4): 18638 i-C4: 10772 Butane (nC4): 24402 n-C4: 10234 isoPentane (iC5): 0 i-C5: 0 Pentane (nC5): 0 n-C5: 0 Acetylene (C2H2): 0 C2H2: 0 Manually Edit Scale Factors << Save Save >> 12:04:16 Wed Aug 9 (File Pos 44) Next Latest Go To... Clear Save Previous Integrate Integrate Range Options Clear Range Integration Calibration Load Factory Cal Save All Start a Single Run Save Field Cal Start Continuous Run Stop Continuous Run Load Field Cal Refresh Record OK Cancel

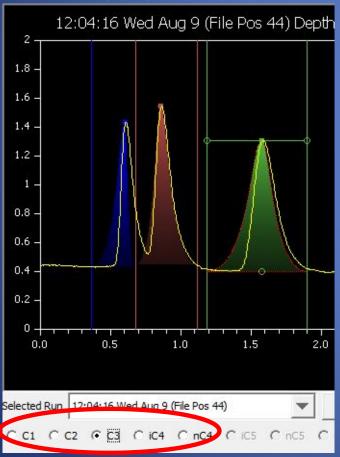
Chromat Charting



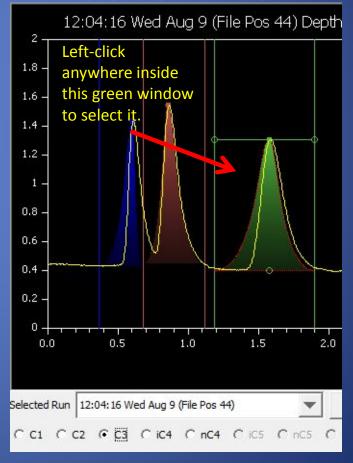


- 17. Set the Retention Windows.
 - a. In the Chromat Charting window, select a Retention Window either by selecting one of the radio buttons for C1-nC4 at the bottom of the Chromat Charting window or left-click anywhere inside the desired Retention Window.

Radio Buttons

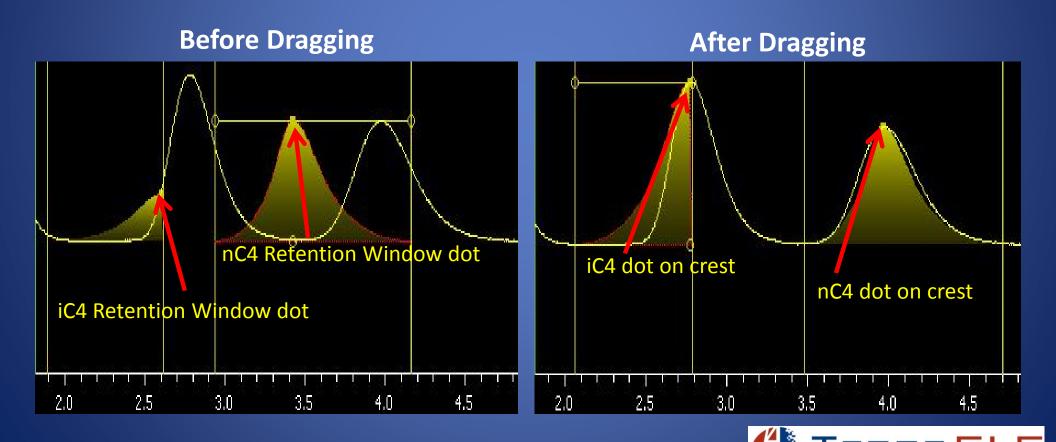


Retention Window

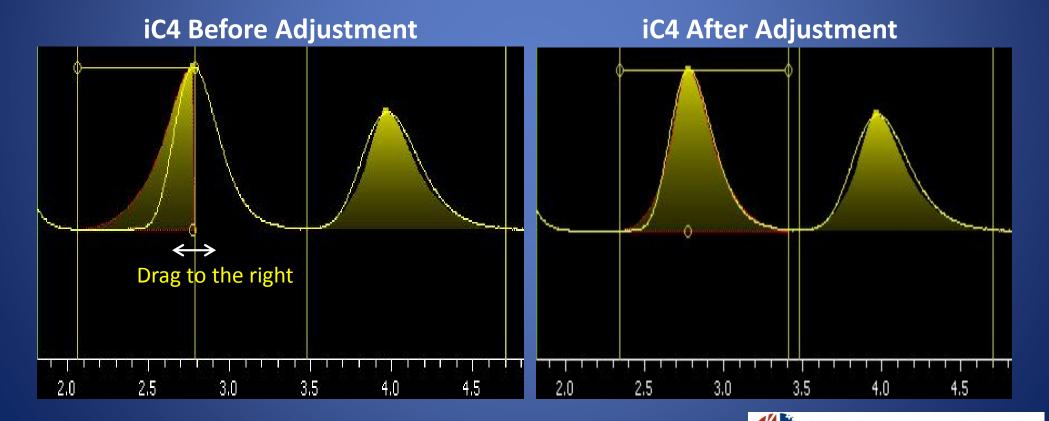




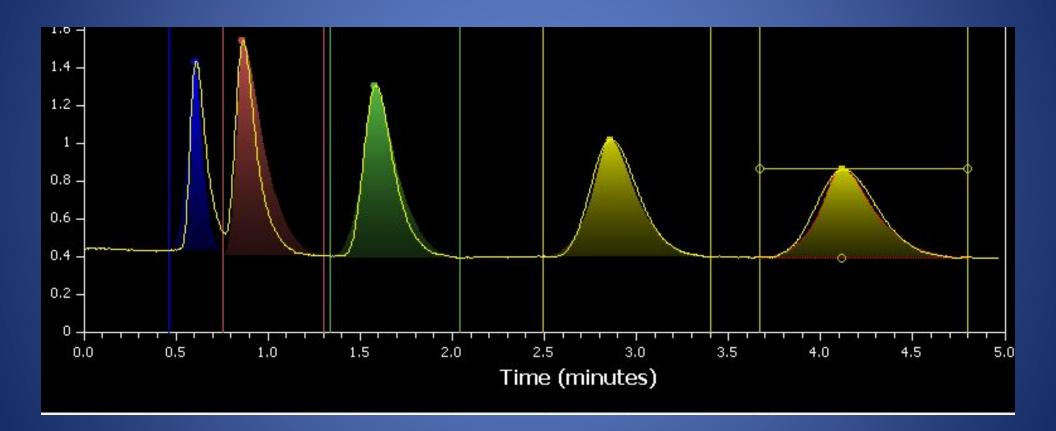
- 17. Set the Retention Windows (cont'd).
 - b. Once a Retention Window is selected, drag the entire window (hold the left-click button and drag at the same time) so that the colored dot in the Retention Window rests at the top of the corresponding peak. Note: The sides of a Retention Window cannot move past a colored dot and can actually move another Retention Window if pushed into it.



- 17. Set the Retention Windows (cont'd).
 - c. After a Retention Window has been dragged into place, adjust the window to fully encompass its corresponding peak by dragging the sides of that window. When a Retention Window is selected, the left and right sides can be adjusted by hovering the cursor over the side of the window until the cursor changes to a double-sided arrow. Once the cursor changes to the double-sided arrow, hold down the left-click button and move the mouse to drag that side of the window into position.



- 17. Set the Retention Windows (cont'd)
 - d. Repeat Steps 17a through 17c for each peak. The final result should look similar to the image below.



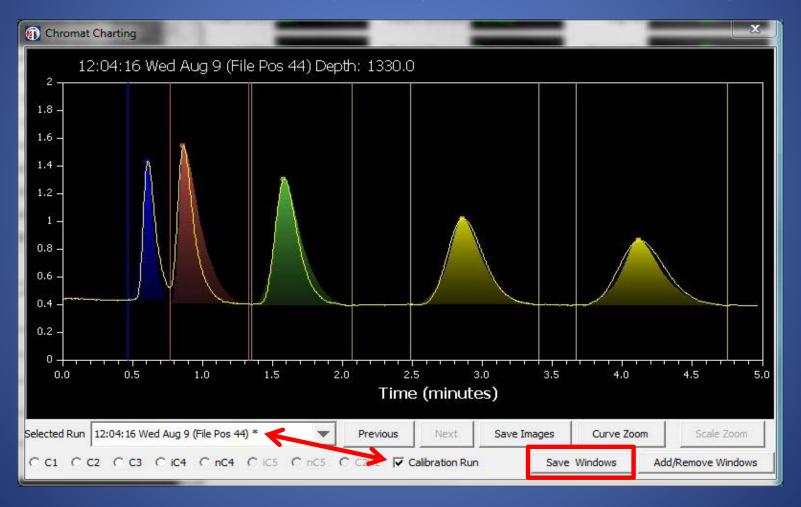


17. Set the Retention Windows (cont'd)

- e. When making the adjustments to the windows, here are some tips.
 - i. Don't make the windows too tight around the peaks. Give some 'wiggle' room.
 - ii. Try to get the right side of the C1 window and the left side of the C2 window right on top of each other. The lines will appear purple the closer they get and will turn red once they are on top of each other.
 - iii. The shaded area doesn't actually matter; it's just there for visual effect to help guide setting the windows.
 - iv. The line that goes across the window represents the highest point within the window. On that line is a dot, which is automatically placed at the highest point. The sides of the retention window **cannot** be adjusted past this point (i.e., the right side of the window cannot move to the left side of this dot).
 - v. Don't set the right side of the nC4 window farther than the 4.8 minute mark. Setting the window past that point can cause a "Swoosh Detection" in later runs if the yellow sparkline terminates within the nC4 Retention Window. If the right side of the nC4 window needs to be set past the 4.8 minute mark, it is best to just increase the Carrier Air Pressure and do another calibration run.



- 18. After the windows are set, click the Save Windows button.
- 19. Check the box for Calibration Run. Checking the Calibration Run box will add an asterisk to the Chromat Record or File Position so that the record can easily be found later on and it adds the functionality to carry the calibration to all subsequent runs.





20. Now, in the Chromat Records window, click the Integration Calibration button to open the Set Test Gas PPM window. Enter the ppm values from the bottle of 1% Blend into the corresponding fields. These values will vary from bottle to bottle. As the values are typed in, new Scale Factor values will be calculated.

Note: If the Calculated Scale Factors do not fall within the following ranges, check the Troubleshooting section.

C1: 9000 to 12000

C2: 8000 to 11000

C3: 9000 to 13000

iC4: 15000 to 30000

nC4: 16000 to 35000

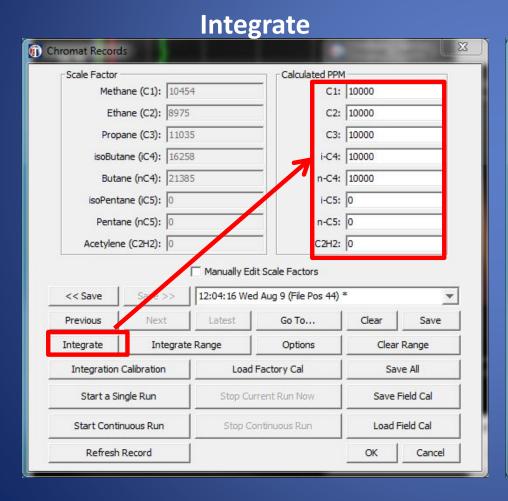


	Input PPMs	Calculated Scale Factor
Methane (C1):	10000	10454
Ethane (C2):	10000	8975
Propane (C3):	10000	11035
isoButane (iC4):	10000	16258
Butane (nC4):	10000	21385
isoPentane (iC5):		0
Pentane (nC5):		0
Acetylene (C2H2):		0

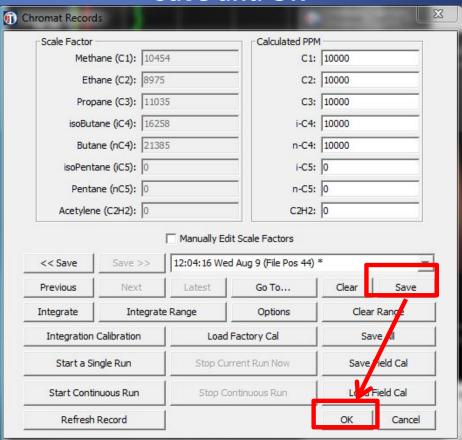
21. Click OK to exit the Set Test Gas PPMs window. The new Scale Factors should now be reflected in the Scale Factor column in Chromat Records window.



- 22. In the Chromat Records window, click the Integrate button. The values in the Calculated PPM column should now reflect what was entered in the Integration Calibration step.
- 23. Click Save and OK.
- 24. Turn the Zero Air knob back to Rig. DONE!



Save and OK





Summary

- 1. Stop Continuous Runs.
- 2. Turn the box to Zero Air.
- 3. Zero the GC filament.
- 4. Hook the bottle of Blend up to the Test Gas port.
- 5. Turn the Test Gas knob to Test Gas.
- 6. Turn the test gas bottle on.
- 7. Adjust the GC flowmeter up to 0.5scfh or higher.
- 8. Wait 30-45 seconds.
- 9. Start a single run.
- 10. Turn the test gas bottle off after the run has started.
- 11. Wait for the run to finish.
- 12. If necessary, make adjustments to the Carrier Air Pressure and redo previous steps.
- 13. Turn the Test Gas knob back to Rig.
- 14. Adjust the GC flowmeter back up to 0.5scfh.
- 15. Adjust the Retention Windows.
- 16. Save the Retention Windows.
- 17. Check the box for Calibration Run.
- 18. Integration Calibration.
- 19. Integrate.
- 20. Save.



Troubleshooting



A bad Chromat Calibration can affect the Scale Factor values that are used to calculate the ppm value of each gas component. High Scale Factor values can affect the chromat readings drastically by exaggerating actual chromat values or by calculating false chromat values when no visible chromat peaks are present in the chromat run. Below are some normal ranges for Scale Factor values for each component. If the Scale Factor values are much different than what is listed below after performing a Chromat Calibration, then something was done incorrectly during the calibration and the calibration should be done again.

Normal Scale Factor Ranges

C1: 9000 to 12000

C2: 8000 to 11000

C3: 9000 to 13000

iC4: 15000 to 30000

nC4: 16000 to 35000



There are several things that can go wrong during a Chromat Calibration, such as starting the single run too soon, not adjusting the GC flowmeter up, not saving the retention windows before doing the Integration Calibration step, a bad GC filament, residual gas left in the regulator, etc. The following examples display what can go wrong during a calibration and how to correct or prevent the issues.

Common Issues

Short Chromat Peaks

High Scale Factor Values

Short C1 Peak

Tall C1 Peak

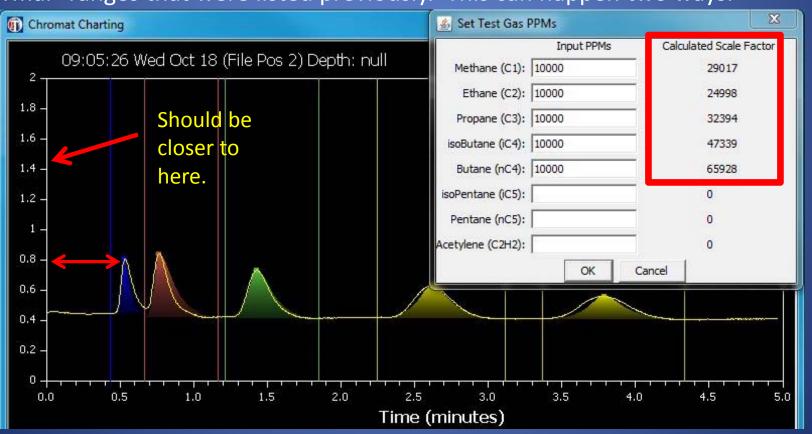
Setting C1 Retention Window Improperly

Recycled Gas



Short Chromat Peaks

In the image below, all of the chromat peaks are shorter than normal. From Base to Peak, the C1 peak starts at about 0.4 and crests at 0.8, making it roughly 0.4 chart divisions tall when it should be right around 1.0 chart divisions tall. This will cause the Calculated Scale Factors to be abnormally high compared to the "normal" ranges that were listed previously. This can happen two ways.





Short Chromat Peaks (cont'd)

Starting the Single Run Too Soon

Step 8 of the calibration instructs to wait 30-45 seconds after the bottle has been turned on before starting a single run in Step 9. This is important because it allows enough time for the Sample Loop to completely fill with the test gas. If not enough time has been allowed to pass before starting the single run, then the Sample Loop won't have enough gas in it. This results in chromat peaks that appear shorter than normal. Continuing on with the rest of the calibration steps with these short peaks will cause higher Scale Factor values to get calculated.

<u>Solution</u>

Go through another Chromat Calibration and be sure to wait the full 30-45 seconds in Step 8.



Short Chromat Peaks (cont'd)

Not Adjusting the GC Flowmeter Up

Step 7 of the calibration instructs to turn the GC flowmeter up to 0.5scfh or higher before counting the 30-45 seconds in Step 8. This is important because not adjusting the flowmeter will restrict the flow of the gas into the Sample Loop. If the flow is restricted by not adjusting the GC flowmeter up, then the Sample Loop might not get completely filled with gas in that 30-45 seconds. This results in chromat peaks that are shorter than normal. Continuing on with the rest of the calibration steps with these short peaks will cause higher Scale Factor values to get calculated.

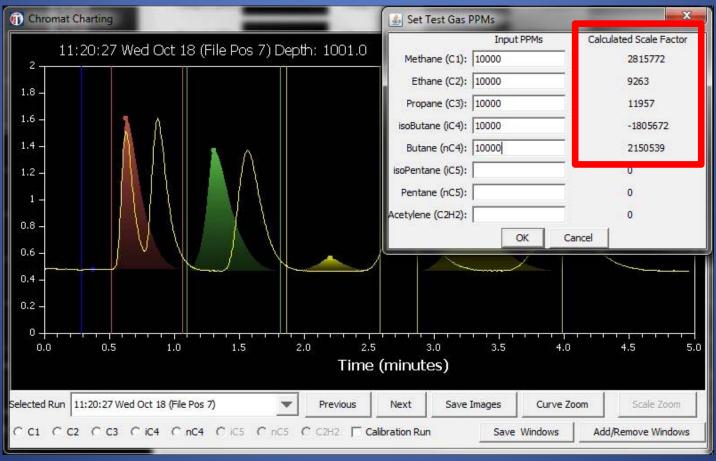
<u>Solution</u>

Go through another Chromat Calibration and be sure to adjust the GC flowmeter up to 0.5scfh or higher in Step 7.



High Scale Factor Values

In the image below, the Calculated Scale Factor values are very high and negative. There are two main reasons why this happens: the Retention Windows were adjusted but not saved before progressing to Step 20 or the Retention Windows were left in bad positions before progressing to Step 20.





High Scale Factor Values (cont'd)

Not Setting and Saving Retention Windows Properly Before the Integration Calibration Step

Step 17 of the calibration discusses adjusting the Retention Window placement, Step 18 instructs to click the Save Retention Windows button, Step 19 instructs to check the Calibration Run checkbox, and Step 20 is the Integration Calibration step. If the Retention Windows are not appropriately adjusted and saved before
Step 20, then Step 20 will do its calculation based on where the windows were set previously. If a Retention Window was in a position where no peak was present, so that nothing but a flat line was within the window, then it will result in an incredibly high Scale Factor value in Step 20 that could be in the millions or could possibly even be a negative value. The high Scale Factor values will falsely calculate chromat data in later runs, even when no chromat peaks are present.

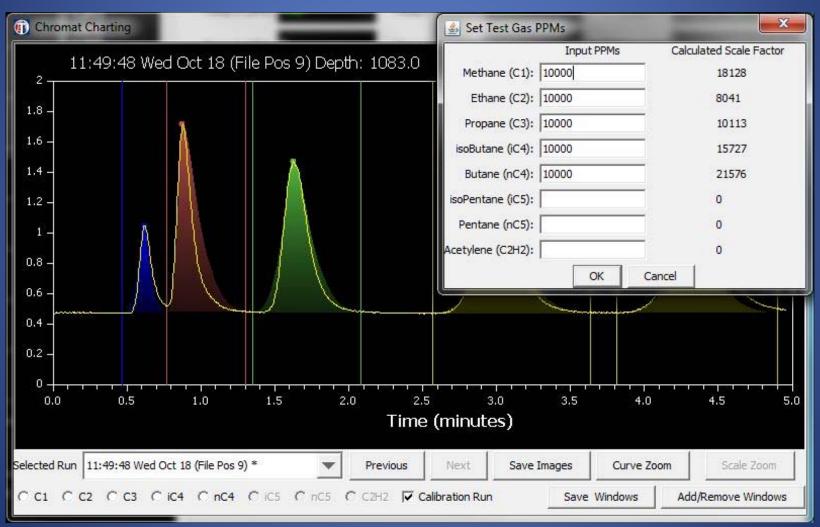
Solution

Set the Retention Windows in the proper positions again (review Step 17) and then be sure to click the Save Windows button first, then check the box for Calibration Run, then move to the Integration Calibration step.



Short C1 Peak

In the image below, the C1 peak is shorter than it normally is in the Chromat Calibration run.





Short C1 Peak (cont'd)

If a GC filament starts degrading, it will lose its sensitivity on the Methane gas first, resulting in a shorter C1 peak in the calibration run. Most of the time the C1 and C3 peaks are very close to the same height or taller on calibrations. If the C1 peak is significantly lower than normal on the calibration run, then it will cause a higher Scale Factor for C1, which will skew the C1 data in normal runs.

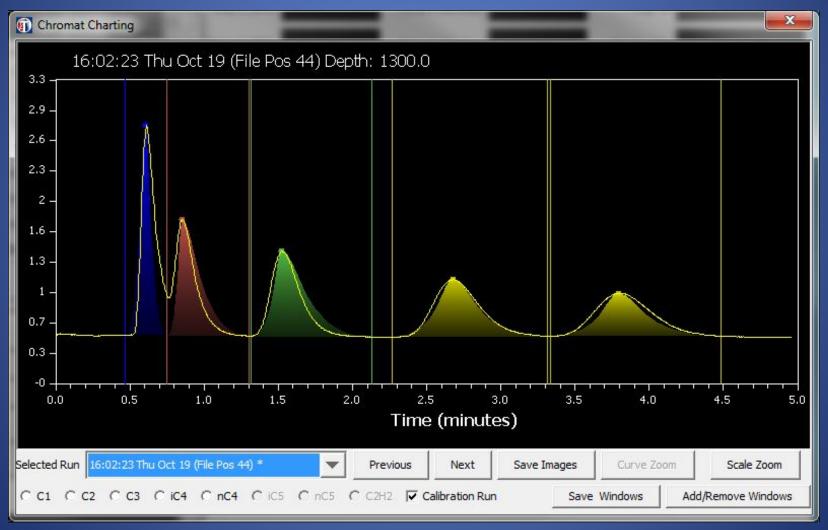
Solution

The GC filament should be changed and another calibration should be completed with the new filament. For help with changing the filament, read the *Changing Filaments* guide.



Tall C1 Peak

In the image below, the C1 peak is taller than it normally is in the Chromat Calibration run.





Tall C1 Peak (cont'd)

If the C1 peak is significantly taller than the C2 peak in the Chromat Calibration run, then, most likely, the 99% Methane Calibration was performed before the Chromat Calibration and there was probably residual test gas left in the regulator from the bottle of 99% Methane. Since there was "extra" Methane in the regulator, it got pushed out into the Chromat Calibration run, resulting in a much taller C1 peak than what was expected. This results in a lower than normal Scale Factor value for C1.

Solution

To prevent this from happening again, perform the Chromat Calibration **before** performing the Total Gas Calibration or purge the regulator before performing the Chromat Calibration using the following steps.

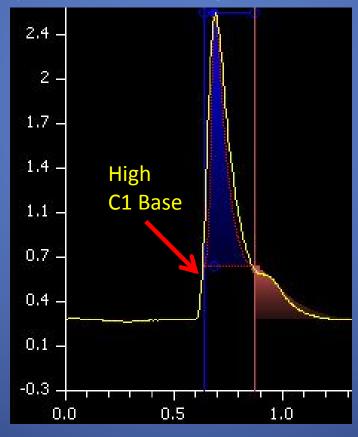
Purge the Gas Regulator

- 1. Attach the gas regulator to the bottle of 1% Blend.
- 2. Leaving the bottle and regulator disconnected from the MLogger, turn the bottle on for 2-3 seconds, letting the gas release into open air, and then turn the bottle off. This will purge the regulator of any residual gases left over from the 99% cal.
- 3. Now, execute the full Chromat Calibration again.



Setting C1 Retention Window Improperly

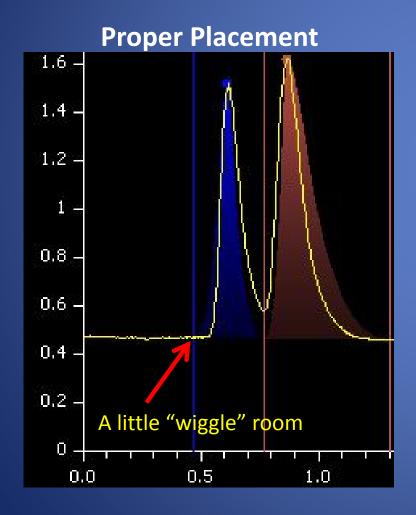
If the left side of the C1 Retention Window is set too close to the C1 peak in the calibration, it can raise the baseline of the integrated area for C1 higher than the actual baseline when larger concentrations of C1 are present because the C1 peak can start to cross over the left side of the window. If the baseline for C1 raises higher than the C2 peak, then C2 will get calculated as O.

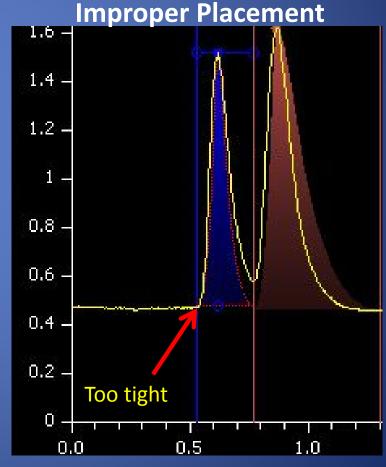




Setting C1 Retention Window Improperly (cont'd)

The images below display proper and improper placement of the C1 Retention Window in a Chromat Calibration run.

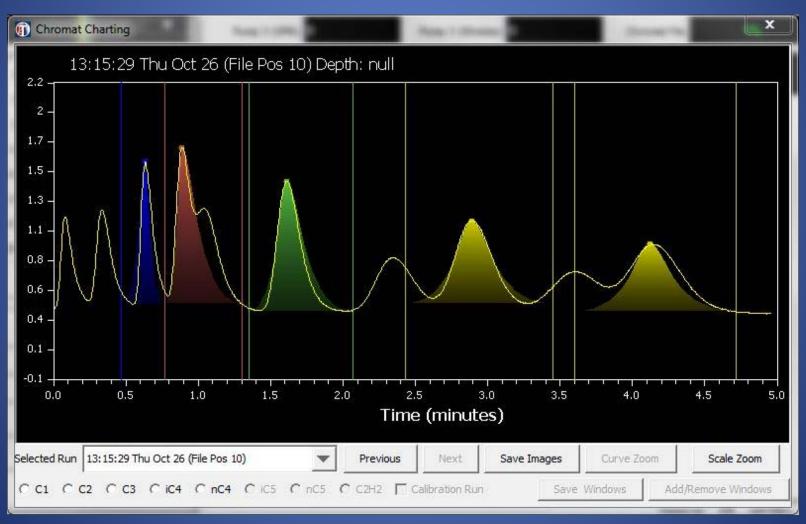






Recycled Gas in Chromat Calibration

The image below shows a Chromat Calibration run with more than the 5 peaks that are expected. This is an example of Recycled Gas.





Recycled Gas in Chromat Calibration (cont'd)

Recycled Gas will occur if one or more exhaust lines are loose or disconnected or if a polyflow line is attached to the Carrier In port on the rear panel of the MLogger and is ran outside with the exhaust lines. In both cases, the exhausted gas will get sucked in by the Carrier In port and will be used as the "carrier gas" for the chromat run. Recycled gas will also occur if a chromat run is started, stopped, and then started again before allowing the gas in the Chromat Column to evacuate. Frozen or plugged Chromat Exhaust lines can cause this too.

Solution

- 1. Check the rear panel of the MLogger and make sure that there is an exhaust line of each exhaust port. A Red Box has 3 exhausts and a Green Box has 4.
- 2. If all the exhaust lines are attached, pull on them to make sure they do not come right out. If they do come out, loosen the polyflow nut, attach the line back to the port, and then tighten the nut.
- 3. Blow the Chromat Exhaust line out to ensure there isn't a blockage.
- 4. If Stop Current Run is pressed while gas is in the Chromat Column, then be sure to allow a full 5 minutes (or whatever the remainder of the run time would have been) before starting another run.